Durability of a biocemented sand when exposed to wetting-drying cycles

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Abstract: Biocementation was adopted to treat a sandy soil against erosion, to be used for slopes protection. Several samples were prepared in the laboratory to be subjected to 10 wetting-drying cycles, to study durability because the treated soil will be exposed to atmospheric actions in real slopes. The durability of the treatment was evaluated by measuring the evolution of the amount of calcium carbonate precipitated with the cycles, besides mineral analysis. The saturated permeability of the soil was measured before and after the 10 cycles. Pocket penetrometer tests were also performed for different atmospheric conditions. The results indicate that the biocement remained in the soil after these cycles. For the amount of biocement precipitated, the water content in the soil had larger influence on the value of the penetration strength than the amount of biocement.

Introduction

Bio-cementation, or MICP (Microbiologically Induced Calcite Precipitation), consists in using biological agents, such as bacteria, to produce calcium carbonate (biocement). When applied in soils, the bacteria adhere to the particles and the biocement precipitated clogs the voids, introduces roughness and forms bonds, resulting in stiffness and strength increment and a decrease in permeability [1]. There are several studies in the literature where this technique was applied to improve strength [2,3], to reduce permeability [4], to mitigate internal erosion in earth dams and levees [5,6], increase resistance against the formation of ravines [7] and to reduce the collapse potential in soils [8]. There are already some successful real field applications described in the literature (for example [9,10]), being relevant to highlight the use of Biocementation to increase the resistance to erosion caused by wind [11] or by surface water in slopes [12].

The study on the durability of this treatment is fundamental to promoting this treatment in real applications. As far as the authors know, durability has been investigated by studying the effect of freeze-drying cycles on biocemented soils [13,14] and improvement against erosion by submersion in water [15]. Durability was investigated in the work presented in this paper. Several samples of the same soil were prepared in the laboratory and treated by

biocementation. Then, they were subjected to 10 wetting-drying cycles in two different environments. The durability of the treatment was evaluated by measuring the evolution of the amount of calcium carbonate precipitated with the cycles, besides mineral analysis. Pocket penetrometer tests were also performed for the two different atmospheric conditions. The durability of this treatment is still not properly investigated and can be a valuable contribution to current knowledge. The different atmospheric conditions promote different degrees of saturation for the soil, which may affect the precipitation of biocement or promote its dilution.

Materials and Methods

Bacteria and feeding solution

Sporosarcina pasteurii or Bacillus pasteurii, is the bacteria species usually adopted because they are easy to find in Nature and are non-pathogenic. They are introduced into the soil with a nutrient solution that contains urea ($CO(NH_2)_2, 0.5M$) and calcium chloride ($CaCl_2$, also 0.5M). The bio-cement production process is based on the hydrolysis of urea (Eq.1). Calcium carbonate ($CaCO_3$) results from the reaction between carbonate ions (CO_3^{2-}) and calcium ions (Ca^{2+}) from calcium chloride (Eq.2).

$$CO(NH_2)_2 + 2H_2O \rightarrow 2NH_4^+ + CO_3^{2-} \quad (1)$$

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3 \quad (2)$$

Calcium carbonate occurs in the form of three minerals, calcite, vaterite, and aragonite. Calcite is the mineral form intended for durability because it is insoluble [16].

The bacteria were growth at 30°C in a culture medium prepared at pH 9.0 with yeast extract and ammonium sulphate, until reaching the concentration of 10^8 cells/mL (optical density at 600nm, OD_{600}=1). The nutrient solution was prepared with calcium chloride (0.5 M), urea (0.5 M), diluted growth medium, sodium bicarbonate and ammonium chloride.

Soil and sample preparation

The soil used classifies as a well-graded sand (SW), with D_{50}=0.65 mm and 1.6 % of material with diameter D< 0.075 mm [17]. The volumetric weight of the particles is 26.1 kN/m^3. Quartz is the main mineral present.

The samples were prepared with some water to simplify their deposition in plastic containers (22 x 13 x 9 m^3). The water content at preparation was 5%, and the dry volumetric weight was 16.5 kN/m^3, corresponding to a void ratio of 0.58. The soil was placed in a 3 cm thick layer, above a 2 cm thick layer of gravel that worked as a drain, being both layers separated by filter paper. The treatment fluids and the water from the wetting-drying cycles were collected in a pipe (Fig 1.a). The surface was smoothed before applying the treatment (Fig 1.b).

a)  b)  c) 

Figure 1- Experimental setup [17]: a) samples prepared with the drainage layer: b) sample surface in the laboratory; c) chamber with high temperature and relative humidity.

Treatment protocol

Several boxes were prepared to be exposed to two different environmental conditions: (i) laboratory environment, with average temperature of 22 ± 2ºC and relative humidity RH= 50 ± 1%, corresponding to average climate conditions in Lisbon area; (ii) Climate chamber (Fig. 1 c), with average temperature of 40 ± 2ºC and relative humidity RH= 95 ± 1%, controlled using an UV light. The biocementation treatment was applied already in those environments, having the soil different initial degrees of saturation.

The treatment was carried out in two phases. In the first, with 3 days of duration, bacteria and the nutrient solution were applied. The total volume applied in each day was the void volume Vv, divided in 1/3 Vv of bacteria solution and 2/3 Vv of nutrient solution (10 minutes waiting period between applying the bacteria and the nutrient solution). In the second, in the following 5 days, only nutrient solution was applied, being the volume equal to the void volume in each day. The application of the different solutions was done using a needleless syringe, allowing control over the dispersion of the treatment across the entire surface This is important to achieve the most homogeneous treatment as possible.

Durability tests

Ten wetting-drying cycles were performed on the samples to study the effect of bio-cement washout due to exposure to atmospheric actions. Such conditions promote different degrees of saturation for the soil, which may affect the precipitation of biocement or even promote its dilution. These cycles were applied one week after the treatment. Each cycle had 3 days of duration. In the first day, tap water was added to the soil, using a syringe for a better distribution in the entire surface. The volume of water was equal to the void volume of the soil and the samples were closer to full saturation (not measured). Wetting rate was slow enough to allow full infiltration of water. After this wetting drainage from the bottom was allowed and then the samples were left drying for two days.

Small pieces of material were extracted before wetting, to measure calcium carbonate content, %$CaCO_3$:

$$\%CaCO_3 = \frac{m_{after} - m_{before}}{m_{before}} \times 100 \quad (3)$$

where m_{before} and m_{after} are, respectively, the dry weights measured before and after washing the samples with acid HCl (concentration 0.5 M), being dried in an oven at 105°C for 24 hours.

Saturated permeability

The saturated permeability was measured in an untreated sample, in a treated sample immediately after the treatment before the cycles, and after the 10 wetting-drying cycles in both environments. A variable water head test was done, in samples extracted from the boxes using 7cm diameter steel rings. The rings with the samples were then installed in a permeameter, which water column above the soil has 10 cm height, being recorded the time necessary for occurring 0.5 cm water lever drop, for a total of 2 cm.

Penetration strength

A pocket penetrometer (CONTROLS Geopocket, model 16-T0161) was used to measure the penetration strength. The tip used had 6.4 mm diameter. This strength was measured in the dry biocemented samples one week after the treatment before the cycles, and after the 10 wetting-drying cycles, in both environments. Two measurements were done in each box.

Mercury intrusion porosimetry tests

Smal cubic pieces of material (1x1x1 cm^3) were extracted from the biocemented samples before the cycles, and after the 10 wetting-drying cycles, in both environments for mercury intrusion porosimetry tests. These pieces were lyophilized before the porosimetry tests. No test was performed in the untreated material because it was not possible to extract intact samples.

Mineral analysis by XRD and Scanning electron microscope SEM

X-ray diffraction (XRD) was performed to confirm the presence of biocement, under the mineral forms of calcite and vaterite. These tests were done in pieces of the biocemented samples before the cycles, and after the 10 wetting-drying cycles, in both environments.

Small pieces of the biocemented samples before the cycles, and after the 10 wetting-drying cycles, in both environments were observed in SEM.

Results and discussion

Calcium carbonate content

The evolution of calcium carbonate content along the cycles is presented in Figure 2. The largest oscillations observed are explained by a large heterogeneity of the treatment in the irrigated area, besides the fact that small samples were collected each time, to reduce disturbance. Average final values found were 4.5% for the sample treated in the laboratory environment, and 4.2% for the sample in the wet chamber. These values were smaller than the initial average values measured, which were 8.2% and 7.7% for the laboratory and wet chamber environments, respectively. The fact that similar results were found for the two environments suggests that the different degrees of saturation of the soil in each environment during the treatment have not interfered with the precipitation of biocement. Therefore, the oscillations of the $\%CaCO_3$ measured along time indicate that some carbonate dissolution might have occurred.

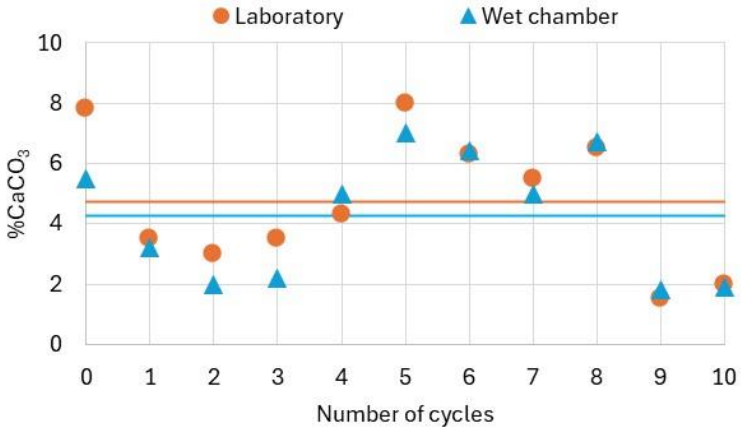

Figure 2: Evolution of calcium carbonate content along the wetting-drying cycles.

Saturated permeability and penetration strength

Table 1 presents the values of the saturated permeability measured, as well as the average values of the penetration strength (measured in three points of each box). The water contents were measured in samples collected closer to the location of the penetration strength measurements. The values of calcium carbonate were not measured and those from Figure 2 cannot be considered because of the large heterogeneity of the treatment.

Concerning saturated permeability, the most significant difference was observed for the samples with and without the treatment. The permeability has increased after the treatment, which was unexpected because the precipitation of biocement is supposed to clog the soil pores. However, this is collapsible soil [8], and for this reason the permeability measured for the

untreated soil has reduced during the measurement. For the treated soil the collapse occurred during the treatment, but also the migration of fines was observed while the bacterial and feeding solutions were added. The differences observed before and after the cycles were not significant if experimental error is considered, and for this reason the wetting-drying cycles appeared not to affect the saturated permeability.

Table 1: Average results of permeability and penetrometer test.

	Without Treatment	Laboratory		Wet chamber	
		Without cycles	After 10 Cycles	Without cycles	After 10 Cycles
Saturated permeability (m/s)	1.11×10^{-6}	1.42×10^{-5}	1.47×10^{-5}	3.01×10^{-5}	1.92×10^{-5}
Average penetration strength (kPa)	167	280	257	243	83
Average water content (%)	3.0	5.1	5.8	13.9	17.9

The average values of the water contents measured have increased after the cycles, maybe because the time allowed for the samples to dry was not enough. This was particularly important for the wet chamber environment.

Although affected by error, the results of the penetration strength tests were consistent. They allowed confirming that penetration strength has increased with the treatment (almost doubled). In addition, penetration strength was strongly dependent on water content, as expected and presented in Figure 3.

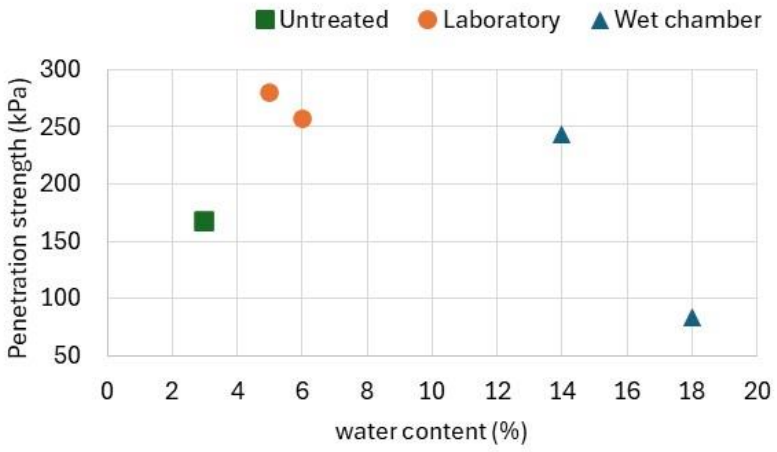

Figure 3: Penetration strength vs water content.

The wetting and drying cycles had some effect on penetration strength as well, reducing it. This could be expected if the evolution of calcium carbonate content reported in Figure 1 is considered, because the average values found at the end of the cycles (4.4% and 4.2%) were smaller than the average values measured before the cycles (8.1% and 7.9%).

Pore size distribution

The pore size distribution curves for the treated materials are presented in Figure 4. The dominant pore size is 90 μm in the laboratory constant before and after the cycles, reducing from 110 μm into 60-90 μm after the cycles in the wet chamber. Other smaller peaks were identified: at 4 μm for all cases, at 0.07 μm only for the samples treated in the wet chamber, before and after the cycles, and at 0.006 μm also for all cases. The last may be experimental error because it is in the very high pressures. It appears that the pores are slightly larger when the treatment was done in the wet chamber, treatment possible because some water was trapped in the soil and no biocement could be precipitated in the pores. The cycles led to a small pore size reduction, possible because of the collapsible nature of the soil. The fact that the large peak displaced into the smallest dimensions in the samples with the highest pore size is in accordance with the collapse behaviour of the soil [8]. This collapse behaviour was not eliminated with the treatment, specially because the amount of biocement precipitated was not very large.

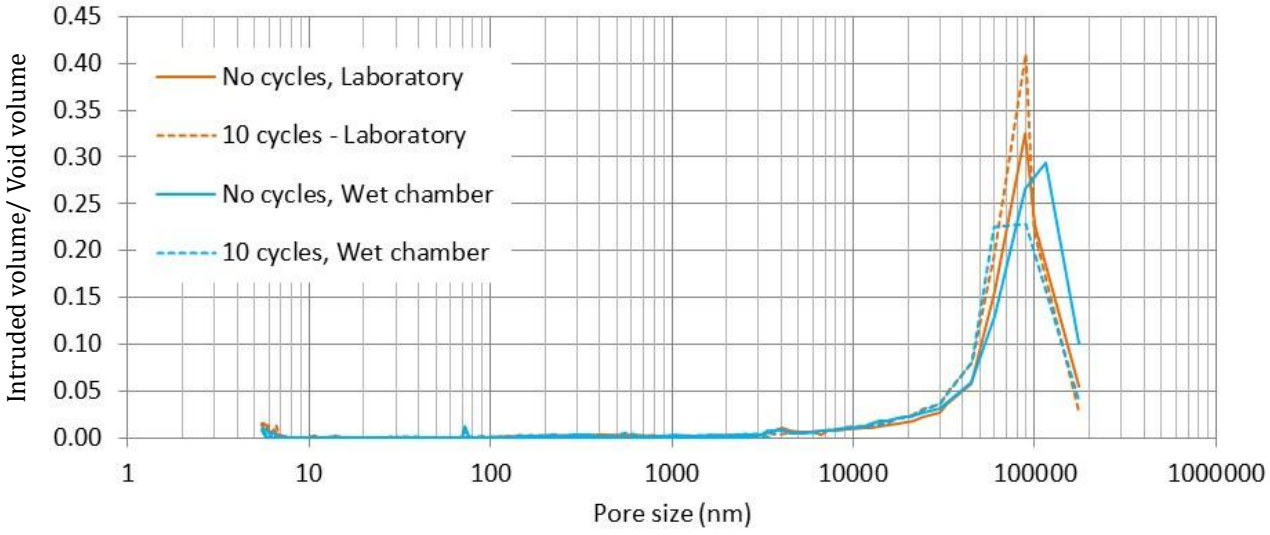

Figure 4: Pore size distribution curves.

XRD results and SEM images

The minerals present identified by XRD analysis confirmed the presence of biocement, because the mineral forms of calcite and vaterite were detected. The other minerals identified, such a quartz and albite, are from the soil. Both biocement minerals were detected before and after

the cycles, therefore the cycles were not able to wash them away. No differences were detected for the samples prepared in the different environments investigated before or after the cycles.

SEM images presented in Figure 5 for the samples before and after the treatment in the two different environments confirm the presence of the biocement minerals. Their morphology is slightly different, as the minerals are irregular when the treatment was done in laboratory environment (temperature of 22 ± 2ºC and RH= 50 ± 1%) while spheres were observed in the wet chamber (temperature of 40 ± 2ºC and RH= 95 ± 1%). None of them are typical calcite minerals, with rhombohedral shape, but they can be attributed to vaterite. Such shapes may be explained by the different temperatures and availability of water during precipitation, and further investigation is required. EDS tests performed on the minerals, not shown, have detected the presence of calcium and oxygen and this is consistent with the presence of biocement.

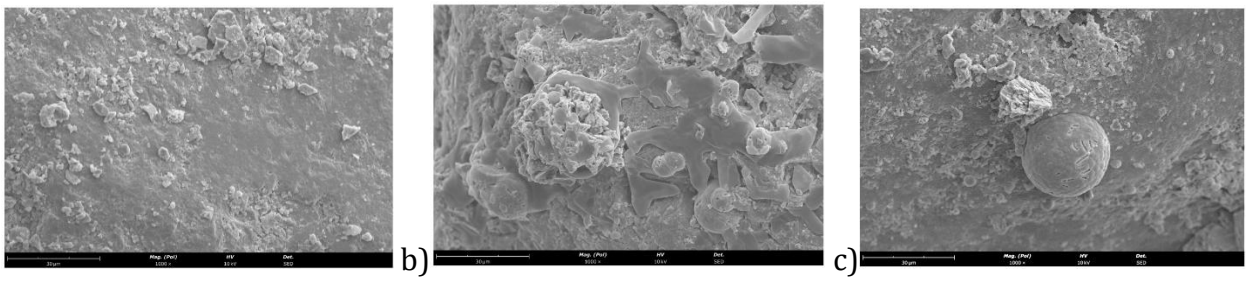

Figure 5: SEM images for the samples without the cycles: a) before the treatment; b) after the treatment in laboratory environment; c) after the treatment in the wet chamber.

Conclusions

Several tests were performed on samples of the same soil treated by biocementation to study the durability of the treatment under 10 wetting-cycles in two different environment conditions: (i) laboratory environment (temperature 22 ± 2ºC, RH= 50 ± 1%), and (ii) climate chamber (temperature of 40 ± 2ºC, RH= 95 ± 1%). The environmental conditions, or the different degrees of saturation of the soil, appeared not to affect the amount of calcium carbonate precipitated nor the durability of the treatment, because similar calcium carbonate contents were measured before and after the cycles. Nevertheless, pore size distribution form MIP tests allowed detecting larger pores when the treatment was done under high relative humidity, possibly because of the presence of liquid water in the pores. Some differences on the shape of the biocement minerals visualized in SEM images were also found for the two different environmental conditions, possible because of water, however this topic requires deeper investigation.

Because the treated soil is a collapsible soil, pore sizes reduced slightly after the 10 wetting and drying cycles applied, for both environments. The amount of biocement appeared to slightly

reduce with the cycles (average values found were 8.1% and 7.9% before the cycles and 4.4% and 4.2% at the end of the cycles), which may indicate that some biocement was washed away. Such measurements were affected by large dispersion because of the treatment procedure adopted, consisting in irrigating the entire area.

The application of 10 wetting-drying cycles may not be enough to investigate durability and further investigation, applying much more cycles, is required. Nevertheless, this number was enough to affect penetration strength because its average value has reduced after the cycles. The reduction was more marked for the samples in the wet chamber, with larger water contents. Although pocket penetrometer tests are not a reliable tool for determining soils strength, their use has provided consistent information on the characteristics of the soil cover after the treatment and successive wetting-drying cycles.

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