



Effect of curing temperature on the soil physical and mechanical properties on clay shale geopolymer fly ash stabilization



Edi Hartono^{1*}, Willis Diana¹, Agus Setyo Muntohar¹, Muhammad Azhar², Lia Indriani¹

¹Department of Civil Engineering, Faculty of Engineering, Universitas Muhammadiyah Yogyakarta, Indonesia

²Department of Road and Bridge Construction Engineering, Politeknik Negeri Tanah Laut, Indonesia

Abstract

Clay shale will have a low strength and durability when exposed to weathering. The properties can be improved by geopolymer stabilization. Geopolymer is the latest method in recent years, and it shows its economic benefits and lower carbon footprint. This study utilizes fly ash-based geopolymer to examine the effect of curing temperatures on the physical modification and strength behavior. The clay shale was taken from the road-side slope of the Bawen-Semarang toll road. The geopolymer was made of fly ash and an activator. The activator ratio was 1:2 for sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃), diluted with water to create a 12 M solution. All specimens underwent a seven-day curing period. Prior to conducting the unconfined compressive strength test, the specimens were subjected to various curing temperatures from 26°C to 60°C. The test result shows that, in general, the soil density increased with the temperature but the soil moisture and volume decreased. The dry density exhibited an increase from 1.66 g/cm³ to 1.84 g/cm³, while the unconfined compressive strength multiplied about 3.5 times. Meanwhile, the moisture content decreases from 19% to 2.5% after curing. The results led to the specimen volume experiencing decrement due to the shrinkage during the curing period. The volume reduces from 67.7 cm³ to 63.5 cm³. In general, temperature play a significant role in enhancing the strength of clay shale stabilized with fly ash-based geopolymer.

This is an open access article under the [CC BY-SA](https://creativecommons.org/licenses/by-sa/4.0/) license



Keywords:

Clay Shale;
Curing Temperature;
Fly Ash;
Unconfined Compressive Strength;

Article History:

Received: January 26, 2024
Revised: February 2, 2024
Accepted: April 25, 2024
Published: January 1, 2025

Corresponding Author:

Edi Hartono
Civil Engineering Department,
Universitas Muhammadiyah
Yogyakarta, Indonesia
Email: edi.hartono@umy.ac.id

INTRODUCTION

Many constructions on clay shale pose serious problems because of the rapid-down of the strength when exposed to the environment. Clay shale comprises 50% to 70% sedimented rock formed mainly by clay minerals and mixed with claystone, soil, dust, or rock with cementation [1]. Clay shale is a highly sturdy sedimentation rock. However, clay shale is highly susceptible to degradation when exposed to atmospheric conditions or subjected to moisture from water [2]. The degradation altered the geotechnical characteristics and reduced the strength and durability of clay shale [2, 3, 4, 5, 6].

In a construction project, slake deterioration on the surface becomes a major problem in the future when the clay shale is exposed due to the excavation [6]. Alatas et al. [4] reported a landslide event caused by the deterioration process at the Sports Training Center in Hambalang Sentul and on the Semarang-Bawen toll road, precisely, at Semarang-Bawen toll road KM. 32+000, the clay shale was exposed to the atmosphere due to the slope-cutting works, leading to a collapse on the clay shale slope.

Chemical stabilization is widely adopted to enhance the physical and geotechnical characteristics of problematic soils. The traditional soil improvement approaches involve replacing

the in-situ soil with materials that has suitable properties, such as concrete, or utilize mechanical reinforcement with geogrids or geotextile [7]. Aside from the conventional method, the soil stabilization method arises by mixing the soil with poor engineering properties with cementitious material to enhance soil strength and ductility, decrease swelling potential, permeability, deformation, and settlement, and increase the weathering resistance [1, 2, 8, 9]. The cementitious material will initiate chemical reactions for cation exchange, carbonation, and pozzolanic activity, thereby enhancing the overall structure of the soil [7][8].

Recently, geopolymers have become a popular option as an environmentally friendly material for improving soil properties. Murmu et al. [10] utilized a 5M NaOH solution mixed with fly ash geopolymer as a material to stabilize black soil. The results demonstrated that ash-based geopolymer is a viable option for stabilization, particularly in preparing highway subgrade and sub-base. Similar findings were reported by Nguyen and Phan [11], who employed geopolymer for soil stabilization in road construction. Compared to cement, fly ash exhibits a significantly lower global warming potential (GWP), with cement ranging from 0.82 to 0.948 kg CO₂ eq/kg, while fly ash ranges from 0.00526 to 0.027 kg CO₂ eq/kg [10]. Nath et al. [12], reported a 36-43% reduction in carbon footprint by replacing Portland cement with fly ash. Furthermore, fly ash-based geopolymer cement production is reported to have a lower carbon footprint, up to 25% less than that of Portland cement [13].

Fly ash has been utilized as a stabilization material for clay shale, and numerous researchers have studied it with different variables and methods [14, 15, 16]. Sumiyanto et al. [14] employed an injection method to stabilize clay shale using a fly ash-based geopolymer. A mixture of fly ash and alkali was injected into compacted clay shale, resulting a remarkable fivefold enhancement in the unconfined compressive strength. A chemical is needed to activate the fly ash. The activator type, quantity, and condition are essential in stabilizing the soil. Fly ash with a calcium content <5% (classified as fly ash type F by ASTM) is unable to form a cementitious product [17]. In contrast to type F fly ash, type C fly ash has high amounts of calcium and produces cementitious products [18]. Cristelo et al. [19] used sodium silicate (SiO₂) and sodium hydroxide (NaOH) as activators for type C and F fly ash to stabilize marlstone, which contains high calcium carbonate

Besides fly ash type, other factors such as geopolymer content, molarity, alkali ratio, and curing temperature play crucial roles in determining the strength of soil stabilized with fly ash-based geopolymer [20]. Hartono et al. [16] investigated the concentration of alkali used as an activator for fly ash-based geopolymer. The study suggests that 12-15M Na₂SiO₃+NaOH concentration is optimal for stabilizing clay shale. However, the correlation between curing temperatures and the performance of clay shale stabilized using fly ash-based geopolymer has not been analyzed. Mapping the interrelation among the influencing factors can specify a more effective stabilization method. This study aims to employ a fly ash-based geopolymer for the stabilization of clay shale and assess the impact of varying curing temperatures on the unconfined compressive strength.

RESEARCH METHOD

Soil Sample

The clay shale used in this study was sourced from the roadside slope of the Bawean-Semarang toll road in Central Java. This type of clay shale has a high mechanical property; however, when exposed to water, it quickly degrades. The soil was collected in large boulders and fractured into smaller fragments until passing through sieve No. 4. Figure 1 shows the clay shale used in the present experiment. The X-ray diffraction indicated that smectite dominated the clay minerals of the soil sample, followed by Illite, Kaolinite, and Chlorite [2]. The soil consists of 93% silt/clay and 7% sand. The particle size distribution of clay shale sample used in this study is shown in Figure 2.



Figure 1. The Clay from Bawean-Semarang Toll Road, Central Java

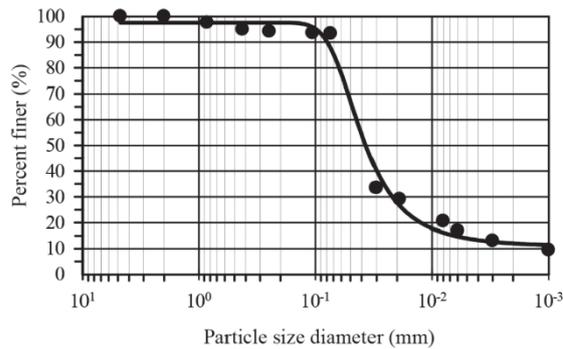


Figure 2. Grain Size Distribution of Clay Shale

The soil is classified as CH based on the Unified Classification System (USCS), with plastic and liquid limits of 57.9% and 28.4%, respectively. Table 1 presents the properties of the tested soil.

Fly Ash and Alkali Activator

A fly ash-based geopolymer is used as a stabilization material to replace cement. The fly ash, a by-product of coal combustion from coal-fired power plants, is classified into N, F, and C types according to ASTM C 618-22 [17]. Fly ash type F is often chosen as a stabilization material due to its pozzolanic properties, containing more than 70% $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ and less than 10% CaO. This study considered using the F-type Fly Ash since it is widely used as a stabilization material for soil; thus, it will be easier to provide in bulk. A combination of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) was used for the activator. The activator mixture is diluted with water to create a 12M mixture, as suggested by Hartono et al. [16]. The ratio of NaOH to Na_2SiO_3 for the activator is 1:2 for each specimen made.

Specimen Preparation

Four groups of specimens were prepared to evaluate the effect of curing temperature. The specimens were prepared by using NaOH and Na_2SiO_3 with a 1:2 ratio as activators. The details of the samples are listed in Table 2.

Table 1. Soil Properties of Clay Shale

Soil Properties	Values
Specific gravity, G_s	2.65
Atterberg limits:	
Liquid limit, LL	58%
Plastic limit, PL	28%
Plasticity index, PI	30%
Grand size distribution	
Sand	7%
Silt/Clay	93%
Optimum moisture content, OMC	19%
Maximum dry density, MDD	1.66
	(g/cm^3)

Table 2. The sample details.

Sample Code	A26	A40	A50	A60
Activator Ratio (NaOH/ Na_2SiO_3)	1:2.0	1:2.0	1:2.0	1:2.0
Molarity	12	12	12	12
Curing Temperature ($^{\circ}\text{C}$)	26	40	50	60
Curing Time (days)	7	7	7	7
Number of Specimens	3	3	3	3

The two numbers represent the curing temperature. Each sample was treated with a different curing temperature. The curing temperatures were variously set from room temperature (26°C), 40°C , 50°C , and 60°C . The sample curing time lasts for seven days.

A boulder of clay shale was crushed until it passed through sieve No. 4 and mixed with fly ash (15% of the total weight). The activator was then added to the mixture (19% of the total weight, considering the optimum moisture content of natural clay shale). The soil-geopolymer mixture was then compacted into a cylindrical mold of 76 mm in height and 38 mm in diameter with a dry density of $1.66 \text{ g}/\text{cm}^3$. After compacting, the specimen was dismantled from the mold and cured in a temperature room for seven days.

Unconfined Compressive Strength Test

The unconfined compression strength (UCS) [21] test was used to determine the mechanical properties of the stabilized soil. After seven days of curing, the specimen was tested on a compression machine. The specimen was loaded until it reached maximum load and failure. The shearing rate of loading was $0.76 \text{ mm}/\text{min}$. The unconfined compressive strength of the sample q_u is calculated by dividing the maximum axial load (F_{max}) by the shearing test by the cross-section area (A), as shown in (1).

$$q_u = \frac{F_{max}}{A} \quad (1)$$

RESULTS AND DISCUSSION

Effect of temperature on the physical properties

Figure 3 shows the condition of the samples after seven days of curing. The dry density of soil increases with the increase of curing temperature. A noticeable increment is shown between the room temperature curing (26°C) and 40°C with $0.08 \text{ g}/\text{cm}^3$. However, the increase until the sample reaches a 60°C curing temperature. The result indicates that the maximum density in this test series is obtained after curing the sample at 60°C . Specimens with higher curing temperatures have a lower moisture content after the curing process, leading to specimen shrinkage, thus reducing the volume.

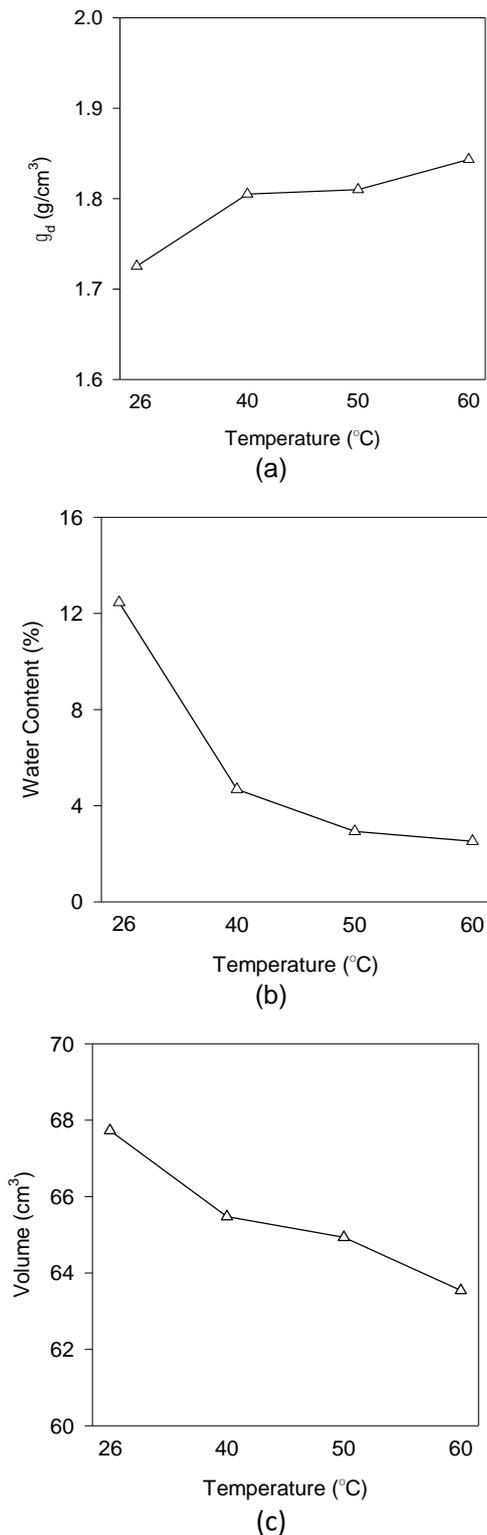


Figure 3. Variation of physical properties of soil stabilized with fly ash-based geopolymer with temperature change (a) dry density, (b) water content (c) volume

The initial average water content of samples is 19% and decreased to 12.5%, 4.7%, 2.9%, and 2.5% for the curing temperatures of 26°C, 40°C, 50°C, and 60°C respectively. Furthermore, the volume is decreased to 67.7 cm^3 , 65.5 cm^3 , 64.9 cm^3 , 63.5 cm^3 for 26°C, 40°C, 50°C, and 60°C respectively.

The decrement rate tends to be gentler as the temperature increases. The most significant moisture content drops between 26°C and 40°C are inversely proportional to the dry density results. The average density increased from 1.66 g/cm^3 to 1.84 g/cm^3 . Similar behavior is also observed in volume changes where the drastic shrinkage happens between 26°C and 40°C. After 40°C, no more significant changes were observed.

Effect of temperature on the unconfined compressive strength

Figure 4 shows the stress and strain curves from the UCS test. The UCS test results show almost identical results for each group of samples, with an exception for the samples cured at 50°C. In general, the q_u increases as the curing temperature increases. However, as shown in Figure 4(c), sample A50-1 has a more rapid increment in the strain for the early loading stage. The peak stress reached 5.8% of strain, the highest value among all specimens. The average q_u is 2.67 MPa, 6.32 MPa, 8.79 MPa, and 9.28 MPa for specimens A26, A40, A50, and A60. The q_u increases multiply about 3.5 times by increasing the temperature from 26°C to 60°C.

Discussion

As shown in Figure 3, the dry density increased as the curing temperature increased. The increment of soil dry density is related to polymer gel formation, which bonds the soil particles and fills the pores and cracks. It is also possible that the loss of moisture content in the polymerization reaction, which becomes more active in higher temperatures, leads to more rapid moisture loss due to evaporation and causes a volumetric shrinkage [22]. Both polymerization and evaporation cause the sample to become denser.

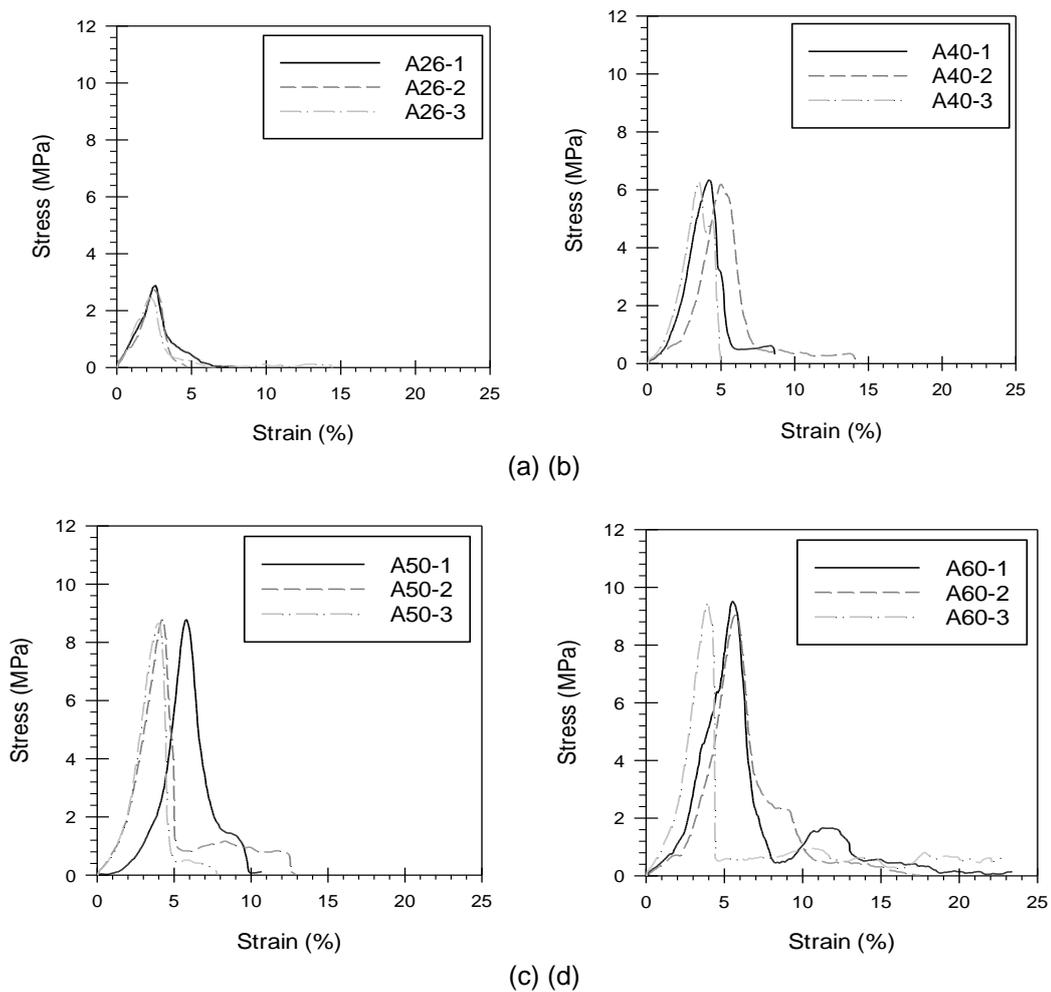


Figure 4. The UCS test result for (a) sample curing at 26°C, (b) sample curing at 40°C, (c) sample curing at 50°C, and (d) sample curing at 60°C.

The process mentioned above is also responsible for the enhancement of mechanical properties. The UCS test results in Figure 4 show that the increment q_u has the same trend as the increment of curing temperature. Leong et al. [23] and Phetchuay et al. [24] mentioned that treating fly ash-based geopolymer in an environment with higher temperatures will lead to a more rapid polymerization reaction in the early stage, resulting in higher early strength. Dissimilar to cement usage as a stabilization material, geopolymers do not form calcium-silicate-hydrates in the soil matrix. Geopolymers utilize an endotherm reaction that absorbs heat from the environment, resulting in polycondensation. Polycondensation is the polymer formation process involving silica and alumina, which leads to the bonding between small molecules and forming alumina-silicate-hydrates. A higher curing temperature and more silica and alumina available to react will lead to more polymerization, thus

increasing the number of polymer gels and the bonding strength [25].

Higher curing temperatures increase the availability of reactive silica and alumina, accelerating polymer gel formation and enhancing the bonding strength of the geopolymer matrix [26]. This improvement leads to increased unconfined compressive strength (UCS) of the treated soil. Optimizing curing conditions is essential for maximizing the benefits of geopolymer stabilization, with ongoing research focusing on the effects of additives and methods for sustainable construction applications [27].

The curve shapes are almost identical, with a larger strain at the peak point for samples prepared under different temperatures. However, this trend is negated in samples cured at 50°C. Moreover, specimen A50-1 shows a more considerable deformation before reaching the peak stress, and it fails.

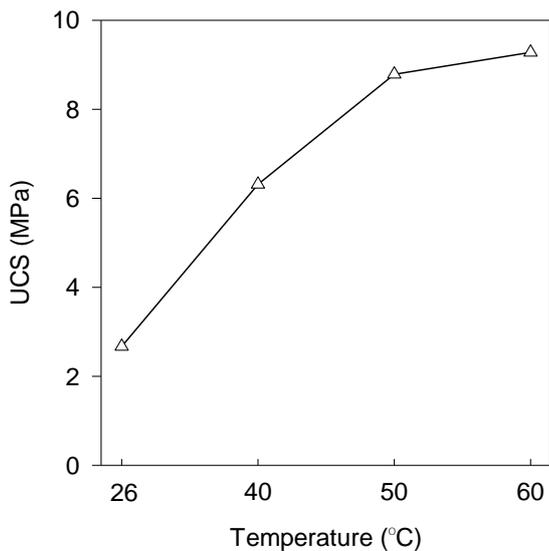


Figure 5. Variation of q_u with temperature

Compared to specimen A50-2, specimen A50-1 has a higher dry density, smaller volume, and smaller water content. In this case, the difference in the initial stiffness might be related to the cracks formed during the curing process. Although many cementitious bonds are formed, the rapid moisture loss during curing also makes the sample prone to tension cracks during shrinkage. These cracks were compressed in the initial loading stage, leading to a larger strain. However, the re-structured dried clay shale and the weakened cementitious bonds could still withstand the applied load, even though the additional NaOH do not produce any noticeable positive influence. In addition, the variation in stress-strain behavior might also happen due to the non-optimal mixing process of soil-fly ash-alkali, leading to a non-homogenic mixture.

In general, as summarized in Figure 5, temperature plays a significant role in enhancing the strength of clay shale stabilized using fly ash-based geopolymer. The incrementation of curing temperature shows a stagnancy in the strength development at 50°C or higher. A more rapid polymerization demands more silica and alumina to react. Thus, more activators are required to achieve greater strength. It should be noted that this outcome results from an early stabilization period. The performance of the samples might be different in the later stage after curing (up to 28 days).

CONCLUSION

The study investigates the effect of curing temperature and alkali activator ratio on clay shale stabilization. The unconfined compression test has been successfully conducted. Based on the

result and discussion in the earlier section, several notes can be pointed out as follows:

1. The dry density of soil is positively correlated with temperature. There is a significant increase of 0.08 g/cm^3 in density between the curing process at ambient temperature (26°C) and 40°C. Nevertheless, the temperature gradually rises until it reaches a curing temperature of 60°C. The test results demonstrate that the highest density in this series of tests is achieved by curing the sample at 60°C.
2. Higher curing temperatures lowered the moisture content after the curing process, leading to specimen shrinkage and thus reducing the volume.
3. The unconfined compressive strength increases as the curing temperature increases. The q_u increases multiply about 3.5 times by increasing the temperature from 26°C to 60°C.

ACKNOWLEDGMENT

This research is a part of the Fundamental Research Grant with contract number 181/E5/PG.02.00.PL/2023. The Ministry of Education, Culture, Research and Technology, the Republic of Indonesia has financially supported the grant. The assistance from the Soil Mechanics Laboratory of the Universitas Muhammadiyah Yogyakarta is highly appreciated.

REFERENCES

- [1] W. Diana, E. Hartono, and A. Muntohar, "The permeability of Portland cement-stabilized clay shale," in *IOP Conference Series: Materials Science and Engineering*, 2019, vol. 650, no. 1: IOP Publishing, p. 012027, doi: 10.1088/1757-899X/650/1/012027
- [2] E. Hartono, S. P. R. Wardani, and A. S. Muntohar, "The behavior of the clay shale stabilized by dry and wet cement mixing method," *Journal of GeoEngineering*, vol. 16, no. 3, 2021, doi: 10.6310/jog.202109_16(3).1
- [3] P. Agung, B. MA Damianto, and Y. Yuwono, "A critical state approach to stability of clay shale for design structures of the Sentul hill, west Java, Indonesia," in *EACEF-International Conference of Civil Engineering*, 2013, vol. 1, no. 1, pp. 027-027, https://eacef.com/?page_id=1383
- [4] I. M. Alatas, S. A. Kamaruddin, R. Nazir, M. Irsyam, and A. Himawan, "Shear strength degradation of Semarang Bawen clay shale due to weathering process," *J. Teknol*, vol. 77, no. 11, pp. 109-118, 2015, doi: 10.11113/jt.v77.6429

- [5] S. K. Haldar, *Introduction to mineralogy and petrology*. Elsevier, 2020, doi: 10.1016/C2019-0-00625-5
- [6] I. A. Sadisun, H. Shimada, M. Ichinose, and K. Matsui, "Study on the physical disintegration characteristics of Subang claystone subjected to a modified slaking index test," *Geotechnical & Geological Engineering*, vol. 23, pp. 199-218, 2005, doi: 10.1007/s10706-003-6112-6
- [7] L. Sharma, N. Sirdesai, K. Sharma, and T. Singh, "Experimental study to examine the independent roles of lime and cement on the stabilization of a mountain soil: A comparative study," *Applied Clay Science*, vol. 152, pp. 183-195, 2018, doi: 10.1016/j.clay.2017.11.012
- [8] P. G. Nicholson, *Soil improvement and ground modification methods*. Elsevier, Butterworth-Heinemann, 2015, doi: 10.1016/C2012-0-02804-9
- [9] E. Adeyanju and C. Okeke, "Clay soil stabilization using cement kiln dust," in *IOP conference series: materials science and engineering*, 2019, vol. 640, no. 1: IOP Publishing, p. 012080, doi: 10.1088/1757-899X/640/1/012080
- [10] A. L. Murmu, N. Dhole, and A. Patel, "Stabilisation of black cotton soil for subgrade application using fly ash geopolymer," *Road Materials and Pavement Design*, vol. 21, no. 3, pp. 867-885, 2020, doi: 10.1080/14680629.2018.1530131
- [11] D. Nguyen and V. Phan, "Engineering properties of soil stabilized with cement and fly ash for sustainable road construction," *International Journal of Engineering*, vol. 34, no. 12, pp. 2665-2671, 2021, doi: 10.5829/ije.2021.34.12c.12
- [12] P. Nath, P. K. Sarker, and W. K. Biswas, "Effect of fly ash on the service life, carbon footprint and embodied energy of high strength concrete in the marine environment," *Energy and Buildings*, vol. 158, pp. 1694-1702, 2018, doi: 10.1016/j.enbuild.2017.12.011
- [13] C. C. Chan, D. Thorpe, and M. Islam, "An evaluation carbon footprint in fly ash based geopolymer cement and ordinary Portland cement manufacture," in *2015 IEEE International Conference on Industrial Engineering and Engineering Management (IEEM)*, 2015: IEEE, pp. 254-259, doi: 10.1109/IEEM.2015.7385647
- [14] Sumiyanto, S. P. R. Wardani, and A. S. Muntohar, "Stabilisation Of Degraded Clay Shale With The Geopolymer Injection Method," *GEOMATE Journal*, vol. 24, no. 104, pp. 19-26, 2023, doi: 10.21660/2023.104.3666
- [15] Sumiyanto, S. P. R. Wardani and A. S. Muntohar, "The Effect of Soil Porosity and Geopolymer Viscosity on Spreading Grouting in Weathered Clay Shale," in *IOP Conference Series: Materials Science and Engineering*, 2021, vol. 1144, no. 1: IOP Publishing, p. 012095, doi: 10.1088/1757-899X/1144/1/012095
- [16] E. Hartono, W. Diana, and F. N. Bahti, "Unconfined compressive strength test on geopolymer fly ash stabilized clay shale," in *E3S Web of Conferences*, 2023, vol. 429: EDP Sciences, p. 04025, doi: 10.1051/e3sconf/202342904025
- [17] *C 618; Standard Specification For Coal Fly Ash And Raw Or Calcined Natural Pozzolan For Use In Concrete*, ASTM, 2019, doi: 10.1520/C0618-23E01
- [18] G. Ferguson, "Use of self-cementing fly ashes as a soil stabilization agent," *Fly Ash For Soil Improvement. ASCE Geotechnical Special Publication No. 36*, 1993, <https://trid.trb.org/View/408499>
- [19] N. Cristelo, S. Glendinning, L. Fernandes, and A. T. Pinto, "Effect of calcium content on soil stabilisation with alkaline activation," *Construction and Building Materials*, vol. 29, pp. 167-174, 2012, doi: 10.1016/j.conbuildmat.2011.10.049
- [20] M. M. A. B. Abdullah, H. Kamarudin, M. Bnhussain, K. N. Ismail, A. Rafiza, and Y. Zarina, "The relationship of NaOH molarity, Na₂SiO₃/NaOH ratio, fly ash/alkaline activator ratio, and curing temperature to the strength of fly ash-based geopolymer," *Advanced Materials Research*, vol. 328, pp. 1475-1482, 2011, doi: 10.4028/www.scientific.net/AMR.328-330.1475
- [21] *D 2166; Standard Test Method for Unconfined Compressive Strength of Cohesive Soil*, ASTM, 2016, doi: 10.1520/D2166_D2166M-16
- [22] P. Ghadir and N. Ranjbar, "Clayey soil stabilization using geopolymer and Portland cement," *Construction and Building Materials*, vol. 188, pp. 361-371, 2018 doi: 10.1016/j.conbuildmat.2018.07.207
- [23] H. Y. Leong, D. E. L. Ong, J. G. Sanjayan, and A. Nazari, "Strength development of soil-fly ash geopolymer: Assessment of soil, fly ash, alkali activators, and water," *Journal of Materials in Civil Engineering*, vol. 30, no. 8, p. 04018171, 2018, doi: 10.1061/(ASCE)MT.1943-5533.0002363

- [24] C. Phetchuay, S. Horpibulsuk, C. Suksiripattanapong, A. Chinkulkijniwat, A. Arulrajah, and M. M. Disfani, "Calcium carbide residue: Alkaline activator for clay–fly ash geopolymer," *Construction and Building Materials*, vol. 69, pp. 285-294, 2014, doi: 10.1016/j.conbuildmat.2014.07.018
- [25] A. Palomo, M. Grutzeck, and M. Blanco, "Alkali-activated fly ashes: A cement for the future," *Cement and concrete research*, vol. 29, no. 8, pp. 1323-1329, 1999, doi: 10.1016/S0008-8846(98)00243-9
- [26] L. O. Agashua, C. Arum, B. D. O. Ayibiowu, and C. M. Ikumapayi, "A Systematic Review Of Geopolymer Materials: Innovations, Prevailing Constraints And Resolutions," *SINERGI*, vol. 28, no. 3, pp. 473-488, 2024, doi: 10.22441/sinergi.2024.3.004
- [27] L. O. Agashua, C. Arum, B. D. O. Ayibiowu, and C. M. Ikumapayi, "Workability And Durability Analysis of Waste Based Geopolymer Concrete," *SINERGI*, vol. 28, no. 1, pp. 199-208, 2024, doi: 10.22441/sinergi.2024.1.020