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Carbonation investigation on atmospherically exposed lime-treated silty soil.

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Abstract

- 12 The effect of carbonation on a one-year atmospherically exposed lime-treated soil structure is investigated.
- 13 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
- 15 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
- 17 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
- 20 confirmed the loss of cementitious compounds due to carbonation, thus, converting the mesopores
- 21 developed due to lime treatment to macropores of larger pore diameter.
- 22 Keywords: lime-treated soil; carbonation; atmospherically exposed; macropores; soil pH; calcium
- 23 carbonates.

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1. Introduction.

- 26 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
- 29 lime was shown to bring significant development in workability and strength [1-8]. There are two basic
- 30 soil-lime chemical reactions that contribute towards the improvement of soil engineering properties. These
- 31 reactions are categorized as short-term or modification reactions and long-term or pozzolanic reactions
- 32 [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
- 34 cementitious compounds due to pozzolanic reactions between soil pozzolana and the calcium from lime.
- 35 This evolution of cementitious compounds improves the long-term hydromechanical performances of soil

36 [1-7].

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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.

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

However, exposing lime-treated structures to an external environment leads to carbonation. Carbonation is defined as a reaction in which CO₂ diffuses through lime-treated soil, gets dissolved in the pore water, reacts with the dissolved Ca²⁺, and produces calcium carbonates (CaCO₃) [10-14]. It is a naturally occurring phenomenon that is instigated when any cementitious or pozzolanic compounds come in contact with air [14]. During carbonation, precipitation of calcium carbonate occurs with the consumption of available lime for pozzolanic reactions in the short term. While in the long term, carbonation was shown to be detrimental due to the conversion of pozzolanic compounds into CaCO₃, which is regarded as a weak cementitious compound [14]. However, the extent of carbonation is shown to be marked by several factors such as temperature, relative humidity (RH), CO₂ concentrations [15], soil type and mineralogy [11,14], binder amount, and curing period [14].

Xu et al. [15] evaluated the evolution of carbonation depth by subjecting lime-treated soils to different temperatures, relative humidity (RH), and CO₂ concentrations. Compacted and cured lime-treated soil was subjected to accelerated carbonation conditions consisting of implementation of temperature varying from 15-30°C, RH between 60-90%, and CO₂ in the range of 5-20%. Based on the investigation, they stated that an environment bearing maximum CO₂ concentration and temperature and minimum RH creates the maximum carbonation depth in a given lime-treated soil. Deneele et al. [14] demonstrated that the evolution of carbonation depth was impacted by the lime content added to the soil. Soil treated with 2% lime was reported to have a carbonation depth that was about two times higher than the corresponding soil treated with 5% lime after 28 days of carbonation.

Bandipally et al. [11] stated that in a given soil-lime composite, the extent of free-lime carbonation is determined by the dominant mineralogy of the soil. Padmaraj and Arnepalli [12] demonstrated that carbonation of free lime at the early curing stage occurs to cause an expansion in the soil due to precipitation of CaCO₃, which exhibits a higher molar volume than lime. Such a phenomenon can induce tensile stress in the lime-treated soil resulting in microcracking and strength reduction. Netterberg and Paige-Green [16] emphasized the importance of sealing lime stabilized pavements layers with another layer soon after construction to avoid carbonation. They reported the deleterious effect of carbonation in disintegrating the upper base course of pavement, thus, causing surface failure and reducing the service life of such structures. Bagonza et al. [17] evidenced a reduction of 45-70% in the strength of 7 years carbonated lime stabilized soil.

Recently, Deneele et al. [14] evaluated the impact of carbonation on the UCS evolution of lime-treated samples cured in carbonated and non-carbonated environments. They showed that lime-treated specimens, when exposed to carbonation, attain a quick gain in UCS owing to increased pore filling by calcium carbonates (CaCO₃), thus, stiffening the soil. However, non-carbonated and cured specimens that have gained a high UCS due to pozzolanic reactions show a decrease in UCS when exposed to carbonation. At the microscale level, carbonation was shown to transform pozzolanic compounds into carbonates, thus,

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

Thus, a part of the above existing studies is focused on the effect of carbonation on the strength evolution, while other brings discussion regarding microstructural modification brought by carbonation on the lime-treated soils at a laboratory scale. However, research on a full-scale lime-treated structure upon exposure to atmospheric CO_2 is limited. Besides, most of these laboratory studies involve the use of CO_2 content varying from 5-20%, which is much higher than the atmospheric CO_2 ($\approx 0.03\%$) to which an in-situ lime-treated soil gets exposed to. Also, a significant difference in the evolution of strength was reported between lime-treated specimens carbonated atmospherically and the one carbonated under controlled laboratory conditions [14]. Thus, there exists essentiality to investigate the carbonation phenomenon in an in-situ lime-treated earth structure subjected to atmospheric stresses.

In this context, this study aims to investigate the evolution of carbonation depth in a full-scale lime-treated experimental embankment, which was exposed to atmospheric CO_2 for a period of over one year. The study is made on specimens sampled at different depths perpendicular to the surface of the embankment. The first part of the study explains the sampling method. Then chemical and microstructural analyses were made on the sampled specimens to investigate the carbonation effect. Finally, discussions are provided to elaborate on the in-situ carbonation effect.

2. Studied embankment, samples, and methods

2.1 Materials of the embankment

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

2.2 Specimens sampling from the embankment

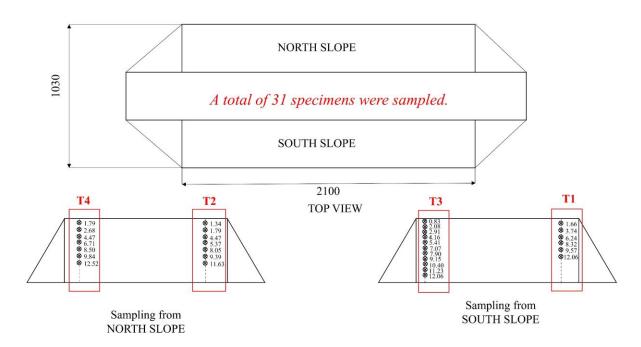
The studied embankment was atmospherically cured for 7 years after treatment. After 7 years, specimens were sampled at different depths starting from the surface towards the core, and the physicochemical, mechanical, and microstructural investigations made were reported in the study of Das et al. [2,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

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

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

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

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



FRONT VIEWS

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

2.3 Specimen preparation and Laboratory tests

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

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

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

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

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

$$D = \frac{4.\gamma \cdot \cos \theta}{p} \tag{1}$$

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

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

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

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

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

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

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

 Table 1

 Test programs with samples identifications and numbers.

Test name	No. of samples	Sample name
pН	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 ⊥ to surface)

3. Results and Discussions

3.1 pH analysis of atmospherically exposed specimens at different depths

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

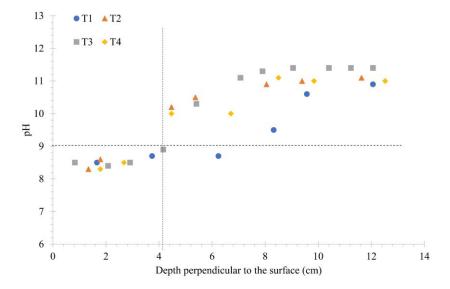


Fig. 2 pH of in-situ sampled lime-treated specimens sampled at different depths.

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

3.2 TGA analysis of atmospherically exposed specimens at different depths

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

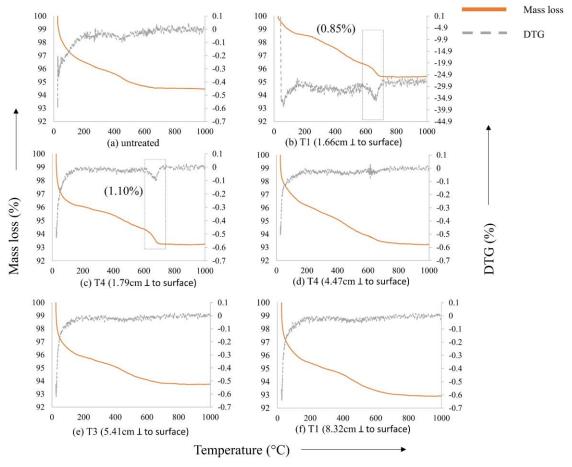
Fig. 3 compares the TGA analysis of untreated and 5 selected lime-treated specimens. The selection of the lime-treated specimens was made in a way that 2 of the specimen's exhibit pH below 9, and the remaining exhibits pH above 9, to highlight the presence of carbonates.

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

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



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Fig. 3 Comparative evaluation of TGA analysis between untreated (a) and lime-treated specimens sampled at different depths (bf).

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

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

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3.3 Pore structure analysis of atmospherically exposed specimens at different depths

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

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Fig. 4a & b presents the pore structure analysis made by MIP and BJH in 5 lime-treated sampled specimens, respectively. The analysis was compared with the pore structure evolution of untreated and a core-sampled (15cm perpendicular to the surface) lime-treated soil presented in the study reported by Das et al. [2] to observe the evolution.



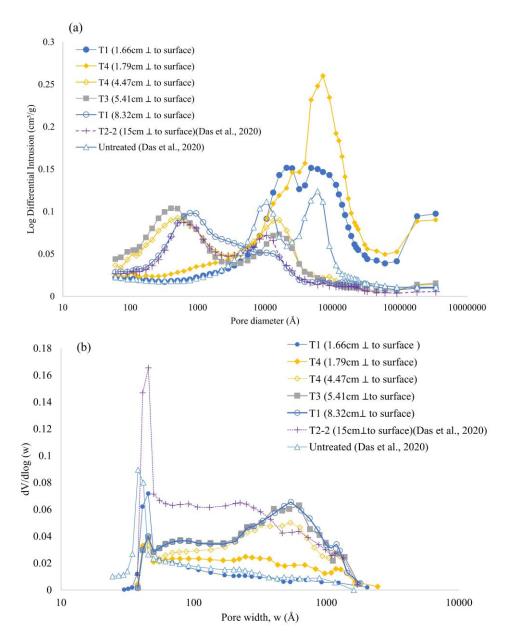


Fig. 4 PSD analyzed by MIP (a) and BJH (b) for untreated and lime-treated specimens sampled at different depths perpendicular to the surface.

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

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

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

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

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

4. Conclusions

- The study investigated the effect of atmospheric exposition on the chemical and pore-structure evolution of 2.5% quicklime-treated sampled specimens after 1 year of atmospheric exposure. Following are the conclusions derived based on the investigation:
 - 1. One year of atmospheric exposure induces carbonation of the 2.5% lime-treated specimens up to a depth of about 4.2 cm perpendicular to the surface. The decrease in pH to a value equal to or less than 9 and mass loss at a temperature corresponding to 650-800°C during TGA analysis confirmed the carbonation of lime-treated soils.
 - 2. Carbonation generated macropore peaks within the pore range 20000 Å to 100000 Å in the limetreated specimens. Mesopores developed due to lime treatment, as observed in the non-carbonated specimens, were not available in the carbonated specimens.
- Thus, the case study shows that one year of atmospheric exposure of a lime-treated structure brought a reduction in soil pH and an increase in soil macropores due to carbonation up to a depth of about 4.2 cm perpendicular to the surface.

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