Strength and Density of Geopolymer Mortar Cured at Ambient Temperature for Use as Repair Material

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Abstract. Geopolymers produced by synthesizing aluminosilicate source materials with an alkaline activator solution promised an excellent properties akin to the existing construction material. This study focused on the effect of various binder to sand ratio on geopolymer mortar properties. Mix design of geopolymer mortar was produced using NaOH concentration of 12 molars, ratio of fly ash/alkaline activator and ratio Na\textsubscript{2}SiO\textsubscript{3}/NaOH of 2.0 and 2.5 respectively. Samples subsequently were cured at ambient temperature. The properties of geopolymer mortar were analysed in term of compressive strength and density at different period which are on the 3\textsuperscript{rd} and 7\textsuperscript{th} day of curing. Experimental results revealed that the addition of sand slightly increase the compressive strength of geopolymer. The optimum compressive strength obtained was up to 31.39 MPa on the 7\textsuperscript{th} day. The density of geopolymer mortar was in the range between 2.0 g/cm\textsuperscript{3} to 2.23 g/cm\textsuperscript{3}. Based on this findings, the special properties promoted by geopolymer mortar display high potential to be implemented in the field of concrete patch repair.

1. Introduction

Geopolymer is an inorganic polymer produced from two main constituents which are the combination of raw materials known as aluminosilicate source containing alumina (Al) and silica (Si) with alkaline activator [1]. The combination of sodium silicate (Na\textsubscript{2}SiO\textsubscript{3}) and sodium hydroxide (NaOH) has been used widely as the alkaline activator to produce geopolymer.

Dissolution of solid aluminosilicate source by alkaline activator and followed with polycondensation to form 3D network of silico-aluminates structures described the geopolymerisation process [2]. A
number of researchers reported excellent properties promoted by geopolymer such as high compressive strength, resistance to steel corrosion, low permeability and high durability [3,4]. Source materials which are rich in silica and alumina such as fly ash, slag, kaolin and metakaolin are commonly used to make geopolymers which promote excellent behaviour and properties. Fly ash is one of the major material approached as the source material of geopolymer due to its commercial and performance advantage. The amount of fly ash generated by electric power plant in Malaysia is increasing each year. Instead of their availability, silica and alumina content is mostly found in fly ash material thus making it the suitable raw material for geopolymer.

Curing temperature also affect the development of mechanical strength in most cementitious systems especially for geopolymer [5]. Geopolymerisation was suggested by previous researchers using a curing temperature less than 60 % for 4-48 hours which is one of the important conditions for the synthesis of geopolymer [3]. However, many researchers have tried to improve the strength development of fly ash geopolymers at ambient temperature [6-8]. The ability of geopolymer mortar to cure at ambient temperature is the advantage especially for concrete repair and rehabilitation [9-11]. Hu et al. [9] investigated the influence of steel slag added in geopolymer and achieved 40 MPa compressive strength and above when cured at ambient temperature and it displayed higher bond strength. Songpiriyakij et al. [10] also studied the rice hush bark ash geopolymer and discovered its high compressive and bonding strength properties. In addition, T. Phoo-ngernkham et al. [11] also studied the potential of geopolymer as repair material and reported that high calcium fly ash geopolymer containing Portland cement cured at ambient temperature exhibited higher compressive strength due to the reaction of calcium in the system. Therefore, the strength of geopolymers are sufficiently high and desirable for some application even when cured at ambient temperature and this can be improved.

Geopolymer mortar is the combination of fly ash, sand and the chosen activator. For the geopolymer formulation, Mustafa et al. [12] discovered that 12 M of NaOH is the optimum compressive strength achieved. In a previous research by Hardjito et al. [13], it was also found that Na2SiO3/NaOH ratio of 2.5 produced the best results. Geopolymer formulation determined by previous researchers was used in this study due to the same class and types of fly ash. However, the addition of sand in geopolymer improved the strength and exhibited different physical properties at different binder to sand ratio. The addition of sand as aggregates to geopolymer is not only economically favourable but also reduces pore density and promotes high strength [14]. C. Kuenzel et al. [15] studied the influence of sand on the mechanical properties of geopolymers and discovered that adding sand aggregate also means adding strong particles to the geopolymer matrix. Thus, sand acts as reinforcements in the geopolymers hence promoting it to the higher strength if properly formulated. The objective of this paper is to determine the effect of various binder to sand ratio on the properties of geopolymer mortar. Determining the optimal binder to sand ratio is important in order to meet the standard and specified mechanical properties of mortars for used as repair material for concrete infrastructure rehabilitation.

2. Experimental

2.1 Material selection
The fly ash was obtained from Manjung Power Station in Lumut, Perak, Malaysia. It is an industrial by product which residue resulting from the combustion of coal. The combination of sodium silicate (Na2SiO3) and sodium hydroxide (NaOH) has been used in this study. The 12 M NaOH was prepared by mixing sodium hydroxide pellets of 97–99 % purity with distilled water. The river sand used in this study having water absorption of 1.6 % and specific gravity of 2.50.

2.2 Samples preparation
The ratio of fly ash/alkaline activator and Na$_2$SiO$_3$/NaOH was fixed at 2.0 and 2.5 for the entire experiment. The preparation of NaOH solution by diluting the NaOH pellets with distilled water in order to produce 12 M NaOH concentration. Meanwhile, the various binder to sand ratio was used in this study. The details of mix design was described in Table 1. The selection of ratio was based on the practicality of the previous researchers of geopolymer mortar and the acceptable workability to be used at various application.

Table 1. Mix design details of geopolymer mortar.

| Sample   | Binder:Sand, wt. ratio | Fly ash, wt. % | Sand, wt. % | 12 M NaOH, wt. % | Sodium silicate, wt. % |
|----------|------------------------|----------------|-------------|-------------------|------------------------|
| Mortar A | 0.25                   | 13.33          | 80          | 1.90              | 4.76                   |
| Mortar B | 0.33                   | 16.54          | 75          | 2.36              | 5.91                   |
| Mortar C | 0.5                    | 22.22          | 67          | 3.17              | 7.94                   |
| Mortar D | 1.0                    | 33.33          | 50          | 4.76              | 11.90                  |
| Mortar E | 2.0                    | 44.45          | 33          | 6.35              | 15.87                  |
| Mortar F | 3.0                    | 50.00          | 25          | 7.14              | 17.86                  |
| Mortar G | 4.0                    | 53.33          | 20          | 7.62              | 19.05                  |
| Paste    | -                      | 66.66          | -           | 9.52              | 23.81                  |

Initially, the sand and fly ash were mixed together in the mixer for 3 minutes. After that, alkaline activator was added in the mixer and the mixing process was continue for another 5 minutes. When the mixture becomes homogeneous, the mixture was moulded using (50×50×50) mm$^3$ according to ASTM C109. The samples were demolded at the age of 1 day and immediately wrapped with vinyl sheet to protect moisture loss and kept in the room at ambient temperature for 3rd and 7th days. The compressive strength and density values were an average of testing of 3 samples for each ratio.

3. Results and Discussion

3.1 Chemical Composition with X-ray fluorescence (XRF)

The chemical composition of the fly ash was obtained by X-ray fluorescence (XRF) (Table 2). The main chemical composition of fly ash is silica oxide SiO$_2$, followed by Fe$_2$O$_3$, CaO, and Al$_2$O$_3$.

Table 2. Chemical composition of fly ash using XRF.

| Chemical | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | TiO$_2$ | CaO | MnO | CuO | K$_2$O | P$_2$O$_5$ | SO$_3$ | SrO |
|----------|---------|-------------|-------------|---------|-----|-----|-----|--------|----------|--------|-----|
| wt.%     | 38.8    | 14.7        | 19.48       | 1.02    | 18.1| 0.16| 0.041| 1.79   | 0.054    | 1.50   | 0.11|

Based on the data, the fly ash is classified as Class F as referred to ASTM C618 where the calcium content (18.1%) which is less than 20% (Table 3). The summation of SiO$_2$ and Al$_2$O$_3$ content is 54% which is the most required composition to produce a good geopolymer [16, 17]. In addition, the sufficient CaO in the fly ash geopolymer mixture is also reported as a suitable source to produce a geopolymer which able to be cured at room temperature [11].

Table 3. Standard classification.

| Chemical                  | wt.% | ASTM C618 (%) |
|---------------------------|------|---------------|
| SiO$_2$ + Al$_2$O$_3$ + Fe$_2$O$_3$ | 72.98| 70.00 min     |
| CaO                       | 18.1 | 20.0 max      |
3.2  **Particle size distribution of sand**
Grading curve described the particle-size distribution of an aggregate as determined by a sieve analysis. The particle size distribution (grading curve) of the sand is shown in Figure 1. The particle size distribution limitation was obtained according to ASTM C33. The standard size sieves for fine aggregate have openings range from 150 μm to 9.5 mm. Grading curve obtained indicates that the particle size distribution of sand is within the limit specified in the standard. The appropriate distribution of sand plays an important role which influence the mechanical properties of geopolymer and its workability.

![Particle size distribution of sand](image)

**Figure 1.** Particle size distribution of sand.

3.3  **Characteristics of geopolymer mortar**
Selection of the appropriate binder to sand ratio helps to ensure workability and performance expectations. Geopolymer mortar shows different behavior at different binder to sand ratio. Physically, each ratio with various binder to sand ratio has some basic characteristics (Table 4). The composition of sand and its grading can influence the characteristics of mortars either in fresh or hardened state. Geopolymer mortar with 0.5 and 1.0 binder to sand ratio display acceptable workability as repair material for patching purposes while the rest were stiff with excessive percentage of sand and slurry due to small percentage of sand.

3.4  **Density**
Density of the geopolymer mortars are summarized in Table 5. The average value of geopolymer mortar density are in the range between 2.0 to 2.23 g/cm³. As the level of aggregate was decreased the density decreased from Mortar A to paste. The density of a geopolymer paste is reported to be below 2 g/cm³ depending on its composition [18].
### Table 4. Physical characteristics of geopolymer mortar.

| Sample          | Binder : Sand, wt. ratio | Characteristics                                                                 |
|-----------------|--------------------------|---------------------------------------------------------------------------------|
| Mortar A and B  | 0.25 and 0.33 (75 - 80) % of sand | Difficult to mix and cast into the mould and not very workable                  |
| Mortar C and D  | 0.5 and 1.0 (50 - 67) % of sand | Easily placed into the mould. Suitable for restoration of concrete spalling.    |
| Mortar E and F  | 2.0 and 3.0 (25 - 33) % of sand | Easy to handle but slurry                                                       |
| Mortar G        | 4.0 (20 % of sand)        | Mixture is too wet, very high workability, not suitable for restoration of concrete spalling. |

The main component of the sand aggregate is quartz which has a density of 2.65 g/cm³ [19]. This factor contributes to the increase of geopolymer mortar from Mortar G containing 20 % sand content to Mortar A which is about 80 % sand content. The denser the sample the better the compressive strength is however, sufficient alkaline activator is required. This is due to the sand which help reduce porosity in the geopolymer mortar thus generally increasing the strength of geopolymer. However, the optimum binder to sand ratio from the compressive test result is the most accurate data to justify the best formulation of the mix design to avoid insufficient alkaline activator for geopolymerisation to take place.
Table 5. Density of geopolymer mortar.

| Sample | Binder:Sand, (wt. ratio) | Sand content (wt. %) | Density (g/cm³) |
|--------|-------------------------|----------------------|-----------------|
| Mortar A | 0.25 | 80 | 2.23 |
| Mortar B | 0.33 | 75 | 2.21 |
| Mortar C | 0.5 | 67 | 2.19 |
| Mortar D | 1.0 | 50 | 2.16 |
| Mortar E | 2.0 | 33 | 2.04 |
| Mortar F | 3.0 | 25 | 2.01 |
| Mortar G | 4.0 | 20 | 2.00 |
| Paste | - | - | 1.94 |

3.5 Compressive strength

The compressive strength values obtained from geopolymer mortar are compared with the compressive strength values obtained from geopolymer paste as shown in Figure 2. Compressive tests on geopolymer under different binder to sand ratio showed that fly ash based geopolymer mortar with a ratio 0.5 (Mortar C) had the highest strength which is 31.39 MPa at the 7th day of curing (Fig. 2). It also promotes higher compressive strength than geopolymer paste which is an increase of 13%.

The compressive strength increased with further increase in the ratio of binder to sand until the ratio of 0.5 (Mortar C). However, the strength starts to decrease after the increase of the ratio. This shows that the compressive strength was affected by the binder to sand ratio. This result can be explained by the effect of sand to the geopolymer system. Sand is a material containing silica. An addition of sand
beyond 50% causes the insufficient content of an activator for geopolymerisation process to take place. This causes the compressive strength of geopolymer mortar to decrease. Sand is responsible for the compressive strength of geopolymer mortar. Increasing sand content without increasing the amount of alkaline activator resulted in a decreasing level of geopolymerisation. It is proposed that compressive strength of geopolymer mortar with high levels of aggregate can be increased by optimizing the amount of alkali and properly distributed in the particle sand size [20].

Particle size distribution of the sand also plays an important role in the strength of the geopolymer mortar. The smaller particles will try to fit in between larger particles. Thus, the interlocking between the aggregate exist and this contributes to the strength of geopolymer mortar. The 0.5 binder to sand ratio was selected as an optimum compressive strength achieved. Therefore this ratio is the optimum compressive strength of geopolymer mortar.

4. Conclusion
The aim of this paper is to determine the best mix design of geopolymer mortar. Thus, the main purpose of this paper which to give an overview on the physical and mechanical properties of geopolymer is done. The various binder to sand ratio acts as an excellent repair material to be developed and applied to solve civil engineering problems. From the behavior of the geopolymer, it is clearly highlighted that the use of sand as fillers in geopolymer has the potential for use as repair material at optimal binder to sand ratio formulation. The geopolymer is compatible with the standard requirements and shows the potential to overcome damaged concrete structures. It is proposed that the compressive strength of geopolymer mortars with aggregate can be increased by optimising the binder to sand ratio and optimising the content of alkaline activator. Therefore, the suitable preparation of geopolymer mortar, 0.5 of binder to sand ratio is recommended on the basis of workability and compressive strength for concrete patch repair. However, the high compressive strength are not yet confirmed to promise good bonding strength between repair materials to concrete substrate. Thus, further analysis is required.

5. References
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