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TECHNICAL NOTE

Stabilization Mechanism for Sands Treated with Organic Acids

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Abstract

Contractors tout the use of organic acids for soil improvement even though the underlying stabilization mechanism is not well understood. Strength, stiffness, and chemical tests were conducted on two sandy materials treated with a commercial organic acid mixture to evaluate the effectiveness and stabilization mechanism involved. After curing for 28 days, tests show a moderate increase in unconfined compressive strength, approximately two-fold, and a large increase in constrained modulus, approximately one order of magnitude. X-ray diffraction and chemical analyses do not suggest traditional pozzolanic reactions as the source of improvements in the treated specimens. Test results suggest the organic acid solution promotes microbe growth and thus an increase in organic matter within the sand skeleton. This reduction in void space increases relative density to levels above the maximum unit weight of the host sand, resulting in higher soil strength and stiffness.

Keywords: *organic acid, stabilization, relative density, microbes*

1. Introduction

Over the years, contractors and construction personnel have advanced soil stabilization technology through experience and innovation than by research and theory. Because of this, many engineers have conducted research on a variety of soil stabilization techniques to verify performance, applicability, and the underlying stabilization mechanisms. Recently, the East Asian engineering community has been inquiring on the applicability of organic acids in soil stabilization. The application of organic acids is touted as being effective and environmentally friendly in a time when professionals are being encouraged to apply more sustainable soil stabilization techniques due to climate change and energy production issues. However, as most organic acid related products are proprietary and can be reformulated, it is difficult to ascertain the effectiveness and stabilization mechanisms involved.

This study explores the engineering effectiveness of a commercial organic acid product on loose granular soils, particularly those that are typically found at shallow depths. Engineering effectiveness was evaluated by measuring the increase in strength and stiffness due to the acid. A brief review on modern soil stabilization techniques in granular materials is presented followed by descriptions in experimental testing and analysis to ascertain how effective organic acid soil stabilization is as well as to help provide some insight into the underlying stabilization mechanisms.

2. Soil Stabilization

Soil stabilization is generally categorized into mechanical and chemical techniques. Mechanical soil stabilization techniques generally involve some form of densification or reinforcement. In terms of densification, Poulos and Hed (1973) and Townsend (1973) have shown that vibratory methods are more effective for cohesionless soils. Accordingly, vibratory techniques in granular materials consist of vibratory compaction rollers (D'Appolonia *et al.*, 1969; Mooney and Rinehart, 2009) and vibratory probe techniques. The most basic vibratory probe technique is termed vibro-compaction, which involves vibrating a probe, or vibroflot, into the ground (Mitchell, 1981; Harder *et al.*, 1984; Dobson, 1987; Kerwin and Stone, 1997; Slocombe *et al.*, 2000). Different names are assigned to vibro-compaction depending on the methodology and type of backfill used. Typically, gravel is used for backfill, resulting in what is more commonly known as a stone column. Another mechanical technique called dynamic compaction involves repeatedly dropping a large weight onto the soil surface. However, the effectiveness of densification decreases

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with depth (Mayne *et al*., 1984; Lukas, 1986; Mitchell and Wentz, 1991; Rollins and Kim, 2010). Compaction grouting involves the injection of a low slump grout under high pressure, which displaces the surrounding granular material resulting in densification (Baez and Henry, 1993; Miller and Roycroft, 2004; El-Kelesh *et al.*, 2012). Ground reinforcement typically involves the insertion of geosynthetics or micropiles to create a composite system that can resist multiple types of loads (Moayed and Naeini, 2012).

Chemical stabilization techniques involve adding chemicals or additives, such as cementitious materials, into the soil. Traditional cementitious materials include cement, lime, fly ash, coal ash, silica fume, and even rice husks (Lin *et al.*, 2007; Chatterjee, 2011). Alternative chemicals include sodium silicate, acrylamide, epoxy resins, and polyurethane (Kazemian *et al.*, 2010). Such materials are generally delivered to the soil deposit via grouting or soil mixing. Although chemical stabilization techniques are great for increasing soil strength, there is concern over the toxicity and sustainability of the more commonly used cementitious materials (Mohanty and Chugh, 2006; Dombrowski *et al.*, 2010; Liu *et al.*, 2012). The stabilization mechanisms in traditional chemical and cementitious grouts are well understood, leading to their widespread use. Non-traditional stabilizers, such as enzymes, have demonstrated varying performance and are utilized due to their cost, applicability, and availability (Scholen, 1992). However, enzymes as stabilizers are usually proprietary in nature with the exact composition and stabilization mechanisms unknown to the public. Additionally, enzyme products are typically reformulated making case history studies and comparisons difficult to interpret.

A third category in stabilization of granular soils is biological. DeJong *et al.* (2006) showed cementation through Microbially Induced Calcite Precipitation (MICP) to increase axial capacity and stiffness for Ottawa sand. The researchers used a common soil microorganism, *bacillus pasteurii*, to break down urea and allow calcium and carbonate ions to precipitate between soil particles. One drawback to this technique is the large amount of ammonia produced, which is harmful to certain ecosystems and human health. Van Paassen *et al.* (2010) used a similar MICP approach to develop a special type of grout while Dove *et al.* (2011) minimized several of the limitations in MICP by utilizing a biologically inspired silicification process to cement Ottawa sand.

3. Experimental Testing

3.1 Materials

A commercially available organic acid was evaluated in this study, which will be referred to as product α. The manufacturer's information on product α describes it as a mixture of different types of organic acids and plant extracts supplied in powder form that when mixed with soils, will break down large clods and increase soil strength. The manufacturer states product α encourages microbe growth and indirectly suggests some form of cementation, but does not give a detailed engineering explanation on the strength increasing mechanism.

Two sands were used for laboratory testing with their index properties summarized in Table 1. One batch was recovered from a site in the Sangwangsimni area of Seoul, South Korea, and is named Sangwangsimni fill. Sangwansimni fill has a fines content, FC = 4%, mean grain size, D_{50} = 1.7 mm, coefficient of uniformity, C_U = 5.1, coefficient of curvature, C_C = 1.1, maximum unit weight, γ_{max} = 16.0 kN/m^3, and minimum unit weight, γ_{min} = 11.2 kN/m^3, making it a poorly graded sand, SP, according to the Unified Soil Classification System, USCS. Interestingly, the fines portion had a liquid limit, LL = 39 and a plasticity index, PI = 17, indicating low plasticity clayey mineralogy. The second sand used for this study was packaged from a local construction materials supplier and is called No. 5 Quartz. This is an artificial silica sand with FC = 0%, D_{50} = 0.71 mm, C_U = 1.8, C_C = 0.9, γ_{max} = 15.8 kN/m^3, and γ_{min} = 12.6 kN/m^3, also making it a SP according to USCS. Specific gravity, G_s, was determined from ASTM D854. Maximum unit weights were determined from the Japanese method, which is similar to ASTM D4253. The Japanese method places dry granular soil in a compaction mold using 5 lifts. Each lift was densified by hitting the base plate of the compaction mold with a hammer 200 times. A standard Proctor compaction mold was used in the tests. The minimum unit weight was determined from dry-tipping, which is also similar to ASTM D5254. Dry-tipping places a finite amount of dry granular soil into a graduated cylinder and slowly rotating the cylinder until a loose column of soil is reached.

Table 1. Soil Material Properties

Material	FC (%)	D_{50} (mm)	C_U	C_C	γ_{max} (kN/m^3)	γ_{min} (kN/m^3)	G_s
Sangwansimni	4	1.7	5.1	1.1	16.0	11.2	2.65
No. 5 Quartz	0	0.71	1.8	0.9	15.8	12.6	2.65

3.2 Preparation

Reconstituted samples were made by mixing an organic acid solution with the previously described sandy materials. For every 100 g of soil, an organic acid solution was made by mixing 1.5 g of product α with 10 g of water. This mixture results in a water content of approximately 10% and a well-concentrated organic acid solution. The organic acid solution was then manually mixed into the sandy batches until the solution was evenly distributed. Treated sands were then placed into Polyvinyl Chloride (PVC) tubes measuring approximately 50 mm in diameter and 100 mm tall using 5 lifts. Each lift was tamped 15 times to ensure a uniform density. Over 50 samples of each sand were created in this way for unconfined compression testing. An additional 25 samples were submerged in a bin to see what effects constant saturation would have on the treated samples. Treated sand was also placed into oedometers to evaluate stiffness through constrained modulus. These samples were constructed in two lifts, each tamped 15 times. For comparison, corresponding sand samples with 10% water content were also placed into PVC tubes and oedometers for testing.

As suggested by the manufacturer, samples were brought out into the sun at least 6 times a week to help with the microbe

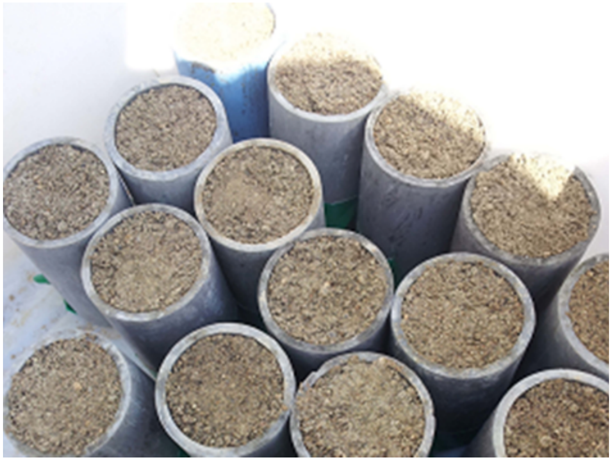

Fig. 1. A Batch of Treated Samples being Taken Out for Sunlight

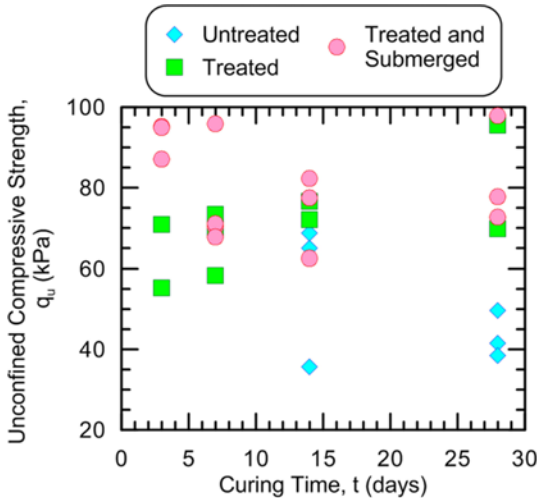

Fig. 2. Unconfined Compressive Strength of Untreated and Treated Sangwangsimni Sand Samples based on Curing Times

growth, which is shown in Fig. 1. The manufacturer also suggested to keep the samples moist and so approximately 1 L of water was distributed to all samples. However, it was impractical to move the submerged samples out into the open, but water was added to maintain a water level above the top of the samples.

As chemical and organic stabilization techniques take time to develop their improvements, compression tests were conducted for samples aged 3, 7, 14, and 28 days old. Samples were tested in an unsaturated state such that matric suction could develop as sand samples would not be able to stand unsupported for an unconfined compression strength test (i.e. cohesion = 0). The submerged samples were also allowed to dry in an electric oven heated to approximately 30-40°C. This temperature would allow some moisture to be retained in the soil sample and thus ready for unconfined compression testing. Samples were extracted from their PVC tubes by carefully applying a plastic disk to cap one end and then lifting the tube above the sample.

4. Experimental Results

4.1 Unconfined Compressive Strength and Deformation

Specimens cured 3, 7, 14, and 28 days were tested for unconfined compressive strength, q_u, with the Sangwangsimni sand results plotted in Fig. 2. The figure shows test results for 3 and 7 day untreated samples to be missing, with the reason being 3 and 7 day untreated and treated samples did not develop enough cohesion to stand alone for testing. Thus, many untreated samples and several treated samples extracted after 3 and 7 days were rendered unusable because they were not able to stand unconfined. However, several 14 and 28 day samples were able to stand for testing, as shown in Fig. 3(a), implying a gain of strength with time perhaps due to mineral precipitation (Baxter and Mitchell 2004). Fig. 2 shows the sand with organic acid treatment to develop an increase in q_u over time, from an average of about 65 kPa after 3 days to an average of about 80 kPa after 28 days. Unfortunately, No. 5 Quartz samples were unsuccessfully extracted from their PVC tubes, as shown in Fig. 3(b), and thus the lack of results.

In terms of deformation, samples cured underwater did not show brittle behavior. Fig. 4 plots the axial strain to failure, ε_f, against curing time and shows no definitive pattern other than samples cured underwater to be less brittle. Additionally, no

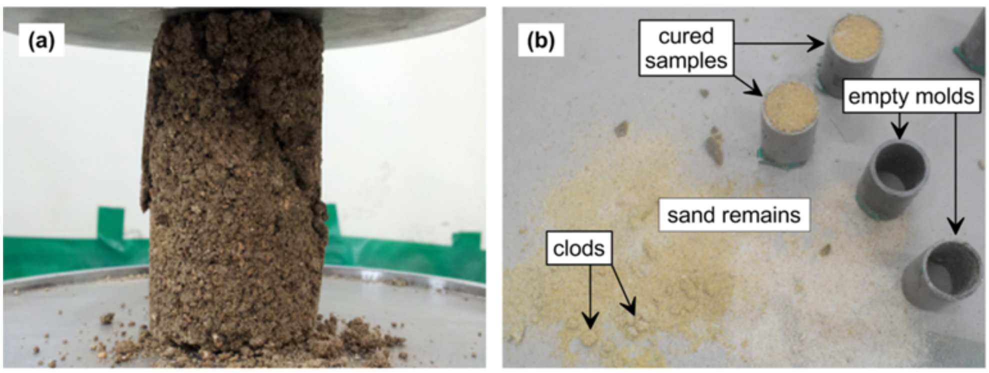

Fig. 3. (a) Sample of Sangwangsimni Sand being Tested, (b) No. 5 Quartz Sand Samples were Unable to Stand Unsupported after Molds were Removed

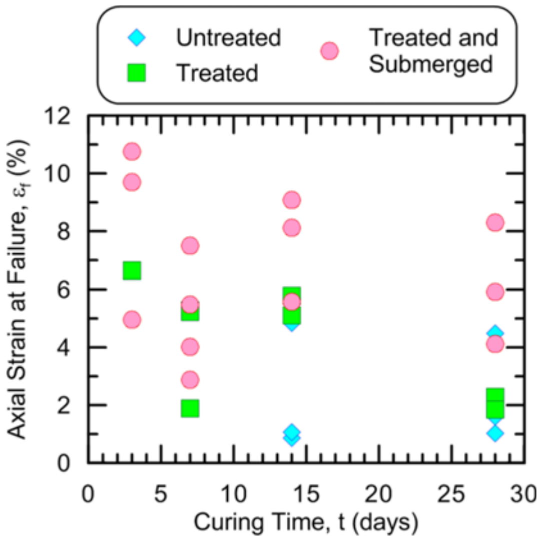

Fig. 4. Axial Strain at Failure of Untreated and Treated Sangwangsimni Sand Samples based on Curing Times

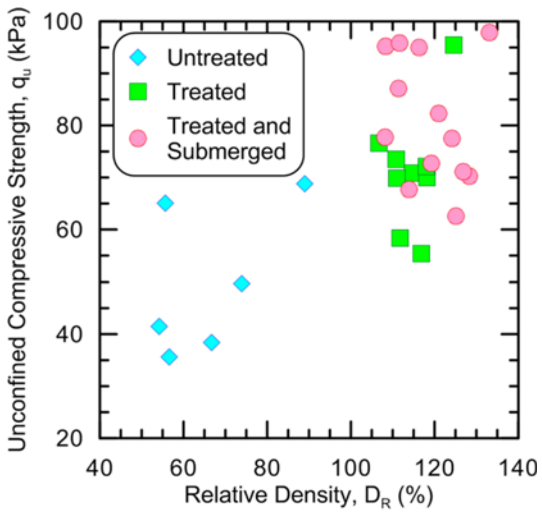

Fig. 5. Unconfined Compressive Strength of Sangwangsimni Sand Samples based on Relative Density

samples cured underwater showed an axial strain at failure to be less than 2%.

In theory and in practice, the strength of cohesionless soils is correlated to its density state, typically measured by relative density, D_R. Fig. 5 plots q_u against D_R and clearly shows higher relative densities lead to higher compressive strengths as expected. The figure also shows a majority of the treated specimens had $D_R > 100\%$.

Even though the figures show a strong correlation between D_R and q_u, theory and testing also show soil strength is highly dependent on effective confining stress, which can be modified through suction. Fig. 6 shows the variation of q_u and ε_f against saturation levels. Fig. 6(a) shows the saturation levels for the treated sands cured underwater to have higher saturation levels and higher q_u relative to the untreated and air cured samples

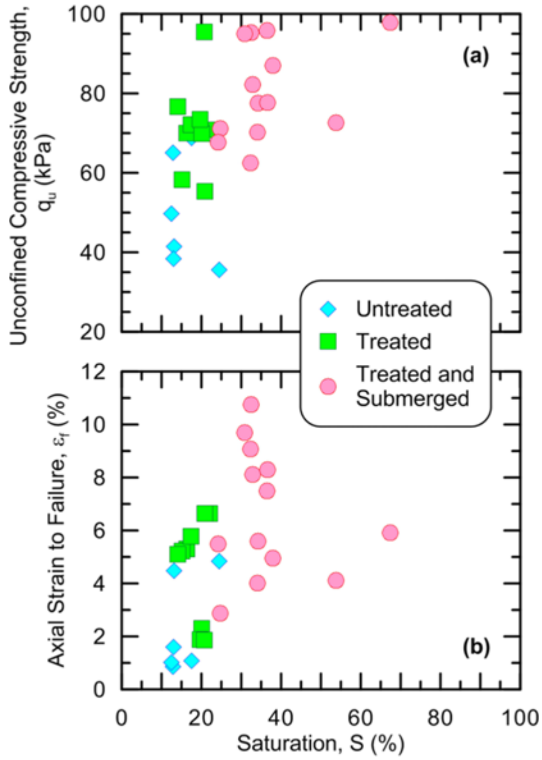

Fig. 6. (a) Unconfined Compressive Strength, (b) Axial Strain to Failure Against Saturation Level for Sangwansimni Sand

while Fig. 6(b) shows similar behavior for ε_f. These results suggest little influence from saturation levels on the untreated and air cured samples and therefore it is reasonable to assume the increases in q_u are correlated to the increase in D_R. Although the sand samples cured underwater show a higher as-tested saturation level, the effect from partial saturation should also be minimal as soil water characteristic curves for sand show soil suction to decrease as volumetric water contents increase. Additionally, researchers have shown that shear strength increases from suction in partially saturated sands is minimal (Shimada, 1998; Farouk *et al.*, 2004).

4.2 Stiffness

The constrained modulus of soil, D_V, was estimated for treated and untreated samples by repeatedly applying a load to oedometer samples for 10 cycles with the results after the 10th cycle plotted in Fig. 7 for the Sangwangsimni sand material. As expected, test results show D_V to be nonlinear and stress dependent. The data show D_V of untreated samples to increase with vertical load, but tend to remain under 5 MPa. Treated samples cured 14 days show a marked increase in stiffness that also increases as vertical load increases. However, acid treated samples cured 28 days show a slight reversal in behavior, that is a decrease in stiffness as vertical load increases, in particular, the D_V estimated from a vertical load of about 60 kPa is much higher than at larger vertical loads. This behavior can be attributed to the soil stabilization mechanism involved, which is explained in

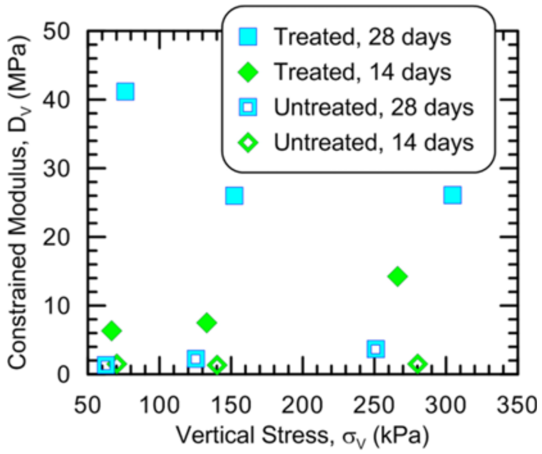

Fig. 7. Constrained Modulus as a Function of Vertical Stress for Sangwangsimni Sand

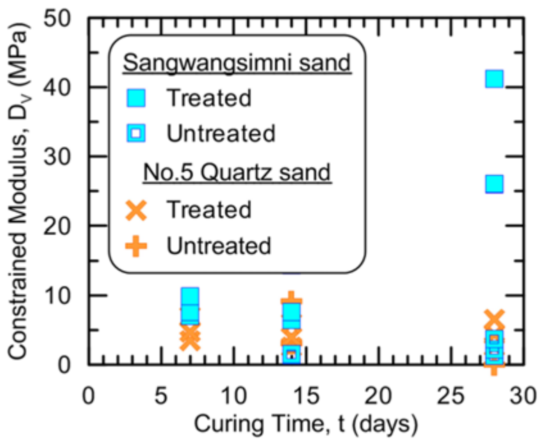

Fig. 8. Constrained Modulus as a Function of Curing Time for Sangwangsimni and No. 5 Quartz Sand

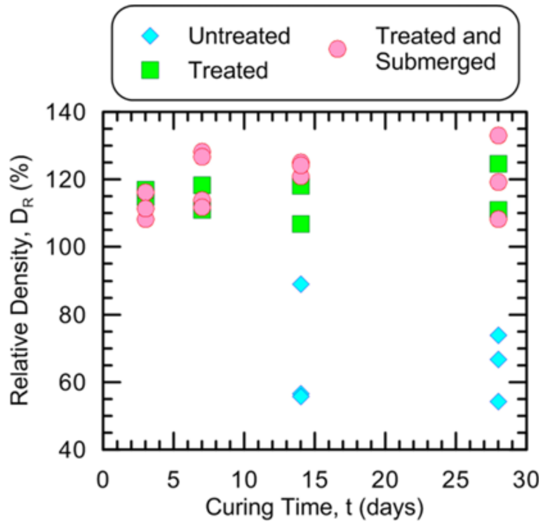

Fig. 9. Relative Density of Untreated and Treated Sangwangsimni Sand Samples based on Curing Times

Fig. 10. Swelling in Treated Samples

the proceeding section.

Since it was shown in a previous section that compressive strength increases for treated samples, Fig. 8 plots the correlation between average D_V and curing times for samples loaded at the lowest vertical stress, σ_V = 60-80 kPa. For Sangwangsimni sand, test results show D_V to increase through time, with a majority of the increase in stiffness attained after being cured for 28 days, which is consistent with the results from Fig. 7. Untreated Sangwangsimni samples did not show a significant increase in stiffness due to curing time. Treated No. 5 Quartz sand samples showed no significant improvement in stiffness and did not demonstrate a correlation with curing time.

5. Stabilization Mechanism

5.1 Physical Testing

Results from compression testing suggest a significant change in D_R, with Fig. 5 showing treated samples to have both higher q_u and D_R relative to untreated samples. Fig. 9 shows the variation in D_R over time and two minor patterns emerge. One is the variability in D_R as curing times increase. The figure shows a well defined range, D_R~105-120%, after 3 days curing time, which progressively widens to D_R~105-130% after 28 days curing time. Admittedly the number of tests is small, but being that the same operator using the same techniques and samples being chosen at random for testing does lower the probability of coincidence. For the Sangwangsimni sand samples to have such a marked increase in D_R, the dry unit weight would need to increase 25-40%.

This significant increase in density is demonstrated in Fig. 10, which shows three treated samples in clear acrylic molds. One sample shows swelling at the top, one sample shows swelling at the bottom, while another sample does not show significant swelling. All submerged samples showed swelling. The Sangwangsimni samples showed more swelling than the No. 5 Quartz materials, while none of the untreated sandy materials exhibited this behavior. Prior to physical testing, all excess material was carefully trimmed off.

This increase in compressive strength and density also reveal a special characteristic that was initially shown in Fig. 7, where the constrained modulus of treated sands after 28 days was higher at relatively lower vertical loads. This observed behavior is a side-effect of the increasing density mechanism. Whatever material that is occupying the void space would also develop some form of cohesion by binding soil particles. These bonds are present at lower loads, but would break at larger loads. How these bonds form was analyzed chemically.

5.2 Chemical Tests

X-Ray Diffraction (XRD) and chemical analyses conducted by external consultants confirm product α to consist of organic acids with no significant amounts of calcium, aluminum, or silicon. The lack of additional calcium, aluminum, or silicon implies an externally driven pozzolanic reaction is not expected, thus eliminating traditional cementation as the strength improvement mechanism. Neither microbes nor substrates were found in the product α batch received, suggesting direct biological stabilization (i.e. DeJong *et al*., 2006) is also unlikely.

During sample preparation, a finite amount of the treated Sangwangsimni fill was put into a small transparent plastic vial and filled with water. Measurements of pH and hardness were taken from day 3 to day 28 and the results are plotted in Fig. 11. The pH of the water in the vial averaged 3.6-3.8 and did not change significantly during the testing period while hardness increased from approximately 800 mg/L to approximately 1,700 mg/L after 28 days. The pH and hardness of the water in the tub used to submerge treated samples were also measured and displayed in Fig. 11. The pH in the tub initially started out at 4 and then increased to 6 after 28 days while the hardness increased from about 200 to 700 mg/L after 28 days. The increase in pH is most likely due to the addition of water to keep the samples submerged and not the uptake of hydrogen ions in a chemical reaction. If a pozzolanic reaction were occurring, the hardness would drop and the pH would significantly increase. Interestingly,

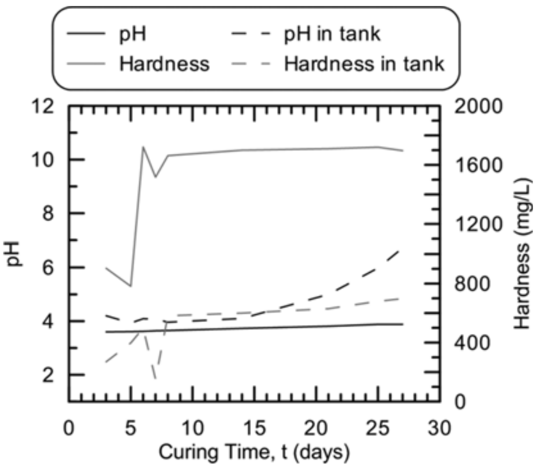

Fig. 11. pH and Hardness from a Small Sample and Water Tub used to Submerge Treated Samples

Table 2. Bacteria Counts for Soil Specimens

Soil Material	Day 0 (CFU/g)	Day 28 (CFU/g)
Sangwangsimni		
Untreated	3×10^6	2×10^6
Treated	3×10^6	8×10^6
No. 5 Quartz		
Untreated	$< 1 \times 10^6$	$< 1 \times 10^6$
Treated	$< 1 \times 10^6$	1×10^6

organic material, such as mold and colored slime, were visible in the solution and on the soil materials in both the vial and tub.

Since organic acids are widely known to fuel microbial activity (Sylvia *et al*., 2005), the role of microbes was explored for the stabilization mechanism. Soil samples were sent to an external consultant to estimate bacteria counts using acridine orange staining and epifluorescence microscopy (Hobbie *et al*., 1977). Results of the bacteria counts, shown in Table 2, indicate the colony-forming-units per gram (CFU/g) of Sangwangsimni materials more than doubled after 28 days when treated with product α. The untreated samples showed little variance in bacteria counts. Not surprisingly, the bacteria counts for the No. 5 Quartz materials were initially low (close to 0) and thus very little growth occurred after curing for 28 days.

In addition to bacteria counts, Loss-On-Ignition (LOI) tests were also conducted on the soil materials. After curing for 28 days, the untreated Sangwangsimni sample had LOI~9% while the treated sample had LOI~22%. The No. 5 Quartz material had LOI~1-2% for both untreated and treated samples.

6. Recommendations

The results show the increase in density state is primarily attributed to the proliferation of microbes and not physical chemical reactions, such as traditional cementation. These microbes use the product α solution as an energy source to multiply and in turn, produce microbial by-products. These by-products occupy the soil void space, to the point of swelling. This is supported by the increases in D_R as curing times increased for the sandy fill samples, but not for the artificial clean sand, where few microbes were found. Thus, for organic acid soil stabilization to work, there must be a sufficient amount of microbes.

Even though product α has shown promise in increasing the q_u of a sandy soil when microbes are prevalent, the amount of improvement may not be satisfactory. ASTM D4609 states that for a treatment to be effective, an increase in q_u of 345 kPa or more must be achieved, which none of the samples in this study demonstrated. Muhunthan and Sariosseiri (2008) showed that for an SP-SM soil material, q_u increased to about 750 kPa from about 200 kPa when 5% cement content was added and cured for 7 days. Anagnostopoulos and Papaliangas (2012) showed a siliceous sand mixed with an epoxy resin to have a 7 day strength of approximately 3,300 kPa while Akbulut and Saglamer (2003) showed a granular soil to have a 7 day strength of approximately 2,700 kPa when mixed with cement, but decreases to about 1,600 kPa when some fly ash is mixed with the cement mixture. Given that granular soils have low unconfined strengths, the strength improvements offered by cement, fly ash, and epoxy resins are much greater than those offered by product α and comparable organic acids. However, an organic acid type stabilizer is much more sustainable and environmentally friendly, as well as economical due to limited material and equipment costs.

7. Conclusions

An organic acid solution made from commercial product α was applied to a fill soil, Sangwangsimni sand, and an artificial clean sand, No. 5 Quartz sand, to evaluate its effect on soil improvement. Unconfined compression strength and oedometer testing revealed an increase in curing times led to general increases in strength, approximately two-fold, and stiffness, approximately one order of magnitude. The variability in data also revealed the difficulties in testing partially saturated specimens.

However, these increases in strength and stiffness were only evident in the fill soil and not with the treated artificial sand. Initial observations showed Sangwangsimni sand samples to slightly swell during curing and 28 day relative densities were over 100%. This study found the increase in density state was caused by an increase in microbes and microbial by-products, fueled by the addition of an organic acid solution, as the stabilization mechanism. This increase in density state, or reduction in void space, would lead to increases in strength and stiffness. Additionally, x-ray diffraction and chemical analyses do not support the hypothesis of traditional cementation processes for the measured improvements from using product α. Thus, microbes are needed for the organic acids to promote any type of soil improvement.

A comparison with other soil stabilization techniques, primarily cement, fly ash, and epoxy resin, show much higher increases in unconfined compressive strength at much shorter curing times relative to organic acids. Although organic acids are inefficient in soil stabilization, they are much more environmentally friendly and economical. Application of an organic acid as a soil stabilization or improvement technique for granular soils would need to be evaluated on a case-by-case basis, with several considerations outlined in this study.

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