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Carbonation investigation on atmospherically exposed lime-treated silty soil.

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Abstract

The effect of carbonation on a one-year atmospherically exposed lime-treated soil structure is investigated. The investigation involves analyzing the chemical characteristics and pore-structure modifications of several specimens sampled up to 12 cm depth perpendicular to the surface. On comparing the analysis with untreated and lime-treated core-sampled specimens, carbonation is found to have occurred on specimens sampled up to 4.2 cm depth perpendicular to the surface. The decrease in soil pH to a value 9 or lower than that and the presence of carbonates confirmed the carbonation effect. At the pore-structure level, carbonation mechanism is found to increase macropores of diameter 20000 Å to 100000 Å, which is missing in the non-carbonated specimens and even greater than the one found at untreated soil. Such an observation confirmed the loss of cementitious compounds due to carbonation, thus, converting the mesopores developed due to lime treatment to macropores of larger pore diameter.

Keywords: lime-treated soil; carbonation; atmospherically exposed; macropores; soil pH; calcium carbonates.

1. Introduction.

Soil treatment by lime is currently a practice employed for improving the low engineering properties of the soil. Often a soil present in a given land chosen for infrastructure development fails to meet the desired strength and workability needed for that specific land development project. Such soil being treated with lime was shown to bring significant development in workability and strength [1-8]. There are two basic soil-lime chemical reactions that contribute towards the improvement of soil engineering properties. These reactions are categorized as short-term or modification reactions and long-term or pozzolanic reactions [8,9]. The former reaction improves the workability of the soil by undergoing hydration and dissolution reactions. While the latter produces chemical bonding within soil fabrics owing to the evolution of cementitious compounds due to pozzolanic reactions between soil pozzolana and the calcium from lime. This evolution of cementitious compounds improves the long-term hydromechanical performances of soil [1-7].

Recently, Akula et al. [1] and Das et al. [2, 3] performed in-situ investigations of a lime-treated hydraulic earth structure and an embankment after several years of atmospheric exposures, respectively.

39 Akula et al. [1] presented the performance of the Friant-Kern Canal in California, which was built with 4%
40 of quicklime treated plastic clays. The Friant-Kern Canal was reported to suffer severe erosion initially.
41 However, after even 40 years after construction, an increase of strength, a reduction in swelling potential,
42 erosion resistance, thus good geo-mechanical stability was reported. Another experimental embankment
43 was shown to exhibit a long-term lime effect even after 7 years of atmospheric exposure in the study
44 reported by Das et al. [2, 3]. The specimens sampled from the embankment were reported to show an
45 average Unconfined Compressive Strength (UCS) of about $3.29 \pm (0.45)$ MPa due to the development of
46 cementitious compounds because of the long-term pozzolanic reactions. Thus, lime was shown to
47 successfully contribute towards the attainment of desired performances of land structures.

48 However, exposing lime-treated structures to an external environment leads to carbonation.
49 Carbonation is defined as a reaction in which CO_2 diffuses through lime-treated soil, gets dissolved in the
50 pore water, reacts with the dissolved Ca^{2+} , and produces calcium carbonates (CaCO_3) [10-14]. It is a
51 naturally occurring phenomenon that is instigated when any cementitious or pozzolanic compounds come
52 in contact with air [14]. During carbonation, precipitation of calcium carbonate occurs with the
53 consumption of available lime for pozzolanic reactions in the short term. While in the long term,
54 carbonation was shown to be detrimental due to the conversion of pozzolanic compounds into CaCO_3 ,
55 which is regarded as a weak cementitious compound [14]. However, the extent of carbonation is shown to
56 be marked by several factors such as temperature, relative humidity (RH), CO_2 concentrations [15], soil
57 type and mineralogy [11,14], binder amount, and curing period [14].

58 Xu et al. [15] evaluated the evolution of carbonation depth by subjecting lime-treated soils to
59 different temperatures, relative humidity (RH), and CO_2 concentrations. Compacted and cured lime-treated
60 soil was subjected to accelerated carbonation conditions consisting of implementation of temperature
61 varying from 15-30°C, RH between 60-90%, and CO_2 in the range of 5-20%. Based on the investigation,
62 they stated that an environment bearing maximum CO_2 concentration and temperature and minimum RH
63 creates the maximum carbonation depth in a given lime-treated soil. Deneele et al. [14] demonstrated that
64 the evolution of carbonation depth was impacted by the lime content added to the soil. Soil treated with 2%
65 lime was reported to have a carbonation depth that was about two times higher than the corresponding soil
66 treated with 5% lime after 28 days of carbonation.

67 Bandipally et al. [11] stated that in a given soil-lime composite, the extent of free-lime carbonation
68 is determined by the dominant mineralogy of the soil. Padmaraj and Arnepalli [12] demonstrated that
69 carbonation of free lime at the early curing stage occurs to cause an expansion in the soil due to precipitation
70 of CaCO_3 , which exhibits a higher molar volume than lime. Such a phenomenon can induce tensile stress
71 in the lime-treated soil resulting in microcracking and strength reduction. Netterberg and Paige-Green [16]
72 emphasized the importance of sealing lime stabilized pavements layers with another layer soon after
73 construction to avoid carbonation. They reported the deleterious effect of carbonation in disintegrating the
74 upper base course of pavement, thus, causing surface failure and reducing the service life of such structures.
75 Bagonza et al. [17] evidenced a reduction of 45-70% in the strength of 7 years carbonated lime stabilized
76 soil.

77 Recently, Deneele et al. [14] evaluated the impact of carbonation on the UCS evolution of lime-
78 treated samples cured in carbonated and non-carbonated environments. They showed that lime-treated
79 specimens, when exposed to carbonation, attain a quick gain in UCS owing to increased pore filling by
80 calcium carbonates (CaCO_3), thus, stiffening the soil. However, non-carbonated and cured specimens that
81 have gained a high UCS due to pozzolanic reactions show a decrease in UCS when exposed to carbonation.
82 At the microscale level, carbonation was shown to transform pozzolanic compounds into carbonates, thus,

83 degrading the initial strength developed. Similar statement was also reported by Vitale et al. [18] in their
84 study made regarding the impact of carbonation on the chemo-mechanical behavior of lime-treated soils.

85 Thus, a part of the above existing studies is focused on the effect of carbonation on the strength
86 evolution, while other brings discussion regarding microstructural modification brought by carbonation on
87 the lime-treated soils at a laboratory scale. However, research on a full-scale lime-treated structure upon
88 exposure to atmospheric CO₂ is limited. Besides, most of these laboratory studies involve the use of CO₂
89 content varying from 5-20%, which is much higher than the atmospheric CO₂ ($\approx 0.03\%$) to which an in-situ
90 lime-treated soil gets exposed to. Also, a significant difference in the evolution of strength was reported
91 between lime-treated specimens carbonated atmospherically and the one carbonated under controlled
92 laboratory conditions [14]. Thus, there exists essentiality to investigate the carbonation phenomenon in an
93 in-situ lime-treated earth structure subjected to atmospheric stresses.

94 In this context, this study aims to investigate the evolution of carbonation depth in a full-scale lime-
95 treated experimental embankment, which was exposed to atmospheric CO₂ for a period of over one year.
96 The study is made on specimens sampled at different depths perpendicular to the surface of the
97 embankment. The first part of the study explains the sampling method. Then chemical and microstructural
98 analyses were made on the sampled specimens to investigate the carbonation effect. Finally, discussions
99 are provided to elaborate on the in-situ carbonation effect.

100

101 **2. Studied embankment, samples, and methods**

102 *2.1 Materials of the embankment*

103 This study involves specimens sampled from the 2.5% quicklime-treated embankment reported in the study
104 presented by Das et al. [2, 3]. The embankment was constructed at Rouen, France, in 2011 using silty soil
105 from Marche-Les-Dames (MLD), Belgium. The main geotechnical properties of the soil and the
106 information regarding the embankment's construction and the surrounding climate can be obtained from
107 Makki-Szymkiewicz et al. [19].

108

109 *2.2 Specimens sampling from the embankment*

110 The studied embankment was atmospherically cured for 7 years after treatment. After 7 years, specimens
111 were sampled at different depths starting from the surface towards the core, and the physicochemical,
112 mechanical, and microstructural investigations made were reported in the study of Das et al. [2, 3].

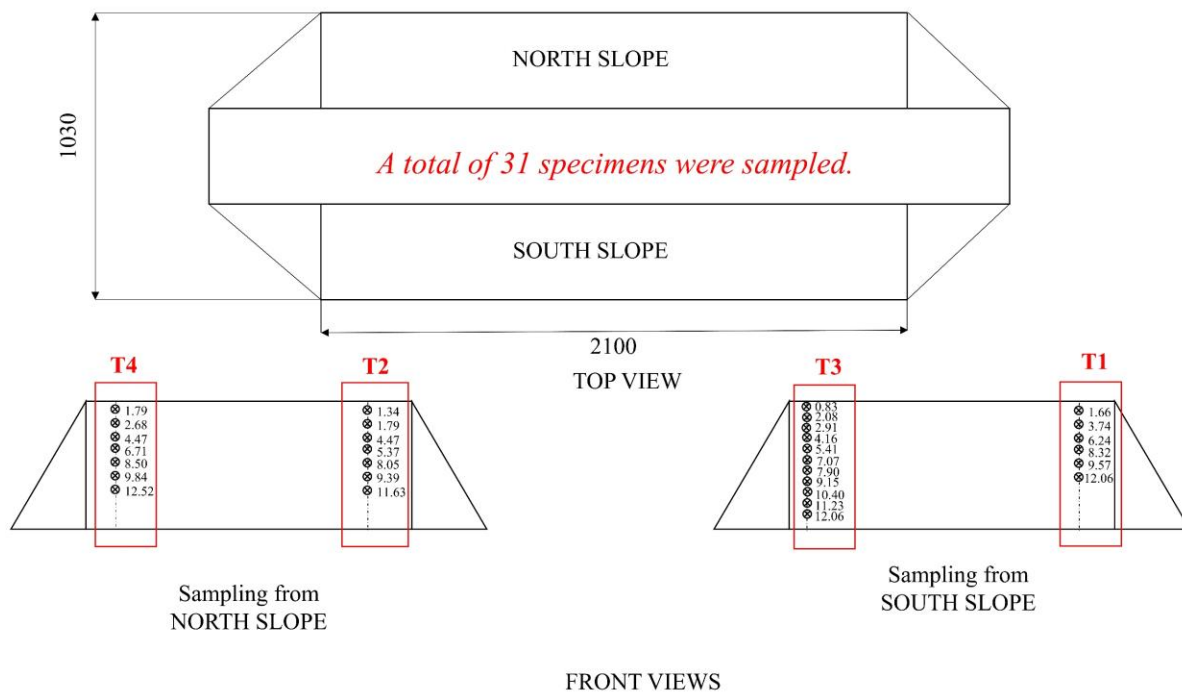
113 Das et al. [2, 3] demonstrated that specimens sampled from the upper layer of the embankment,
114 *i.e.*, a layer up to 12cm depth perpendicular to the surface, showed loss of lime-treated soil pH and water
115 content due to soil-atmospheric interactions. After sampling few specimens within this 12 cm depth, the
116 upper layer was removed. Then compacted specimens were sampled throughout the core, which was shown
117 to report a pH value within 11.08 to 11.66 and the presence of cementitious compounds through scanning
118 electron microscope analysis, thus, evidencing the persistence of lime effect throughout the core.

119 After removal of the upper layer, the core of the embankment was exposed to the atmosphere for
120 another 1 year, to investigate the effect of atmospheric interaction on the core, which was previously
121 reported to bear high pH and cementitious compounds. From the one-year atmospherically exposed core
122 surface layer, specimens were then sampled at different depths up to 12-13 cm perpendicular to the surface.

123 Specimens were sampled manually and was a tedious process provided the lime-treated soils were too hard
 124 to break.

125 Fig. 1 shows the sampling of specimens at different depths from the slope of the embankment
 126 facing North and South. A total of 31 loose specimens were sampled from the 4 trenches, T1, T2, T3, and
 127 T4, and the sampling was made as per convenience. The designation and location of the trenches presented
 128 were similar to the previous study reported by Das et al. [2, 3]. Sampled specimens were then carefully
 129 transported to the laboratory for analysis.

130
 131
 132



133
 134 **Fig. 1** Schematic diagram presenting the top and front view of the full-scale embankment and sampling of specimens from trench
 135 T1, T2, T3, and T4 at different depths normal to the surface. (All units in cm)
 136

137 2.3 Specimen preparation and Laboratory tests

138 All the 31 sampled specimens were subjected to pH tests conducted as per ASTM D4972-19 [20].

139 Then, a carefully selection of specimens was done to perform microstructural investigations:
 140 Thermogravimetric analysis (TGA), Mercury Intrusion Porosimetry (MIP), and Brunauer–Emmett–Teller
 141 (BET) analysis. All specimens were previously lyophilized.

142 TGA analysis was made to detect the presence and evolution of carbonates. The analysis consists
 143 of recording the mass change in a subjected powdered sample as a function of temperature between 20 to
 144 1000°C under a controlled atmosphere [14]. The mass loss indicating the decarbonation of carbonates in
 145 lime-treated soil is known to be recorded in temperature within 650 to 800°C [13].

146 Both MIP and BET tests were conducted to analyze the pore structure of the specimens. The MIP
 147 test involves the progressive intrusion of mercury at pressure p (MPa) into the lyophilized and evacuated

148 specimens. Knowing the pressure required to force the mercury into a cylindrical pore of diameter, D , pore
 149 sizes can be obtained using the Washburn equation (Equation 1) [21].

150
 151

$$152 \quad D = \frac{4 \cdot \gamma \cdot \cos \theta}{p} \quad (1)$$

153

154 D is the diameter of the entrance pore where mercury intrudes, γ is the surface tension of mercury, and θ
 155 represents contact angle.

156 During BET test, nitrogen gas was intruded in evacuated and lyophilized samples. The intrusion
 157 and extrusion phenomenon of nitrogen produces isotherms, which are then used to analyze pore structure
 158 through a method called Barrett-Joiner-Halenda pore (BJH) method. Pore structure is determined by using
 159 the Kelvin equation (Equation 2) [22].

160
 161

$$162 \quad r_k = \frac{2 \cdot V_m \cdot \gamma \cdot \cos \theta}{R \cdot T \cdot \ln \left(\frac{p}{p_0} \right)} \quad (2)$$

163

164 where r_k is the radius of curvature of the condensed gas inside the pore, γ is the surface tension, V_m is the
 165 molar gas volume of an ideal gas, θ is the contact angle, and R is the gas constant.

166 This study classifies pore structure based on the International Union of Pure and Applied
 167 Chemistry (IUPAC) [23], which categorizes the pore-width as macropores ($> 500 \text{ \AA}$), mesopores ($20\text{-}500$
 168 \AA), and micropores ($< 20 \text{ \AA}$). It is worth noting that all the analyses made are compared with untreated
 169 specimens to highlight the evolution in lime-treated soil.

170 MIP and BJH were shown to elaborate the evolution of macropore and mesopores distinctively in
 171 lime-treated soil in the study reported by Das et al. [2]. Thus, both these techniques were employed herein
 172 to investigate the carbonation effect on pores of different ranges.

173 Table 1 summarizes the complete testing program with the corresponding identifications and
 174 numbers of specimens reported in this study.

175

176 **Table 1**

177 Test programs with samples identifications and numbers.

| Test name | No. of samples | Sample name |
|-----------|----------------|---|
| pH | 31 | T1, T2, T3, T4 |
| TGA | 5 | T1 (1.66cm \perp to surface), T4 (1.79cm \perp to surface), |
| MIP | 5 | T4 (4.47cm \perp to surface), T3 (5.41cm \perp to surface), |
| BET | 5 | T1 (8.32cm \perp to surface) |

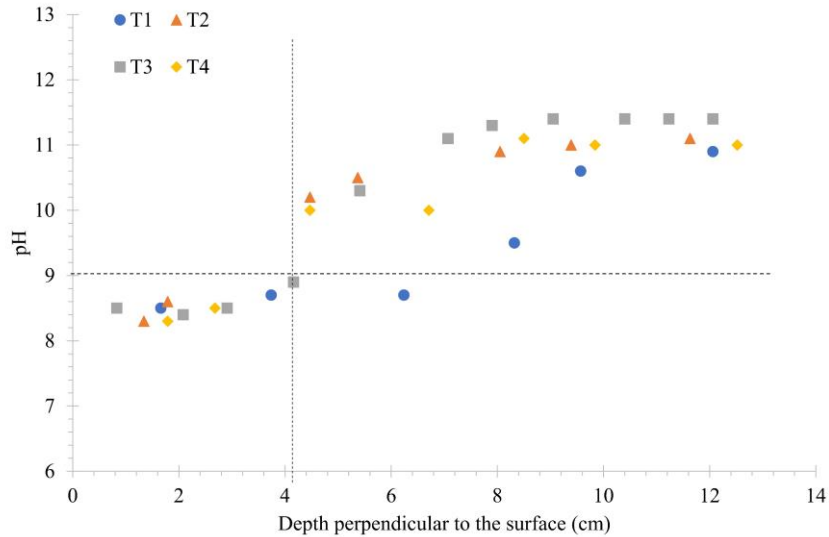
178
 179
 180

181 **3. Results and Discussions**

182 *3.1 pH analysis of atmospherically exposed specimens at different depths*

183 Fig. 2 presents the pH measured in all the 31 lime-treated specimens sampled at different depths and from
184 different trenches, perpendicular to the surface of the embankment. The average pH of the untreated silty
185 soil was about 8.5, as reported in the study of Das et al. [2].

186



187

188 **Fig. 2** pH of in-situ sampled lime-treated specimens sampled at different depths.

189

190 Studies showed that lime treatment enhances the pH of the soil above 12 [8,9], and Xu et al. [15]
191 reported that carbonation of lime-treated soil reduced the pH of the soil below 9. Considering this, it can be
192 seen in Fig. 2 that about 10 specimens sampled in the depths ranging from 0 to 4.2 cm perpendicular to the
193 surface exhibit pH values below 9. Remaining shows pH above 9, which were sampled below 4.2 cm depth
194 perpendicular to the surface.

195

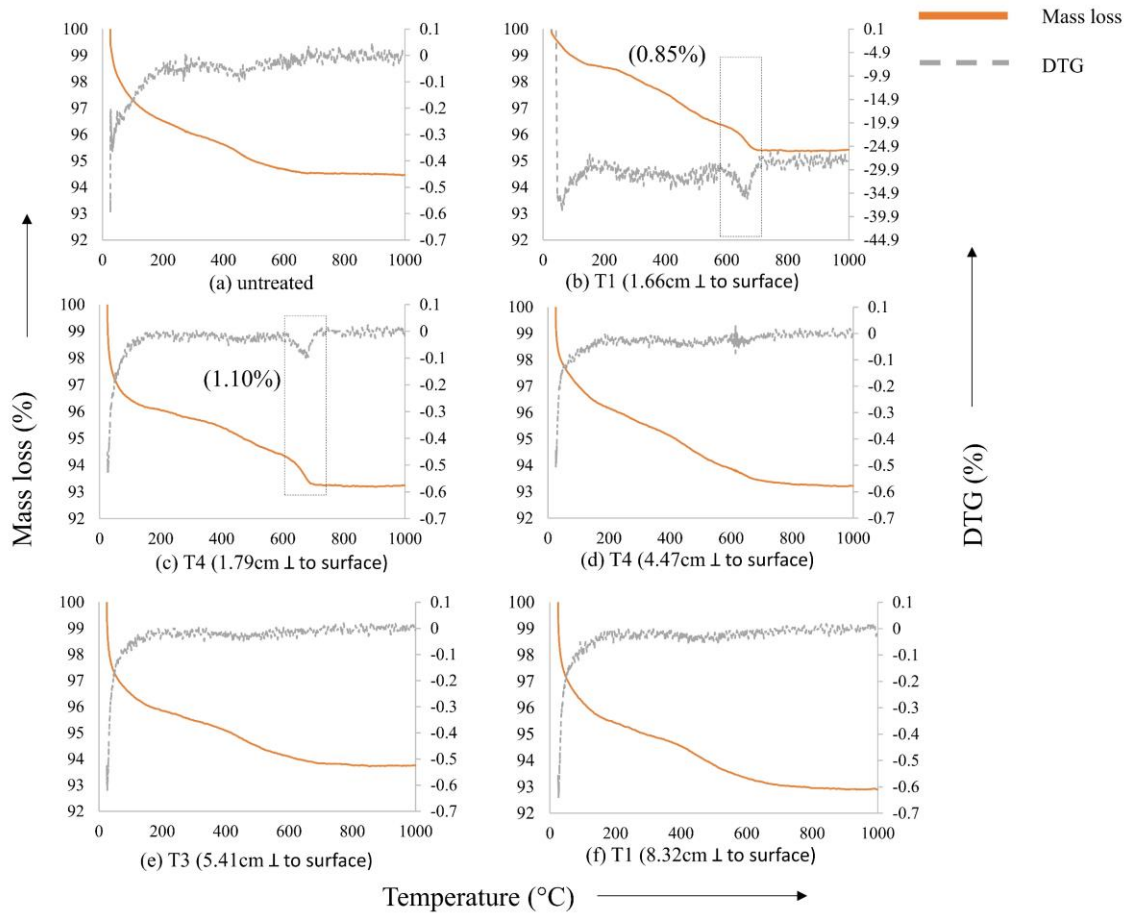
196 *3.2 TGA analysis of atmospherically exposed specimens at different depths*

197 The pH below 9 in about 10 specimens in Fig. 2 could predict the presence of carbonates, as stated in
198 previous studies. In order to confirm this, TGA analysis is presented in Fig. 3.

199 Fig. 3 compares the TGA analysis of untreated and 5 selected lime-treated specimens. The selection
200 of the lime-treated specimens was made in a way that 2 of the specimen's exhibit pH below 9, and the
201 remaining exhibits pH above 9, to highlight the presence of carbonates.

202 On comparing Fig. 3(b) & (c) with Fig. 3(a), a mass loss of 0.85% and 1.10% was observed in the
203 temperature corresponding to 650-800°C in specimens T1 (1.66 cm) and T4 (1.79 cm) perpendicular to the

204 surface. Such a feature indicates the presence of carbonates in specimens T1 (1.66 cm) and T4 (1.79 cm)
 205 perpendicular to the surface, as demonstrated in the study reported by Vitale et al. [18].
 206



207
 208 **Fig. 3** Comparative evaluation of TGA analysis between untreated (a) and lime-treated specimens sampled at different depths (b-
 209 f).
 210

211 At the same time, no such feature was observed in specimens presented in Fig. 3(d), (e), and (f),
 212 sampled at depths 4.47 cm, 5.41cm, and 8.32 cm perpendicular to the surface, respectively. The TGA
 213 analysis of these specimens remained similar to the untreated soil (Fig. 3a).

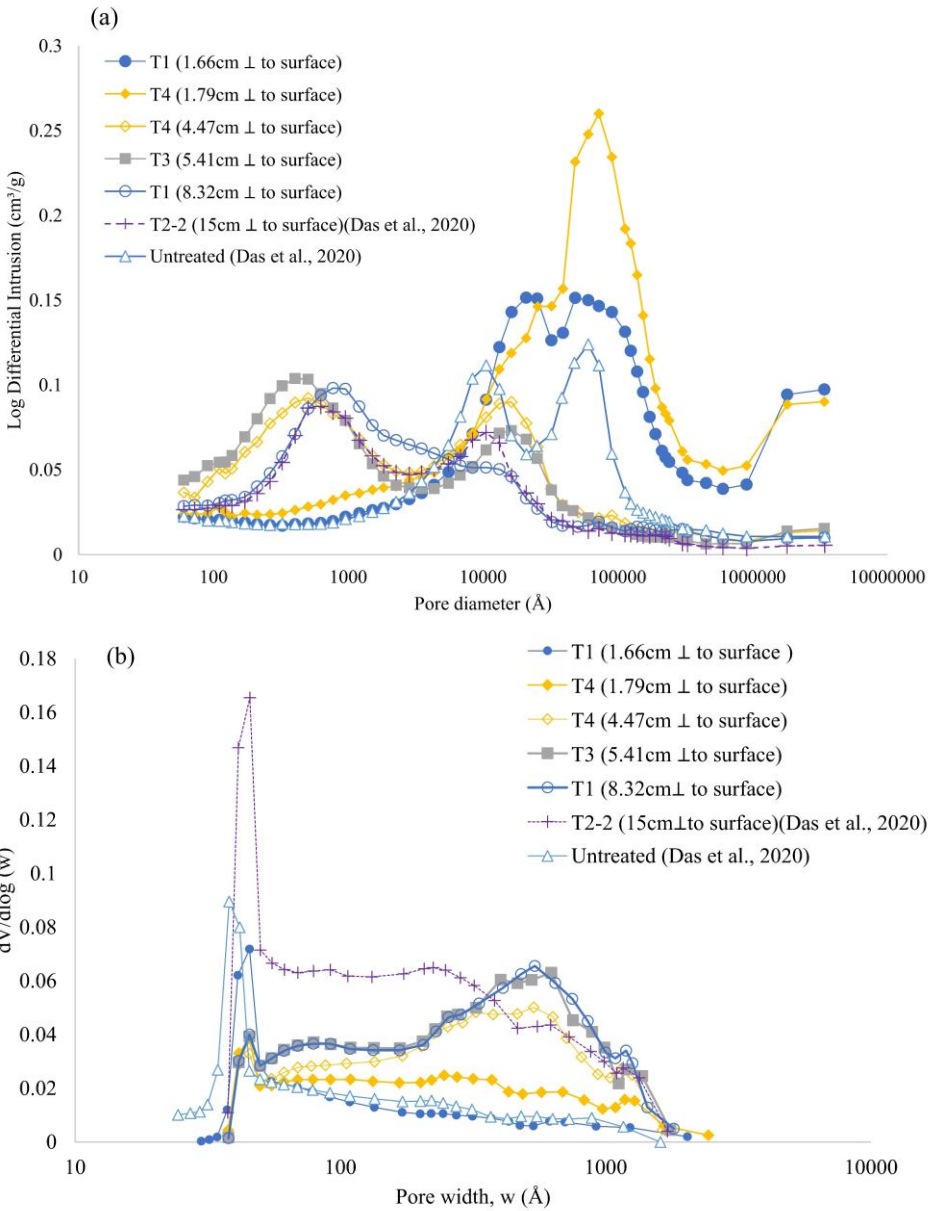
214 Thus, from Fig. 2 and Fig. 3, it can be concluded that the carbonation effect occurred in specimens
 215 sampled at depth up to about 4.2 cm perpendicular to the surface during the one year of atmospheric
 216 exposure.
 217

218 3.3 Pore structure analysis of atmospherically exposed specimens at different depths

219 Specimens subjected to TGA analysis were then subjected to MIP and BET tests to evaluate the impact of
 220 carbonation on the pore structure modification of the soils.

221 Fig. 4a & b presents the pore structure analysis made by MIP and BJH in 5 lime-treated sampled
 222 specimens, respectively. The analysis was compared with the pore structure evolution of untreated and a

223 core-sampled (15cm perpendicular to the surface) lime-treated soil presented in the study reported by Das
 224 et al. [2] to observe the evolution.
 225



226
 227
 228 **Fig. 4** PSD analyzed by MIP (a) and BJH (b) for untreated and lime-treated specimens sampled at different depths perpendicular
 229 to the surface.
 230

231 Comparing Fig. 4a and 4b, it was observed that MIP results distinctively highlight the distribution
 232 of macropores, while BJH, the mesopores.

233 In Fig. 4a, two bimodal peaks at macropore sizes of about 10000 Å and 60000 Å were observed in
 234 the untreated soil. In the lime-treated non-carbonated sampled specimens, as per Figs. 2 & 3, *i.e.*, T4 (4.47
 235 cm ⊥ to surface), T3 (5.41 cm ⊥ to surface), T1 (8.32 cm ⊥ to surface), no such macropore peaks were
 236 observed. A similar feature was observed in the core-sampled lime-treated soil T2-2 (15 cm ⊥ to surface).
 237 Instead, maximum intensities of mesopores availability were present in the core sampled T2-2 specimen,

238 followed by the non-carbonated specimens. Such an evolution was due to development of cementitious
239 compound as a consequence of pozzolanic reactions due to lime treatment [2-9].

240 However, in the carbonated lime-treated sampled soils T1 (1.66 cm \perp to surface) and T4 (1.70 cm
241 \perp to surface), new macropore peaks were observed within the pore range 20000 Å to 100000 Å. Such larger
242 macropores were missing both in the untreated and the non-carbonated lime-treated specimens. At the same
243 time, almost negligible mesopores were observed in the carbonated specimens and were almost similar to
244 the untreated soil. It is worth noting that the representation of the specimens as carbonated and non-
245 carbonated in the texts are made based on the analysis obtained in Fig. 2 & 3.

246 This presence of large macropores in the carbonated specimens can be attributed to the
247 development of CaCO₃ as a consequence of carbonation based on previous investigations [10, 12, 19].
248 During the early stage of atmospheric curing, transformation of available free lime to calcite might have
249 occurred on the surface. Since the molar volume of CaCO₃ is reported to be greater than portlandite [12,18].
250 Thus, this might have caused tension within soil particles and induced cracking [12]. Such a phenomenon
251 explains the present development of macropores in the carbonated specimens even greater than the
252 untreated specimen. Again, the absence of mesopores in the carbonated specimens can be attributed to the
253 transformation of cementitious compounds into calcite, which was reported to create larger pore diameter
254 by Deneele et al. [14].
255

256

257 **4. Conclusions**

258 The study investigated the effect of atmospheric exposition on the chemical and pore-structure evolution
259 of 2.5% quicklime-treated sampled specimens after 1 year of atmospheric exposure. Following are the
260 conclusions derived based on the investigation:

- 261 1. One year of atmospheric exposure induces carbonation of the 2.5% lime-treated specimens up to a
262 depth of about 4.2 cm perpendicular to the surface. The decrease in pH to a value equal to or less
263 than 9 and mass loss at a temperature corresponding to 650-800°C during TGA analysis confirmed
264 the carbonation of lime-treated soils.
- 265 2. Carbonation generated macropore peaks within the pore range 20000 Å to 100000 Å in the lime-
266 treated specimens. Mesopores developed due to lime treatment, as observed in the non-carbonated
267 specimens, were not available in the carbonated specimens.

268 Thus, the case study shows that one year of atmospheric exposure of a lime-treated structure brought a
269 reduction in soil pH and an increase in soil macropores due to carbonation up to a depth of about 4.2 cm
270 perpendicular to the surface.

271

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273

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278

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