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Experimental Study to Determine an EICP Application Method Feasible for Field Treatment for Soil Erosion Control

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ABSTRACT

The end goal of this research is assessing the feasibility of using enzyme induced carbonate precipitation (EICP) to create a cemented top layer to control runoff erosion in sloping sandy soil. The paper presents the results of an experimental study of bench-scale tests on EICP-treated sands to determine a treatment method feasible for field placement for this application. The soils tested were two natural sands and Ottawa 20-30 sand used as control. The EICP application methods were percolation by gravity, one-step mix-compact, and two-step mix-compact. Other conditions considered were pre-rinsing the sand prior to treatment, adjusting soil pH prior to treatment, and changing the EICP solution concentration. Promising results for this field application were obtained using the two-step mix-compact when the soil was first mixed with the urease enzyme solution before compaction. Considering that the EICP reaction starts once all components are added, this method would ensure that the reaction does not take place before the protective layer of treated soil has been installed. The effect of pre-rinsing the natural sand was not consistent throughout the testing conditions and its role in improving soil cementation in natural sand needs further study.

INTRODUCTION

Erosion of sands is an important issue in geotechnical engineering. Civil engineering and transportation projects frequently involve creating soil slopes and embankments susceptible to runoff erosion that need protective measures (Krenitsky et al. 1998; Álvarez-Mozos et al. 2014a, 2014b; Luo et al. 2013). Various methods, such as grass covers, hydroseeding, compost, polymers, geotextiles and geocells, have been employed to stabilize soils and ultimately provide a remedy to the soil erosion problem (Smets et al. 2007; Montoya 2012; Ao et al. 2016). Some of

these soil stabilization techniques are highly energy intensive in terms of material production or installation and consume natural resources. In addition, some of these erosion control methods are associated with environmental concerns (Montoya 2012). Although grass covers and hydroseeding have been shown to be effective for soil loss control, including in steep slopes, they do not provide immediate protection to the newly constructed embankment or project; seeds can be washed away by runoff before germination, leaving the slope unprotected. In addition, establishing plant growth in dry climates is very difficult (Cerdà 2007; Smets et al. 2007; Luo et al. 2013). The production of geosynthetics usually involves the use of nonrenewable resources; studies have also shown that geotextiles may be effective at controlling runoff under moderate rainfall intensities but not as effective under high rainfall intensities (above 47 mm/h) (Won et al. 2012; Luo et al. 2013). Thus, it is important to develop alternative techniques to control soil erosion caused by water runoff that are cost-effective, environmentally friendly, and sustainable.

Enzyme induced carbonate precipitation (EICP) is a biogeotechnical process in which calcium carbonate is precipitated from the hydrolysis of urea. EICP is a process inspired in the microbially induced carbonate precipitation (MICP), which uses microbes to generate urease enzyme that serves as a catalyst to stimulate the precipitation reaction between an aqueous solution of calcium chloride and urea (Hamdan et al. 2013; Kavazanjian and Hamdan 2015). The EICP is a soil improvement technique with several applications such as enhancing bearing capacity, mitigating fugitive dust, and controlling runoff erosion (DeJong et al. 2013; Hamdan et al. 2013). Some of the advantages of EICP as a ground improvement method compared to the current practice include use of natural materials, smaller environmental impact (e.g., less greenhouse gas emissions), flexibility to serve as temporary and permanent solution, and adaptability to different project conditions and climates. Extensive research has been documented and is ongoing on the use of EICP to stabilize soil for geotechnical applications (Hamdan et al. 2013; Kavazanjian and Hamdan 2015; Hamdan and Kavazanjian 2016).

The research described in this paper is part of a project that aims at developing a runoff erosion control method using the EICP technique. The goal is to create a cemented top layer to control runoff erosion in sandy soil slopes, particularly in areas with arid and semiarid climate. Towards that end, this paper presents results of an experimental study using bench-scale tests on EICP-treated natural sands to identify a treatment method feasible for field placement for this application. Several EICP application techniques and pre-treatment conditions were assessed.

DESCRIPTION OF MATERIALS

The soils tested were two natural quartz sands and standard Ottawa 20-30 sand. The natural sands were labeled herein as NS1 and NS2 and the Ottawa sand was labeled as OT20-30. Sample NS1 was obtained from a quarry in southern New Mexico and is commercially available as stucco sand. NS1 has coefficients of uniformity (C_u) and curvature (C_c) of 2.67 and 1.31, respectively, and contains less than 1% fines (i.e., material passing sieve No. 200) based on results from mechanical sieving. Sample NS2 was collected from the embankment slope of a

ramp of Interstate 10 (I-10) and Avenida de Mesilla interchange (Las Cruces, New Mexico). NS2 has $C_u = 2.50$, $C_c = 1.00$, and about 3% fines. These natural sands were selected because they meet the gradation requirements for highway embankments and are soils susceptible to runoff erosion in unprotected slopes. Ottawa 20-30 sand is a standard quartz sand with most of its particles within the size range defined by sieves No. 20 and 30 (0.85 mm and 0.60 mm, respectively) and contains no fines. It has $C_u = 1.19$ and $C_c = 0.95$. All three sands are classified as poorly graded sands (SP) according to the Unified Soil Classification System (USCS) (ASTM International 2011). The particle size distribution curves of the sands are shown in Figure 1.

The EICP solution was composed of urea (Sigma-Aldrich ReagentPlus[®] grade), calcium chloride dihydrate ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, ACS grade), urease enzyme (Fisher Scientific low-grade jack bean urease), and laboratory grade nonfat powder milk. The EICP solution with 1 mol (1 M) concentration contained 1 M urea, 0.67 M CaCl_2 , and 3g/L urease enzyme (200 ml solution contains 12.02 g of urea, 19.7 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 0.6 g of urease enzyme, and 0.8 g of nonfat powder milk). In this study, the EICP solution with 2 mol (2 M) concentration contained 2 M urea, 1.34 M CaCl_2 , and 3g/L urease enzyme. The EICP solution was prepared by mixing a solution of calcium chloride and urea, and then adding this to a solution of non-fat powder milk and urease enzyme. The urease enzyme catalyzes the hydrolysis of urea that causes precipitation of calcium carbonate in the soil.

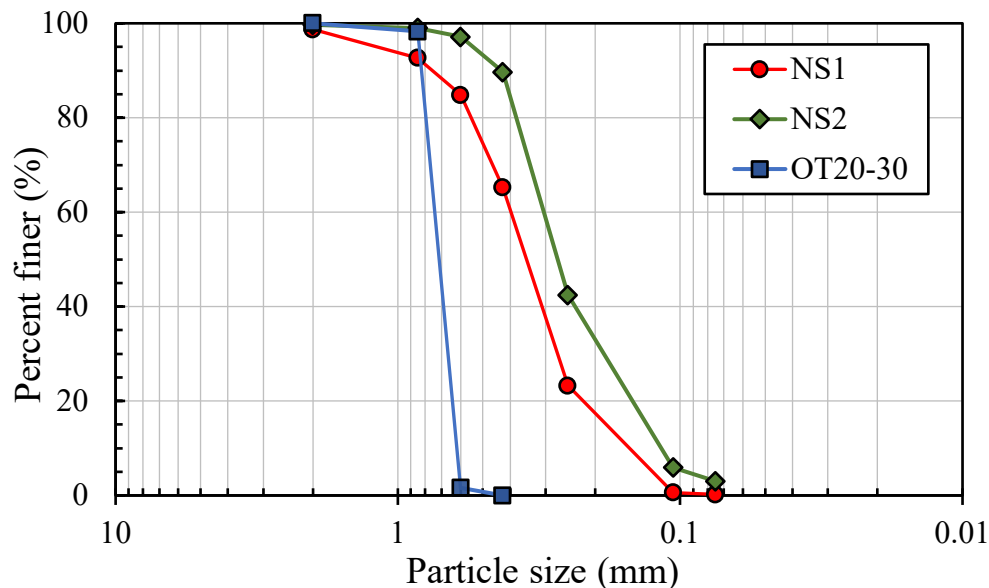


Figure 1. Particle size distribution curves for the tested sands.

METHODOLOGY

Treatment methods. In this study, the treatment application methods considered were percolation of the EICP solution by gravity, one-step mix-compact, and two-step mix-compact. The goal was to explore the potential of these methods for use in the field to create a top layer for runoff erosion control in sandy soils. In the laboratory tests, percolation by gravity involved

placing the sand in a mold. The EICP solution was poured on the top of the specimen and allowed to percolate into the soil by gravity without accumulating on the surface. A syringe was used to control easily the rate of application of the solution. The percolation process lasted approximately 5 minutes. One-step mix-compact consisted of mixing the sand with the EICP solution inside the mold and immediately compacting and forming the specimen. The mixing lasted approximately 3 minutes and compacting lasted approximately 1 minute.

In the two-step mix-compact technique, the components of the EICP solution were not mixed prior to the application (i.e., the solution of calcium chloride and urea and the solution of powder milk and urease enzyme were not combined outside the specimen). Instead, one of the two solution components was first mixed with the sand with a spatula in a small bowl; once the wet sand was placed and compacted into the mold to the target relative density (D_r), the other part of the solution was percolated. Separating the two solution components prevented early precipitation of calcium carbonate before the specimen was formed. Two sequences were tried for the two-step mix-compact method. For Sequence 1, the sand was mixed with 100 ml of the urea and calcium chloride solution; the wet sand was then compacted into the mold, and finally 100 ml of the urease solution was percolated to induce precipitation and cementation. Sequence 2 started by mixing the sand with 100 ml of urease solution; the wet sand was compacted into the mold to form the specimen, followed by percolating 100 ml of the urea and calcium chloride solution. In any of these methods, the total amount of solution applied was the same and corresponded to the estimated pore volume of the sand specimen. In both cases, the percolation lasted 4 to 5 minutes and the entire preparation process lasted approximately 11 minutes.

Pre-treatment conditions. The initial conditions of the soil at the site, such as pH, may affect the EICP reaction and consequently the effectiveness of the treatment (e.g., type and amount of cementation, spatial distribution of precipitation at the pore-scale and at the layer-scale, and strength of the cemented sand). The recommended pH range of the solution for EICP treatment is 7.5 to 9.5 because this is the pH range most amenable to calcium carbonate precipitation (Knorr 2014). The chemistry of mineral particles in the sand may also affect the cementation. Selected tests were performed to explore the effect of some pre-treatment conditions that may help improve the treatment results in natural sands that resist to EICP cementation. The treatment conditions considered in this exploratory program were pre-rinsing the sand with deionized water prior to treatment, adjusting the soil pH prior to treatment, and changing (increasing) the EICP solution concentration. Pre-rinsing of the sand was done by placing the mass of dry sand necessary to prepare a specimen in a medium-size bowl and then adding enough deionized water to cover the sand; after stirring with a spoon a few times, the sand was allowed to soak for two hours at room temperature. The sand was stirred again before draining the water carefully not to lose any of the sand. These steps were repeated three more times without soaking time. The washed sand was oven-dried and cooled to room temperature before preparing the specimen. The pH adjustment was performed by adding a slightly acidic solution to the sand after pre-rinsing and before oven drying the sand.

Specimen preparation. The specimens were formed in molds with dimension of 10.2 x 10.2 x 5.1 cm (4 x 4 x 2 in.) (Figure 2). The molds consisted of lateral walls and were completely open at the top and bottom. The molds could be disassembled to remove the cemented specimen. To support the sand inside the mold during specimen preparation and retain the solution within the soil for longer time, a plastic sheet was wrapped tightly around the mold bottom and fastened around the mold sides with an elastic band. All specimens were compacted to a target D_r of 55% (medium dense sand). Specimen prepared by percolation method were formed by pouring sand into the mold by dry pluviation using a funnel, maintaining a drop height of approximately 76 mm (3 in.). To densify the sand, the mold was shaken back and forth 20 times over the laboratory counter. Specimens prepared with one-step mix-compact method were compacted by tamping the wet soil 15 times in one layer using a rectangular wooden rod. For two-step mix-compact method, the specimens were compacted in two layers by tamping the wet soil 15 times per layer using a rectangular wooden rod. Care was taken to distribute the pressure uniformly throughout the area. These procedures were determined through initial trials to find the compaction energy required to obtain the target D_r and a specimen of approximately 5.1 cm (2 in.) in height. After treatment, the specimens were cured for 5 days before removing the plastic sheet from the mold bottom and disassembling the mold. The cemented specimens were soaked in and rinsed with deionized water for at least an hour to remove ammonia byproduct and determine mass loss. The specimens were allowed to air dry in the laboratory.



Figure 2. Molds with EICP treated specimens.

Test series. The experimental program included three test series. In Series 1, the specimens of NS1 and OT20-30 were treated with 200 ml of 1 M EICP solution. This series was used to assess the feasibility of achieving cementation in natural sand (NS1) and compare it to the cementation in Ottawa sand obtained in prior testing and documented in the literature (Hamdan et al. 2013; Kavazanjian and Hamdan 2015; Almajed et al. 2019). In Series 2, the specimens of NS1 and OT20-30 were treated with 200 ml of 2 M EICP solution. Series 2 was used to assess whether increasing (doubling) the molar concentration of the EICP solution could improve considerably

the cementation of natural sand. These series included seven batches each, representing different combinations of pre-treatment and EICP treatment application methods as listed in Table 1. In Series 1 and 2, four and three replicates respectively were made per condition for each sand, for a total of 98 specimens.

Series 3 focused on the two-step mix-compact method with Sequence 2 to assess if the cementation could be improved compared to Sequence 1. Series 3 also included natural sand NS2 in addition to NS1 and OT20-30. The pre-treatment conditions are shown in Table 2. Series 3 included three replicates per batch for a total of 15 specimens.

Table 1. Treatment conditions in Series 1 and 2 on OT20-30 and NS1.

Batch number	Batch label	Pre-treatment condition	EICP treatment method
1	B1	None	Percolation
2	B2	None	One-step mix-compact
3	B3	None	Two-step mix-compact, Sequence 1
4	B4	Pre-rinsing	Percolation
5	B5	Pre-rinsing, pH adjustment	Percolation
6	B6	Pre-rinsing	One-step mix-compact
7	B7	Pre-rinsing	Two-step mix-compact, Sequence 1

Table 2. Treatment conditions in Series 3 on OT20-30, NS1, and NS2.

Batch number	Batch label	Pre-treatment condition	EICP treatment method
1	B1	None	Two-step mix-compact, Sequence 2
2	B2	Pre-rinsing ^a	Two-step mix-compact, Sequence 2

^a For NS1 and NS2 only.

RESULTS AND DISCUSSION

The visual inspection of the specimens after curing and rinsing indicated that the results of Series 1 and 2 were very similar for a given condition, method, and sand type when comparing the size and shape of the cemented mass. Figure 3 shows representative specimens of Series 1 for OT20-30 and NS1 shortly after rinsing. Ottawa 20-30 specimens cemented throughout for all methods and conditions (B1, B2, B4, B5, B6) except for two-step mix-compact with Sequence 1 (B3, B7). Percolation (B1, B4, B5) and one-step mix-compact (B2, B6) produced the best results for NS1 in terms of size of the cemented mass. During visual inspection, no significant difference in cementation was noticed without or with pH adjustment of the sand as a pre-treatment (B4 and B5 respectively). It was also found that pre-rinsing the sand with a slightly acidic solution did not change considerably the pH of the sand to affect (positively or negatively) the cementation process. Comparing B1, B2 and B3 (no pre-rinsing) with B4, B6 and B7 (pre-

rinsing) for NS1, it was found that pre-rinsing with deionized water helped achieve slight to moderate improvement in terms of the total cemented mass in NS1 when the EICP application method was two-step mix-compact with Sequence 1 (B7) but did not lead to a noticeable improvement when the treatment method was percolation (B4) or one-step mix-compact (B6). Despite a slight improvement with sand pre-rinsing, NS1 specimens prepared with two-step mix-compact with Sequence 1 produced disappointing results (See Figures 3j and 3n). For OT20-30, two-step mix-compact with Sequence 1 produced consistently a relatively thin cemented layer located halfway between the top and bottom of the specimen (B3 and B7) (See Figures 3c and 3g). The formation of a thin cemented layer at the middle of the mold was likely related to the specimen preparation method but this could not be confirmed.

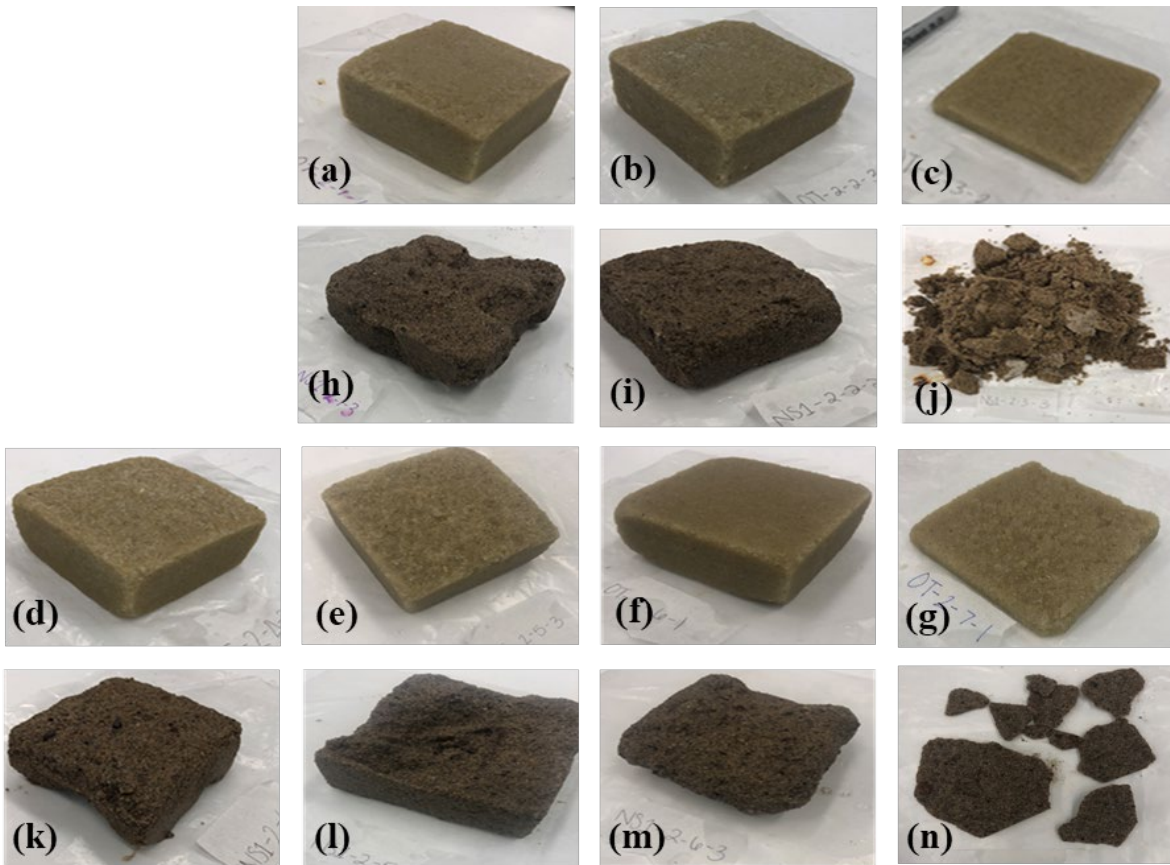


Figure 3. Representative specimens of Series 1: (a) through (g) are OT20-30 specimens; (h) through (n) are NS1 specimens. Testing conditions: (a) and (h) are B1, (b) and (i) are B2, (c) and (j) are B3, (d) and (k) are B4, (e) and (l) are B5, (f) and (m) are B6, (g) and (n) are B7.

After the discouraging results of two-step mix-compact with Sequence 1, Series 3 inverted the sequence of application and mixing into the soil of the two parts of the EICP components (referred to as Sequence 2). Two-step mix-compact with Sequence 2 improved cementation of OT20-30 and NS1 sand (Figure 4) compared to two-step mix-compact with

Sequence 1 (Figure 3) for the same sands and pre-treatment conditions. The improvement in cementation was much more important when NS1 was pre-rinsed. For a visual reference, compared OT20-30 specimens in Figures 3c and 3g with Figure 4a, and NS1 specimens in Figures 3j and 3n with Figures 4b and 4c. Significant cementation was also obtained for NS2 specimens without and with pre-rinsing (B1 and B2, respectively). In Series 3, the mass loss of NS1 was approximately 90% without pre-rinsing (Figure 4b) and 40% with pre-rinsing (Figure 4c). The mass loss of NS2 was approximately 50% without and with pre-rinsing (Figures 4d and 4e, respectively). Pre-rinsing did not have a significant effect on cementation of NS2. The percent loss of fines due to pre-rinsing was 1.2% for NS1 and 2.0% for NS2.

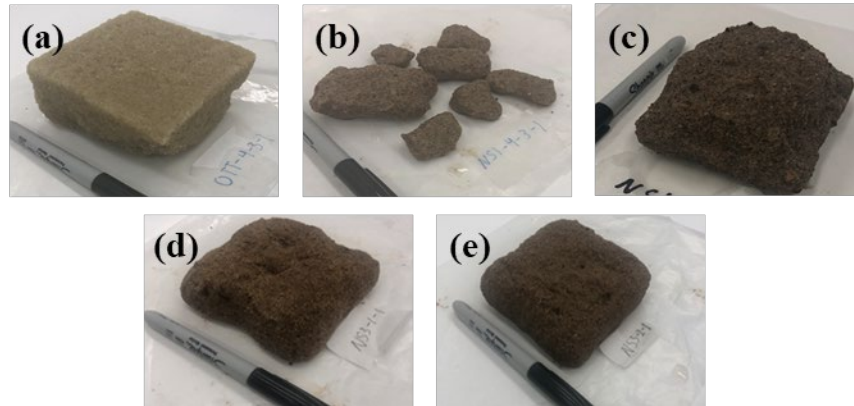


Figure 4. Representative specimens of Series 3: (a) is OT20-30 specimen; (b) and (c) are NS1 specimens; (d) and (e) are NS2 specimens. No pre-rinsing (B1): (a), (b), and (d). Pre-rinsing (B2): (c) and (e).

CONCLUSIONS

This paper presented the results of a laboratory study on EICP-treated natural sands to identify a treatment method feasible for field placement of a top cemented layer for runoff erosion control in sandy slopes. Several EICP application techniques and pre-treatment conditions were considered. Pre-rinsing OT20-30 sand did not make any difference in cementation regardless of the application method. This was expected because Ottawa 20-30 is a clean sand composed mostly of quartz grains. Pre-rinsing NS1 did not make a difference in cementation (determined visually) for percolation by gravity and one-step mix-compact methods but did improve cementation for two-step mix-compact method with Sequence 1. The pH adjustment did not result in visual improvement of the cementation and was not considered a factor to explore further. Also, increasing EICP concentration from 1 M to 2 M did not result in improvement of the cementation for a given sand and method; this indicates that using the lower concentration is satisfactory in future testing because it implies lesser material costs when the method reaches field deployment. Further testing, such as mechanical strength tests, microscopy study, and carbonate content determination, is necessary to further identify differences in cementation among the conditions considered.

The two-step mix-compact method with Sequence 2 produced significant cemented specimens in NS1 and NS2 sands as well as in OT20-30 sand used as control. This method will be further evaluated using other natural sands and scaling up toward field scale. This method provides flexibility considering that the EICP reaction occurs relatively quickly (as soon as 5 to 10 minutes) once all components are added (Hamdan and Kavazanjian 2016). Mixing the sand with one part of the solution, then placing the wet sand on the slope surface to be treated, and finally percolating the other part of the solution would ensure that the reaction takes place in the treated soil.

Finally, several factors like chemical composition of the sand, fines content and mineralogy, and sand gradation may be responsible for the varying results obtained for the natural sands. Some elements such as magnesium have been found to be inhibitors of calcium precipitation and they were found in small amounts in the natural sands tested. Further investigation will be performed to assess the effects of these factors.

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