

## Influence of wetting fluids on the compressive strength, physicochemical, and pore-structure evolution in lime-treated silty soil subjected to wetting and drying cycles

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g fluid, thus, affecting its durability. However, studies regarding such impact are limited. In this study, the influence of different wetting fluids on the microstructural, physicochemical modifications, and Unconfined Compressive Strength (UCS) evolution of lime-treated soil is investigated. Lime-treated soils are subjected to wetting and drying cycles using 0.10 M and 0.60 M NaCl solutions, demineralized water, and Methyl Methacrylate. The results show that despite the soil being exposed to several wetting and drying cycles, the UCS evolved positively compared to the reference specimen. However, this evolution varies with the types of wetting fluids the specimens are exposed to. The NaCls-and Methyl Methacrylate-subjected soils showed an increase in UCS up to about 3 times, while the UCS increase is about 2 times in

the corresponding demineralized water subjected soil. Similarly, the extent of the variation of the final pH and the cumulative calcium concentrations measured in the effluent is marked by the wetting fluids available in the surrounding medium. At the pore structure level, the presence of NaCl solutions and Methyl Methacrylate solvent as a wetting fluid was observed to enhance the evolution of pores smaller than 3000 Å. However, such a phenomenon is less significant in the demineralized water-subjected soil. Thus, the study confirms that the type of wetting fluid plays an important role in the evolution of lime-treated soil during the wetting and drying cycles.

Keywords: Wetting and drying cycles; physicochemical; microstructure; Lime-treated soil; wetting fluids.

#### **1. Introduction**

Improvement of soil characteristics such as strength, bearing capacity, elastic modulus, etc., through lime treatment is a current practice in geotechnical engineering. Such an improvement is a consequence of combined modifications brought through chemical and mechanical stabilizations. Two basic chemical reactions called short-term and long-term pozzolanic reactions occur during chemical stabilization. The short-term reactions bring physical improvement in the treated soil by reducing the plasticity and increasing the workability of the soil [1-3]. The long-term pozzolanic reactions create cementitious compounds such as Calcium Silicate Hydrates (C-S-H), Calcium Aluminate Hydrates (C-A-H) and modify the soil fabrics [4-13]. The mechanical stabilization involves the execution process, such as the soil-binder mixing process, using an appropriate binder and water contents during compaction and mixing, implementing the accurate compaction conditions [6, 14-15].

So far, few field investigations have reported the sustainability of earth structures attained through such improvements [5, 8, 16-19]. Akula et al. [16] studied the performance of the Friant-Kern Canal in California, the United States, which was built with 4% quicklime treated plastic soil after 40 years from treatment. Based on the investigations, they have reported increased long-term strength, reduction in swelling potential, erosion resistance, thus indicating a desired geo-mechanical stability of the lime-treated

structure. Dawson and McDowell [17] monitored the Unconfined Compressive Strength (UCS) of a pavement built with 3% lime-treated clayey gravel. After 14 years from construction, a UCS value ranging from 2.07 MPa to 4.14 MPa was obtained, which was observed to be 6 to 12 times higher than the one obtained from the untreated soil. Jung et al. [18] observed an increase in resilient modulus from 96.5 MPa to 703.2 MPa in a 4% lime-treated expansive soil pavement after 11 years from construction. Recently, Das et al. [5,8] demonstrated the long-term impact of 2.5% quicklime lime treatment on an experimental embankment exposed to a damp climate for 7 years. UCS evaluation of core-sampled specimens was made, which showed an average value of  $3.29 \pm (0.45)$  MPa despite being the exposure to such a severe climate. Such significant evolution in strength was explained in detail through physicochemical and microstructural investigations.

Thus, the above in-situ studies have evidenced the successive improvement brought by lime treatment. However, an exposed lime-treated hydraulic earth structures, particularly the surface cover is often prone to several drastic environmental situations. For instance, when hydraulic earth structure remains permanently in contact with water, it is subjected to several fluctuations in water level [20-23], which induces ingression or egression of water in the structure. Such impact on the lime-treated structure was evaluated at a laboratory scale by subjecting the treated soil to successive wetting and drying cycles as per the procedure mentioned in ASTM D 559 [24]. Subjection of lime-treated soil to severe wetting and drying cycles was shown to bring deterioration in the improved pore-structure of the soil attained from lime treatment [25]. Such a deterioration was reported to bring total loss in improved hydromechanical performances of the lime-treated soil [26-27].

However, during the wetting phase of the wetting and drying cycles, infiltration of water occurs into the subjected soil, which interacts with the treated soil components. Since natural water can be constituted of several organic compounds or inorganics compounds or a mixture of both, which control the pH level of the water [28-29], thus, the chemical nature of the infiltered water can modify the improvement of the lime-treated soil. Soil pH in a lime-treated structure contributes greatly towards the maintenance of the long-term performance of the concerned structure, as shown by Das et al. [5,8]. Hence, subjecting the 76 lime-treated soil to extreme wetting and drying cycles accompanied by a wetting fluid of different chemical 77 nature might impact the soil pH and can modify the long-term performance. Such a possibility remained 78 less investigated.

In this context, the present study is focused on investigating the influence of different wetting fluids on the behaviors of lime-treated soil. Four different wetting fluids that are commonly found to be used in the laboratory [30-33], and that exhibit divergent nature from each other were selected. They were an organic solvent: methyl methacrylate (MMA); a low and a high salt concentration solution: 0.10 M NaCl and 0.60 M NaCl solutions; and demineralized water (DW). DW was used as a reference fluid since it is commonly employed in almost all kinds of studies associated with lime-treated soil [7,10,14,15,34].

It is worth noting that the selected fluids do not directly represent the in-situ fluids. However, they fulfill the necessity of investigating the effect of the different chemical natured fluids on the behaviors of lime-treated soil. The first part of the study demonstrates the influence of different wetting fluids on the UCS evolution of lime-treated silty soil at the end of 5 wetting and drying cycles. Later the influence of wetting fluids on the evolution of physicochemical properties and microstructural modifications were presented.

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#### **2. Materials and Methodologies**

#### 93 2.1 Soil, Lime, and fluid properties

The soil used was silty soil that has been imported from Marche-Les-Dames (Belgium). The soil consists of 12% clay and 82% silt fraction. The liquid limit is 31%, and the plasticity index of the soil ranges between 8 to 12. The Methylene blue value is 2.5 g/100 g, which was determined as per the standard ASTM C1777-20 [35]. The mineralogy of the soil, determined by X-ray diffraction analysis, consists of Illite, Kaolinite, and Chlorite as clay minerals along with Quartz and Feldspars [33].

Quicklime (CaO) was used for soil treatment. It consists of 90.9% of available CaO and a reactivity
(*t*<sub>60</sub>) of 3.3 min, which complies with the specifications of the requirements of standard NF EN 459-1. The

Lime Modification Optimum (LMO) of the silt, which defines the minimum lime content required to initiate the pozzolanic reactions [37], was determined by Eades and Grim test as per ASTM D 6276-99a [38]. The LMO was found to be 1% by weight of lime. Lime content, slightly higher than LMO, *i.e.*, 2.5% lime was used for soil preparation since soil prepared at LMO was shown to have minimum contribution towards long term improvement of lime-treated soil [34]. The pH of the chosen wetting fluids is presented in Table 1.

Table 1

The pH of the fluids

Fluids ID	pН
MMA	5.80
0.10 M NaCl	8.41
0.60 M NaCl	8.74
DW	7.40

#### 2.2 Sample preparations

The maximum dry density and Optimum Moisture Content (OMC) of the 2.5% lime-treated silt obtained as per ASTM D698-91 [39] were 17.1 kN/m<sup>3</sup> and 18.5%, respectively. 

Air-dried silt was sieved using 5 mm-sieve and was then mixed with distilled water at a water content slightly higher than the OMC, *i.e.*, at the wet moisture content (WMC) (=1.1\*OMC). Compacting the soil at WMC allows maintaining a compaction moisture content similar to the study reported by Das et 52 117 al. [5,8], which involves performances of an in-situ embankment built with the present soil configuration. The obtained soil paste was placed in sealed plastic bags for about 24 hours to allow moisture content homogenization. The wet soil was then mixed with 2.5% CaO and was placed aside for 1 hour before

compaction. This procedure of soil preparation was as per GTS Technical Guide for soil treatment [40], which is also used for in-situ construction of lime-treated structures in France.

Cylindrical lime-treated specimens of dimensions 0.10 m height and 0.05 m diameter were prepared by Standard static compaction at WMC. The static compaction was performed by compressing the specimens placed inside the compaction mold from top and bottom, as demonstrated by Holtz et al. [41].

A total of 10 specimens was prepared, which includes two duplicates for each soil configuration. After compaction, specimens were wrapped in plastic film and cured for 28-days at a laboratory temperature of  $20 \pm 1$  °C.

#### 2.3 Laboratory tests

On completion of the curing period, specimens were subjected to 5 cycles of wetting and drying as per the procedure mentioned in ASTM D559 using the four different fluids. According to ASTM D559 process, specimens are required to be alternately wetted for 5 hours at room temperature and then be dried for 43 hours in the oven at 71°C. Fig. 1 presents a picture showing the placement of the specimens in four different fluids for 1<sup>st</sup> wetting. In Fig.1, 35 g NaCl corresponds to 0.60 M NaCl, referred herein. 



Fig. 1 Lime-treated specimens placed in four different fluids for 1<sup>st</sup> wetting during the W-D cycles

The mass and volume of each specimen after each cycle were recorded using a weighing machine and Vernier caliper, respectively. Specimens were periodically turned during the wetting and drying cycles to ensure homogeneity.

After completion of the 5<sup>th</sup> wetting and drying cycle, each specimen was subjected to UCS test, which involves using of a mechanical press with a load sensor of 10 KN. The application of the load to the specimens was made at a constant axial displacement rate of 1 mm/min.

After UCS, the measurement of the water content of each UCS-subjected specimen was conducted by oven drying at 105° [42]. Specimens were collected after the UCS test and were crushed to measure the suction and pH of the soil using the WP4C Dewpoint Potentiometer and as per the procedure mention in ASTM D4972-19 [43], respectively. The final pH of the fluids that remained in contact with the specimens up to the 5<sup>th</sup> wetting phase of the wetting and drying cycle, *i.e.*, the effluent, was determined by a Water Quality Meter.

About 50 ml of the collected effluent at the end of 5<sup>th</sup> wetting cycle was filtered using 0.45  $\mu$ m syringe and then were subjected to Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) analysis. ICP-OES was done for determining the elementary concentrations of Calcium (*Ca*) in order to investigate the leaching of lime under the influence of W-D cycles and the wetting fluids.

Pore Size Distribution (PSD) was analyzed by Mercury Intrusion Porosimetry (MIP) test and Barrett-Joiner-Halenda (BJH) method [44]. MIP and BJH were shown to provide elaborative evolution of macropores and mesopores, respectively, in lime-treated soil as performed by Das et al. [5,8]; hence, both the methods are used herein to investigate the pore modification extensively under the impact of wetting and drying cycles using different fluids. The procedure of the MIP test and BJH method can be referred to in Romero and Simms [45] and Westermarck [46], respectively. The analysis was made on the freeze-dried samples gathered from the 28-days cured specimen and the specimens obtained at the end of 5<sup>th</sup> wetting and drying cycle.

Discussions regarding the classification of pores in this study were provided as per the classifications given by the International Union of Pure and Applied Chemistry (IUPAC) [47]. IUPAC classifies pores based on their pore-width as macropores (> 500 Å), mesopores (20-500 Å), and micropores (< 20 Å).

#### **3. Results**

This section presents the UCS, physicochemical and microstructural evolution in the lime-treated specimens after being subjected to 5 wetting and drying cycles using different wetting fluids. All the evolutions are presented with the respective results obtained with the 28 days cured soil, which was considered as the reference specimen.

#### 176 3.1 UCS evolution at the end of wetting and drying cycle

177 Fig. 2 presents the evolution of UCS in the 5 wetting and drying cycles subjected lime-treated soils.

178 The UCS of the 28-days cured reference specimen was 0.80 MPa. After 5 successive cycles of

179 wetting and drying, the UCS increased by about 3 times in the specimens subjected to NaCl solutions and

180 MMA solvent. At the same time, the increase in UCS was about 2 times for the DW-subjected specimen.





Fig. 3 Soil suction and soil pH measured at the end of 5 W-D cycles along with the reference 28 days cured soil.

The soil suction obtained in the reference 28 days cured soil was 0.49 MPa (Fig. 3a). After 5 wetting and drying cycles, the soil suction measured was in the range of 290 to 320 MPa for all the specimens subjected to different wetting fluids. The corresponding water content of these specimens is presented in Table 2.

Table 2. Water content measured during suction measurement.

Specimens	Water content during suction		
specificits	measurement (%)		
0.10 M NaCl	0.46		
MMA	0.53		
0.60 M NaCl	0.55		
DW	0.46		
28-days cured	20.1		

The pH of the reference specimen was 11.86 (Fig. 3b). After 5 wetting and drying cycles, the minimum decrease in soil pH with respect to the initial soil pH was obtained for 0.60 M NaCl subjected soil, which was 11.2. The soil pH obtained for 0.10 M NaCl, and MMA subjected soil was 10.39 and 10.71, respectively. The maximum decrease in soil pH was observed for the DW-subjected soil, which was 9.77.

#### 208 3.3 pH and Ca concentration evolution in the effluent collected at the end of wetting and drying cycle

The final pH of the effluent, measured at the end of the 5<sup>th</sup> cycle is presented in Fig. 4 by comparing the same with the initial pH of the respective fluids.



Fig. 4 Final pH measured in the effluents and compared with the initial pH of the respective fluids at the end of 5 W-D cycles.

The final pH obtained from the effluent gathered from the MMA solvent that was in contact with the specimens till the end of 5 wetting and drying cycles remained unchanged compared to the initial pH of the MMA solvent. This pH increased by about 10% in the 0.10 M NaCl and 0.60 M NaCl solutions and

increased by about 20% in DW after being in contact with the lime-treated soil compared to the initial pH of the respective solutions.

Table 3 presents the cumulative concentration of *Ca* analyzed in the effluent collected at the end of 5 wetting and drying cycles. In the effluent obtained from the specimen subjected to MMA solvent, the cumulative Ca concentration was below the limit that can be detected during the ICP-OES test, *i.e.*, < 0.2mg/l; hence no Ca concentration was found. The maximum cumulative Ca concentration was measured in the effluent gathered from the soil submitted to 0.60 M NaCl, followed by the one submitted to 0.10 M NaCl solution. The effluent collected from the DW subjected lime-treated soil gave the minimum value of cumulative Ca concentration, which was 49.97 mg/l. 

Table 3. Cumulative (Cum.) concentration of Ca analyzed in the effluents at the end of 5 wetting and drying cycles.

	Soil ID	Cum. Concentration of Ca (mg/l)	-
	MMA	< 0.20	-
	0.10 M NaCl	142.90	
	0.60 M NaCl	232.48	
	DW	49.97	
			-
3.4 Pore structure evolu	ution at the end o	f wetting and drying cycle	
3.4.1 Pore size determin	nation by MIP		
The evolution of pores is	n all the specimen	s at the end of 5 wetting and drying cyc	le was determined by MIP
and was then compared	with the untreated	d and the reference specimens in Fig. 5.	
		12	



**Fig. 5** Comparative evaluation of PSD in lime-treated specimens subjected to 0.10 M NaCl (a), MMA (b), 0.60 M NaCl (c), and DW (d) at the end of 5 W-D cycles with the untreated and the reference specimens by MIP.

According to Fig. 5, untreated compacted specimens showed bi-modal PSD with a peak at macropores diameter  $10^4$  and  $10^5$  Å. After 2.5% quicklime treatment and 28-days of curing, no significant presence of macropores greater than  $3 \times 10^4$  Å was observed, and significant evolution of pores smaller than 3000 Å was observed due to the formation of cementitious bonding. Such an observation was in accordance with the studies reported by Das et al. [5-10].

Specimens subjected to 0.10 M NaCl and 0.60 M NaCl solutions showed similar evolution of pore structure at the end of 5 wetting and drying cycles compared to the pore structure of the reference specimen (Fig. 5a & c). The macropore peak at  $10^3$  Å present initially in the reference specimen disappear, and a new mesopore peak at about  $10^2$  Å was observed in both the NaCl subjected soils. Similar to the NaCl subjected soils, MMA subjected soil showed the reduction of macropore peak presence at  $10^3$  Å and generation of mesopore peak at about  $5 \times 10^2$  Å compared to the reference specimen (Fig. 5b).

At the same time, DW-subjected specimen showed a decrease in pores smaller than 3000 Å, which was formed due to lime treatment in the reference specimen (Fig. 5d).

#### 3.4.2 Mesopore volume determination by BJH

The evolution of cumulative pore volume in the mesopore range 24-250 Å was analyzed by BJH at the end of 5 wetting and drying cycles and is presented in Fig. 6 by comparing with the reference and untreated soil.



Fig. 6 Comparative evaluation of Cumulative (Cum.) pore volume evolution in the mesopore range 24-250 Å in specimens subjected to different fluids at the end of 5 W-D cycles with the reference and untreated soil by BJH.

Fig. 6 shows that compared to the reference soil, a significant cumulative pore volume in the mesopore range 24-250Å was present in the MMA subjected soil, followed by the specimens subjected to NaCl solutions. On the contrary, the cumulative pore volume in the mesopore range 24-250Å decreased in
the DW subjected soil compared to the reference soil.

#### 269 4. Discussions

From the results, it is evident that the unconfined compressive strength, chemical, and microstructural evolution of a similar configured lime-treated soil varies based on the types of wetting fluids the specimens were exposed to.

Although the specimens underwent five successive W-D cycles, the UCS evolved positively in all the lime-treated specimens compared to the reference 28 days cured soil (Fig. 2). This extent of unconfined compressive strength evolution in the specimens varied based on the wetting fluids they were subjected to; the maximum being obtained with the NaCls- and MMA-subjected soils, with a less significant difference of 0.10 to 0.50 MPa UCS values between respective soils. The evolution of unconfined compressive strength was comparatively lower in the DW-subjected specimen.

Similar to the UCS evolution, the extent of the average soil pH evolution at the end of 5 wetting and drying cycles also varied with the type of wetting fluids they were subjected to (Fig. 3b). The average soil pH recorded with all the 5 wetting and drying cycles subjected soils was comparatively lower than the soil pH recorded with the reference 28 days cured soil. This decrease in the average soil pH can be linked with the loss of OH<sup>-</sup> ions from the soil during the wetting-drying cycles. Such a phenomenon was confirmed by the 10% and 20% rise in final pH in the NaCl solutions and the DW, respectively (Fig. 4). However, the final pH of the effluent obtained from MMA-subjected soil remained constant (Fig. 4).

The difference in average soil pH was minimum between the 0.10 M NaCl, MMA, and 0.60 M NaCl subjected soils and the reference specimen and remained between 10.39 to 11.2 (Fig. 3b). However, the maximum decrease was observed in the DW subjected soil, where the pH recorded was about 9.77. This decrease explains the release of more OH<sup>-</sup> ions from the DW-subjected soil, thus increasing the final pH of the effluent by 20%, as seen in Fig. 4.

On observing Table 3, the differences observed in the measured cumulative calcium concentrations between the effluents gathered from the 0.10 M NaCl-, 0.60 M NaCl- and DW-subjected specimens again emphasized the influence of the nature of the surrounding medium. The maximum cumulative calcium concentration occurred to be in the effluent obtained from the 0.60 M NaCl subjected soil, followed by 0.10 M NaCl-subjected soil and then DW subjected soil (Table 3). However, similar to the constant final pH attained in the effluent obtained from the MMA-subjected soil (Fig. 3b), no significant cumulative calcium concentration was recorded (Table 3).

In addition to the changes brought in the strength and chemical properties of the lime-treated soil owing to subjection to different wetting fluids, modifications in the soil pore structure also occurred. The MIP analysis presented in Fig. 5 evidenced the decrease in macropore at peak 10<sup>3</sup> Å and initiation of mesopore in the NaCl-and MMA-subjected soil compared to the reference soil. The significant initiation of mesopore evolution was confirmed in the BJH analysis, which provides a cumulative pore volume evolution in the mesopore range 24-250 Å in Fig. 6. The maximum evolution of cumulative pore volume in the mesopore range 24-250 Å in the MMA subjected soil indicates the certain probability of MMA interacting with the lime-treated soil components. However, such a feature was missing in the DW subjected specimen (Fig. 5 & Fig. 6). Such a difference indicates that subjecting lime-treated soil to NaCls solutions and MMA solvent favored the mesopore evolution compared to DW. The evolution of mesopores in lime-treated soil was shown to be a benefit towards the long-term performances of lime-treated soil in the studies reported by Das et al. [5-10].

Except for the strength, chemical, and microstructural modification, it's the average soil suction that remained the same regardless of the wetting fluids the specimens were subjected to (Fig. 3a). Such an evolution indicates that the evolution of high soil suction in the range of 290 to 320 MPa for all the W-D cycles subjected specimens was triggered mainly by oven-drying at 71°C, which reduced the water content in the soil almost to zero (Table 2).

#### 316 Conclusions

The influence of different wetting fluids on the unconfined compressive strength, physicochemical, and microstructural evolution of 2.5% quicklime treated silty soil during five successive W-D cycles were evaluated. Based on the investigation, the following conclusions are derived:

1) Regardless of the types of wetting fluids lime-treated soil was subjected to, the unconfined compressive strength evolved at the end of 5 wetting and drying cycles compared to the reference 28 days cured soil. The extent of unconfined compressive strength evolution varied with the types of wetting fluids the specimens were exposed to. About 3 times higher unconfined compressive strength was obtained with limetreated specimen subjected to sodium chloride solutions and methyl methacrylate solvent with an insignificant difference of 0.10 to 0.50 MPa UCS values between respective soils. The evolution of unconfined compressive strength was comparatively lower in the DW-subjected specimen.

2) The average pH of the lime-treated soils decreased during the wetting and drying cycles compared to the reference soil pH. This decrease in average soil pH was from 11.86 to 11.2 and 10.39 for the 0.60 M-and 0.10 M-NaCl-subjected soils, respectively. For MMA-subjected soil, the average soil pH decreased to 10.71. However, a comparatively greater decrease in average soil pH up to 9.77 occurred in the DW-subjected soil, thus, leading to a comparatively higher increase in the final pH of the effluent.

3) The release of calcium from the lime-treated soil occurs to be governed by the nature of wetting fluids available in the surrounding medium. Maximum calcium was released from the 0.60 M NaCl subjected specimen followed by the 0.10 M NaCl subjected specimen, and then DW subjected soil. No calcium was released in the effluent of the MMA-subjected soil during the wetting and drying cycles.

4) Mesopore evolution in wetting and drying cycles subjected lime-treated soil was dependent on the type
of fluids the specimens were exposed to. NaCl solutions and MMA solvent subjected specimens, showed
additional development of mesopores compared to the 28 days cured reference specimens. However, DW

subjected specimens showed a reduction in the pores smaller than 3000 Å formed in the reference 28 days cured soil due to lime treatment.

5) The type of wetting fluids has a less significant effect on the suction evolution of the wetting and drying cycles subjected lime-treated soils. The evolution of soil suction observed at the end of 5 wetting and drying cycles in all specimens was high and equivalent, which is attributed to the increased soil grain-to-grain contact owing to a total loss in water content during oven-drying at 71°C.

The study highlights that the type of wetting fluids to which a lime-treated soil was subjected brings significant modifications in the UCS, physicochemical, and microstructural evolution during the wetting and drying cycles. The extent of this modification was unique for each type of wetting fluid. Thus, this study shows the necessity of the reproduction of laboratory fluids that are a close representation of the real fluids available in nature while evaluating the performances of a lime-treated structure. However, deep investigations regarding the chemical interactions of the wetting fluids and subjected specimens are needed, which is the future scope of this study.

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1	Influence of wetting fluids on the compressive strength, physicochemical, and pore-				
2	structure evolution in lime-treated silty soil subjected to wetting and drying cycles.				
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7					
8	Highlights				
9	• Effect of the nature of wetting fluids on compacted lime-treated soil is investigated.				
10	• Lime-treated soil is submitted to wetting-drying cycles using different liquids.				
11	• Unconfined compressive strength increased irrespective of the nature of wetting fluids.				
12	• Extent of compressive strength increased varies by the nature of subjected wetting fluids.				

• Extent of chemical and pore-structure evolution varies by the nature of subjected wetting fluids

14

#### 15 Abstract

16 The long-term benefit brought in lime-treated soil can be altered by the nature of the surrounding wetting 17 fluid, thus, affecting its durability. However, studies regarding such impact are limited. In this study, the influence of different wetting fluids on the microstructural, physicochemical modifications, and 18 19 Unconfined Compressive Strength (UCS) evolution of lime-treated soil is investigated. Lime-treated soils 20 are subjected to wetting and drying cycles using 0.10 M and 0.60 M NaCl solutions, demineralized water, 21 and Methyl Methacrylate. The results show that despite the soil being exposed to several wetting and drying 22 cycles, the UCS evolved positively compared to the reference specimen. However, this evolution varies 23 with the types of wetting fluids the specimens are exposed to. The NaCls-and Methyl Methacrylate-24 subjected soils showed an increase in UCS up to about 3 times, while the UCS increase is about 2 times in the corresponding demineralized water subjected soil. Similarly, the extent of the variation of the final pH and the cumulative calcium concentrations measured in the effluent is marked by the wetting fluids available in the surrounding medium. At the pore structure level, the presence of NaCl solutions and Methyl Methacrylate solvent as a wetting fluid was observed to enhance the evolution of pores smaller than 3000 Å. However, such a phenomenon is less significant in the demineralized water-subjected soil. Thus, the study confirms that the type of wetting fluid plays an important role in the evolution of lime-treated soil during the wetting and drying cycles.

32 **Keywords:** Wetting and drying cycles; physicochemical; microstructure; Lime-treated soil; wetting fluids.

33

#### 34 **1. Introduction**

Improvement of soil characteristics such as strength, bearing capacity, elastic modulus, etc., through lime 35 36 treatment is a current practice in geotechnical engineering. Such an improvement is a consequence of 37 combined modifications brought through chemical and mechanical stabilizations. Two basic chemical 38 reactions called short-term and long-term pozzolanic reactions occur during chemical stabilization. The 39 short-term reactions bring physical improvement in the treated soil by reducing the plasticity and increasing 40 the workability of the soil [1-3]. The long-term pozzolanic reactions create cementitious compounds such 41 as Calcium Silicate Hydrates (C-S-H), Calcium Aluminate Hydrates (C-A-H) and modify the soil fabrics 42 [4-13]. The mechanical stabilization involves the execution process, such as the soil-binder mixing process, 43 using an appropriate binder and water contents during compaction and mixing, implementing the accurate 44 compaction conditions [6, 14-15].

So far, few field investigations have reported the sustainability of earth structures attained through such improvements [5, 8, 16-19]. Akula et al. [16] studied the performance of the Friant-Kern Canal in California, the United States, which was built with 4% quicklime treated plastic soil after 40 years from treatment. Based on the investigations, they have reported increased long-term strength, reduction in swelling potential, erosion resistance, thus indicating a desired geo-mechanical stability of the lime-treated 50 structure. Dawson and McDowell [17] monitored the Unconfined Compressive Strength (UCS) of a pavement built with 3% lime-treated clayey gravel. After 14 years from construction, a UCS value ranging 51 52 from 2.07 MPa to 4.14 MPa was obtained, which was observed to be 6 to 12 times higher than the one 53 obtained from the untreated soil. Jung et al. [18] observed an increase in resilient modulus from 96.5 MPa 54 to 703.2 MPa in a 4% lime-treated expansive soil pavement after 11 years from construction. Recently, Das 55 et al. [5,8] demonstrated the long-term impact of 2.5% quicklime lime treatment on an experimental 56 embankment exposed to a damp climate for 7 years. UCS evaluation of core-sampled specimens was made, 57 which showed an average value of  $3.29 \pm (0.45)$  MPa despite being the exposure to such a severe climate. 58 Such significant evolution in strength was explained in detail through physicochemical and microstructural 59 investigations.

60 Thus, the above in-situ studies have evidenced the successive improvement brought by lime 61 treatment. However, an exposed lime-treated hydraulic earth structures, particularly the surface cover is 62 often prone to several drastic environmental situations. For instance, when hydraulic earth structure remains 63 permanently in contact with water, it is subjected to several fluctuations in water level [20-23], which 64 induces ingression or egression of water in the structure. Such impact on the lime-treated structure was 65 evaluated at a laboratory scale by subjecting the treated soil to successive wetting and drying cycles as per the procedure mentioned in ASTM D 559 [24]. Subjection of lime-treated soil to severe wetting and drying 66 67 cycles was shown to bring deterioration in the improved pore-structure of the soil attained from lime 68 treatment [25]. Such a deterioration was reported to bring total loss in improved hydromechanical 69 performances of the lime-treated soil [26-27].

However, during the wetting phase of the wetting and drying cycles, infiltration of water occurs into the subjected soil, which interacts with the treated soil components. Since natural water can be constituted of several organic compounds or inorganics compounds or a mixture of both, which control the pH level of the water [28-29], thus, the chemical nature of the infiltered water can modify the improvement of the lime-treated soil. Soil pH in a lime-treated structure contributes greatly towards the maintenance of the long-term performance of the concerned structure, as shown by Das et al. [5,8]. Hence, subjecting the lime-treated soil to extreme wetting and drying cycles accompanied by a wetting fluid of different chemical
nature might impact the soil pH and can modify the long-term performance. Such a possibility remained
less investigated.

In this context, the present study is focused on investigating the influence of different wetting fluids on the behaviors of lime-treated soil. Four different wetting fluids that are commonly found to be used in the laboratory [30-33], and that exhibit divergent nature from each other were selected. They were an organic solvent: methyl methacrylate (MMA); a low and a high salt concentration solution: 0.10 M NaCl and 0.60 M NaCl solutions; and demineralized water (DW). DW was used as a reference fluid since it is commonly employed in almost all kinds of studies associated with lime-treated soil [7,10,14,15,34].

It is worth noting that the selected fluids do not directly represent the in-situ fluids. However, they fulfill the necessity of investigating the effect of the different chemical natured fluids on the behaviors of lime-treated soil. The first part of the study demonstrates the influence of different wetting fluids on the UCS evolution of lime-treated silty soil at the end of 5 wetting and drying cycles. Later the influence of wetting fluids on the evolution of physicochemical properties and microstructural modifications were presented.

91

#### 92 **2. Materials and Methodologies**

#### 93 2.1 Soil, Lime, and fluid properties

The soil used was silty soil that has been imported from Marche-Les-Dames (Belgium). The soil consists of 12% clay and 82% silt fraction. The liquid limit is 31%, and the plasticity index of the soil ranges between 8 to 12. The Methylene blue value is 2.5 g/100 g, which was determined as per the standard ASTM C1777-20 [35]. The mineralogy of the soil, determined by X-ray diffraction analysis, consists of Illite, Kaolinite, and Chlorite as clay minerals along with Quartz and Feldspars [33].

Quicklime (CaO) was used for soil treatment. It consists of 90.9% of available CaO and a reactivity
(*t*<sub>60</sub>) of 3.3 min, which complies with the specifications of the requirements of standard NF EN 459-1. The

Lime Modification Optimum (LMO) of the silt, which defines the minimum lime content required to initiate the pozzolanic reactions [37], was determined by Eades and Grim test as per ASTM D 6276-99a [38]. The LMO was found to be 1% by weight of lime. Lime content, slightly higher than LMO, *i.e.*, 2.5% lime was used for soil preparation since soil prepared at LMO was shown to have minimum contribution towards long term improvement of lime-treated soil [34]. The pH of the chosen wetting fluids is presented in Table 1.

- 107 Table 1
- 108 The pH of the fluids

Fluids ID	рН	
MMA	5.80	
0.10 M NaCl	8.41	
0.60 M NaCl	8.74	
DW	7.40	

109

110

#### 111 2.2 Sample preparations

The maximum dry density and Optimum Moisture Content (OMC) of the 2.5% lime-treated silt obtained
as per ASTM D698-91 [39] were 17.1 kN/m<sup>3</sup> and 18.5%, respectively.

Air-dried silt was sieved using 5 mm-sieve and was then mixed with distilled water at a water content slightly higher than the OMC, *i.e.*, at the wet moisture content (WMC) (=1.1\*OMC). Compacting the soil at WMC allows maintaining a compaction moisture content similar to the study reported by Das et al. [5,8], which involves performances of an in-situ embankment built with the present soil configuration. The obtained soil paste was placed in sealed plastic bags for about 24 hours to allow moisture content homogenization. The wet soil was then mixed with 2.5% CaO and was placed aside for 1 hour before compaction. This procedure of soil preparation was as per GTS Technical Guide for soil treatment [40],
which is also used for in-situ construction of lime-treated structures in France.

122 Cylindrical lime-treated specimens of dimensions 0.10 m height and 0.05 m diameter were prepared 123 by Standard static compaction at WMC. The static compaction was performed by compressing the 124 specimens placed inside the compaction mold from top and bottom, as demonstrated by Holtz et al. [41].

125 A total of 10 specimens was prepared, which includes two duplicates for each soil configuration. 126 After compaction, specimens were wrapped in plastic film and cured for 28-days at a laboratory temperature 127 of  $20 \pm 1$  °C.

128

129

#### 130 2.3 Laboratory tests

On completion of the curing period, specimens were subjected to 5 cycles of wetting and drying as per the procedure mentioned in ASTM D559 using the four different fluids. According to ASTM D559 process, specimens are required to be alternately wetted for 5 hours at room temperature and then be dried for 43 hours in the oven at 71°C. Fig. 1 presents a picture showing the placement of the specimens in four different fluids for 1<sup>st</sup> wetting. In Fig.1, 35 g NaCl corresponds to 0.60 M NaCl, referred herein.

136





Fig. 1 Lime-treated specimens placed in four different fluids for 1<sup>st</sup> wetting during the W-D cycles

The mass and volume of each specimen after each cycle were recorded using a weighing machine
and Vernier caliper, respectively. Specimens were periodically turned during the wetting and drying cycles
to ensure homogeneity.

After completion of the 5<sup>th</sup> wetting and drying cycle, each specimen was subjected to UCS test, which involves using of a mechanical press with a load sensor of 10 KN. The application of the load to the specimens was made at a constant axial displacement rate of 1 mm/min.

After UCS, the measurement of the water content of each UCS-subjected specimen was conducted by oven drying at 105° [42]. Specimens were collected after the UCS test and were crushed to measure the suction and pH of the soil using the WP4C Dewpoint Potentiometer and as per the procedure mention in ASTM D4972-19 [43], respectively. The final pH of the fluids that remained in contact with the specimens up to the 5<sup>th</sup> wetting phase of the wetting and drying cycle, *i.e.*, the effluent, was determined by a Water Quality Meter.

About 50 ml of the collected effluent at the end of 5<sup>th</sup> wetting cycle was filtered using 0.45  $\mu$ m syringe and then were subjected to Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) analysis. ICP-OES was done for determining the elementary concentrations of Calcium (*Ca*) in order to investigate the leaching of lime under the influence of W-D cycles and the wetting fluids.

156 Pore Size Distribution (PSD) was analyzed by Mercury Intrusion Porosimetry (MIP) test and 157 Barrett-Joiner-Halenda (BJH) method [44]. MIP and BJH were shown to provide elaborative evolution of 158 macropores and mesopores, respectively, in lime-treated soil as performed by Das et al. [5,8]; hence, both 159 the methods are used herein to investigate the pore modification extensively under the impact of wetting 160 and drying cycles using different fluids. The procedure of the MIP test and BJH method can be referred to 161 in Romero and Simms [45] and Westermarck [46], respectively. The analysis was made on the freeze-dried samples gathered from the 28-days cured specimen and the specimens obtained at the end of 5<sup>th</sup> wetting 162 163 and drying cycle.

Discussions regarding the classification of pores in this study were provided as per the classifications given by the International Union of Pure and Applied Chemistry (IUPAC) [47]. IUPAC classifies pores based on their pore-width as macropores (> 500 Å), mesopores (20-500 Å), and micropores (< 20 Å).

168

#### 169 **3. Results**

170

This section presents the UCS, physicochemical and microstructural evolution in the lime-treated specimens after being subjected to 5 wetting and drying cycles using different wetting fluids. All the evolutions are presented with the respective results obtained with the 28 days cured soil, which was considered as the reference specimen.

175

#### 176 3.1 UCS evolution at the end of wetting and drying cycle

177 Fig. 2 presents the evolution of UCS in the 5 wetting and drying cycles subjected lime-treated soils.

178 The UCS of the 28-days cured reference specimen was 0.80 MPa. After 5 successive cycles of

179 wetting and drying, the UCS increased by about 3 times in the specimens subjected to NaCl solutions and

180 MMA solvent. At the same time, the increase in UCS was about 2 times for the DW-subjected specimen.





182

Fig. 2 Unconfined Compressive Strength obtained after 5 W-D cycles.

184 Thus, the maximum UCS was obtained with the NaCls- and MMA-subjected soils. The evolution of 185 unconfined compressive strength was comparatively lower in the DW-subjected specimen.

186

#### 187 3.2 Soil suction and soil pH evolution at the end of W-D cycle

The soil suction and soil pH measured at the end of 5 wetting and drying cycles along with the reference 28 days cured soil is presented in Fig. 3. Thyagaraj and Salini [48] reported that the type of pore fluid in a given compacted soil can induce change in soil's pore-structure which modifies the soil suction. Hence, along with soil pH, soil suction is also reported herein.





Fig. 3 Soil suction and soil pH measured at the end of 5 W-D cycles along with the reference 28 days cured soil.

The soil suction obtained in the reference 28 days cured soil was 0.49 MPa (Fig. 3a). After 5 wetting
and drying cycles, the soil suction measured was in the range of 290 to 320 MPa for all the specimens
subjected to different wetting fluids. The corresponding water content of these specimens is presented in
Table 2.

201	Table 2.	Water conte	ent measured	l during	suction	measurement.

Specimens	Water content during suction		
	measurement (%)		
0.10 M NaCl	0.46		
MMA	0.53		
0.60 M NaCl	0.55		
DW	0.46		
28-days cured	20.1		

The pH of the reference specimen was 11.86 (Fig. 3b). After 5 wetting and drying cycles, the minimum decrease in soil pH with respect to the initial soil pH was obtained for 0.60 M NaCl subjected soil, which was 11.2. The soil pH obtained for 0.10 M NaCl, and MMA subjected soil was 10.39 and 10.71, respectively. The maximum decrease in soil pH was observed for the DW-subjected soil, which was 9.77.

#### 208 3.3 pH and Ca concentration evolution in the effluent collected at the end of wetting and drying cycle

The final pH of the effluent, measured at the end of the 5<sup>th</sup> cycle is presented in Fig. 4 by comparing the same with the initial pH of the respective fluids.

211



212

Fig. 4 Final pH measured in the effluents and compared with the initial pH of the respective fluids at the end of 5 W-D cycles.

The final pH obtained from the effluent gathered from the MMA solvent that was in contact with the specimens till the end of 5 wetting and drying cycles remained unchanged compared to the initial pH of the MMA solvent. This pH increased by about 10% in the 0.10 M NaCl and 0.60 M NaCl solutions and increased by about 20% in DW after being in contact with the lime-treated soil compared to the initial pHof the respective solutions.

Table 3 presents the cumulative concentration of *Ca* analyzed in the effluent collected at the end of 5 wetting and drying cycles. In the effluent obtained from the specimen subjected to MMA solvent, the cumulative *Ca* concentration was below the limit that can be detected during the ICP-OES test, *i.e.*, < 0.2mg/l; hence no *Ca* concentration was found. The maximum cumulative *Ca* concentration was measured in the effluent gathered from the soil submitted to 0.60 M NaCl, followed by the one submitted to 0.10 M NaCl solution. The effluent collected from the DW subjected lime-treated soil gave the minimum value of cumulative *Ca* concentration, which was 49.97 mg/l.

227

Table 3. Cumulative (Cum.) concentration of Ca analyzed in the effluents at the end of 5 wetting and drying cycles.

Cum. Concentration of Ca (mg/l)
< 0.20
142.90
232.48
49.97

229

230

#### 231 3.4 Pore structure evolution at the end of wetting and drying cycle

#### 232 **3.4.1** *Pore size determination by MIP*

233 The evolution of pores in all the specimens at the end of 5 wetting and drying cycle was determined by MIP

and was then compared with the untreated and the reference specimens in Fig. 5.





Fig. 5 Comparative evaluation of PSD in lime-treated specimens subjected to 0.10 M NaCl (a), MMA (b), 0.60 M NaCl (c), and
 DW (d) at the end of 5 W-D cycles with the untreated and the reference specimens by MIP.

According to Fig. 5, untreated compacted specimens showed bi-modal PSD with a peak at macropores diameter  $10^4$  and  $10^5$  Å. After 2.5% quicklime treatment and 28-days of curing, no significant presence of macropores greater than  $3 \times 10^4$  Å was observed, and significant evolution of pores smaller than 3000 Å was observed due to the formation of cementitious bonding. Such an observation was in accordance with the studies reported by Das et al. [5-10].

Specimens subjected to 0.10 M NaCl and 0.60 M NaCl solutions showed similar evolution of pore structure at the end of 5 wetting and drying cycles compared to the pore structure of the reference specimen (Fig. 5a & c). The macropore peak at  $10^3$  Å present initially in the reference specimen disappear, and a new mesopore peak at about  $10^2$  Å was observed in both the NaCl subjected soils. Similar to the NaCl subjected soils, MMA subjected soil showed the reduction of macropore peak presence at  $10^3$  Å and generation of mesopore peak at about  $5 \times 10^2$  Å compared to the reference specimen (Fig. 5b).

- At the same time, DW-subjected specimen showed a decrease in pores smaller than 3000 Å, which was formed due to lime treatment in the reference specimen (Fig. 5d).
- 253

#### 254 3.4.2 Mesopore volume determination by BJH

The evolution of cumulative pore volume in the mesopore range 24-250 Å was analyzed by BJH at the end of 5 wetting and drying cycles and is presented in Fig. 6 by comparing with the reference and untreated soil.

258



Fig. 6 Comparative evaluation of Cumulative (Cum.) pore volume evolution in the mesopore range 24-250 Å in specimens

subjected to different fluids at the end of 5 W-D cycles with the reference and untreated soil by BJH.

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Fig. 6 shows that compared to the reference soil, a significant cumulative pore volume in the mesopore range 24-250Å was present in the MMA subjected soil, followed by the specimens subjected to NaCl solutions. On the contrary, the cumulative pore volume in the mesopore range 24-250Å decreased in
the DW subjected soil compared to the reference soil.

268

#### 269 4. Discussions

From the results, it is evident that the unconfined compressive strength, chemical, and microstructural evolution of a similar configured lime-treated soil varies based on the types of wetting fluids the specimens were exposed to.

Although the specimens underwent five successive W-D cycles, the UCS evolved positively in all the lime-treated specimens compared to the reference 28 days cured soil (Fig. 2). This extent of unconfined compressive strength evolution in the specimens varied based on the wetting fluids they were subjected to; the maximum being obtained with the NaCls- and MMA-subjected soils, with a less significant difference of 0.10 to 0.50 MPa UCS values between respective soils. The evolution of unconfined compressive strength was comparatively lower in the DW-subjected specimen.

Similar to the UCS evolution, the extent of the average soil pH evolution at the end of 5 wetting and drying cycles also varied with the type of wetting fluids they were subjected to (Fig. 3b). The average soil pH recorded with all the 5 wetting and drying cycles subjected soils was comparatively lower than the soil pH recorded with the reference 28 days cured soil. This decrease in the average soil pH can be linked with the loss of OH<sup>-</sup> ions from the soil during the wetting-drying cycles. Such a phenomenon was confirmed by the 10% and 20% rise in final pH in the NaCl solutions and the DW, respectively (Fig. 4). However, the final pH of the effluent obtained from MMA-subjected soil remained constant (Fig. 4).

The difference in average soil pH was minimum between the 0.10 M NaCl, MMA, and 0.60 M NaCl subjected soils and the reference specimen and remained between 10.39 to 11.2 (Fig. 3b). However, the maximum decrease was observed in the DW subjected soil, where the pH recorded was about 9.77. This decrease explains the release of more  $OH^{-}$  ions from the DW-subjected soil, thus increasing the final pH of the effluent by 20%, as seen in Fig. 4. On observing Table 3, the differences observed in the measured cumulative calcium concentrations between the effluents gathered from the 0.10 M NaCl-, 0.60 M NaCl- and DW-subjected specimens again emphasized the influence of the nature of the surrounding medium. The maximum cumulative calcium concentration occurred to be in the effluent obtained from the 0.60 M NaCl subjected soil, followed by 0.10 M NaCl-subjected soil and then DW subjected soil (Table 3). However, similar to the constant final pH attained in the effluent obtained from the MMA-subjected soil (Fig. 3b), no significant cumulative calcium concentration was recorded (Table 3).

298 In addition to the changes brought in the strength and chemical properties of the lime-treated soil 299 owing to subjection to different wetting fluids, modifications in the soil pore structure also occurred. The MIP analysis presented in Fig. 5 evidenced the decrease in macropore at peak  $10^3$  Å and initiation of 300 301 mesopore in the NaCl-and MMA-subjected soil compared to the reference soil. The significant initiation of 302 mesopore evolution was confirmed in the BJH analysis, which provides a cumulative pore volume evolution 303 in the mesopore range 24-250 Å in Fig. 6. The maximum evolution of cumulative pore volume in the 304 mesopore range 24-250 Å in the MMA subjected soil indicates the certain probability of MMA interacting 305 with the lime-treated soil components. However, such a feature was missing in the DW subjected specimen 306 (Fig. 5 & Fig. 6). Such a difference indicates that subjecting lime-treated soil to NaCls solutions and MMA 307 solvent favored the mesopore evolution compared to DW. The evolution of mesopores in lime-treated soil 308 was shown to be a benefit towards the long-term performances of lime-treated soil in the studies reported 309 by Das et al. [5-10].

Except for the strength, chemical, and microstructural modification, it's the average soil suction that remained the same regardless of the wetting fluids the specimens were subjected to (Fig. 3a). Such an evolution indicates that the evolution of high soil suction in the range of 290 to 320 MPa for all the W-D cycles subjected specimens was triggered mainly by oven-drying at 71°C, which reduced the water content in the soil almost to zero (Table 2).

#### 316 Conclusions

The influence of different wetting fluids on the unconfined compressive strength, physicochemical, and microstructural evolution of 2.5% quicklime treated silty soil during five successive W-D cycles were evaluated. Based on the investigation, the following conclusions are derived:

1) Regardless of the types of wetting fluids lime-treated soil was subjected to, the unconfined compressive strength evolved at the end of 5 wetting and drying cycles compared to the reference 28 days cured soil. The extent of unconfined compressive strength evolution varied with the types of wetting fluids the specimens were exposed to. About 3 times higher unconfined compressive strength was obtained with limetreated specimen subjected to sodium chloride solutions and methyl methacrylate solvent with an insignificant difference of 0.10 to 0.50 MPa UCS values between respective soils. The evolution of unconfined compressive strength was comparatively lower in the DW-subjected specimen.

2) The average pH of the lime-treated soils decreased during the wetting and drying cycles compared to the reference soil pH. This decrease in average soil pH was from 11.86 to 11.2 and 10.39 for the 0.60 M-and 0.10 M-NaCl-subjected soils, respectively. For MMA-subjected soil, the average soil pH decreased to 10.71. However, a comparatively greater decrease in average soil pH up to 9.77 occurred in the DW-subjected soil, thus, leading to a comparatively higher increase in the final pH of the effluent.

3) The release of calcium from the lime-treated soil occurs to be governed by the nature of wetting fluids available in the surrounding medium. Maximum calcium was released from the 0.60 M NaCl subjected specimen followed by the 0.10 M NaCl subjected specimen, and then DW subjected soil. No calcium was released in the effluent of the MMA-subjected soil during the wetting and drying cycles.

4) Mesopore evolution in wetting and drying cycles subjected lime-treated soil was dependent on the type
of fluids the specimens were exposed to. NaCl solutions and MMA solvent subjected specimens, showed
additional development of mesopores compared to the 28 days cured reference specimens. However, DW

subjected specimens showed a reduction in the pores smaller than 3000 Å formed in the reference 28 days
cured soil due to lime treatment.

5) The type of wetting fluids has a less significant effect on the suction evolution of the wetting and drying cycles subjected lime-treated soils. The evolution of soil suction observed at the end of 5 wetting and drying cycles in all specimens was high and equivalent, which is attributed to the increased soil grain-to-grain contact owing to a total loss in water content during oven-drying at 71°C.

The study highlights that the type of wetting fluids to which a lime-treated soil was subjected brings significant modifications in the UCS, physicochemical, and microstructural evolution during the wetting and drying cycles. The extent of this modification was unique for each type of wetting fluid. Thus, this study shows the necessity of the reproduction of laboratory fluids that are a close representation of the real fluids available in nature while evaluating the performances of a lime-treated structure. However, deep investigations regarding the chemical interactions of the wetting fluids and subjected specimens are needed, which is the future scope of this study.

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