MORPHOLOGICAL AND ENVIRONMENTAL CHARACTERIZATION OF LIME SLUDGE/FLY ASH STABILIZED SUB-BASE MATERIALS

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ABSTRACT*: In chemical stabilization, cement and lime can be replaced with fly ash (FA) and lime sludge (LS) in the soil treatment. Therefore, the stabilized soil by the latter industrial by-products has to undergo morphological and environmental characterization for further assessment. In this work, morphological characterization thru scanning electron microscopy (SEM) was conducted on six subbase samples treated with FA (15 to 50% utilization) and LS (held constant at 10%). This is to validate internally the utilization of the latter materials in the stabilization process of the said samples. Further, the US EPA 1311 method or the Toxicity Characteristics Leaching Procedure was also employed on the samples to assess if they are environmentally safe to use or not as determined by the presence of heavy metals; cadmium (Cd), lead (Pb), cobalt (Co) and zinc (Zn) thru atomic absorption spectroscopy (AAS). Results revealed thru SEM that the treated samples were composed of a combination of spherically- and irregularly-shaped particles. The spherically-shaped particles are the FA particles whereas the irregularly-shaped, those of LS. However, the US EPA 1311 results showed that the treated samples passed the requirements for cadmium and lead according to TCLP standard limits. However, for Philippine standards such as those in DAO 2016-08 and DOH Administrative Order 2017-0010, all the samples did not pass especially for the lead requirement. The amounts of the LS may be varied, this time, in treating the samples instead of FA to produce a more environmentally-sound subbase material when assessed thru US EPA 1311. This can be further validated too thru SEM characterization when there are more irregularly-shaped particles in the micrographs than those of fly ash particles.*

Keywords: chemical stabilization, lime sludge, fly ash, sub-base

1. INTRODUCTION

In chemical stabilization, cement, lime, asphalt, and even fly ash are required to mix with soil for stabilization purposes. Among the chemical substances mentioned, the most common in soil treatment are cement and lime [1]. Over-dependence on the latter chemicals have led construction cost in soil stabilization to remain high. Therefore, there has to be a sustainable and cost-efficient method along the way. And the best option to do it is to replace the latter chemicals with potential raw materials in the soil stabilization.

Cement and lime are replaced with industrial and agro-industrial waste by-products as stabilizers in the stabilization process as evident in previous studies owing to the best option. Among the by-products that are used as stabilizers are lime sludge [2-7], hypo sludge [8-11], rice husk ash [12-15], and fly ash [7, 16-20].

With the utilization of these industrial by-products in soil treatment, the soil properties should never be compromised but rather be improved such as the bearing capacity, water permeability, or risk of liquefaction [21]. The improvement of the soil properties such as strength and durability can lead them to be completely suitable for construction as purposed [22]. In subbases or bases, for example, improving the geotechnical properties of the pavement layers is vital for the appropriate purpose and proper function of the structures. The success of pavements also depends on the underneath layers like these bases, subbases, or subgrades [3].

Locally, fly ash generated from a coal power plant at the municipality of Villanueva, Northern Mindanao, Philippines, is currently utilized along with lime sludge to stabilize road subbases [7]. It is believed to be the first of its kind work combining fly ash of the host town with lime sludge from a sugar milling plant in Bukidnon, Philippines, in chemical stabilization. Because of the continuous generation of fly ash in the plant, the host town has been utilizing it for construction purposes; as well as the neighboring towns [7, 23-24]. However, in the utilization, the environmental properties of these construction materials were not determined.

Although the best option for reducing fly ash generation at the plant is its massive utilization for construction materials such those of other agro-industrial wastes [5-6, 8, 20, 24-27], there has to be an assessment that these materials are environmentally-safe to use or not. This is because fly ash contains heavy metals [28-29]. Hence, the US EPA 1311 method or the Toxicity Characteristics Leaching Procedure (TCLP) is applied to these construction materials with fly ash.

In this work, the US EPA 1311 method was conducted to subbase samples stabilized with lime sludge and fly ash. Further, morphological characterization was also employed on the treated samples to validate the utilization of the two raw materials thru their particles as revealed by scanning electron microscopy. The combination of these raw materials was already tested as resources for the chemical stabilization of road subbase materials and has resulted in an impact on their geotechnical properties [7] as in those other studies which also used resource combinations for a specific purpose [6, 8, 24-26, 30-32]. Therefore, it is just appropriate to also assess their morphological and environmental properties also as construction materials.

2. MATERIALS AND METHODS

2.1. Characterization of Raw Materials

Lime sludge was collected from a sugar milling company in Bukidnon, Philippines whereas the fly ash was sourced out from a coal power plant in Villanueva, Misamis Oriental, Philippines. They were air-dried and sieved using the no. 200. For chemical composition, raw samples were sent to Ostrea Mineral Laboratories, Inc. in Laguna, Philippines for oxide analyses. On the other hand, samples were sent to the Chemistry Analytical Research Laboratory of the Ateneo de Davao University for surface morphological and elemental analyses using Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy (SEM-EDS), respectively.

2.2. Preparation of Blended Samples

Some variations of blended samples are presented in Table 1 below. Type 1 Portland cement available in the market was used. This was also sieved using the no. 200 before mixing with soil, lime sludge, fly ash, and water. As stipulated in the Philippines' Department of Public Works and Highways (DPWH) Blue Book (2013), the amount of cement to be added to the soil aggregate shall be from 6 – 10 mass percent of the dry soil. Therefore, the maximum amount of cement added was within 1% for the sets of the mixture. The fly ash was utilized up to 50%. For the lime sludge, the utilization was held at 10% all throughout. The raw samples were thoroughly mixed until a uniform color was observed. The prepared blended samples were tested in a separate study for compaction and California Bearing Ratio (CBR) to evaluate their capacities as sub-base materials.

Table 1. Some Variations of Mixtures

Soil Samples	Aggregate Base/Subbase Course	(%OPC + %LS + %FA)
A	100% Soil	1%OPC + 10%LS + 15%FA
B	100% Soil	1%OPC + 10%LS + 20%FA
C	100% Soil	1%OPC + 10%LS + 25%FA
D	100% Soil	1%OPC + 10%LS + 30%FA
E	100% Soil	1%OPC + 10%LS + 40%FA
F	100% Soil	1%OPC + 10%LS + 50%FA

2.3. Morphological Characterization of the Lime Sludge/Fly Ash Treated Subbase Samples

The Scanning Electron Microscopy (SEM) of the treated samples was conducted at the Chemistry Analytical and Research Laboratory in Ateneo de Davao University, Davao City, Philippines. The SEM was employed to view and assess the morphologies of the treated samples in 1000X and 5000X magnifications, respectively, to validate the utilization of the fly ash and lime sludge in the stabilization of road subbase materials.

2.4. Conduct of US EPA 1311 Method to Lime Sludge/Fly Ash Treated Subbase Samples

The Toxicity Characteristics Leaching Procedure (TCLP) or the US EPA 1311 method was also conducted for the treated samples. This is to assess the potential of the toxic metals to be leached when it will be finally disposed of in sanitary landfills that they should not exceed the regulatory limits cited by the Philippines' Department of Environment and Natural Resources (DENR) Administrative Order 2016-08 (DAO 2016-08) and Department of Health (DOH) Administrative Order 2017-0010 as analyzed by Atomic Absorption Spectrophotometer (AAS). The 1.5-gram samples were then dissolved with glacial acetic acid. The amount of such extraction fluid was equal to 20 times the masses of the said samples. Leaching was done for 18 hours. After leaching, the leachates were separated from the solid phases by filtering through a certain micron filter. The AAS analyses of the leachates for cadmium (Cd), cobalt (Co), zinc (Zn), and lead (Pb) were conducted at the Water Laboratory of the University of San Carlos, Cebu City, Philippines.

3. RESULTS AND DISCUSSION

3.1. Characteristics of Lime Sludge and Fly Ash

Both the fly ash and the lime sludge underwent oxide and SEM characterizations to validate their utilization of the sub-base samples as stabilizers.

Table 2 below displays the oxide composition of the fly ash sample used in the study. It is clearly evident that fly ash is rich with mainly SiO_2, Al_2O_3, Fe_2O_3, and CaO. According to ASTM C 618, the fly ash is a Class C type because the total content in wt% of the SiO_2, Al_2O_3, and Fe_2O_3 is 61.61% greater than 50%. Further, the % CaO is 31.44% greater than 10%. This is in consonance with previous studies that also obtained Class C-type fly ash [23, 33-36]. A Class C type of fly ash has both pozzolanic and cementitious properties relative to its purpose in construction. On the other hand, Table 3 presents the oxide composition of the lime sludge sample. It can be observed that the sample is rich in SiO_2 and CaO, as in the previous studies [37-40]. The oxides present in the samples actually indicate their potential utilization in construction.

Table 2. Oxide Composition of Fly Ash

Oxide	Wt. %	Method
SiO_2	30.32	Gravimetric
Al_2O_3	19.96	Direct Nitrous Oxide-Acetylene Flame
Fe_2O_3	11.33	Direct Air-Acetylene Flame
CaO	31.44	Direct Nitrous Oxide-Acetylene Flame
MgO	0.80	Direct Air-Acetylene Flame
Na_2O	4.19	Direct Air-Acetylene Flame
K_2O	0.52	Direct Air-Acetylene Flame
SO_4	0.075	Gravimetric

The surface morphologies of both fly ash and lime sludge samples in both 1000 X and 5000X magnifications are shown in Figs. 1 and 2 below. It can clearly be seen that fly ash is internally composed of predominantly spherically-shaped particles [23-24, 41-43]. On the other hand, the lime sludge sample is consisting of irregularly-shaped particles that are clustered and/or agglomerated [39-40, 44]. In this study, SEM analysis of the treated samples was obtained. So later, in the treated samples, the aforementioned spherically-shaped and irregularly-shaped particles of both fly ash and lime sludge, respectively, will be distinguished.

Table 3. Oxide Composition of the Lime Sludge

Oxide	Wt. %	Method
SiO_2	18.76	Gravimetric
Al_2O_3	0.29	Direct Nitrous Oxide-Acetylene Flame
Fe_2O_3	8.78	Direct Air-Acetylene Flame
CaO	6.41	Direct Nitrous Oxide-Acetylene Flame
MgO	0.31	Direct Air-Acetylene Flame
Na_2O	0.09	Direct Air-Acetylene Flame

Moreover, the EDS spectra of both fly ash and lime sludge samples were also obtained. The EDS would reveal elemental analyses of the two samples. This technique can determine the presence of heavy metals in the samples. There should be no heavy metals in the treated samples in order that they are environmentally safe to use as purposed. In this study, the US EPA 1311 method or the Toxicity Characteristics Leaching Procedure (TCLP) was conducted on treated samples to assess the presence of heavy metals in them. Therefore, the EDS analyses of the samples would help explain the said assessment.

The EDS spectra of the samples are shown in Figs. 3 and 4. It is clearly revealed that the fly ash has traces mainly of Si, Fe, Ca, and O. This is in support of the oxides revealed in Table 2. However, the lime sludge sample has mainly the elements, Si, Ca, and O. Therefore, the said sample has mainly oxides of Si and Ca and is in correspondence to its oxide analysis revealed in Table 3. Now there are no heavy metals stipulated in the samples as in their EDS spectra. But the most reliable method for elemental analysis is the liquid method. So, the US EPA 1311 method has to be conducted to verify the presence of heavy metals which may or may not be likely to occur in the treated samples.

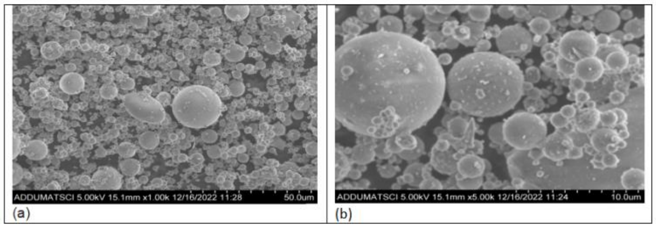

Fig. 1. Morphologies of the fly ash sample at a) 1000 X magnification; b) 5000 X magnification

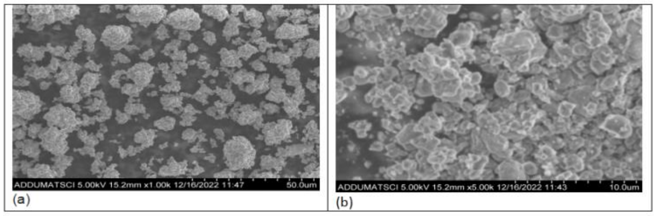

Fig. 2. Morphologies of the lime sludge sample at a) 1000 X magnification; b) 5000 X magnification

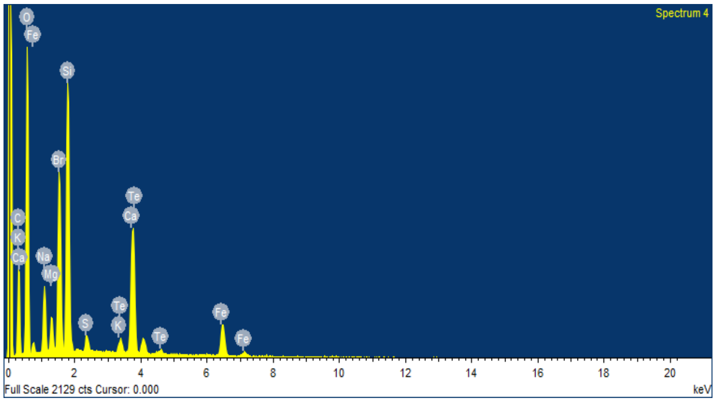

Fig. 3. EDS Spectra of the fly ash sample

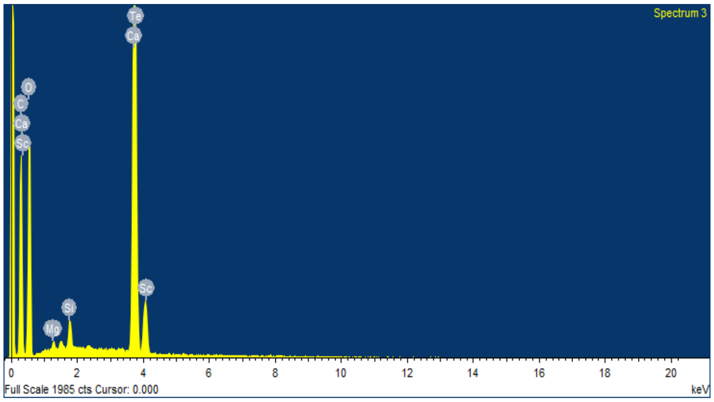

Fig. 4. EDS Spectra of the lime sludge sample

3.2. Surface Morphologies of Lime Sludge/Fly Ash Treated Subbase Samples

The surface morphologies of the treated samples were also obtained through Scanning Electron Microscopy (SEM). This is to validate and assess that the raw materials, fly ash, and lime sludge, were really employed as stabilizers to subbase samples in the study. Thus, as revealed in Figs. 1 and 2, the treated samples should have a combination of particles that are spherically- and irregularly shaped.

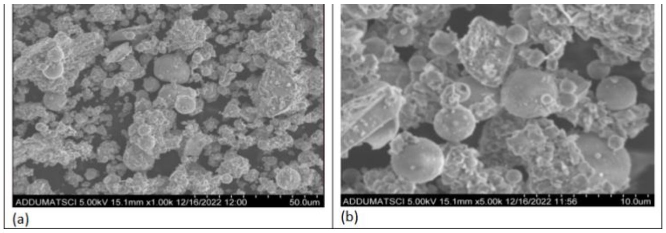

Fig. 5. Morphologies of 10% LS - 1% OPC - 15% FA treated sample at a) 1000 X magnification; b) 5000 X magnification

Fig. 5 shows the SEM morphologies of 10% LS - 1% OPC - 15% FA treated samples at 1000X and 5000X magnifications, respectively. In general, it can be observed that the sample is a combination of spherically- and irregularly-shaped particles. These spherically-shaped particles which are seen dominant all throughout are the fly ash particles whereas the irregularly-shaped particles that form agglomeration are the lime sludge particles. Fly ash particles are predominantly spherical [23-24, 41-43]. On the other hand, lime sludge particles are irregularly-shaped and show agglomerated or clustered structures [39-40, 44]. The distribution of the spherically-shaped particles is a little greater than the irregularly-shaped because the sample was treated with 15% fly ash and 10% lime sludge.

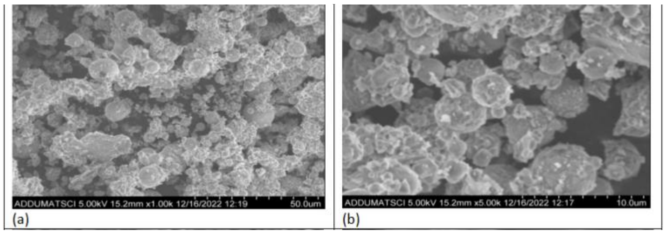

Fig. 6. Morphologies of 10% LS - 1% OPC - 20% FA treated sample at a) 1000 X magnification; b) 5000 X magnification

Fig. 6 shows SEM morphologies of 10% LS - 1% OPC - 20% FA treated samples in 1000X and 5000X magnifications, respectively. The sample is still composed of more predominantly spherically-shaped particles than irregularly-shaped particles as it was treated with 20% FA which is greater than that of 10% LS.

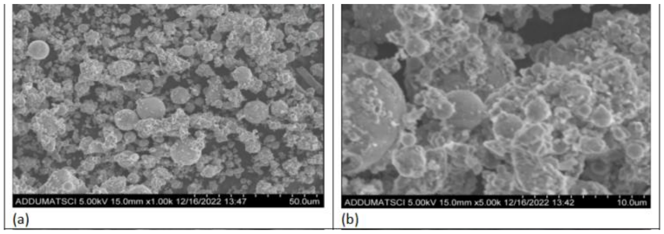

Fig. 7. Morphologies of 10% LS - 1% OPC - 25% FA treated sample at a) 1000 X magnification; b) 5000 X magnification

In Fig. 7, the SEM morphologies in all magnifications applied show still a combination of spherically-shaped particles and irregularly-shaped particles. More spherically-shaped particles are observed than agglomerated irregularly-shaped particles. This is so because the sample was treated with 25% FA and 10% LS. Some irregularly-shaped particles can be observed on top of the bigger spheres, especially in Fig. 4.3b.

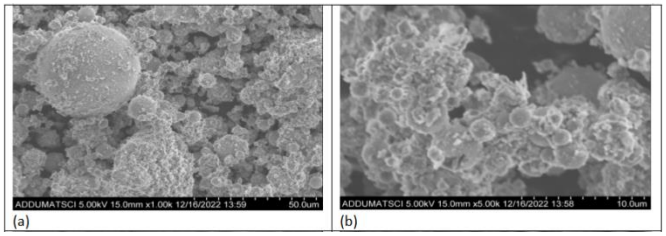

Fig. 8. Morphologies of 10% LS - 1% OPC - 30% FA treated sample at a) 1000 X magnification; b) 5000 X magnification

In Fig. 8, the sample was treated with 30% FA and still 10 %LS. Thus, it can still be observed that the spherically-shaped particles are more predominant than the irregularly-shaped ones. There are also spherically-shaped particles that are having with them on their surfaces these irregularly-shaped ones.

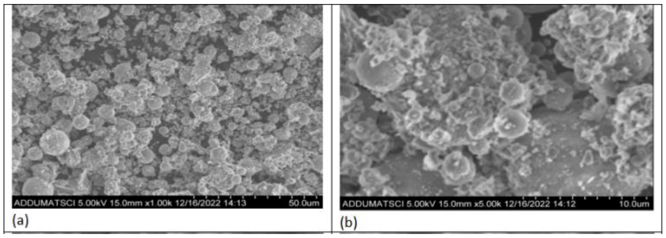

Fig. 9. Morphologies of 10% LS - 1% OPC - 40% FA treated sample at a) 1000 X magnification; b) 5000 X magnification

In Fig. 9, it can be observed in all magnifications that the spherically-shaped particles are more prevalent than the irregularly-shaped ones. This is because, this time, the %FA utilized was increased to 40% already. It is evident also that most of the spherically-shaped particles do have in their surfaces some of these agglomerated irregularly-shaped ones.

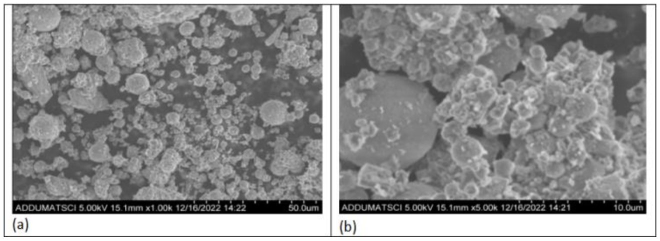

Fig. 10. Morphologies of 10% LS - 1% OPC - 50% FA treated sample at a) 1000 X magnification; b) 5000 X magnification

Lastly, Fig. 10 shows that both big and small spherically-shaped particles are still more prevalent than the irregularly-shaped ones in all magnifications. This time, the sample was treated with 50% FA and still 10% LS. It can also be observed that bigger and even smaller spheres carry some irregularly-shaped particles.

3.3. Environmental Analyses of Lime Sludge/Fly Ash Treated Samples

The Toxicity Characteristics Leaching Procedure (TCLP) or the US EPA 1311 Method was conducted for the treated samples. The TCLP test was conducted to determine if the stabilized materials are environmentally safe to use by assessing the contents of the heavy metals present in the sample such that they should not exceed the standard limits. This study is limited to the analyses of cadmium (Cd), cobalt (Co), zinc (Zn), and lead (Pb) from the leachates through Atomic Absorption Spectroscopy (AAS).

According to Table 4, the contents of cadmium and lead are very much lower in all of the treated samples considering their TCLP standard limits. They basically pass the minimum requirements. However, cobalt and zinc have no standard TCLP limits. Since cadmium and lead satisfied the minimum requirements, the samples are environmentally safe to use.

Table 4. Heavy Metal Concentrations in the Treated Samples as Compared to TCLP Standard Limits

Heavy Metals	TCLP Standard Limits for Heavy Metals (mg/L)	Heavy Metals in the LS/FA Treated Samples (mg/L)						Remarks
		Sample A: 10% LS - 1% OPC - 15% FA	Sample B: 10% LS - 1% OPC - 20% FA	Sample C: 10% LS - 1% OPC - 25% FA	Sample D: 10% LS - 1% OPC - 30% A	Sample E: 10% LS - 1% OPC - 40% FA	Sample F: 10% LS - 1% OPC - 50% FA	
Cadmium (Cd)	1.00	0.02	0.02	0.02	< 0.008	< 0.008	< 0.008	Passed
Lead (Pb)	5.00	0.67	0.70	0.51	0.93	0.94	0.89	Passed
Cobalt (Co)	-	0.61	0.81	0.86	0.82	1.06	0.83	-
Zinc (Zn)	-	1.03	1.53	1.16	1.44	1.76	1.58	-

Table 5 shows heavy metals in the samples as compared to the standard limits stipulated in the Philippines’ Department of Environment and Natural Resources (DENR) Administrative Order 2016-08 (DAO 2016-08) for Class AA water bodies. According to DAO 2016-08, Class AA water is intended for waters having watersheds, which are uninhabited and /or otherwise declared as protected areas. From the table, only samples D, E & F passed for cadmium. However, for lead, all the samples failed the assessment. Not all standard limits are complied with, therefore, the treated subbase samples are not environmentally safe.

Table 5. Heavy Metal Concentrations in the Treated Samples as Compared to Philippines’ DENR Administrative Order 2016-08 (DAO 2016-08) Standard Limits for Class AA Water Body

Heavy Metals	DAO 2016 Standard Limits for Heavy Metals in Class AA Water (mg/L)	Heavy Metals in the LS/FA Treated Samples (mg/L)						Remarks
		Sample A: 10% LS - 1% OPC - 15% FA	Sample B: 10% LS - 1% OPC - 20% FA	Sample C: 10% LS - 1% OPC - 25% FA	Sample D: 10% LS - 1% OPC - 30% FA	Sample E: 10% LS - 1% OPC - 40% FA	Sample F: 10% LS - 1% OPC - 50% FA	
Cadmium (Cd)	0.003	0.02	0.02	0.02	< 0.008	< 0.008	< 0.008	Passed for Samples D, E & F
Lead (Pb)	0.01	0.67	0.70	0.51	0.93	0.94	0.89	Failed
Cobalt (Co)	-	0.61	0.81	0.86	0.82	1.06	0.83	-
Zinc (Zn)	2	1.03	1.53	1.16	1.44	1.76	1.58	Passed

In Table 6, a comparison of the Philippines' Department of Health (DOH) Administrative Order 2017-0010 standard values for inorganic chemical parameters of drinking water is shown. Based on the table, only samples D, E & F passed the limit for cadmium. On the other hand, all of the treated samples did not pass for lead. Therefore, the samples did not completely pass the standard limits.

Table 6. Heavy Metal Concentrations in the Treated Samples as Compared to Philippines’ DOH Administrative Order 2017-0010 Standard Limits

Heavy Metals	DOH Admin. Order 2017-0010 Standard Limits for Heavy Metals (mg/L)	Heavy Metals in the LS/FA Treated Samples (mg/L)						Remarks
		Sample A: 10% LS - 1% OPC - 15% FA	Sample B: 10% LS - 1% OPC - 20% FA	Sample C: 10% LS - 1% OPC - 25% FA	Sample D: 10% LS - 1% OPC - 30% FA	Sample E: 10% LS - 1% OPC - 40% FA	Sample F: 10% LS - 1% OPC - 50% FA	
Cadmium (Cd)	0.003	0.02	0.02	0.02	< 0.008	< 0.008	< 0.008	Passed for Samples D, E & F
Lead (Pb)	0.01	0.67	0.70	0.51	0.93	0.94	0.89	Failed
Cobalt (Co)	-	0.61	0.81	0.86	0.82	1.06	0.83	-
Zinc (Zn)	-	1.03	1.53	1.16	1.44	1.76	1.58	-

The presence of toxic heavy metals in any fly ash-based products can be greatly associated with the percentage of fly ash in the utilization. The higher the amount of fly ash added, the higher the possibility of having heavy metals in the final products. This is so because fly ash inherently contains toxic metals [29]. In this study, fly ash was actually incorporated with cement and lime sludge to stabilize soils. However, the addition of both cement and lime sludge basically did not prevent the heavy metals to be seen in the samples, especially for cadmium and lead as in Tables 5 and 6; even though the addition supposedly would increase the oxide contents in the mixtures and thus, would immobilize the heavy metals in the stabilized system.

4. CONCLUSIONS

The SEM morphologies showed that the treated samples are composed of a combination of spherically-shaped and irregularly-shaped particles. The spherically-shaped particles are the fly ash particles and the irregularly-shaped pertain to the lime sludge. The SEM characterization validated the utilization of the two solid wastes, fly ash, and lime sludge. On the other hand, the US EPA 1311 test results showed that the treated samples passed the requirements for cadmium and lead according to TCLP standard limits. However, for Philippine standards stipulated in DAO 2016-08 and DOH Administrative Order 2017-0010, all these samples did not pass especially for the lead requirement. Therefore, the samples are not totally environmentally-safe to use for road subbase applications. It can be recommended to vary also the amounts of the lime sludge to the fly ash in treating the samples. And this can be further validated too thru SEM morphological characterization when there are more irregularly-shaped particles, this time, in the micrographs than those of fly ash particles. In this case, more environmentally sound subbase materials are produced especially when assessed thru US EPA 1311 method.

5. ACKNOWLEDGEMENT

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