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FLY-ASH-BASED GEOPOLYMER AS STABILIZER FOR SILTY SAND EMBANKMENT MATERIALS

*Jonathan R. Dungca[1] and Edward Ephrem T. Codilla II[2]

[1,2]Civil Engineering Department, De La Salle University, Philippines

*Corresponding Author, Received: 10 June 2017, Revised: 29 July 2017, Accepted: 10 Dec. 2017

ABSTRACT: Fly ash in the recent decades has become abundant resulting in the improper waste disposal. However, it is also known to be a precursor to the process of geopolymerization which is categorized under waste utilization. Geopolymer has become known for being on par with cement and other chemical stabilizers in terms of strength. It has been used in numerous studies focusing on concrete and on soil stabilization. This study proposes the use of fly-ash-based geopolymer as a stabilizer for silty sand embankment material wherein the process of synthesizing geopolymer is through the dry-mix method. The dry-mix method is another way of producing geopolymer, requiring the aluminosilicate precursors and alkali activators to be in its dry state, and adding water to the dry-mix would result in geopolymerization. The geopolymer was applied on Silty Sand to verify its reaction with the soil and to obtain the effective mix that would enable the soil to be stabilized for embankment use. The effectiveness of this method in soil stabilization was tested through the following tests, the CBR Test (ASTM D1833) and the UCS Test (ASTM D2166). Results have shown improvements of the geopolymer-stabilized soil in terms of the CBR index and the UCS value. The effective geopolymer concentration was found to be at 30% geopolymer concentration which produced the highest increase for the stabilized soil with a maximum CBR index of 34.32% and a maximum UCS value of 1349.74 kPa.

Keywords: Fly Ash, Dry-mix Geopolymer, Embankment Materials, Soil Stabilization

1. INTRODUCTION

In geotechnical works, a site is surveyed whether soil conditions meet the design criteria. However, most commonly, sites designated for earthworks do not reach the minimum standards, such as those with soft, highly compressible, or expansive soils lacking the desired strength for loading during construction or for their serviceability [1]. For this reason, such soils are enhanced through soil stabilization, wherein the mechanical properties of the soil are improved by applying materials that have cementitious properties or are considered to be binder materials [2]. The most common soil stabilizers used nowadays are OPC (ordinary Portland cement) and lime. However, due to the CO_2 (Carbon Dioxide) emissions produced during the manufacturing of aforementioned soil stabilizers, which contribute to greenhouse gases, other soil stabilizers are being sought out and recommended. Studies and tests are being conducted in synthesizing alternative and eco-friendly soil stabilizers from waste materials, which can allow a decrease in CO_2 emissions and, simultaneously decrease cost in earthwork construction [3], [4].

Fly ash is an industrial waste produced from the combustion of coal into fuel for power generation [5]. Globally, fly ash produced by factories and thermal power plants have been increasing for the past few years. The mass production of fly ash causes disposal problems and an increase in expenses for storage in available landfills [6], [7]. This eventually poses a threat to the environment if it is not properly managed [8]. Consequently, several studies [9], [10], [11], [12], [13], [14], [15], [16] have been conducted, which is aimed towards recycling fly ash and utilizing it in various applications.

An alternative to stabilizing soil is by introducing geopolymer. Geopolymer is a product of the alkali activation of aluminosilicate materials present in industrial waste materials such as fly ash, red mud, and furnace slag [1]. The waste material used in the study to synthesize geopolymer was fly ash.

2. MATERIALS AND METHODS

2.1 Soil

The soil used in the study was obtained from Taguig City. It has a specific gravity of 2.50. The soil passing the No. 40 sieve has a liquid limit (LL) of 26 and a plasticity index (PI) value of 4, which classifies the soil to be slightly plastic as per ASTM D4318. Referring to Fig. 1, the particle size analysis performed on the soil indicates that the soil has varying grain sizes since it is composed of 18% gravel, 55% sand, and 13% silty fines. According to the Unified Soil Classification System (USCS) and the American Association of

State Highway and Transportation Officials (AASHTO) Soil Classification System, the soil is classified as Silty Sand (SM) with Gravel and as Silty Gravel and Sand (A-2-4), respectively. The standard Proctor test was also performed on the soil to determine its maximum dry unit weight ($\gamma_{d,max}$) and optimum moisture content (OMC), which were 17.61 kN/m^3 and 15.15%, respectively.

To determine the corresponding AASHTO rating of the soil, the California bearing ratio (CBR) test was performed. The soil, with a CBR index of 1.68%, was rated as *Poor* for *Subgrade* use. The unconfined compression strength (UCS) test, however, could not be performed as the soil could not form UCS samples; these samples crumbled after being removed from the UCS molds. The two strength tests, UCS and CBR, were later on used as bases in verifying the soil stabilization caused by the geopolymer in comparison to the results of the unstabilized soil.

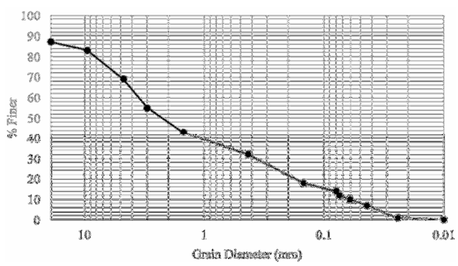

Fig.1 The particle size distribution of the soil

2.2 Fly Ash

The fly ash used in geopolymer synthesis was obtained from a coal-fired power plant in Mindanao. The X-ray Fluorescence (XRF) test was performed on the fly ash to determine its chemical compounds as shown in

Table 1. As per ASTM C168, it is classified as Class F. To test whether the fly ash is safe for use in the environment with regards to leaching, the Toxicity Characterization Leaching Procedure (TCLP) and Heavy Metal test was performed. Table 2 shows that the fly ash is a non-hazardous material as its heavy metal content does not exceed the permissible limits as per TCLP standards.

Table 1 XRF results of the fly ash

Chemical Compounds	Fly Ash (%)
Al_2O_3	26.50
SiO_2	36.50
Fe_2O_3	16.20
SiO_2 plus Al_2O_3 plus Fe_2O_3	79.30
SO_3	8.20
CaO	10.70

Table 2 TCLP results of the fly ash

Heavy Metals	Permissible Limit	Fly Ash
Cadmium	1.000	Not Detected
Chromium	5.000	Not Detected
Lead	5.000	Not Detected
Iron	0.300	0.06
Copper	1.000	0.02
Manganese	0.050	Not Detected
Zinc	5.000	0.008

2.3 Geopolymer Synthesis By Dry Mix Method

Soil stabilization should be practical when applied in the field. For geopolymer, this is solved by using the dry mix method. This method requires all materials to be in their dry state when initially mixed together. Afterwards, water is added to this mixture as the final step, which will result in geopolymerization.

The alkali activators used in the study were Sodium Hydroxide (NaOH) pellets and Sodium Silicate ($NaSi_2O_3$) pellets. In other studies [1], [17], [18], the optimal range of values for activators to fly ash ratio, $NaSi_2O_3$ to NaOH ratio, and NaOH concentration were obtained; the values applied in this study were 0.4, 14 M, and 2, respectively. Three geopolymer concentrations were tested to stabilize the soil. These were in the values of 10%, 20%, and 30% by percent weight of the sample batch.

Prior to preparing the specimens, the geopolymer paste was produced. In the synthesis of the geopolymer paste, the dry alkali activators and the fly ash were initially mixed together using a mixer. Afterwards, water was then added and mixed for approximately 10 minutes. The total water required was considered as the water required to reach the NaOH concentration (14 M) and the OMC (15.15%) of the soil. The said procedure is shown in Fig.2. This geopolymer paste was then added to the soil to produce that stabilized specimens.

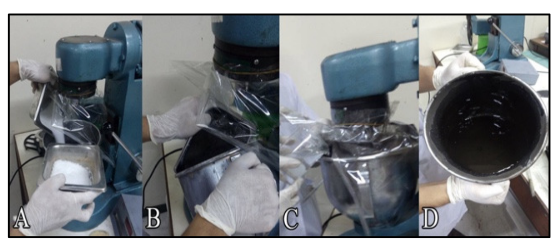

Fig.2 The dry mix method: (A) dry alkali activators, (B) fly ash, (C) water, (D) geopolymer paste

2.4 Soil Stabilization with Geopoloymer and Characterization Tests

2.4.1 UCS and CBR sample preparation and testing

Five samples were made for each geopolymer concentration; a total of 15 samples per strength test was synthesized. The UCS samples of the stabilized soil were compacted into cylindrical PVC molds (40 mm x 100 mm). Prior to testing, these were cured at room temperature for 28 days. For the CBR samples which were unsoaked, the stabilized soil was compacted into the CBR molds at 5 layers with 56 blows per layer to obtain a 95% of the desired $\gamma_{d,max}$ as per ASTM D1883. The CBR samples were cured for 7 days prior to testing.

2.4.2 Statistical analysis of mechanical properties of geopolymer stabilized soil

The statistical analysis of the data was performed in the Design Expert software v7.0. The partial sum of squares Type III analysis of variance (ANOVA) was applied to test the presence of a main effect between the interactions of the model terms in a model equation. The model order (linear, quadratic, or cubic) was selected based on the coefficient of determination (R^2) value; whichever model had the highest R^2 value indicated a great fit for the data points. Each term of the model equation was determined to be significant if its probability p (Prob > F) was less than 0.05. Moreover, the null hypothesis is focused on the lack of fit, as it is assumed that there is no lack of fit for the data points in a linear equation. Small p values, those that are less than 0.05, indicating that the null hypothesis is rejected and that a model of higher order is required. To verify the model equations, equality graphs were made and the correlation value (r) between the predicted and actual values was calculated.

2.4.3 Morphological characteristics

The SEM (scanned electron microscopy) imaging test was applied on the fly ash, the soil, and the samples of the stabilized soil. These were used to verify the formation of geopolymer gels since the dry mix method was used which could affect the rate of geopolymerization. Moreover, the stabilized soil with 28 days of curing was used in the SEM imaging test to better observe the formation of geopolymer gels when compared to the SEM images of the fly ash and the soil. Portions of each sample, with a mass of 10 to 15 grams, were oven-dried before being tested.

2.4.4 Fourier Transform Infrared (FTIR) spectroscopy

The FTIR was performed to further prove the geopolymerization in the dry-mix method. The samples used in the FTIR tests were the fly ash, the soil, the pure geopolymer, and the 30% geopolymer concentration stabilized soil. The results were used for comparison of the spectra of the samples to check for changes when geopolymer is applied to the soil.

3. RESULTS AND DISCUSSION

3.1 Mechanical Properties of Geopolymer-Stabilized Soil

In the UCS test, the soil being predominantly of sand could not be tested solely due to sand being cohesionless as shown in Fig.3. When geopolymer was applied starting at 10% geopolymer concentration, UCS samples were able to be produced; these samples started to have cohesive characteristics. The geopolymer acts as a binder for the sand particles and fills the gaps between these particles. Moreover, the geopolymer gel formation creates a network that binds the soil particles and fly ash particles together creating a stronger network of particles. Assessing the results in Table 3, the samples with 10% geopolymer concentration had a UCS (q_u) value of 78.29 kPa (Medium consistency), with 20% it increased to 247.48 kPa (Very Stiff), and with 30% it significantly increased to 1349.74 kPa (Hard).

Fig.3 The UCS samples of the pure soil.

Table 3 UCS testing results of the stabilized soil

Geopolymer Concentration	10%	20%	30%
Unconfined Compressive Strength, q_u (kPa)	78.29	247.48	1349.74
Consistency	Medium	Very Stiff	Hard

These q_u values are comparable to a study using the wet mix approach of synthesizing geopolymer stabilizers. This study [18] used slag-based geopolymer to stabilize clayey soil which resulted in a q_u value of 1223 kPa at 25% geopolymer concentration. Other studies [14], [15] which stabilized expansive soil using fly ash resulted to increase in q_u value by 200 to 1400 kPa. In comparison to a study which used cement stabilization, q_u values ranged from 500 kPa (5% cement) to 2150 kPa (20% cement) [19] for stabilized dredged sediments composed of clay and silt.

Considering that these were stabilized with cement, the fly-ash-based geopolymer can be considered to have an advantage over cost since fly ash is free as it is a waste material. At 30% geopolymer concentration, similar UCS values were obtained. Moreover, it is said that the hardness of geopolymer is twice as much higher than cement which indicates that geopolymer is less deformable and has a higher brittle behavior [20]. This behavior was actually observed for the stabilized soil at 30% geopolymer concentration wherein the cured UCS samples were physically similar to concrete during its UCS testing as shown in Fig.4.

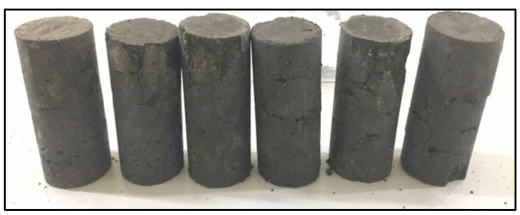

Fig.4 The stabilized soil samples at 30% geopolymer concentration after UCS testing

Similarly, the CBR index of the soil improved as it was stabilized with geopolymer. The CBR index of the stabilized soil increased as the geopolymer concentration increased as shown in Table 4. As previously discussed, the geopolymer creates a stronger soil structure since it acts as a binder between the fly ash and the soil. At 10% and 20% geopolymer concentration, the stabilized soil had CBR indices of 9.89% and 16.24%, respectively, which classifies it as subbase material. At 30% geopolymer concentration, the stabilized soil can be used as a base material as its CBR index has reached 34.32%. With this said, the highest increase in CBR index is 32.64% when compared to the pure soil, which is 1.68%. This is slightly similar to a local study [11] and, studies in Nigeria [12], Germany [13], Sudan [15], USA [16] which applied fly ash on several types of soil. These studies had results wherein the CBR indices increased by 30% to 55%. This suggests the possible use of the dry mix geopolymer for stabilizing soil based on the improved CBR indices of the pure soil as the improvements are similar to other studies.

Table 4 CBR results of the stabilized soil

Geopolymer	10%	20%	30%
CBR Index (%)	9.89	16.24	34.32
General Rating	Fair	Fair	Good
Uses	Subbase	Subbase	Base, Subbase

3.2 ANOVA Analyses and Geopolymer Equations

The factor in the equations is the geopolymer concentration ranging from 0% to 30%, wherein the 0% value is considered as the pure soil. The independent variables are the strength properties which are q_u and CBR Index.

$$q_u = 3135.75G - 37564.80G^2 + 140365G^3 \quad (1)$$

$$CBR\ Index = 2.77 + 25.64G + 257.66G^2 \quad (2)$$

where q_u is the UCS value (kPa), unsoaked CBR Index (%), and G is the geopolymer concentration (%/100). The data points of both models for Eq. (1) and Eq. (2) have p (Prob > F) values of less than 0.0001 and indicate the rejection of the null hypothesis. This means that the data points for both the q_u and CBR Index have a lack of fit in a linear trend and require a higher order polynomial.

The R^2 value of the model equations were 0.9856 for Eq. (1) and 0.9123 for Eq. (2). These denote a good fit for the data points in terms of their respective model equations. Referring to Fig.5 and Fig.6, the equality graphs of both equations are observed to have high r values between the predicted values and the actual values. This implies that the predicted and actual values have a very strong correlation which verifies both model equations.

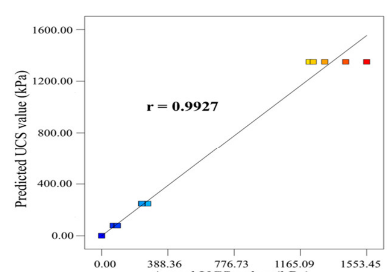

Fig.5 The equality graph of the UCS values

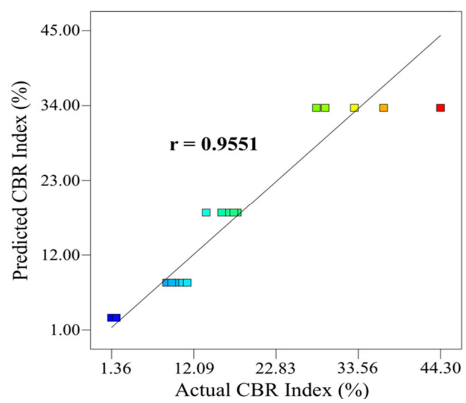

Fig.6 The equality graph of the CBR indices

3.3 SEM of Geopolymer-Stabilized Soil

The SEM images in Fig.7 show how the geopolymer gel formation spread throughout the microstructure as the geopolymer concentration increases. At 10% and 20% geopolymer concentration, the fly ash and the soil particles are still visible with the presence of geopolymer gels. These geopolymer gels have bound the spherical particles of the fly ash together with the soil particles [21]. Notably, the 30% geopolymer gels barely show any particles of both the fly ash and the soil particles. The particles of the precursors have been completely replaced by the geopolymer matrix caused by the increased formation of geopolymer gels. The microstructure shows a compact structure wherein the particles are not separated; they are connected together in the geopolymer matrix. Moreover, the 30% geopolymer concentration shows the crystalline-like structure [1], [22] which is commonly found in geopolymer. These verify that geopolymerization occurs in the dry mix method of synthesizing geopolymer stabilizers.

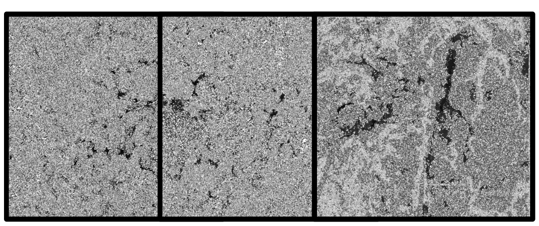
Fig.7 The SEM images of SM at geopolymer concentrations of 10% (left image), 20% (center), and 30% (right) at 2000x magnification

3.4 FTIR of Geopolymer-Stabilized Soil

The FTIR spectra in Fig.8 show how the spectra of untreated soil change when geopolymer is applied. The stretched bands at wavenumbers from 800 to 1200 cm^{-1} are considered as the phase change of fly ash when geopolymerization takes place. These peaks of the bands at that range (800-1200 cm^{-1}) show a behavior of shifting to the right or a shifting to lower wavelengths from the untreated to treated soil caused by geopolymerization [22]. Moreover, the bands of the untreated soil at 600 to 800 cm^{-1}, as it is treated with geopolymer, shows the vibrations similar to that of the pure geopolymer. This indicates the reaction of bending and stretching of silicate frameworks [23].

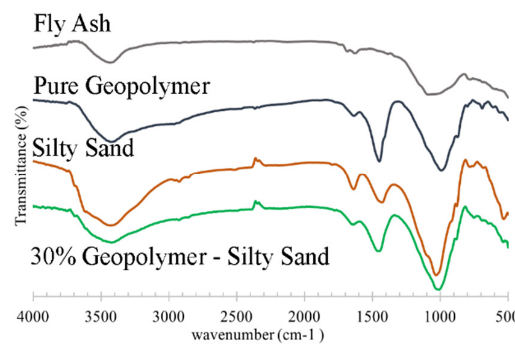

Fig.8 The FTIR spectra of the fly ash, the pure geopolymer, the soil, and the stabilized soil.

4. CONCLUSION

The study is aimed towards testing an alternative method of synthesizing a geopolymer soil stabilizer, which can practically be applied in the field of construction. The SEM images have qualitatively verified that geopolymerization occurs in the dry mix method. It has shown the formation of geopolymer gels in the stabilized soil. These gels are characterized by their crystalline-like structure and their binder-like feature, connecting and surrounding the fly ash particles and soil particles. Moreover, the SEM images of the pure soil and the stabilized soil shows how the geopolymer changes the morphology of the pure soil into a denser soil structure. As for the FTIR results, these have further confirmed the geopolymerization taking place in the stabilized soil which was evident in the change in peaks within the spectra.

The synthesized geopolymer in the study is considered to be the most effective at the concentration level of 30% since both mechanical properties, UCS and CBR Index, increase as the geopolymer concentration is increased from 10% to 30%. For the UCS results, the improvements are considered to be significant. The application of geopolymer on the soil has allowed the stabilized soil to be molded into UCS samples as the soil does not have cohesive characteristics due to its sand content. The q_u values, from 10% to 30%

geopolymer concentration, increase significantly from 78.29 kPa to 1349.74 kPa. As an embankment material, the geopolymer has improved the performance of the soil based on the CBR Index which has increased to a value of 34.32%. The pure soil is rated as *Poor* for *Subgrade* use, whereas the stabilized soil has improved to *Good* for *Base* and *Subbase* use.

The geopolymer, based on these results, has the possibility of being an alternative to conventional soil stabilizers such as cement and lime. The main reason for this is that the geopolymer in the study was synthesized through a different approach rather than the method of using liquid activators, which allows easier application in the field. Focusing on geopolymerization, this further concludes that geopolymerization can still occur in the dry mix method.

5. RECOMMENDATION

The study has used a limited range of values for geopolymer concentration due to the consideration that higher concentration values are costlier due to requiring more alkali activators. However, since these are necessary for obtaining an optimal concentration value, it is recommended for future studies to take into consideration concentration values ranging from 40% to 90%. This will produce a geopolymer concentration that can maximize soil stabilization.

The method of testing the CBR property of the stabilized soil was only through the unsoaked CBR. The soaked CBR was not tested due to time constraints. The soaked CBR is significant as it simulates the strength of soil when saturated or during events of the flood. This is why it is recommended to test the geopolymer stabilizer in terms of the soaked CBR.

The geotechnical properties of the stabilized soil were not tested in the study. The geotechnical properties such as gradation and permeability are considered to be significant properties. These may change when soils are stabilized with geopolymer. As such, it is recommended to test these properties to further discover the extent of the improvement of geopolymer soil stabilization.

6. ACKNOWLEDGEMENTS

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