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Research Article

Durability and Microstructural Properties of Triple-Binder-Stabilized Marine Deposit Clay

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Abstract: Marine clay deposits are commonly found worldwide. Considering the cost of dumping and the related environmental concerns, an alternative solution involving the reuse of soils that have poor conditions is crucial. In this research, the authors examined the durability of marine deposited clays and compiled a corresponding database. The use of slag alone as a binder, at any percentage, increased the accumulated mass loss (ALM) up to 2%. However, the use of lime as the third binder seemed to accelerate the chemical reactions associated with the hydration of clay and cementitious material and to enhance the chemical stability, i.e., samples that included both lime and slag experienced the same ALM as samples treated with cement only. Scanning electron microscopy analysis confirmed the durability improvements of these clays. The proposed unconfined compressive strength and accumulated mass loss relationship yielded practical approximation for the fine- and coarse-grained soils blended with up to three binders until 60 days of curing.

Keywords: reuse, soil, X-ray techniques, binder, cement, durability, microstructure

1. Introduction

Marine clay deposits found worldwide especially in coastal regions. This research investigated the marine clays disseminated beside the Mediterranean and northern coast the Cyprus. Rapid development of construction on those formations comprise mostly of digging and dumping of soil. Environmental pollution during digging and transportation is one of the activities that affect mankind and the ecosystem. Additionally, the huge quantity of excavated soil affects the overall cost of shipping and controlling. Thus, companies try to manage the problems allowing for environmental friendly solutions. Considering the structural integrity, utilization or re-use of untreated marine deposited clay for sub-base construction on highways poses engineering problems. The widely adopted solution is to manage the digging soil to the nearest site or landfill. However, allowing for transportation between the quarrying plant and the excavation area can lead to a large amount of CO_2 emission and increased overall cost. The problems associated with those activities can be minimized by the optimization of the performance of excavated soils, which might reduce the cost and the negative effect on the environment. Every project needs different solutions for the management of excavated soil. The previous studies suggested alternative managing strategies for quarried soils, including using them on-site, reusing excavated materials, pre-treating before use in construction, storing them for future consideration, and using them as landfill cover applications [1,2] Furthermore, Magnusson et al. [3] reported that reusing excavated soil could save as much as 14 kg of carbon dioxide per ton. Additionally, the study by Capobianco et al. [4] stated that the treatment of such soils is more beneficial than digging and dumping.

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This study proposed to reuse the copper slag, which is extensively available at the Cyprus Mining Cooperation (CMC) site in the Lefke Region of Cyprus, as a cement replacement. The available slag is left over from the copper mining operations that ended in 1974 and is available in bulk form, having been haphazardly dumped around the Lefke Region. The serious concerns regarding the use of those materials are trace element contamination and their leaching properties. Nevertheless, many authors have reported that such materials are used at considerably lower rates compared to the levels prescribed by the international environmental authorities [5]. Zain et al. [6] prepared samples composed of 10% copper slag as a cement replacement. The results showed that the penetration of the trace elements did not exceed the normal rates. Another study revealed that the penetration of heavy metals (copper, nickel, lead, and zinc) ions from copper slag in large volume was found to be lower compared to the prescribed limits of the international authorities [7].

On the other hand, many researchers reported that copper slag do not show pozzolanic properties [8–10]. Moura et al. [11] studied the mechanical behavior of concrete containing 10% copper slag. They reported that compressive strength of concrete composed of copper slag had lower than reference concrete up to 91 days. However, other researchers mentioned concrete incorporating with copper slag shows cementitious property and pozzolanicity of copper slag increases the strength [12–16]. Additionally, another study assessed the pozzolanicity of clay composed of cement and copper slag [17]. The scanning electron microscopy results revealed that composites exhibited a pozzolanicity. They concluded that at low cement content (30% dry weight of soil), the strength decreased with the copper slag amount. However, an increased the testing period and cement replacement level (50% dry weight of soil), the strength significantly improved with copper slag amount.

Few studies have considered the engineering properties of copper slag blended with clay and cement [18]. However, no studies have considered the durability properties of such composite. The resistance to chemical attack of composites to resist its stability and structural integrity over a long period of exposure to a severe environment defined as durability [19]. The durability of silty clay-cement combination of composites studied in one research. The authors found that the loss in mass of samples decreased with increasing cement amount [20]. Furthermore, another research showed that the lime addition could be decreased and have a positive effect on the ongoing of wet–dry cycles over a long exposure [21]. Consoli et al. [22] and Consoli and Tomasi [23] investigated the porosity/cement and porosity/lime as durability parameters of soil composed of cement to evaluate the durability indices by considering the weight loss after several wet–dry cycles.

Choquette et al. [24] examined the mineralogy and microstructure of lime-treated Canadian marine clays. Their results revealed that incorporating lime significantly caused the clustering of soil samples. The flocculated arrangement being preserved by cementitious bond development between the particles. The authors correspondingly proposed calcium oxide (lime) incorporation to clayey soil which results in the formation of plate-like morphology. They reported that this can increase the bulk volume of the small pores and more space available between the clay-cement particles. Additionally, they stated that the modification in microstructure as a result of the lime addition agreed well with the mechanical properties of clay. Many researchers analyzed the cement-clay microstructural modification with scanning electron microscopy (SEM), reporting decrease in deflocculation level at a high amount of cement [25,26].

Until now, Ekinci et al [18] studied the stabilized Marine clay that considered the effect of triple blinder (cement + copper slag + hydrated lime) on strength properties and their effect on microstructure. However, composites composed of triple binder have not been examined in terms of durability. To address this research gap, in the current study, hydrated lime was incorporated with copper slag in cement-stabilized soil as a replacement to facilitate pozzolanic reactions. In addition, a porosity triple binder index for soil-cement mixes in terms of mass loss was examined for the first time, revealing a correlation between the mass loss and the unconfined compressive strength. This research could thereby enable the reduction of the amount of excavated soil through the assessment of the effects of soil disposal on the environment and on CO_2 emission caused by transportation.

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Additionally, incorporating various amount of copper slag with cement could potentially decrease the impact of global warming potential and the number of accumulated copper slags at the site.

2. Materials and Methods

Basic characteristic tests such as sieve analysis, specific gravity and Atterberg limits were evaluated based on the international standards in accordance with the corresponding ASTM D2487-17 [27]. The results of these tests are presented in Table 1. The clay was designated as inorganic low-to-medium-plastic clay (CL) [28]. The grain size distribution is shown in Figure 1. The soil is composed of clay, silt, and sand with a percentage of 49, 19 and 32, respectively. Furthermore, the XRF test results showed that this clay is rich in SiO_2, Al_2O_3, and CaO.

Table 1. Physical properties of Marine deposited clay, Hydrated Lime and Copper Slag.

Properties	Marine Clay	Hydrated Lime	Copper Slag
Liquid limit (%)	40	-	-
Plastic limit (%)	21	-	-
Plasticity index (%)	19	-	Nonplastic
Specific gravity	2.61	2.17	3.45
Fine gravel (4.75 mm < diameter < 20 mm) (%)	0	0	0
Coarse sand (2.00 mm < diameter < 4.75mm) (%)	2	0	10
Medium sand (0.425 < diameter < 2.00 mm) (%)	3	0	82
Fine sand (0.075 mm < diameter < 0.425 mm) (%)	27	5	8
Silt (0.002 mm < diameter < 0.075 mm) (%)	19	90	0
Clay (diameter < 0.002 mm) (%)	49	5	0
Mean particle diameter (mm)	0.0035	0.02	0.9
USCS class	CL	ML	SP

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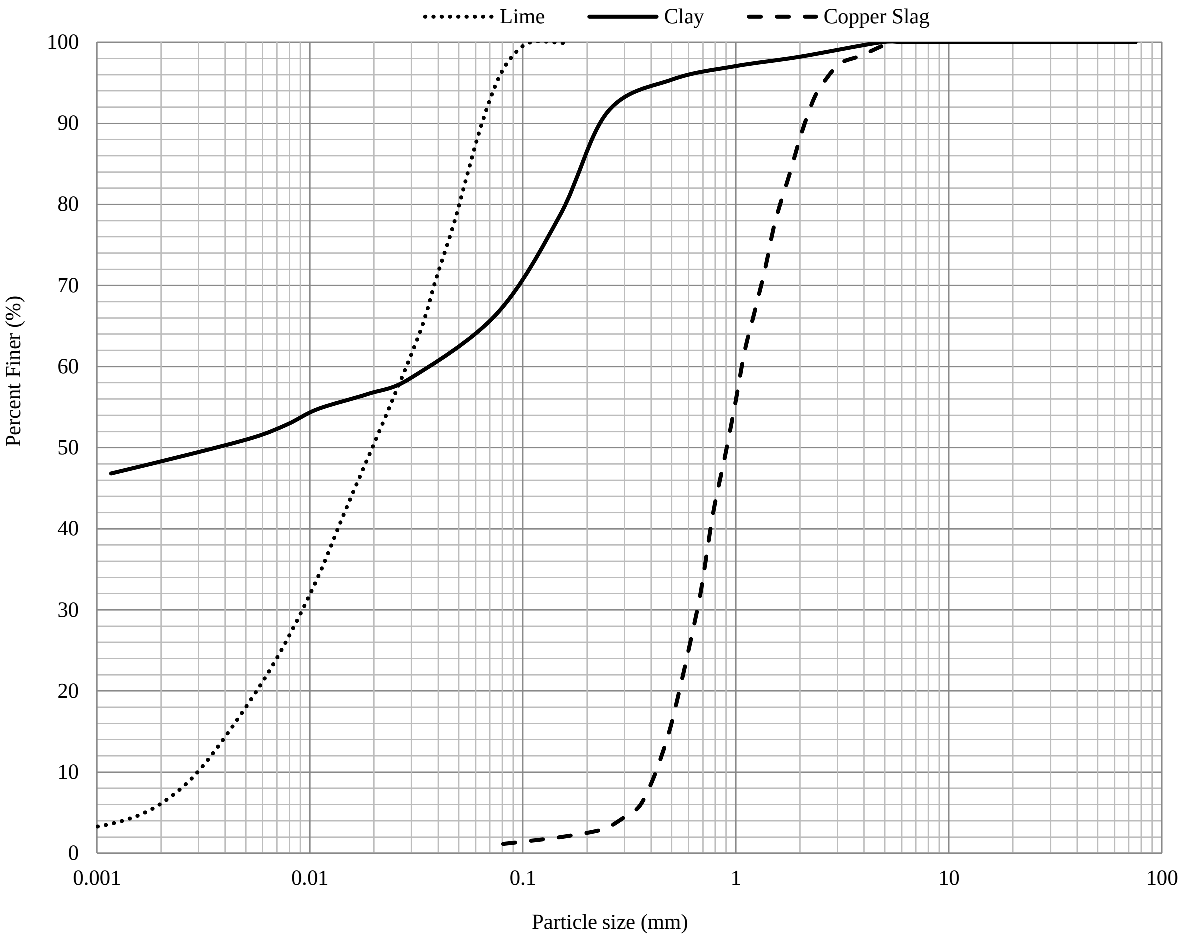

Figure 1. Grain size distribution of studied Clay, Copper Slag and Hydrated Lime.

Copper slag was collected from an abandoned mine in the Lefke region in Northern Cyprus. After performing the characterization tests, the slag was classified according to the USCS as poorly graded sand (SP), and its specific gravity was 3.45. The X-ray spectroscopy analysis allowed us to determine the main components of the slag; 43.5% Ferrous Oxide, 32.8% Silicon Oxide, 8.3% aluminum Oxide, 4.0% CaO, and 2.6% SO_3.

Type I cement having specific gravity 3.12 and the Blaine fineness is 289 m^2/kg was used. The chemical composition of cement is presented in Table 2.

Hydrated lime contains mostly Calcium Oxide (71% CaO), obtained from local manufacturer in Cyprus. The particle size distribution is shown in Figure 1. The physical properties of all used materials is presented in Table 1.

Table 2. Chemical analysis of Portland Cement, Hydrated Lime, and Copper Slag. (EN197-1)

Compound	Portland Cement (%)	Lime (%)	Copper slag (%)
SiO_2	21.2	-	32.5
Al_2O_3	5.1	0.38	8.3
Fe_2O_3	2.5	0.3	43.5
CaO	64.7	70.89	4
MgO	0.9	1.95	-
K_2O	0.2	-	-

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SO_3	1.5	-	2.6
loss in ignition	2.5	24.59	-

To investigate the effects of clay treatment on durability, cylindrical samples of 50 mm diameter and 100mm height were prepared. First, the amounts of materials were calculated from the targeted dry unit weight. They were then measured and dry mixed for at least 5 minutes to achieve uniformity. After that, water was introduced gradually while the mixing process continued. After ensuring the homogeneity of the mixture, it was transferred to a split mold and statically compressed to achieve the required dry density. Upon the completion of the mixing and compressing, the prepared specimens were transferred to a curing room in which they were kept for the required curing time [29].The preparation data for all samples are presented in Table 3.

Table 3. Details of molding and curing data.

Soil type	Cement contents (%)	Copper Slag Content (%)	Hydrated Lime Content (%)	Molding dry unit weight (kN/m3)	Curing periods (days)	Test Type
Marine Deposited Clay	7, 10 and 13	-	-	14.0, 16.0	7, 28, 60	UCS, Wet-Dry Cycles
	7, 10 and 13	10%	-	14.0, 16.0	7, 28, 60	UCS, Wet-Dry Cycles
	7, 10 and 13	-	5%	14.0, 16.0	7, 28, 60	UCS, Wet-Dry Cycles
	7, 10 and 13	10%	5%	14.0, 16.0	7, 28, 60	UCS, Wet-Dry Cycles, SEM

2.1. Durability tests

Durability tests were conducted according to ASTM D 559 [30] for durability testing of marine deposited clays stabilized using various binders. These tests were used to evaluate the mass loss of composites through 12 wetting and drying cycles. Every cycle began by complete immersion of the specimens in water for 5 h and the samples were then dried in an oven for two days. Subsequently, samples were brushed with a wire brush using a pre-calibrated controlled load of about 15 N.

2.2. Compressive strength test

Strength tests were conducted to samples after wetting and drying cycles. Tests conducted according to ASTM C39 [31]. A fully automatic testing machine having 20 kN capacity was used. The failure load was recorded for every samples and average of three samples were used. Based on the procedure, if the single sample compressive strength deviates 10% from the average, the sample discarded and a new sample was prepared. Thus, the variation of the experimental results was completely eliminated.

2.3. Microstructural investigation

Scanning electron microscopy (SEM) was conducted to evaluate the influences of clay treatment with cement, slag, and lime on the microstructure at on 7, 28, and 60 days curing. First, a small dried piece of the specimens was attached to the aluminum stubs. Silver paint was applied. After that, the gold coating was applied. The magnifications of the SEM images ranged from 3500x to 7500x.

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3. Results and discussion

3.1. Durability tests

Figure2a illustrates accumulated mass losses (ALM) of marine deposited clay soil–cement, soil–cement–copper slag, soil–cement–hydrated lime, and soil–cement–copper slag–hydrated lime blends after 12 wetting and drying cycles. The binder contents, distinct dry unit mass, and curing regime for the laboratory-produced samples are summarized in Table 3. A sound polynomial fit of ALM versus η/Xiv0.32 after durability tests could be obtained, as shown in Equation 1. Thus, it was observed that the adjusted porosity/binder index can be used to predict durability with up to a triple binder.

$$ALM = 0.031\left(\eta/{X_{iv}}^{0.32}\right)^2 + 0.864\left(\eta/{X_{iv}}^{0.32}\right) + 0.867,\ \ R^2 = 0.89, \tag{1}$$

The US Army Corps of Engineers (USACE) technical manual [32] states that the maximum permissible mass loss of clay soils after 12 cycles (wet-dry) is 6% of the initial specimen weight for soil stabilization of pavements. In this study, these wetting and drying requirements were satisfied for η/Xiv0.32 of less than about 24. The shaded section in Figure 2a contains the data for the specimens that satisfied this requirement, which are all soil–cement–hydrated lime and soil–cement–copper slag–hydrated lime blends.

Finally, the unconfined compressive strength versus accumulated mass loss after 12 cycles are shown in Figure 2b for the above marine deposited clays stabilized with clay soil–cement, soil–cement–copper slag, soil–cement–hydrated lime, and soil–cement–copper slag–hydrated lime blends. Unique second-order polynomial relationships with reasonable prediction can be obtained from Equation. 2 for such blends.

$$ALM = 131.52(ALM\%)^2 - 2090.9\,(ALM\%) + 9265.5,\ \ R^2 = 0.84, \tag{2}$$

Figure 2b also shows that the durability requirement of the USACE [32] for clay soils is only satisfied by the blends with unconfined compressive strengths greater than 1400 kPa. This finding also defined a lower boundary for achieving satisfactory durability of such blends in terms of strength.

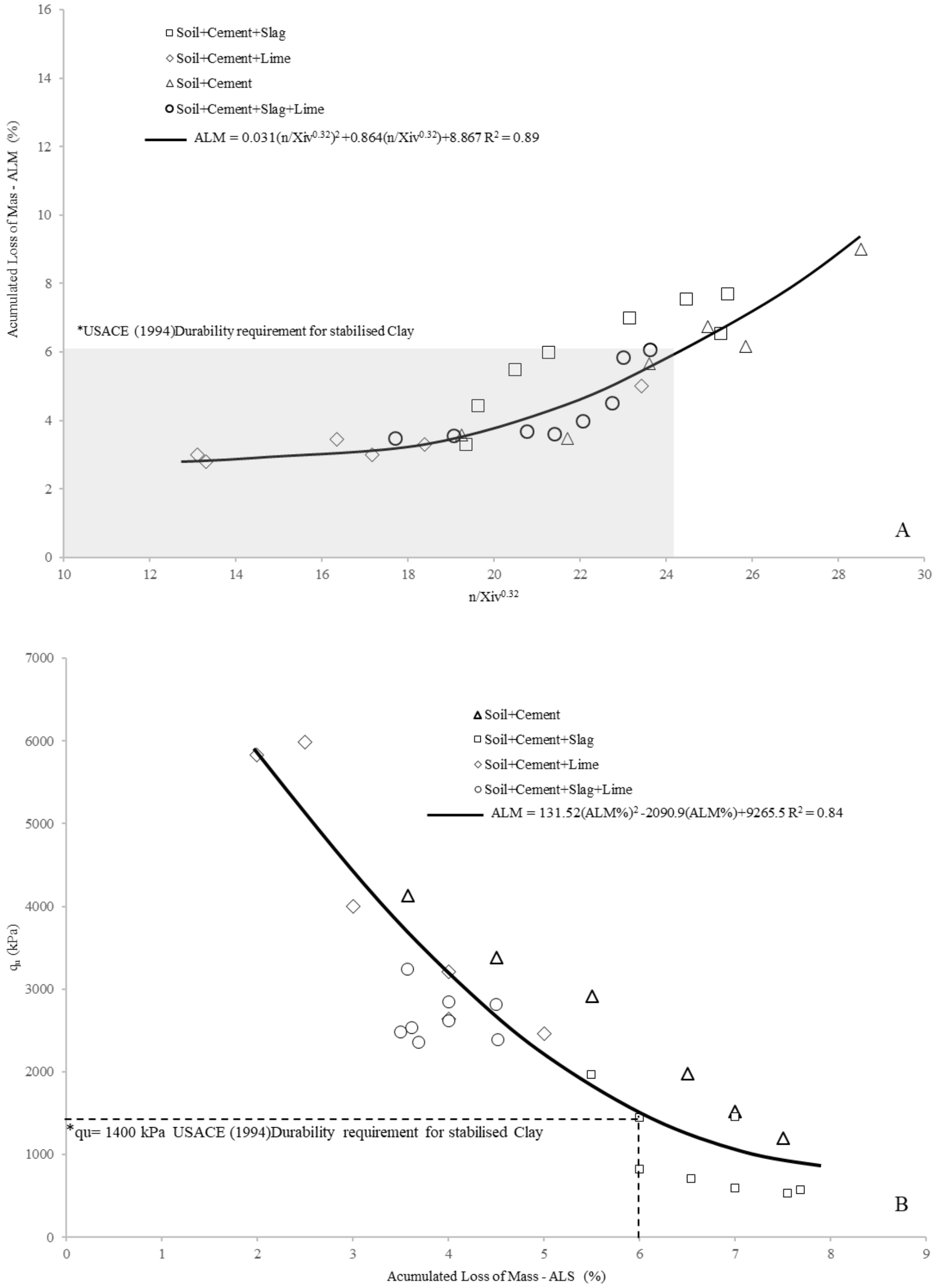

Figure 2. (a) ALM versus adjusted porosity/binder index, (b) ALM considering twelve wet-dry cycles verses unconfined compressive strength(qu), for all tested blends, dry unit weights considering 28 days of curing.

3.2. Microstructural Analysis (SEM)

SEM test results performed after compressive strength tests for the composites incorporated with marine deposited clays at the 7, 28, and 60 days are shown in Figure 3. Those SEM results revealed that no cementitious bonds developed in the untreated clay due to the lack of soil-matrix (bond between the soil, aggregate, and cementitious compounds) cementing bonds. Additionally, as seen in Figures (Figures 3 a-d), the tested marine deposited clay is rich in calcium carbonate and contains “hollow like structures”

The clay-cement mixtures were formed from the main chemical reaction of cement and the secondary reactions of the calcium-silicate-hydrate (CSH) product formation during the pozzolanic

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reaction of soil-cement mixtures. Figure 3b shows the products of hydration, needle-like crystals, and the products that resulted from calcium silicate hydrates between the soil particles. Based on the SEM micrographs, the products had high aspect ratios. This confirms the formation of CSH needles in the bulk volume as a result of high strength and thus reduced ALM. Ettringite is a stable product with needle-like crystals having a hexagonal cross-section that formed easily from samples because of the high void ratios. This formation also caused the expansion and ALM increase at later time-points. However, it seems that the ettringite fills the pores in the matrix during the hardening period of 7 to 60 days and the pore space decreases significantly between those curing periods.

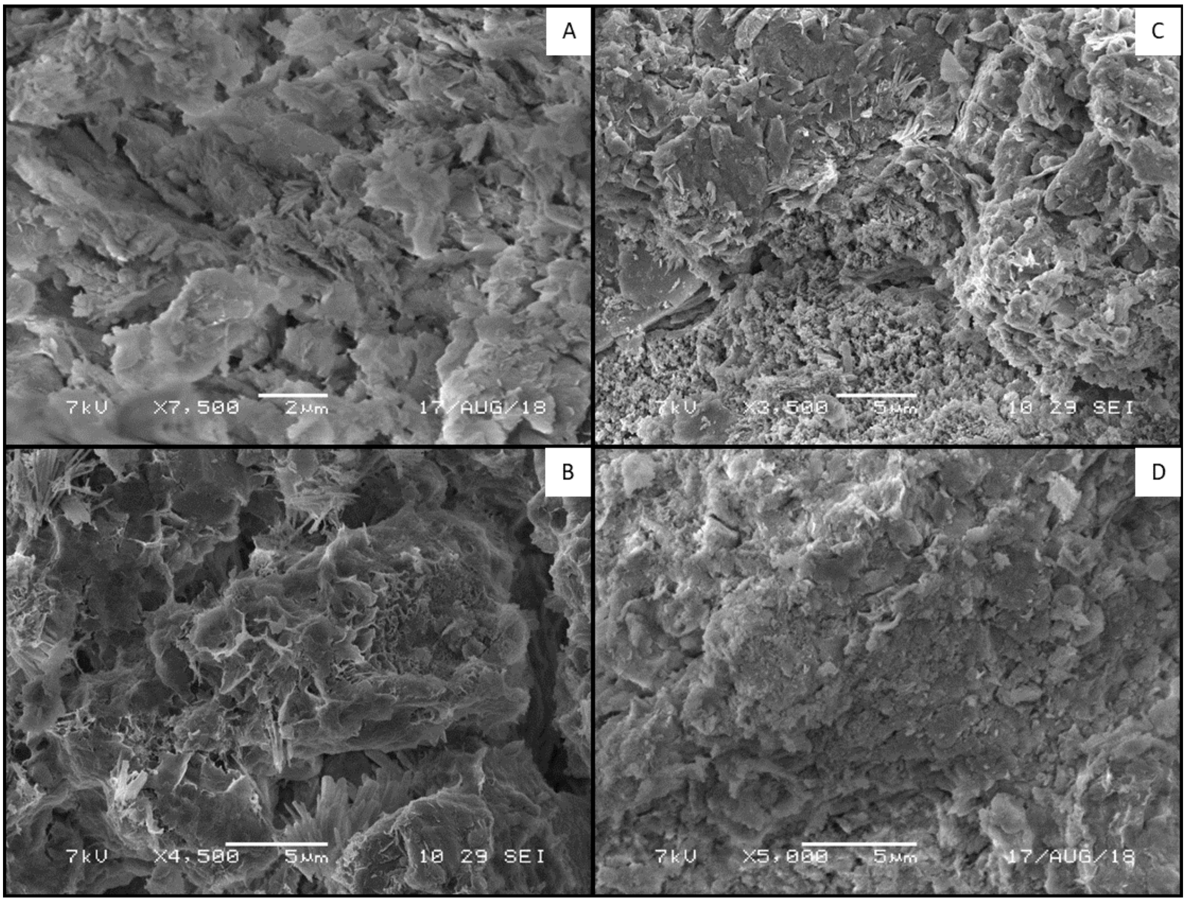

Figure 3. The scanning electron microscopy images, conducted on (a) untreated, (b) 7 days, (c) 28 days and (d) 60 days, cured cement + Copper Slag and Hydrated Lime blend marine deposited clays.

The clay compounds, silica, and alumina react with Ca^{2+} and form CSH-calcium aluminate hydrate (CAH) during pozzolanic reactions. These hydrate products grow and harden, thus, improving the ALM and strength of the clay-cementitious mixtures over hardening. After the 60-day curing period, the pores were filled with CSH gel, as shown in Figure 3c. Particle aggregation is observable due to the cation exchange reactions caused by introducing lime and slag. This aggregation reduces the “thickness of the double layer” between the clay particles and the attraction between particles, forcing the particles to move closer and initiating the particle aggregation phase. As the curing period increased, the porosity of the specimens reduced due to cementation, and a further improvement in ALM is observable. It can be assumed that day 7 is the beginning of curing and the development of slag–lime reactions and corresponding pore spaces, as illustrated in Figure 2, where the porosities of the specimens cured for 7 days are higher and the porosity decreases with curing and with the addition of slag and lime. This can be seen in Figures 3c and 3d that show the completed particle aggregation.

Figure 3d also shows silica and alumina reaction where the cementing property is more obvious. This is due to the secondary reaction development after 60 days indicating a reduction in portlandite (CH) due to the presence of copper slag and cement because of their reuse and transformation to

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secondary CSH at this stage of hydration. This feature is not evident in Figure 3b, as the reaction is in its early stage and CH is more dominant because the CH crystals are absorbed in a later stage since the copper slag reacts in a later stage. This characteristic is observable in the ALM reduction and strength development as well. Furthermore, hydrated lime addition seams to activate pozzolanic reactions at earlier stages, leading to a reduction in ALM.

4. Conclusions

This study examined the durability of the deposited marine clay when treated with cement, copper slag, and hydrated lime. The following can be concluded from the study:

- The proposed adjusted porosity/binder index proved that can be used to predict durability with up to three-component binder.
- The incorporation of hydrated lime to cement – slag treated soils improved the durability of the composites and ensured the satisfaction of weight loss and minimum strength according to the requirements of USACE.
- SEM pictures revealed the formation of "needle-like crystals" with a high aspect ratio between the particles that resulted from the primary hydration. These crystals are responsible for the improvement in UCS and ALM.
- As the curing period increased, the specimens' porosity reduced due to Pozzolonic reactions, and a further improvement in ALM was observable.
- The incorporation of hydrated lime seemed to accelerate the pozzolanic reactions at earlier stages, resulting in a reduction in ALM.

5. Recommendations

In this study, a formula to predict the Accumulated loss of mass when adding a triple binder was successfully created. Further research can be conducted to validate this formula when using pozzolanic materials different than Copper Slag.

Author Contributions: MH and AE conceived the study and were responsible for the scheduling and performing the experimental study. EA was responsible for microstructure interpretation. AE and MH wrote the first draft of the article and EA overlooked and finalized the study.

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