The Effect of Soil Porosity and Geopolymer Viscosity on Spreading Grouting in Weathered Clay Shale

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Abstract. Fly ash geopolymer is a potential material for the stabilisation of clay shale. Therefore, this research determines the effect of soil porosity (n), binder activator ratio (f/a), and geopolymer viscosity on the injected grout volume (V_i) and soil-grout column of weathered clay shale. The research was carried out using fly ash as a geopolymer binder, dissolved in Na_2SiO_3 and NaOH(10M) with an activator ratio of 1:1. The binder activator ratios were 0.50, 0.75, 1.0, and 1.25, with injection applied in the compacted clay shale using three dry densities corresponding to the porosity of 0.42, 0.48, and 0.54. Furthermore, a cylindrical specimen with 46 mm diameter and 100 mm height was used to carry out this test. A hole with a diameter of 12 mm and 80 mm depth was bored at the centre of the specimen to model a fracture grouting method, which was injected with the geopolymer by applying a pressure of 100 kPa for 20 minutes. The research found that the higher the binder activator ratio, the lesser the viscosity obtained; therefore, the grout easily penetrated the pores between the soil particles. As a consequence, more volume of geopolymer could be injected in soil. Binder activator ratio lesser than 0.75 was ineffective for the injection method, and binder activator ration between 1.00 - 1.25 was recommended for the grouting injection method. A new equation to determine the grout diffusion was proposed based on this experiment, as written in the equation:

\[
\left( \frac{R_i}{R_0} \right)^{n/f/a} = K \left( \frac{V_i}{\pi \cdot R_0^2 \cdot L} \right)^{f/a}.
\]

1. Introduction
Clay shale is classified as a weak rock, with low durability due to the weathering process [1], controlled by the amount and type of macrocrack in the rock [2]. However, clays shale's strength tends to rapidly change when exposed to the wetting - drying or soaking – desiccating cycles. Furthermore, the desiccated clay shale's strength increases when the content is dry and decreases to a softened value when wet [3-6]. Supandi et al. [7] stated that the decrease in strength was rapid due to its exposure for more than 200 days. According to Stark and Duncan [4], Irsyam et al. [5], Miščević and Vlastelica [8], dam, road, and slope are some of the problems that cause serious damage in construction. In Indonesia, the problem raised from clay shale disintegration was more common due to the climatic condition [9]. Therefore, numerous efforts have been carried out to improve the clay shale's durability and strength, one of which is cement stabilisation [10-13]. Previous studies focused mainly on determining effective cement content and the mixing method to improve the properties of the clay shale. However, the significant use of cement increases the environmental impact on carbon dioxide emissions. Initially, geopolymers were developed for concrete; however, it is now used in place of cement for ground improvement[14-16]. Geopolymers are inorganic polymers consisting of aluminosilicate elements,
which are cement-like material consisting of alkali-activated materials [17]. The most common precursors used in geopolymer synthesis are fly ash with low calcium content and calcined clays, such as metakaolin. The alkaline activators (NaOH, or KOH) mixed with sodium silicate (Na$_2$SiO$_3$) or potassium silicate (K$_2$SiO$_3$) can react effectively with the amorphous SiO$_2$ and Al$_2$O$_3$ in fly ash to form binder N-A-S-H during the geopolymerisation phase [18].

The use of geopolymers has been studied for grouting in deep soil mixing [14,16,19-21]. However, studies on clay shale have not yet been conducted. Therefore, it is a viable option to enhance disintegrated or degraded strength. A non-degraded clay shale has a low porosity of less than 20% [22], which increases to 54% after degradation [23]. Therefore, geopolymer grout in clay shale fills the fracture of clay shale layers, with numerous factors, such as water to binder ratio, porosity, and pressure responsible for the success of grout injection [24-25]. These factors also control the uniformity or spreading area of the geopolymer grout during the injection. This study investigates the effect of soil porosity and binder activator ratio on spreading the geopolymer injection in a remoulded clay shale. A model of fracture grouting method was applied in the laboratory with the geopolymer viscosity represented by the ratio of fly ash and alkali activator, namely binder activator ratio (f/a).

2. Experiment Method

2.1 Materials

The clay shale was taken from Limbasari Purbalingga, Central Java, as shown in figure 1, while Table 1 presents its index properties. The soil consisted of almost 93% fines, which are identified as low plasticity clay (LL < 50%, and PI > 0.73(LL-20)). From the XRF test, the main chemical elements of clay shale were SiO$_2$ (53%), Al$_2$O$_3$ (20%), CaO (15%), Fe$_2$O$_3$ (7%), and K$_2$O (1, 6%), as shown in table 2. The fly ash was collected from the coal power plant of PJB Tanjungjati in Jepara, Central Java, with the chemical composition shown in table 2. Geopolymers was made of fly ash (type F) as alkali binder and alkaline activator solution dissolved in Na$_2$SiO$_3$ and NaOH(10M) with a ratio of 1:1. Furthermore, the binder activator ratios (f/a) used were 0.50; 0.75; 1; and 1.25. Table 3 presents the unit weight of geopolymers.

![Figure 1. Clay shale from the location.](image-url)
| Parameter                          | Value |
|-----------------------------------|-------|
| Specific gravity, G_s             | 2.52  |
| Maximum dry density, MDD (kN/m^3)| 15.2  |
| Optimum moisture content, OMC (%) | 18.6  |
| Liquid limit, LL (%)              | 44.4  |
| Plastic limit, PL (%)             | 23.9  |
| Plasticity index, PI (%)          | 20.5  |
| Fines fraction (%)                | 92.9  |
| Coarser fraction (%)              | 7.10  |

**Table 2. Chemical elements of clay shale and fly ash.**

| Materials  | SiO_2 | Al_2O_3 | Fe_2O_3 | CaO | MgO | K_2O | SO_3 |
|------------|-------|---------|---------|-----|-----|------|------|
| Clay shale | 53    | 20      | 7       | 15  | 1.4 | 1.6  | 1.1  |
| Fly ash    | 51    | 29      | 11      | 4.7 | 1.6 | 0.9  | 0.8  |

**Table 3. Unit weight of geopolymer.**

| Binder activator ratio (f/a) | Unit weight (kN/m^3) |
|------------------------------|----------------------|
| 0.5                          | 19.5                 |
| 0.75                         | 18.8                 |
| 1.0                          | 18.1                 |
| 1.25                         | 17.6                 |

2.2 Specimens Preparation and Testing Procedures

The clay shale fragments were dried, pulverised, and passed through no. 4 mesh (< 4.75 mm). In addition, a PVC tube of 46 mm in diameter and 100 mm in height was used to prepare the specimens in three variations of density (R_c), namely 75%, 85%, and 95% of the MDD. The dry soil was mixed with water at its OMC with a significant amount of slurry gradually poured in the PVC tube and statically compacted using a hydraulic pressure jack. After compaction, the specimen was extruded, with the weight and sizes measured to evaluate its density. The specimen was then put in the injection tube, as shown in figure 2a. A hole with a diameter of 12 mm and 80 mm depth was bored at the centre to model a fracture grouting method, as shown in figure 2b. The geopolymer was injected into the hole by applying a pressure of 100 kPa for 20 minutes, which is suitable for producing a grouted column, as shown in figure 2c. The injected grout volume (V_i) was used to estimate the spread of geopolymer injection (R_i), as written in equation (1).

![Figure 2](image-url)
\[ R_i = \sqrt{\frac{V_i - \pi \cdot L \cdot R_b^2}{n \cdot \pi \cdot L} + R_b^2} \] 

where \( R_i \) is the radius of grout spread, \( n \) is the specimen porosity, \( R_b \) and \( L \) are the radius and depth of the injection hole, respectively. Figure 3 presents a schematic illustration of the process used to determine the radius of grout spread. The \( V_i \) was determined by subtracting the weight before and after injection, divided by the geopolymer's unit weight.

Figure 3. A schematic illustration to determine the radius of grout spread.

During injection, the grout intrudes into the surrounding soil pores and bound its particles to form a column. In this research, the volume of grout column (\( V_{cg} \)) was determined by two methods. The first is the jar test (method B), which was carried out by immersing the specimen in water for 20 minutes. During this process, the grout column remained stable, while the unstabilised fragment degraded, with the \( V_{cg} \) determined by measuring the weight in water. The second method was conducted by applying a phenolphthalein solution (method C) to determine the carbonation in cement-treated sand [26]. The specimen was cut at a depth of 25, 50, 75, and 95 mm with the surface of each depth uniformly sprayed by phenolphthalein, as shown in figure 4a. The area containing alkaline reacts with the geopolymer, and the colour turns to magenta, as shown in figure 4b. This colour change indicates the presence of alkalinity reaction in the surrounding soil mass. The magenta area was detected by a colours filtering method in ImageJ software, with figure 4c indicating the image's readiness to be analysed using image processing techniques. The \( V_{cg} \) is the sum of volume at each cutting depth, with the grout column (\( R_{cg} \)) radius calculated from \( V_{cg} \), where the length equals the hole depth (\( L = 80 \) mm). The \( R_{cg} \) was assigned as an average radius of grout stabilisation.

Figure 4. Measurement of injection spreading (a) cutting surface of the specimen, (b) colour change after spraying phenolphthalein, (c) colour filtering by image processing method.
3. Results and Discussion

3.1 Effect of Binder Activator Ratio and Soil Porosity on the Grout Diffusion
This research was carried out to determine the amount of geopolymer injection spread in the surrounding soil. A series of the test was performed on the different porosity \((n)\) and binder activator ratio \((f/a)\), with the specimen porosity calculated from the obtained dry density. The corresponding porosity was 0.54, 0.48, and 0.42 for 75%, 85%, and 95% of the MDD, respectively, with the injected grout volume, \(V_i\), shown in table 4. In general, the volume of grout injected in the soil increases with a rise in soil porosity and the binder activator ratio. Furthermore, a rise in \(f/a\) indicates that the geopolymer was less viscous, therefore, the grout easily flowed in pores between the soil particles. Consequently, more volume of grout can be injected in soil with the distance of injection calculated in a radial direction \((R_i)\) using equation (1). For the given \(V_i\) in table 4, the correlation among the \(R_i\), \(n\), and \(f/a\) is shown in figure 5, with the correlation represented by multiple linear equations (2). The variance analysis shows that the \(f/a\) \((p < 0.0001)\) is more significant than \(n\) at a significant value of 0.0002 to affect the \(R_i\).

In a geopolymer, activator solution is the second most important component capable of influencing the kinetics of the reaction [27]. A higher binder activator ratio increased the polymerisation that plays an essential role in the kinetic, structure, and composition of an initial gel [17]. Murmu et al. [28] reported that the use of \(f/a > 1.5\) is not recommended because it decreases the strength of the geopolymer stabilised soil. Theoretically, equation (1) shows that the volume of grout injection and soil porosity has an essential influence on the geopolymer spread. The other investigator indicated the effect of grouting volume on the injection distance [29].

\[
R_i = 4.63 f/a + 12.89n \tag{2}
\]

Table 4. The volume of geopolymer grout injection in specimen.

| Binder activator ratio | \(V_i\) (mm\(^3\)) at various soil porosity |
|------------------------|------------------------------------------|
| \(f/a\)               | \(n = 0.42\) | \(n = 0.48\) | \(n = 0.54\) |
| 0.50                   | 12,328      | 13,127       | 15,084       |
| 0.75                   | 11,877      | 16,456       | 18,918       |
| 1.00                   | 15,189      | 17,592       | 25,027       |
| 1.25                   | 17,851      | 22,667       | 27,768       |

3.2 Soil-Grout Column Profile
A general product of grout injection is a soil-grout column due to the cementation reaction between the soil and grouting materials, as shown in figures 6 and 7. The dimension of the soil-grout column represents the distance of grout spread and uniformity of injection. When a grout with a lower \(f/a\) ratio was used (\(f/a\) of 0.5) as shown in table 4, less penetration into the soil pores occurs due to its high viscosity, which remains instead a homogeneous mass. The success effect of grouting influences the injectability of the chemical slurry, pore size, and porosity of the surrounding soil [30]. Table 4 shows that specimens with higher porosity have a larger volume of the soil-grout column.

Furthermore, the column profile in figures 6 and 7 indicates that a higher binder activator ratio leads to a wider soil-grout column formation. Ghadir and Ranjbar [15] reported that the chemical reaction between geopolymer and soils is depended on the activator concentration and binder content. Therefore, higher mechanical strength is obtainable at a higher pH where the alkali activator increased. At this phase, the volume of soil that reacted with a geopolymer was higher and made a larger soul-grout column, therefore, its strength increased with a rise in column size [16,21]. The silica and alumina content in clay shale enhances the geopolymerization process shown in table 2. Canakci et al. [16] determined the increasing ratio of Si/Al in the geopolymer mixture, which generally leads to a rise in strength.

Figures 6 and 7 show the soil-grout column's profile section measured by Method B (jar test) and C (phenolphthalein solution). The soil-grout columns measured from jar test and phenolphthalein solution are defined as approximate appearance columns, respectively. The calculated volume of each soil-grout columns \((V_{cg})\) is shown in table 5, with the volume 13 percent lesser than the approximate column.
\( V_{cg(\text{Method } B)} = 0.87 \ V_{cg(\text{Method } C)} \) with a correlation coefficient \( r^2 = 0.89 \). The result indicates that the phenolphthalein solution can be applied to determine grout spread in soil, however, this method is limited to the use of laboratory investigation for a small specimen.

**Figure 5.** Radius of injection spread with various \( f/a \) and \( n \).

**Figure 6.** Soil-grout column profiles with soil porosity \( n = 0.42 \) (a) \( f/a = 0.5 \) (b) \( f/a = 0.75 \) (c) \( f/a = 1.0 \), and (d) \( f/a = 1.25 \).

**Figure 7.** Soil-grout column profiles with soil porosity \( n = 0.54 \) (a) \( f/a = 0.5 \) (b) \( f/a = 0.75 \) (c) \( f/a = 1.0 \), and (d) \( f/a = 1.25 \).
Table 5. The volume of geopolymer grout injection in the specimen.

| Binder activator ratio | Method | $V_{cg}$ (mm$^3$) at various soil porosity | $n = 0.42$ | $n = 0.48$ | $n = 0.54$ |
|------------------------|--------|------------------------------------------|------------|------------|------------|
| 0.50                   | B      | 25,229                                   | 26,232     | 29,322     |            |
|                        | C      | 22,751                                   | n/a        | 30,690     |            |
| 0.75                   | B      | 39,721                                   | 48,233     | 48,048     |            |
|                        | C      | 41,631                                   | n/a        | 72,432     |            |
| 1.00                   | B      | 62,055                                   | 73,687     | 88,197     |            |
|                        | C      | 72,722                                   | n/a        | 109,282    |            |
| 1.25                   | B      | 80,980                                   | 86,643     | 99,319     |            |
|                        | C      | 72,624                                   | n/a        | 112,128    |            |

In general, the figures show that the soil-grout column was formed in a larger size as an increase in binder activator ratio. The column size indicates the spreading of grout injection in soil, therefore, there is a correlation between the injected grout volume ($V_i$) and the radius of the soil-grout column ($R_{cg}$) as shown in figure 8a. This empirical correlation has a coefficient value of 0.86 ($r^2 = 0.74$). However, the correlation has not considered the other influence variable, such as soil porosity ($n$), binder activator ratio ($f/a$), diameter ($R_b$), and depth ($L$) of the injection hole. Therefore, figure 8b enhanced the correlation with non-dimensional variables as given in equation (3) to (5).

Figure 8. (a) Correlation between the volume of grout injection and radius of the soil-grout column, (b) Normalised correlation as a function of $n, f/a, R_b, L$.

\[
R \approx V
\]

\[
\left( \frac{R_i}{R_b} \right)^{n \cdot f/a} = K \left( \frac{V_i}{V_b} \right)^{n \cdot f/a}
\]

\[
\left( \frac{R_i}{R_b} \right)^{n \cdot f/a} = K \left( \frac{V_i}{\pi \cdot R_b^2 \cdot L} \right)^{n \cdot f/a}
\]

Where $K$ is a constant of 1.12 obtained from empirical correlation, as shown in figure 8b. The equation was developed from a theoretical basis, which means that when the soil is impervious ($n \approx 0$) or the grout material is near solid ($f/a \approx 0$), it cannot spread wider than the injection hole, $R_{cg} = R_b$. The
proposed equation (5) provides a better correlation than equation (1) therefore, mathematically, it has the ability to resolve the singularity in equation (1) assuming the soil is impervious ($n = 0$). However, the grout diffusion was also controlled by injection pressure [30] and soil fracture [25], which was not involved in equation (5).

4. Conclusion

In conclusion, the laboratory investigation on fly ash geopolymer uses to stabilise weathered clay shale has been preliminarily performed and discussed in this study. The research showed that the grout volume injected in the soil increases with a rise in soil porosity and the binder activator ratio. Furthermore, the higher the binder activator ratio, the lesser the viscosity obtained. Therefore, the grout is easily penetrated in pores between the soil particles, thereby allowing more geopolymer volume into the soil. Binder activator ratio lesser than 0.75 was ineffective for the injection method, while those between 1.00 - 1.25 were recommended. Subsequently, the variance analysis shows that the binder activator ratio was more significant than porosity affecting the grout diffusion. A new equation to determine the grout diffusion was proposed based on this experiment, as written in equation (5)

$$\frac{R}{R_b} = K \left( \frac{V}{\pi \cdot R_b^2 \cdot L} \right)^{a/f/a}.$$ 

5. References

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