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

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 shallow loose granular soils. A brief review on modern soil stabilization techniques in granular materials is presented followed by experimental testing and analysis to help provide some insight into the underlying stabilization mechanism.

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 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 methane produced, which is considered a greenhouse gas. 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.

Experimental Testing

Materials and Preparation

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.

X-ray diffraction (XRD) and chemical analyses of product α revealed it to consist of organic acids with no significant amounts of calcium, aluminum, or silicon. This implies an externally driven pozzolanic reaction is not expected. Neither microbes nor substrates were found in the product α batch received, suggesting microbes inherent in the soil will be needed to produce the purported strength increase.

Two sands were used for laboratory testing. One batch was recovered from a site in the Sangwangsimni area of Seoul, South Korea, and is named Sangwangsimni fill. Sangwansimni fill has FC = 4%, D_{50} = 1.7 mm, C_U = 5.1, C_C = 1.1, γ_{max} = 16.0 kN/m^3, and 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 LL = 39 and a 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. Maximum unit weights were determined from the Japanese method, which is similar to ASTM D4253 (2001). The Japanese method places dry granular soil in a compaction mold using 5 lifts. Each lift is densified by hitting the base plate of the compaction mold with a hammer 200 times. A standard Proctor compaction mold was used in our tests. The minimum unit weight was determined from dry-tipping, which is also similar to ASTM D5254 (2006). 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.

Reconstituted samples were made by mixing an organic acid solution with the previously described sands. 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 sand 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 compressive strength (UCS) 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 estimate 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 growth. 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, UCS and 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 should not be able to stand unconfined for an UCS test. The submerged samples were also allowed to air 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 UCS 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.

Results and Analysis

Initial Observations

Two observations warrant discussion regarding the samples used for testing. Samples prepared in PVC tubes and in oedometers were seen to slightly swell from the onset of curing. A small bulge developed at the top, and sometimes bottom, of each sample, which later had to be trimmed off . Secondly, 3 and 7 day untreated and treated samples did not develop enough matric suction or cementation 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.

Unconfined Compressive Strength

Specimens aged 3, 7, 14, and 28 days were tested for unconfined compressive strength, q_u, with the Sangwangsimni sand results plotted in Fig. 1. The figure shows test results for 3 and 7 day untreated samples to be missing, with the reason being described in the previous section. However, 14 and 28 day samples were able to stand for testing, implying a gain of strength with time perhaps due to mineral precipitation (Baxter and Mitchell 2004). Fig. 1 shows the sand with organic acid treatment to develop an increase in q_u over time, from about 65 kPa after 3 days to about 80 kPa after 28 days. However, samples that were cured underwater show higher q_u than the untreated and treated samples, with no apparent dependence on time. Hollowed symbols in Fig. 1 and subsequent figures represent soil specimens that reached q_u at an axial strain, $\varepsilon_f < 2\%$, suggesting brittle failure.

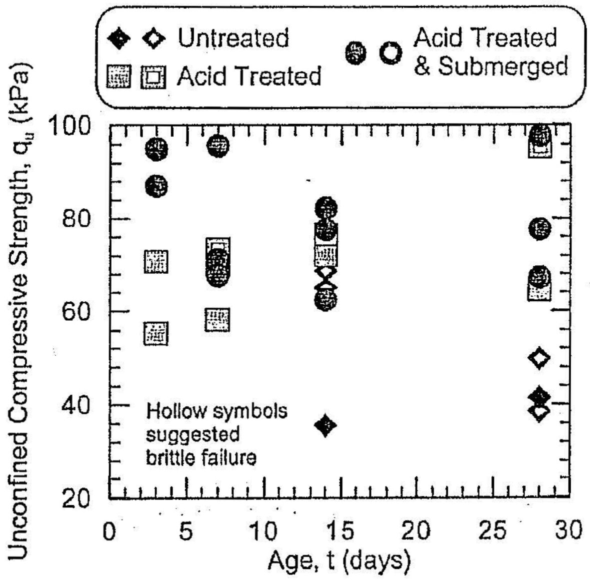

Fig. 1. Unconfined compressive strength of untreated and treated Sangwangsimni sand samples based on curing times.

With the exception of the submerged samples, both untreated and acid treated samples imply specimens that experienced brittle failure to develop larger strengths. Samples cured underwater did not show brittle behavior. Fig. 2 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 samples cured underwater showed an axial strain at failure to be less than 2%.

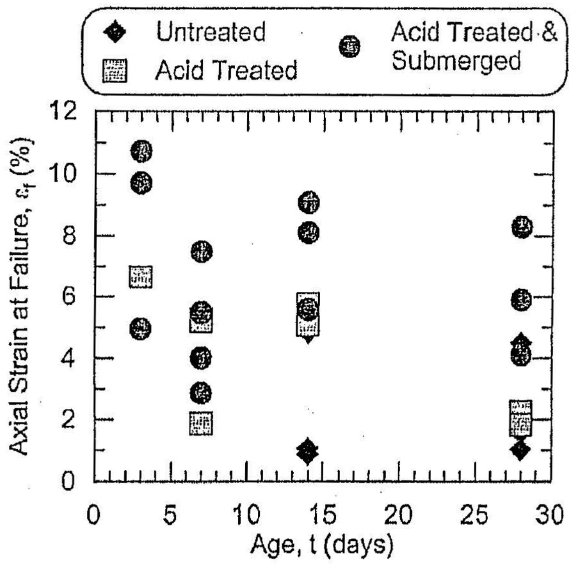

Fig. 2. Axial strain at failure of untreated and treated Sangwangsimni sand samples based on curing times.

In theory and in practice, the strength of cohesionless soils is correlated to its density state, typically measured by relative density, D_R. Fig. 3 plots q_u against D_R and clearly shows higher relative densities lead to higher unconfined compressive strengths. Interestingly, sands treated with an organic acid tended to show D_R > 100%. However, Fig. 4 shows the variation in D_R over time and two minor patterns emerge. One is the wide 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 ~ 90-130% 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.

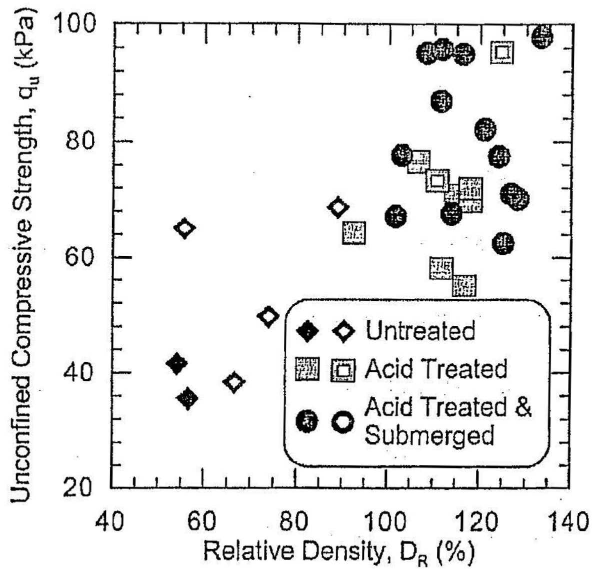

Fig. 3. Relative density of untreated and treated Sangwangsimni sand samples based on curing times.

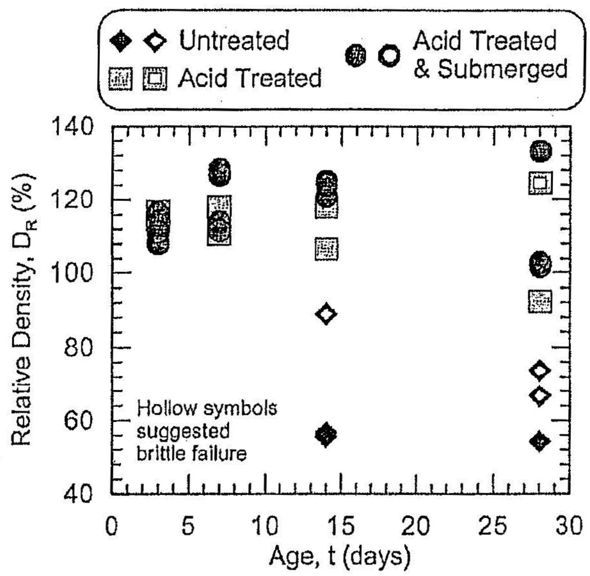

Fig. 4. Relative density of untreated and treated Sangwangsimni sand samples based on curing times.

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. 5 shows the variation of q_u and ε_f against saturation levels. Fig. 5a 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 while Fig. 5b 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 relative density. 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).

Unfortunately, No. 5 Quartz samples were unsuccessfully extracted from their PVC tubes and thus the lack of results. However, it was observed that the No. 5 Quartz sand exposed at the ends of the PVC tubes turned light brown after some time in the oven.

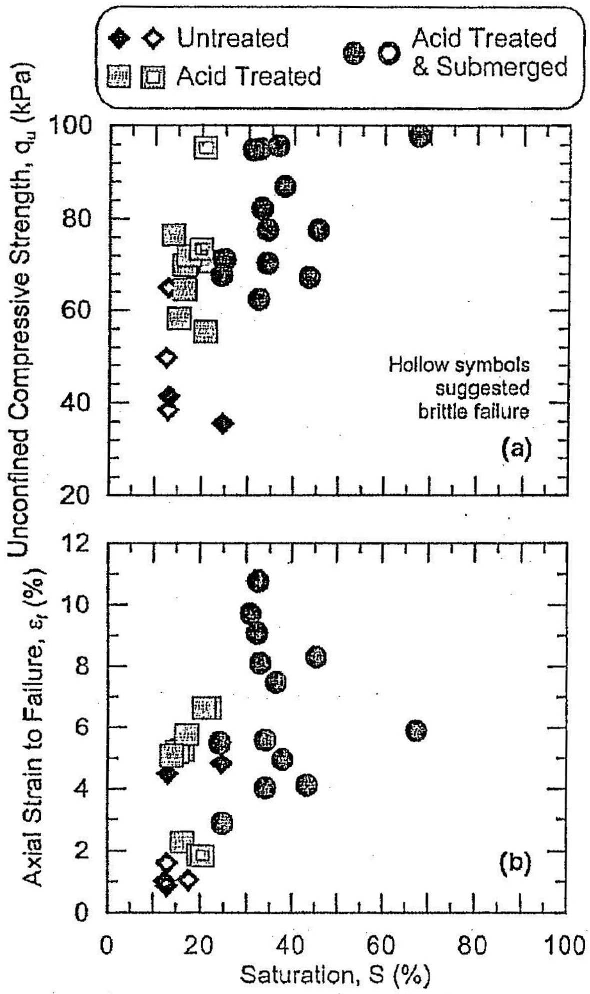

Fig. 5. (a) Unconfined compressive strength and (b) axial strain to failure against saturation level.

Constrained Modulus

The constrained modulus of soil, D_V, is defined as the change in vertical stress over the change in vertical strain when lateral strains are zero, which is more commonly known as the 1D compression modulus of soil. The constrained modulus was estimated for treated and untreated samples by repeatedly applying a load to oedometer samples for 10 cycles and is plotted in Fig. 6 for the Sangwangsimni sand. As expected, test results show D_V to be nonlinear and stress dependent, with D_V being larger for treated samples. It is not known why D_V decreases as the vertical loading stress is increased.

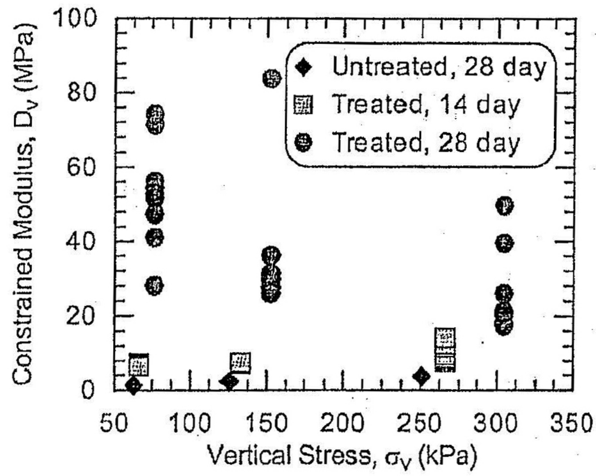

Fig. 6. Constrained modulus as a function of vertical stress for Sangwangsimni sand.

Since it was shown in the previous section that relative density increases for treated samples, Fig. 7 plots the correlation between average D_V and D_R for samples loaded at the lowest vertical stress, σ_V = 60-80 kPa. For Sangwangsimni sand, test results show D_R to increase through time, which is consistent with the UCS test results, and that the treated 28 day sample has a larger D_V on average than the 28 day untreated sample. Additionally, No. 5 Quartz sand test results are shown for comparison. Even though there is a small increase in D_R from 3 to 28 day tests, the D_V decreased from approximately 18 to 3 kPa. Moreover, the results imply the change in D_V is much greater for the Sangwangsimni sand than for the artificial No. 5 Quartz sand.

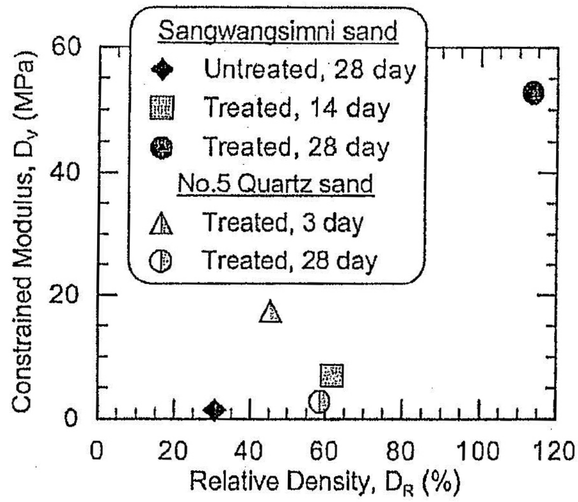

Fig. 7. Constrained modulus as a function of relative density for Sangwangsimni and No. 5 quartz sand.

Chemical Tests

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. 8. Hardness is the concentration of cations in water. The pH of the water in the vial averaged 3.6-3.8 and did not change much during the whole 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. 8. 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. However, it is unknown as to why the water hardness increased during the first 10 days of curing and then stabilized afterwards. The hardness should drop if pozzolanic reactions were occurring.

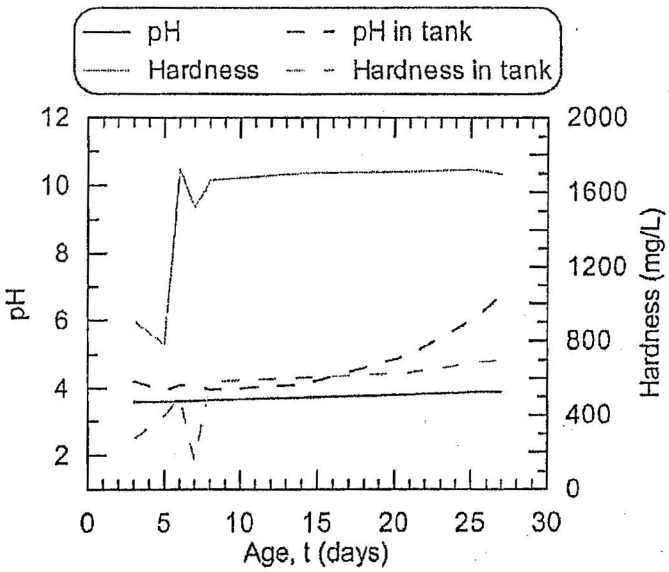

Fig. 8. pH and hardness from a small sample and water tub used to submerge treated samples.

Mechanism

Results from unconfined compression testing, oedometer testing, and elementary chemical testing revealed the primary stabilization mechanism to be an increase in density state. This increase in density state, as measured by D_R, is apparently brought upon by the growth of microbes inherent to the soil sample and their by-products. The growth in microbes is fueled by the addition of an organic acid solution (Sylvia et al. 2005), product α, and the larger microbe population will result in more microbial by-products to occupy the void space. This is supported by the measured increases in D_R as curing times increased for the sandy samples taken from a site, but not for the artificial clean sand. Thus, for organic acid stabilization to work in sandy soils, there must be microbes.

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 proposes 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 through void space reduction.

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