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Mechanical and physicochemical study of a cemented sand exposed to wetting and drying cycles

Etudes mécanique et physico-chimique d'un sable cimenté soumis à des cycles hydriques

A. Wassermann, A. Abdallah, O. Cuisinier*
LEMTA – Université de Lorraine – CNRS – Nancy, France

C. Diliberto, J.-M. Mechling
IJL - Université de Lorraine – CNRS – Nancy, France

**Olivier.Cuisinier@univ-lorraine.fr*

ABSTRACT: The core objective of this study was to analyze the impact of wetting/drying cycles on the mechanical behavior of a cement-treated sand. A comprehensive microstructural and physicochemical analysis were also performed to determine the physicochemical processes occurring in the samples along the cycles. Two types of wetting and drying cycles were applied to a 4%-cement treated sand. The results showed that although the cycles only slightly altered mechanical performance, they induced major mineralogical transformations in the cementitious phases. The results underline the importance of a protocol of imposing wetting/drying cycles for a more accurate assessment of the long-term performance of treated soils.

RÉSUMÉ: L'objectif principal de cette étude était d'analyser l'impact des cycles d'humidification/séchage sur le comportement mécanique d'un sable traité au ciment. Une analyse microstructurale et physicochimique complète a également été réalisée pour déterminer les processus physicochimiques se produisant au cours des cycles. Deux types de cycles hydriques ont été appliqués à un sable traité au ciment à 4 %. Les résultats ont montré que bien que les cycles n'altèrent que faiblement la performance mécanique, ils induisent des transformations minéralogiques majeures des phases cimentaires. Les résultats soulignent l'importance du protocole de cycles d'humidification/séchage imposé pour une évaluation plus précise de la performance à long terme des sols traités.

Keywords: Soil stabilisation; shear strength; dilatancy; microstructure; durability.

1 INTRODUCTION

Soil stabilization with hydraulic binders is employed to improve soil properties for the construction of earth structures such as road bases and subbases, dikes, and backfills. Several authors have shown that wetting/drying cycles can alter the mechanical behaviour of treated soils. The extent of the degradation is a function of several parameters, such as the initial dry density of the stabilized soil, the moisture content, mineralogy, and the amount of binder. An important additional factor is the method employed to apply wetting and drying cycles in laboratory studies. Numerous approaches for imposing drying/wetting cycles have been employed with varying durations for the wetting and drying phases, wetting techniques (such as capillary rise or immersion), and drying phase temperatures (air-drying or oven-drying). Most earlier studies applied cycling conditions derived from the ASTM D559 (ASTM 2015) standard, which suggests immersing for 5 hours in water at room temperature, followed by 42 hours of oven-drying at 71°C. However, this method can be considered to be aggressive compared to solicitations endured on-site by geotechnical structures since it includes full immersion of the samples and complete drying under temperature. Some authors evaluated the impact of the amplitude of the wetting and drying cycle; for instance, by applying suction control techniques. They reported that mechanical performance degradation increased with the magnitude of the wetting/drying cycles (e.g. Menaceur et al., 2021). The impact of the wetting protocol should also be carefully considered. The immersion of samples in water can also induce progressive leaching of the treatment product over time, which may result in lowering the soil performance in the long term. Thermal conditions are also of primary importance since the setting reactions of cement are impacted by temperature.

Varying experimental conditions may also trigger different alteration mechanisms associated with the cycles leading to significant behaviour change compared with what could occur in situ. Some authors also highlighted the role of carbonation on the long-term behaviour of stabilized soils. The extent of the degradation caused by carbonation is a function of temperature and relative humidity (Vitale et al. 2021).

In this context, the main purpose of this study was to analyse the impact of the drying protocol on the ageing of cement-treated sand exposed to wetting and drying cycles at different scales. The analysis of the mechanical behaviour of the samples was associated with an investigation of the microstructure after the cycles, and completed by a physico-chemical analysis of the cement-treated sand.

2 MATERIALS AND METHODS

2.1 Sample preparation

The selected soil is a sand sampled in the eastern part of France. According to the French classification system (GTR, 2023), it is classified as an S1-type soil, and in accordance with the Unified Soil Classification System, it is categorized as an SW soil. The D_{50} is 0.8 mm. The Portland cement chosen for this study is CEM I 52.5 N. This cement has a specific gravity of 3.15. The sand and cement were hand-mixed, and then water was added to reach a water content of 7%. Once the mixture was homogeneous, the blend was compacted in three layers with a piston and a manual press in a two-part mould (100 mm high and 50 mm wide). The targeted dry unit weight was 17 $kN.m^{-3}$.

2.2 Wetting and drying cycle protocol

To assess the impact of the intensity of the cycles on the mechanical behaviour, two different methods of wetting/drying cycles were used. The ASTM standard D559 (ASTM, 2015) served as the basis for the type-I cycle. The samples were wetted by being submerged in water at room temperature for 8 hours and dried in an oven for 16 hours at 65 °C. The type-II cycle protocol was inspired by the literature (Mehenni, 2015). Water immersion for 48 hours was used to achieve the wetting process. The drying phase was conducted in a climatic chamber at a temperature of 20 °C and a relative humidity of 50% for 120 hours.

The main difference between the two cycles is the drying protocol. Compared to the type-I cycle, the type-II cycle was thought to be less aggressive. Its purpose was to impose weathering in accordance with the environmental conditions that an engineered structure is anticipated to be exposed to over the course of its lifespan.

2.3 Triaxial tests

Triaxial testing campaign was carried out with consolidated drained (CD) tests that were run on a conventional triaxial system apparatus. In this paper, only the results obtained with one confining effective pressure of 100 kPa are introduced. The shearing velocity was fixed at 0.1 mm/min. More details about the experimental protocol are available in Wassermann et al. (2022).

2.4 Physico-chemical test protocols

Microlevel observations were performed on fresh breaks on the sample using a scanning electronic microscope (SEM). Secondary electron (SE) images were processed at an accelerating voltage of 15 kV and a working distance of 15mm. The SEM used in this study was a TESCAN® Vega3. The samples were stored in a desiccator after the cycles or after curing time before being oven-dried at 65°C. Then, they were cut into 1 to 2cm cubic pieces, and the surface was metallized. The X-ray diffraction technique (XRD) and thermogravimetric analysis/differential thermal analysis (TGA/DTA) were used to track any alteration of the cementitious phases during the cycles.

2.5 Experimental program

Specimens prepared with 4% cement were submitted to wetting/drying cycles. Each specimen was duplicated with a control specimen that was made at the same time and kept in sealed containers at a controlled room temperature. While the test sample was put through wetting-drying cycles, the control specimen was not subjected to any solicitation. The triaxial tests were then performed on the same day on the two samples after 1, 3, 6, 9, 12, 18 and 24 cycles. For the type-I cycles, the whole procedure required almost one month of experiments, while it required 6 months for the type-II cycles. The physico-chemical analyses and the microstructural investigations were performed on samples exposed to 24 cycles as well as on a sample cured in a sealed container.

3 MECHANICAL BEHAVIOUR

For a 4%-cement-treated sand after a 14-day cure at a constant water content, the maximum deviatoric stress q_{max} has been determined to be close to 1550 kPa by performing 4 triaxial tests. This value will be taken as a reference to quantify the impact of the cycles.

After one cycle, q_{max} was equal to 1146 kPa and 1268 kPa for the type-I and type-II cycles (Figure 1 & 2). The first cycle decreased the maximum deviatoric stress by 26% for the type-I cycle and by 18% for the type-II cycle compared to the reference specimen cured at a constant water content. Subsequent cycles led to a slight increase in q_{max}. For both cycle types, there was an increase in q_{max} after the first three cycles, up to 1376 kPa for the type-I cycles and to 1323 kPa for the type-II cycles. For the type-I cycles, the maximum deviatoric stress then decreased until the 12th cycle, reaching a value of 1022 kPa. Then, until the 24th cycle, the maximum deviatoric stress tended to stabilize. One can notice a decrease in the secant modulus at 50% strength, E_{50}, with cycles.

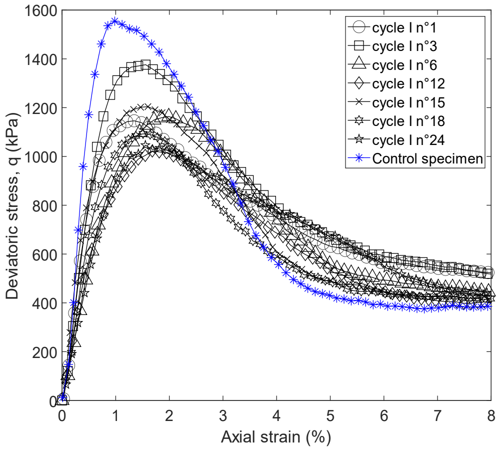

Figure 1. Influence of the wetting–drying cycles on the stress–strain behaviour after type-I cycles.

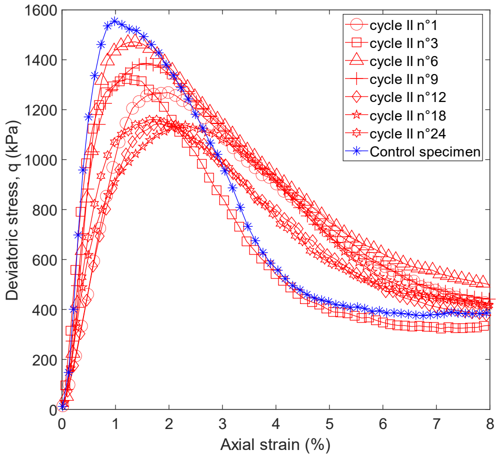

Figure 2. Influence of the wetting–drying cycles on the stress–strain behaviour after type-II cycles.

Concerning the type II cycles, q_{max} continued to increase until the 6th cycle to reach a value of 1472 kPa. Then, it decreased until the 12th cycle down to 1145 kPa (-26% compared to the control specimen). Similarly to the type-I cycles, the values of the maximum deviatoric stress remained constant until the 24th cycle (1138 kPa after 18 cycles and 1160 kPa after 24). A comparison of the maximum deviatoric stresses after each cycle is shown in Figure 3. A decrease in the E_{50} stiffness with cycles can also be noticed.

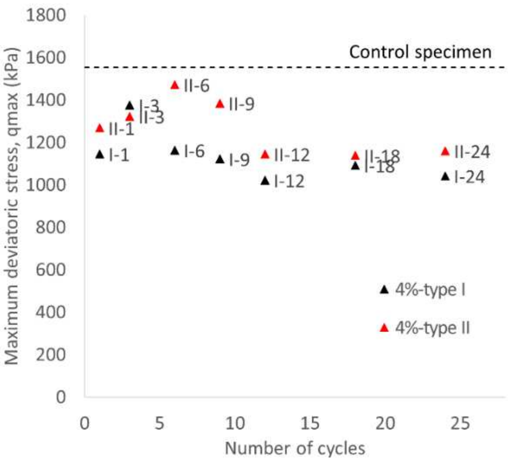

Figure 3. Influence of the type and the numbers of wetting–drying cycles on the maximum deviatoric stress.

4 SEM OBSERVATIONS

The cementitious gel seems to homogeneously encapsulate the surface of the sand grains and create a coating on the grains (Figure 4a and b). After 24 type-I cycles, the porosity remains open with pores of approximately a few tens of micrometers (Figure 4c and d). At 350X magnification, the cementitious paste still seems to coat the sand grains. Some cracks are noticeable all over the surface and are observed in what appeared to be secondary compounds. These cracks are more visible at a magnification of 1000X. Additionally, at this magnification, some needle-shaped compounds of approximately 20 µm in length were detected inside the pores. After 24 type-II cycles the porosity seems more closed than in the other samples (Figure 4e and f). At a higher magnification factor, needle-shaped components are found; they seem to be more frequent than after type-I cycles and cover almost the entire surface of the observed quartz grains. Additionally, the needles appear longer than 20 µm, indicating that they are likely to still be developing in the pores.

5 THERMOGRAVIMETRIC ANALYSIS

For the sample cured at a constant water content, there is a peak at approximately 110 °C to 180 °C, which corresponds to the decomposition of hydrated cementitious compounds such as ettringite or CSH and also residual water (Figure 5). There is a peak at approximately 450 °C, which is the identification peak for portlandite decomposition. The TGA for the samples after cycles (types I and II) showed that there is an important mass loss between 600 °C and 800 °C, which corresponds to the loss of carbonates ($CaCO_3$) such as calcite. In the case of the sample after 24 type-II cycles, there is a large peak in the range of 110 to 180 °C, indicating the presence of cementitious compounds such as ettringite. This peak is less visible after the type-I cycles. For the two samples after the cycles, there is no peak at approximately 450°. The amount of calcite is higher after type-II cycles than after type-I cycles, which is in good agreement with the XRD observations.

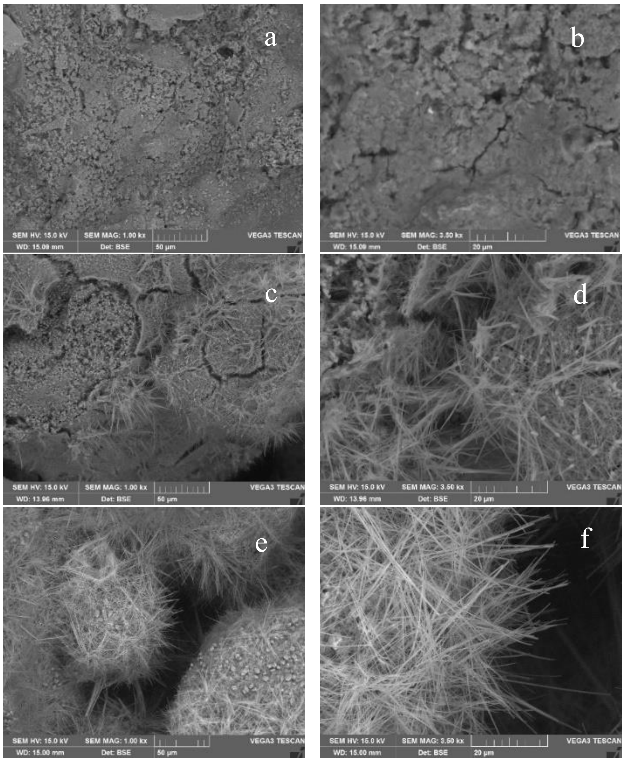

Figure 4. Microstructure of the samples before (a & b) and after 24 cycles I (c & d) and 24 cycles II (e & f).

6 CONCLUSION

The aim of this study was to better understand the processes induced by a large number of wetting/drying cycles, both mechanical and physicochemical. The results showed that the cycles induced a limited degradation of the mechanical macroscopic behaviour of the sand samples after the 24th cycle. Despite this limited impact of the cycles at the macroscale, the physico-chemical analysis and the microscopy showed that the cycles had a strong impact on the binding phases with two major processes associated: carbonation and the formation of ettringite.

Modifying the wetting and drying processes not only slightly impacted the macromechanical behaviour but also the microstructure by influencing the physico-chemical processes involved. Indeed, hydraulic binders are sensitive to several external conditions, such as temperature or humidity variations, that could promote processes such as carbonation. This phenomenon was evidenced in the samples after the wetting and drying cycles.

This study demonstrated that the intense mineralogical transformations that affected the bonding only had a limited effect on the macroscopic mechanical behaviour of the cement-treated soil. It also highlighted the importance of the cycling conditions. Indeed, it has been shown that the type of cycles has an impact on both the mechanical behaviour and its degradation as well as on the extent of ettringite formation and carbonation.

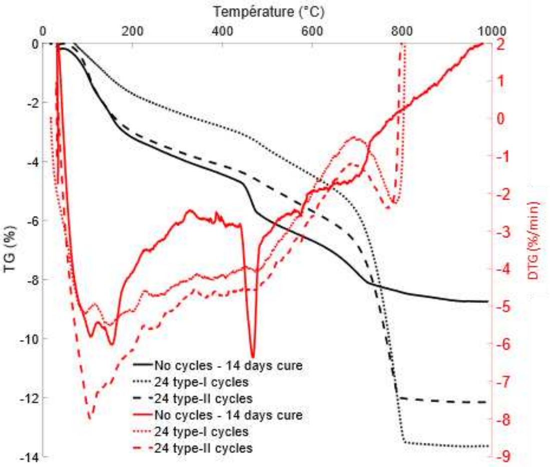

Figure 5. Thermogravimetric analysis curves after curing at constant water content and after the cycles.

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