The processing, properties and optimum mix of fly ash based - self compacting geopolymer concrete

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Abstract. The development of concrete technology toward environmentally friendly concrete has become increasingly interest area. The production of Portland cement has a significant impact on greenhouse emissions. The fact triggers the research and the development of eco-friendly concrete, one of which is geopolymer concrete. The implementation of concrete casting sometimes faces the construction that requires the casting process without vibration. It is then when the self-compacting concrete technology was developed. Concerning the two main issues of eco-friendly self-compacting concrete mention above, fly ash based-self compacting Geopolymer concrete has become a research study and develops in the last decades. This paper identifies the process, properties, an optimum mix of fly ash-based self-compacting geopolymer concrete. The properties in this research are the workability of fresh concrete and compressive strength at 7 days. Compressive strength is reported with several NaOH molarities, Alkaline to fly ash ratio, curing time, Curing temperature, Superplasticizer, and Extra Water. Examination of the workability is done with a slump flow test, V-Funnel test, and L-Shape box test. The result shows the optimum mix of fly ash based self-compacting geopolymer concrete.

1. Introduction
The growth of the world demands massive infrastructure development. Concrete is one of the most widely used materials in construction. This increases the demand for the use of Portland cement as one of the concrete-forming materials. Portland cement itself is not an environmentally friendly material. Cement production has consumed large amounts of natural resources, and also releases large amounts of carbon dioxide (CO₂) into the atmosphere, which causes greenhouse emissions. Cement Industry Trend Report reported that world cement production has reached 4.2 billion tons in 2016. Indonesia was on the fifth of the world's largest cement production country, which is 74 million tons a year [1].

Each ton of cement production contributes 1 ton of CO₂ to the atmosphere, so the global cement industry contributes 11.73% of the total global greenhouse gas emissions (35.8 Gigatons) in 2016 [2, 3]. This was increased compared to the data in 2002 which was only amounted to 7% [4]. Therefore, the replacement of Portland cement with fly ash could be an alternative way to reduce greenhouse emissions due to cement production dramatically. The development of technology in the field of construction grows rapidly every year, both in terms of design and construction methods. This requires the development of concrete technology that responds to the existing construction problems, one of which is self-compacting concrete. Self-compacting concrete is concrete that has a high slump value, good flowability, filling ability and is able to compact itself so it does not require a process of
compaction with vibration. The composition of cement that is needed in mix design of Self Compacting Concrete (SCC) is higher than the normal concrete [5]. Therefore, various studies were done to find replacement material that has cementitious characteristics. In the last decades, research began to answer the two main issues above, the self-compacting concrete that is formed from no cement but cementitious material, such as fly ash. Fly ash is a waste of coal-burning from an electric steam power plant. Replacing cement with fly ash in SCC is called Fly ash based-self compacting geopolymer concrete. This paper identifies the processing, properties and optimum mix of fly ash based Self Compacting Geopolymer Concrete (SCGC).

2. Early research on self compacting geopolymer concrete
The study was examined the compressive strength and characteristic of the low calcium fly ash based-self compacting geopolymer concrete [6]. The essential workability properties of the freshly SCGC were evaluated. The study has also reported the effect of additional water, curing time and temperature of curing on the compressive strength of the SCGC. The results showed that additional water in the concrete mixture plays an important role. Higher curing time and temperature would also result in a higher compressive strength. The study examined the effects of superplasticizer and molarity of alkaline solution sodium hydroxide on the compressive strength and microstructure of the fly ash-based SCGC. The results showed that the workability and compressive strength were increased in accordance with increasing dosage of superplasticizer. Strength was increased and fatigue was decreased along with increasing molarity of NaOH solution from 8M to 14M. Increased Interface Transition Zone (ITZ) and Microstructure along with the promotion of SP and increased concentrations from 8M to 12M were also identified [7]. The study also investigated the effect of time and temperature of curing on the compressive strength of fly ash-based SCGC. The experiment was conducted by varied curing time and temperature at the range of 24-96 hours and 60°C-90°C. The characteristics of SCGC misconduct were also examined. The result showed that the length of curing time at higher temperatures produce higher compressive strength. [8]. The research has conducted an experiment to determine the effect of superplasticizer and the amount of extra water on the strength and the workability of fly ash based SCGC. Experiments were conducted by a varied amounts of extra water and superplasticizer dosages. Increase the amount of extra water and superplasticizer was resulted in workability increasing as well. However, the addition of water beyond 15% resulted in bleeding and decreasing of concrete compressive strength. Fly ash based SCGC’s compressive strength is decreased significantly after additional water quantities exceeded 12% by mass of fly ash [9]. The study reported the results of laboratory tests conducted to determine the effect of sodium hydroxide concentration on the workability and compressive strength of the fly ash based-SCGC. The test results indicated that the variation of sodium hydroxide concentration had an effect on SCGC workability. With the increasing concentrations of sodium hydroxide, the performance of fresh concrete is slightly reduced; however, the compressive strength actually increases. Concrete samples with sodium hydroxide concentrations of 12M produced maximum compressive strength [10].

3. Experimental work

3.1. Material used

3.1.1. Fly ash
The material that is used is low calcium Fly Ash (Class F) which was obtained from the steam power plant in Amurang, South Minahasa Regency, North Sulawesi Province, Indonesia, with the composition as in Table.

3.1.2. Alkaline activator
The combination of sodium silicate and sodium hydroxide is used to help the chemical reaction to aluminum and silica in fly ash. Sodium Silicate \((\text{Na}_2\text{SiO}_3)\) is gel-shaped, while Sodium Hydroxide \((\text{NaOH})\) is flake-shaped with 99% of purity.

**Table 1. Chemical composition of fly ash as determined by XRF**

| Oxides       | Mass (%) | Requirement ASTM C 618 Class F |
|--------------|----------|---------------------------------|
| \(\text{SiO}_2\)  | 38.8     | -                               |
| \(\text{Al}_2\text{O}_3\)  | 15.9     | -                               |
| \(\text{Fe}_2\text{O}_3\)  | 36.8     | -                               |
| \(\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3\)  | 91.5     | Min 70%                         |
| \(\text{CaO}\)  | 3.50     | -                               |
| \(\text{MgO}\)  | 0.24     | Max 5%                          |
| \(\text{TiO}_2\)  | 0.97     | -                               |
| \(\text{K}_2\text{O}\)  | 2.28     | -                               |
| \(\text{Na}_2\text{O}\)  | 0.03     | Max 1.5%                        |
| \(\text{SO}_3\)  | 0.55     | Max 5%                          |

3.1.3. Superplasticizer
Superplasticizer is used to increase the required workability of fly ash based-SCGC. This study uses SIKA® Viscocrete 10 superplasticizer.

3.2. Tests
- Examination of aggregate characteristic
- Examination to the chemical composition of fly ash by XRF test
- Examination of workability using slump flow test, V-Funnel test & L-Shaped Box test
- Examination of compressive strength

4. Result and discussion

4.1. The processing of fly ash based – self-compacting geopolymer concrete

4.1.1. Mixing process of fly ash-based geopolymer concrete
The mixing process includes the following series of activities:
- First, make a solution of sodium hydroxide \((\text{NaOH})\) and water, the proportion of solution with molarity, as planned. The sodium hydroxide solution is allowed for 24 hours.
- Second, make alkaline fluid in the container. The alkaline fluid used in this study was a mixture of sodium silicate \((\text{Na}_2\text{SiO}_3)\) and a solution of sodium hydroxide \((\text{NaOH})\). Measure accordingly to the calculation, then mixed for ± 10 minutes until they are homogeneous.
- Third, materials are measured in accordance with mixed design (coarse aggregate and fine aggregate under SSD conditions) and then prepare the necessary tools.
- Then, Mix coarse aggregate, fine aggregate and fly ash into the concrete mixer.
- After that, mix the alkaline solution, water, and Viscocrete-10, then pour the mixture into the concrete mixer and stir thoroughly until they are homogeneous.
- Repeat steps 1 to 5 with another geopolymer concrete mixtures

4.1.2. Slump flow test
The slump flow test aims to determine the flowability and filling ability of fresh self-compacting concrete. The working steps of this slump flow testing method are as follows:
- First, prepare the slump flow tools by watering the tool surface.
Second, place the slump flow tool (Abram’s cone) just like slump testing on a normal concrete. The cone is placed on a wide flat board. The board is used as a base for concrete to flow properly without any obstacles;

After the concrete mix is ready, the fresh concrete mixture is poured into the Abram’s cone to complete volume and no stabbing of the concrete mixture is applied;

Then, the Abram's cone was lifted slowly and constantly. Concrete flow should not be interrupted;

The data which is collected merely by means of final diameter value of the distribution of fresh concrete mix (maximum diameter produced) until the concrete is not flowing (silent) or SFmax.

The slump flow test result may observe the workability condition of the concrete mixture. Mind the following conditions: 1) the homogeneity of the concrete, which is seen when the concrete condition does not turn up to segregation; 2) no bleeding, and fine aggregate shall be evenly distributed. The standard for self-compacting concrete, SFmax ranges from 550-850 mm [11].

4.1.3. V-Funnel test
V-Funnel test is useful for measuring the ability to flow of the fresh concrete mix, where the filling ability can be seen. In addition, testing with V-Funnel can be used to determine the segregation resistance. The design of the V-Funnel tool may indicate that the concrete mixture is blocked or not. If the coarse aggregate composition in the concrete mixture is too much, it will take longer for the concrete mix to flow. In operation, V-Funnel testing is relatively easy to apply in the field because it does not require special skills in the implementation. The results of the test with the V-Funnel method are mutually supportive of the results of the slump cone test. In testing with V-Funnel tool, there is limitation where Self Compacting concrete categories as good filling ability. Limitations in this V-Funnel tool, concrete mixtures categorized as self-compaction concrete if all mixture has exhausted in ≤ 25 seconds [11].

4.1.4. L-Shaped box test
L-Shaped box, also called the Swedish L-Shaped box, is an L-shaped tool made of iron. This tool serves to test the passing ability of self-compacting concrete. The horizontal and vertical directions are limited by an iron cover seal that can be opened by pulling it up. There is steel reinforcement in front of the bulkhead that serves to test the ability of concrete mixture in passing the reinforcement. Passing ability requirement that must be fulfilled by self-compacting concrete is the passing ratio that is seen from the height of concrete flow in the horizontal direction (H2 / H1) ≥ 0.8 with 2 or 3 rebars [12].

4.1.5. Molding
The molding process includes the following series of activities:

- The walls of the formwork are cleaned and lubricated.
- The fresh mixed concrete is incorporated into formwork without vibration.

4.1.6. Hardening
Unlike Portland cement that produces high hydration heat, geopolymer materials require additional activation energy to speed up the polymerization process. This is because the resulting heat is less high. In order for the hardening process to be proceeding rapidly, the test specimen must be inserted into the oven at 70° Celsius for 48 hours to harden. The hardened concrete is indicated when the concrete is not collapsed as it was taken out from formwork.

4.1.7. Curing
The curing of geopolymer concrete is different from Portland cement concrete, i.e. after exiting the oven, the tested object is left at room temperature for 7 days. Specimens that have been left at room
temperature for 7 days are coded to facilitate the classification and to measure the specimens for the examination of the weight of the concrete volume.

4.1.8. Compressive strength test
The test is carried out by loading pressure until the test object collapses. Compressive strength was tested on seven days by using a universal testing machine (UTM). The test specimen is cubic with size 100x100x100 mm$^3$. Each variant of the mixture is represented by 3 pieces of the specimen.

4.2. The properties of fly ash-based geopolymer concrete

4.2.1. Compressive strength
Experiments were performed using a test object that contains fly ash, alkaline liquid (sodium hydroxide and sodium silicate) and fine aggregate. The variation of Molaritas NaOH obtains optimum molar concentration for the self-compacting Geopolymer concrete. The results can be seen in table 2.

| Molarity | 8 M | 12 M | 14 M |
|----------|-----|------|------|
| Object number |     |      |      |
| 1 | 2,94 | 6,12 | 7,48 |
| 2 | 4,16 | 6,70 | 7,34 |
| 3 | 4,44 | 6,09 | 7,59 |

From the table 2, it is found that the higher the molarity of NaOH is, the higher the result of compressive strength is, but because the compressive strength was produced at 12 M and 14 M increments is not significant, it does not proceed to higher molarity, and takes 14 M as the optimum value. After molarity is obtained, the optimal binding of geopolymer binder is explored, by varying the ratio of sodium hydroxide and sodium silicate to Fly Ash. The results can be seen in table 3.

| Alkalin / fly ash ratio | 0.3 | 0.5 | 0.7 | 0.8 | 0.9 |
|------------------------|-----|-----|-----|-----|-----|
| Object number |     |      |      |      |      |
| 1 | 2,21 | 4,75 | 6,94 | 8,20 | 5,33 |
| 2 | 2,14 | 4,62 | 6,72 | 7,91 | 4,02 |
| 3 | 2,17 | 5,42 | 6,83 | 8,11 | 5,89 |

From table 3, we can see the ratio range as recommended in literature study, which is 0.3; 0.5 and 0.7. The results show that the compressive strength increases up to 0.8 and then decreases at 0.9 so that the ratio of the alkaline mixture to fly ash is optimally selected at 0.8. After obtaining the mixture of binder and Molarity, then the experiment with various times and temperatures of the curing gets the time and the temperature that give optimum compressive strength results. The results can be seen in table 4.

| Temperature Curing time | 60°C | 70°C | 80°C | 90°C |
|------------------------|------|------|------|------|
| Curing time | 24 H | 48 H | 24 H | 48 H | 24 H | 48 H | 24 H | 48 H |
| Object number |     |      |      |      |      |      |      |      |
| 1 | 7,48 | 10,20 | 6,94 | 13,80 | 7,00 | 8,40 | 8,18 | 10,52 |
| 2 | 7,34 | 9,33 | 6,72 | 13,02 | 7,34 | 7,93 | 9,61 | 12,42 |
| 3 | 7,59 | 9,37 | 6,83 | 12,39 | 6,90 | 8,12 | 10,11 | 11,72 |
From Table 4, it can be seen that the Optimum Pressure is obtained from the oven treatment at 70° C for 48 Hours. After obtaining the optimal binder mixture, the experiments are then continued by binding the mixture with aggregate using the superplasticizer Viscocrete 10 in the manufacture of self-compacting geopolymer concrete. See table 5.

Table 5. Compressive strength in various superplasticizer doses

| Superplasticizer | 2,5% | 3% | 4% | 5% | 6% |
|------------------|------|----|----|----|----|
| Object number    | Compressive Strength (MPa) |
| 1                | 10,20 | 12,80 | 12,01 | 10,82 | 9,60 |
| 2                | 9,33  | 12,10 | 12,32 | 11,07 | 9,68 |
| 3                | 9,37  | 12,18 | 11,43 | 10,64 | 9,50 |

From Table 5, it can be seen that the addition of superplasticizer to 3% content of fly ash mixture and alkaline solution is directly proportional to the increase of concrete compressive strength, but decreased thereafter to 3%.

4.2.2. Workability

The examination of workability was done with 3 testing methods. The first is the slump flow checking, by varying the ratio of extra water/fly ash and the addition of superplasticizer until it finds a mixture that meets the self-compacting concrete standard that is at maximum distribution value more than 550 mm. After finding the dose of superplasticizer and extra water that can produce the value of slump flow distribution > 550 mm, which is at dose superplasticizer 3% and the ratio of extra water/fly ash equal to 0.25, then the viscosity with V-Funnel Test and Passing ability with L-shaped is tested.

From the results of filling ability testing using slump cone, it was obtained 560 mm SFmax value, as a result on the addition of extra water 0.25 by mass of fly ash and 3% superplasticizer dose. This indicates that the self-compacting geopolymer concrete mixture has fulfilled the requirements determined by the Efnarc association, ie 550 - 850 mm, in SF1 class according to efnarc classification. The terms of filling ability and viscosity specified in the V-Funnel test is ≤ 25 seconds. In this study, the value that is obtained from the V-Funnel test is 9.58 seconds, so it meets the recommended perimeter conditions in the viscosity class VS2 / VF2 with the limit condition of 9-25 seconds.

From the result of the passing ability test using L-shaped box test, the value of blocking ratio H2 / H1 is 0.87 seconds. This shows that the mixed of self-compacting geopolymer concrete mixture has fulfilled the requirements determined by the Efnarc association, i.e ≥ 0.8 on the passing ability class PA1 with 2 rebars. The values of F-L20 and F-L40 - which were obtained in this study (in 2 secs and in 4 sec) illustrate the consistency of flow velocity that occurs in fresh concrete.

4.3. The Optimum mix of fly ash based self-compacting geopolymer concrete

The composition of the fly ash based self-compacting geopolymer concrete refers to mix design by Memon et al (2011), Gumalang et al (2016) [12], including Trial & Error during an experiment. The optimum results are shown as follows:

- Class F Fly Ash in dry condition (OD)
- Coarse aggregate and fine aggregate in saturated surface dry condition (SSD)
- The concentration of Sodium Hydroxide: 12M-14M
- Alkaline/Fly Ash ratio: 0,8
- Superplasticizer Viscocrete 10: 1%-3%
- Extra Water/ Fly Ash ratio: 0,12-0,3
- The curing process in the oven for 48 hours at temperature 70°C.

The mix design above produces workability that meets the efnarc standard, which is SFmax ≥ 550 mm and the average compressive strength is 20 MPa in 7 days.
5. Conclusion
This paper is presented the study of fly ash based- self-compacting geopolymer concrete. From the experimental results which are reported in this paper, the following conclusions are drawn:

- Higher molarity of sodium hydroxide solution results in a higher compressive strength of fly ash based on self-compacting geopolymer concrete.
- The activator-to-fly ash ratio, by mass of 0.80 produced the highest compressive strength.
- The curing temperature which produced the highest compressive strength is 70°C.
- 48 hours curing time produced the highest compressive strength.

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