Geopolymer Mix in Accordance to Design of Experiment (DOE) Method

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Abstract. This paper presents an experimental study conducted to assess the capability of using DOE method to achieve particular targeted strength for geopolymer concrete. Targeted strength of 30 MPa, 40 MPa and 60 MPa were used in this study. Cube samples with dimension of 100 mm were used for compressive strength test. Similar DOE method calculation as in normal concrete mix were calculated for all the raw material ratios in geopolymer concrete mix. Parameters such as characteristic strength, standard deviation (s), margin (m), target mean strength (f_m), free-water/cement ratio (w/c), slump, relative density of aggregate (SSD), concrete density (D), percentage grading of fine aggregate passing 600 μm sieve, and proportion of fine aggregate were determined according to the respective concrete mix design. However, only characteristic strength, f_m and w/c varied in each targeted strength mix design while the other parameters were kept constant. In order to yield geopolymer concrete, the used of cement and water in DOE form were fully substituted with the used of fly ash and alkaline activator respectively. Final strength gained from test result for the respective 30 MPa, 40 MPa and 60 MPa were 74.6 MPa, 84.5 MPa and 97.7 MPa. Therefore, it was noted that geopolymer mix yielded higher strength compared to the targeted strength by using the DOE method calculation.

1. Introduction

Concrete is the most used construction material all over the world while Ordinary Portland Cement (OPC) is one of its most essential components. Production of one ton of OPC releases one ton of carbon dioxide (CO_2) and requires a large amount of energy. Fly ash, abundantly available by-product of thermal power plant (TPP) is posing great environmental problems through its disposal landfill. The presence of Silica (Si) and Alumina (Al) chemical components in the fly ash make it as a geopolymer that can bind with alkaline activator through a chemical process known as geopolymerization which can produce a binding material.

Basically, geopolymer is a kind of inorganic polymer produced by the reaction of aluminosilicate materials with alkaline solution [1-2]. Geopolymer has shown many excellent properties such as high early strength, good resistance against acid and sulfate attacks as well as good performance in high temperature [3-7]. Figure 1 shows the SEM images of original activated fly ash.

Previous study done by Zarina et al. [8] concluded that the fly ash-based geopolymer concrete produced higher strength compared to Ordinary Portland Cement (OPC) concrete at 1, 7 and 28 days.

The strength of concrete is basically affected by both the aggregate properties and the characteristics of the new cement paste that is developed during the maturing of concrete. Similarly, the strength of geopolymer concrete is highly influenced by the proportions and properties of the constituent materials that make the geopolymer paste [9].
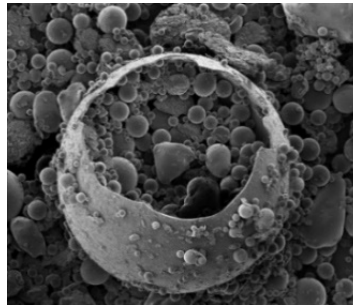
Figure 1. SEM images of original activated fly ash [10].

According to the ASTM C-618 [11], there are two classes of coal combustion fly ash namely Class F and Class C. Class C fly ashes are high-lime fly ashes which typically contain CaO in excess of 10% up to 40%. Meanwhile, Class F fly ashes are low-lime fly ashes which generally contain less than 10% CaO. Due to high CaO content, Class C fly ashes participate in both cementitious and pozzolanic reactions whereas Class F fly ashes predominately participate in pozzolanic reaction during the hydration process. According to the study done by Xu and Deventer [12], the most common alkaline liquid used in geopolymerisation is a combination of sodium hydroxide (NaOH) and sodium silicate [13-16]. Furthermore, a study by Arjunan et al. [17] revealed that NaOH in low concentration was the most effective chemical activator for low calcium fly ash.

2. DOE method for geopolymer mixture concrete

DOE Method used to differentiate the properties of 30 MPa, 40 MPa and 60 MPa concrete targeted strength were filled in the concrete mix design forms. Parameters included in the concrete mix design form were characteristic strength, standard deviation (s), margin (m), target mean strength (f_m), free-water/cement ratio (w/c), slump, relative density of aggregate (SSD), concrete density (D), percentage grading of fine aggregate passing 600 μm sieve, and proportion of fine aggregate.

For 30 MPa geopolymer concrete strength, characteristic strength used was 30 N/mm^2 while its standard deviation, s was 8 N/mm^2. M, f_m, and w/c were 13.12 N/mm^2, 43.12 N/mm^2 and 0.47 respectively. 10-30 mm slump, 2.6 SSD and 2400 kg/m^3 D were used as well. Percentage grading of 70% fine aggregate passing 600 μm sieve with 25% proportion of fine aggregate were used.

Meanwhile, the changing factors for 40 MPa concrete targeted strength were its characteristic strength, f_m and w/c which were 40 N/mm^2, 53.12 N/mm^2 and 0.46 respectively. Other factors included s, m, slump, SSD, D, percentage grading of fine aggregate passing 600 μm sieve, and proportion of fine aggregate were similar as 30 MPa targeted strength.

As in 60 MPa targeted strength, only characteristic strength, f_m and w/c were varied which were 60 N/mm^2, 73.12 N/mm^2 and 0.44 respectively. Other parameters were kept constant as well.

3. Properties of binder used

The raw materials used to yield geopolymer concrete were fly ash, alkaline activator, coarse aggregate and fine aggregate. Class F fly ash from Sejingkat power plant in Kuching, Sarawak was used as a substituent of cement in geopolymer concrete as shown in Figure 2 [18].

The combination of NaOH and sodium silicate (Na_2SiO_3) were used as the alkaline activator. NaOH pellets was dissolved using distilled water to form NaOH solution. 10M of NaOH solution with 2.5 Si:OH ratio were used [18-20].

In order to prepare 10M of NaOH solution, 34.87% of NaOH pellets and 65.13% of distilled water were used. The solution was prepared at least 24 hours prior to mixing [18, 21]. Figure 3 shows the granulated NaOH and Na_2SiO_3 solution.

Figure 2. Class F fly ash used for geopolymer concrete mix.

| (a) | (b) |

Figure 3. Alkaline activator consist of (a) sodium hydroxide **(**NaOH) in pellets form and (b) sodium silicate (Na_2SiO_3) solution.

4. Experimental study

4.1. Mixing process
Following the DOE method, respective ratios by mass for aggregates and binder were determined as tabulated in Table 1. Dry mix, which contain of coarse aggregates and sand were mixed first for about three minutes before added in the fly ash which were then mixed for another three minutes. After adding the solution into the dry mix, mixing process was continued for another five minutes to ensure all the materials were entirely mixed together [22]. The fresh geopolymer concrete then can be casted into the mould as shown in Figure 4.

Figure 4. Fresh geopolymer concrete.

Table 1. Aggregate:Binder ratio for geopolymer mix.

Target strength (MPa)	Aggregate		Binder		Aggregate: Binder ratio
	Coarse aggregate (kg)	Fine aggregate (kg)	Fly ash (kg)	Alkaline activator (kg)	
30	1394	465	340	160	3.72
	1859		500		
40	1419	473	348	160	3.72
	1892		508		
60	1425	475	364	160	3.63
	1900		524		

4.2. Casting and curing

100mm x 100mm x 100mm cube moulds were used to cast the geopolymer concrete. The fresh geopolymer concrete was poured into the mould in two layers with 35 tamping for each layer. The samples were then being oven cured for 24 hours with temperature of 80°C [18, 20].

After that, the samples were removed from the oven and left to cool in room temperature before demoulding. For each 30 MPa, 40 MPa and 60 MPa targeted strength, three cube samples were prepared and tested. In total, there were nine cube samples casted to be tested.

4.3. Compressive strength test

After demoulding, all the samples were tested for compression under 250 kN capacity loading machine in displacement control at a rate of 1 mm/min until failure [18]. Figure 5 shows a sample was being tested.

Figure 5. Sample for 40 MPa targeted strength was being tested.

5. Results and discussion

5.1. DOE method and geopolymer mixture concrete

Based on DOE method, the mass ratio for each material were determined. Aggregate:binder ratios for gepolymer mix were tabulated as in Table 1. Significantly, higher target strength required higher aggregate:binder ratio. This is because increased in mass of aggregates for higher concrete strength was needed to ensure that the geopolymer concrete able to achieved the targeted strength. Therefore, the higher mass of aggregates contained able to enhanced the strength of geopolymer concrete itself.

Table 2 shows the ratio between coarse and fine aggregates (CA:FA), coarse aggregate to fly ash ratio (CA:FAs), fine aggregate to fly ash ratio (FA:FAs) as well as alkaline activator to fly ash ratios (AA:FAs) for all the respective target strength values.

Table 2. Ratios for geopolymer concrete mix.

Target strength (MPa)	CA:FA	CA:FAs	FA:FAs	AA:FAs
30	3.00	4.10	1.37	0.47
40	3.00	4.08	1.36	0.46
60	3.00	3.91	1.30	0.44

Based on Table 2 above, it showed that all the target strengths used the same ratios for coarse and fine aggregate which was 3.00. However, both CA:FAs and FA:FAs ratios showed a fluctuation as the concrete target strength increased. Thus, it can be concluded that the highest CA:FAs and FA:FAs ratios were used for 30 MPa targeted strength. By referring to the ratios, highest targeted geopolymer strength, which was 60 MPa utilized lowest CA:FAs, FA:FAs and AA:FAs ratios which were 3.91, 1.30 and 0.44 respectively.

Results for compressive strength of geopolymer concrete obtained from the compression test were tabulated and analyzed. Figure 6 shows a sample after being tested while Table 3 shows the compressive strength of geopolymer concrete for the respective targeted strength.

Figure 6. Sample for 40 MPa targeted strength after being tested.

Table 3. Average strength achieved for 30 MPa, 40 MPa and 60 MPa targeted strength.

Targeted strength (MPa)	Average strength achieved (MPa)
30	74.6
40	84.5
60	97.7

From Table 3, it can be seen that the average strength achieved for geopolymer concrete was respectively higher than its targeted strength. The highest average strength yielded was 97.7 MPa with the lowest ratios usage of CA:FAs, FA:FAs and AA:FAs. Higher aggregates contained by mass in the mixed were needed to increase the concrete strength significantly. Higher amount of aggregates in a specific concrete also indicated stronger concrete ability to withstand external load. Lower AA:FAs represented higher amount of alkaline activator used in the concrete mix. 0.44 of AA:FAs ratio contained higher liquid which could optimized the geopolymerization process and causing the wet geopolymer concrete to be easily compacted.

6. Summary

From the laboratory results, it can be concluded that highest geopolymer concrete average strength which was 97.7 MPa can be achieved by using 3.91 CA:FAs, 1.30 FA:FAs and 0.44 AA:FAs ratios. By referring to the concrete mixed design form, 60 MPa targeted strength required characteristic strength, f_m and w/c of 60 N/mm^2, 73.12 N/mm^2 and 0.47 respectively. Other parameters which were standard deviation, m, slump, SSD, D, percentage grading of fine aggregate passing 600 μm sieve and proportion of fine aggregate used similar values as in the 30 MPa and 40 MPa targeted strength.

Compressive strength test of geopolymer concrete using DOE method was perceived to be slightly differ from the actual targeted concrete strength. Precisely, all average strengths achieved were higher compared to the targeted strength. Therefore, the method can be improvised to achieve the absolute targeted strength in yielding geopolymer concrete.

7. References

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