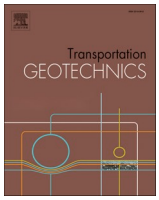
Transportation Geotechnics 44 (2024) 101174



Contents lists available at ScienceDirect

Transportation Geotechnics

journal homepage: www.elsevier.com/locate/trgeo





Carbonation of a lime treated soil subjected to different curing conditions

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ARTICLE INFO

Keywords:
Carbonation
Quicklime
Pozzolanic reaction
Soil treatment
Earthworks

ABSTRACT

Although the lime treatment of soils has been used in many earthworks' projects, limited studies have been devoted to the quantification of their carbonation. This is not only important regarding the impact on the final properties, it is also critical to quantify it when evaluating the carbon footprint of infrastructures. This work investigates the carbonation of a silt treated with 1, 2 and 4% quicklime, exposed to different carbon dioxide conditions: 1% (C1), atmospheric concentration (C2), and 0% (C3). The carbonation depth, mechanical performance and carbonation rate were determined. The results showed that carbonation occurred at a different rate for each condition. A minimum average carbonation rate of respectively 59, 44 and 34 % for 1, 2 and 4% lime addition was measured even in the less exposed conditions. This phenomenon can be attributed to the early carbonation occurring during sample preparation, particularly during the 4-hours mellowing period before compaction.

Introduction

Lime-treatment of soils has been used all over the world for more than 80 years. It is considered by many today to be the reference technology for earthworks when the subgrade does not have the appropriate geotechnical properties in order to withstand the imposed loads from buildings and infrastructure constructed on it, such as roads, railways, airports, dams and levees, or industrial platforms [12]. More precisely, it is heavily used in the context of wet and clayey soils that cause trafficability and workability problems at the construction stage and create a risk of swelling/shrinkage for the final building or infrastructure. Given its wide use all over the world, lime-treatment of soils is well documented and many guides have been published in order to give engineers the necessary technical background for using it for many projects [234].

The way lime addition works is generally explained in a 2-step sequence, separating between a short-term soil improvement and a long-term stabilization effect. The short-term soil improvement [256] is essentially due to a combination of 2 mechanisms: the first mechanism occurs when quicklime (calcium oxide - CaO) is used as a reactant for soil treatment. This is generally the case when wet soils are being considered and is typical of the way soils are treated in Northern Europe (Belgium, France, UK…). In this case, quicklime hydration proceeds in an exothermic way that reduces the water content of the soil:

$$CaO + H_2O \rightarrow Ca(OH)_2 + \text{heat}(1)$$

The second mechanism of soil improvement is a consequence of the basic character of hydrated lime (calcium hydroxide – $Ca(OH)_2$) and the good water solubility of calcium ions. The presence of lime changes the ionic strength of the interstitial water and induces clay particle flocculation [1]. As a consequence, the lime-treated soil becomes less susceptible to changes in moisture content, as reflected by a higher bearing capacity, a better compactibility and a lower swelling/shrinkage potential, that can be completely neutralized [127].

In the long-term, and provided the lime content is above the so-called lime-fixation point [128910], the pH of the interstitial water reaches values close to 12.5. This high alkalinity enhances the solubility of silica and alumina [12]. Pozzolanic reactions can then proceed, greatly increasing the strength of the lime-treated soil as a consequence of the formation of cementitious hydration products such as calcium silicate hydrates (C-S-H) and calcium alumino-silicates hydrates (C-A-S-H). Compressive strengths values above 5 MPa can be reached with 2–4 % lime addition after a few months when the treated-soil is reactive, which is the case of most soils with Plasticity Index (PI) over 10 [12].

So, the fundamentals of lime-treatment of soils seemed to be fully understood a few years ago and guides reflect this basic understanding of the processes.

However, recent focus on the effect of lime carbonation showed that this picture is indeed incomplete. Hydrated lime can react with carbon

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https://doi.org/10.1016/j.trgeo.2023.101174
Received 7 September 2023; Received in revised form 4 December 2023; Accepted 5 December 2023
Available online 6 December 2023
2214-3912/© 2023 Published by Elsevier Ltd.

dioxide (CO_2) according to the following equation (equation 2):

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \quad (2)$$

Very limited studies have been published on the carbonation of lime in real lime-treated embankments. In a study published in 2019, Haas and Ritter reported on the chemistry of old lime-treated samples cored until a depth of 10 m from A3 motorway embankments in southeast Germany [11]. Using carbon dioxide measurements and available lime quantification based on NF EN 459–2 [12], they showed that of the 2.5 % quicklime used 34 years ago for the treatment of the local silt (PI 11–18), 47 % had transformed into hydration products through pozzolanic reaction, 37 % was present in neo-formed calcium carbonate ($CaCO_3$) due to carbonation, and 16 % still remained as available hydrated lime [11]. Calcium carbonate was easy to measure because the original soil had a very limited amount of native calcium carbonate (2.3 %). They also showed that the compressive strength remained high with values up to 6 MPa after 34 years.

A second study by Akula et al. (2020) reported that lime in samples taken 1 m deep from the lime-treated banks of the Friant-Kern canal in California, was carbonated to an extent of 34 % [13]. The lime-treatment of the local clays (Porterville clay with PI 23–36) was performed at 3 or 4 % quicklime addition back in the 1970 s [14] and the samples thus were almost 50 years old. Lime carbonation was measured by Thermo Gravimetric Analysis (TGA measuring mass loss vs temperature), which was made easier by the fact that the untreated clay had essentially no native carbonates. Interestingly, the order of magnitude found for the extent of carbonation of lime in California for the treatment of a clay, i.e; (34 %) is remarkably close to the value found in Germany for the treatment of a silt (37 %).

Some laboratory studies have also been performed: Deneele et al. (2021) [15] focused on a Belgian silt (PI 5–11) treated in the lab with 2 and 5 % quicklime addition and cured for up to 6 months under three different conditions: (i) sealed bag curing at room temperature (25 °C) limiting the contact with air (ie no carbon dioxide), (ii) outside conditions in the city of Paris (average air moisture content of 65 % relative humidity (RH), atmospheric carbon dioxide close to 420 ppm and spring external temperatures from 11 to 18 °C), and (iii) high carbon dioxide content thanks to the use of a curing chamber with controlled temperature (30 °C), moisture (70 % RH), and carbon dioxide level (10 %). Using mostly thermogravimetric analyses (TGA), after 6 months of exposure, they measured average carbonation rates around 30, 70 and 80 % respectively for the three different curing conditions previously described. Clearly, the samples with little carbon dioxide exposure still could reach a carbonation extent not unlike the one reported in the previously cited field studies [1113]. Here, TGA measurements were facilitated by the fact that the treated soil had no native calcium carbonate content.

As a result, the limited published literature on the topic highlights that carbonation is an important part of the chemical processes at stakes in soil treatment with lime. Some studies mentioned carbonation in the context of soil treatment with lime [16171819], but without quantifying the amount of lime that transformed into calcium carbonate and even less the carbonation kinetics. It has been quite overpassed until now because it is not so easy to measure, and most studies were performed in the lab with sealed bags, a procedure minimizing its effects. However, it has become very important to quantify because the exact degree of carbonation has a strong impact on the carbon footprint of infrastructures. Denayer et al. (2019) [20] have shown that taking into account 37 % of carbonation for lime, as suggested from the Haas-Ritter study [11], can lower the carbon footprint of lime-treated embankments by ~ 15 %. Therefore, correctly measuring the extent of carbonation of lime-treated soils has a double interest: first, there is a fundamental issue, regarding the importance of carbonation vs pozzolanic reactions in the strength development of lime-treated soils. Second, documenting the "normal" extent of carbonation of lime in this application is fundamental in order to perform complete life cycle assessments of lime-treated embankments [2021].

In this context this carbonation study was built in order to see whether curing of lime-treated soils in lab conditions, following a procedure adapted from the work by Deneele et al (2021)[15], could confirm the numbers observed in former studies. In addition, the chosen soil had a significant native limestone content, making it more complex to precisely determine the extent of carbonation. The way the study was constructed, the chosen materials and the results obtained are firstly presented. Then, the results are discussed in light of former works. Suggestions are finally proposed to further study this topic of major interest for researchers and practitioners of soil-treatment as well as those involved in sustainability and climate change.

Materials and methods

Characterization of the soil

The soil used in this study was a silty soil from a clay brick plant in the south of France. The characteristics of this soil are presented in Table 1. The methylene blue value (VBS) that allows the semi-quantitative determination of the clay content of a soil was measured according to NF P 94–068 [22]. The Atterberg limits (liquid limit and plastic limit) was determined according to the standard EN ISO 17892–12 [23]. The particle diameter below which 90 % of the particles pass (d90) was determined by measuring the particle size distribution (PSD) using a COULTER laser granulometry type LS 13 320. In addition, Table 2 shows the chemical composition of the soil determined by X-ray fluorescence (XRF) using a S4-Pioneer equipped with 4 KW generator and Rh anode.

Given the results shown in Table 1, the soil classifies as a medium plasticity fine soil (F2) according to the standard NF EN 16907–2 [24] and to the newly updated French technical guide for earthworks (GTR for "Guide Technique Remblais") [25]. In the French context, F1 and F2 soils are generally called silts, when F3 and F4 soils are usually called clays.

The mineralogical composition of the soil was determined by powder X-ray diffraction (XRD) analysis using a Bruker D2 apparatus with Cu Kα radiation. Three different analysis were performed on the soil, using at each time 1 g of material, in order to appreciate the repeatability. The X-ray patterns were acquired in the 2θ (10–80°) with a step size of 0.02° and 1 s per step. The main mineralogical phases found in the soil were: calcite, quartz, and clayey phases such as kaolinite, illite, in addition to dolomite and clinochlore as shown in Fig. 1.

ThermoGravimetric Analysis (TGA) and Derivative ThermoGravimetry (DTG) were conducted on the untreated soil to estimate the native calcium carbonate ($CaCO_3$) content. The soil was first homogenised and then 5 different samples were collected in order to perform 5 TGA analysis to obtain a range of calcium carbonate contents in the samples. A Mettler Toledo TGA testing machine was utilized using 100 to 150 mg of material and the thermal analysis was performed within a temperature range from 40 to 1000 °C. The heating program was divided into 3 segments: linear at 3 °C/min between 40 and 105 °C, isothermal for 20 min at 105 °C and then linear at 10 °C/min between 105 and 1000 °C.

Due to the similarity in the TGA/DTG results for the 5 soil samples, Fig. 2 shows only one result for the sake of clarity. The DTG curve shows two main peaks, the first one at 450 °C related to the decomposition of the organic matter and kaolinite and the second peak at 750 °C corresponding to the decarbonation of calcium carbonates [1315].

The quantification of the mass fraction of native calcium carbonates $x_{CaCO3,0}$ in the soil is needed to calculate the carbonation rate of the soil and to separate the neo-formed carbonates (formed after compaction and exposure to different conditions as described in section 2.2) from the native one originally presents in the soil.

The initial mass fraction of calcium carbonates $x_{CaCO3,0}$ in the soil is calculated according to equation (3) below:

Table 1
Properties of the silt used in this study.

	Methylene blue value (g/100 g) (VBS)	Liquid limit (LL) (%)	Plastic limit (PL) (%)	Plasticity index (%)	d90
Method	NF P 94–068	EN ISO 17892–12	EN ISO 17892–12	EN ISO 17892–12	Laser granulometry
Silt	3.4	32.3	16.6	15.7	<73 μm

Table 2
XRF analysis of the silty soil.

Element	O	Ca	Fe	Al	Si	Mg	K	Na	Ti
Percentage	54.8	8.0	3.2	9.7	20.9	1.0	1.7	0.2	0.4

$$x_{CaCO_3,0} = \frac{\Delta m}{m_0} \cdot \frac{M_{CaCO_3}}{M_{CO_2}} \quad (3)$$

Where:

- Δm represents the mass loss of the soil between 750 and 800 °C as measured by TGA
- m_0 is the initial dry soil mass in the TGA experiment,
- M_{CaCO_3} and M_{CO_2} are the molar masses for calcium carbonate (100 g/mol) and carbon dioxide (44 g/mol) respectively.

With this definition, the average native $CaCO_3$ in the soil was 19.7 %

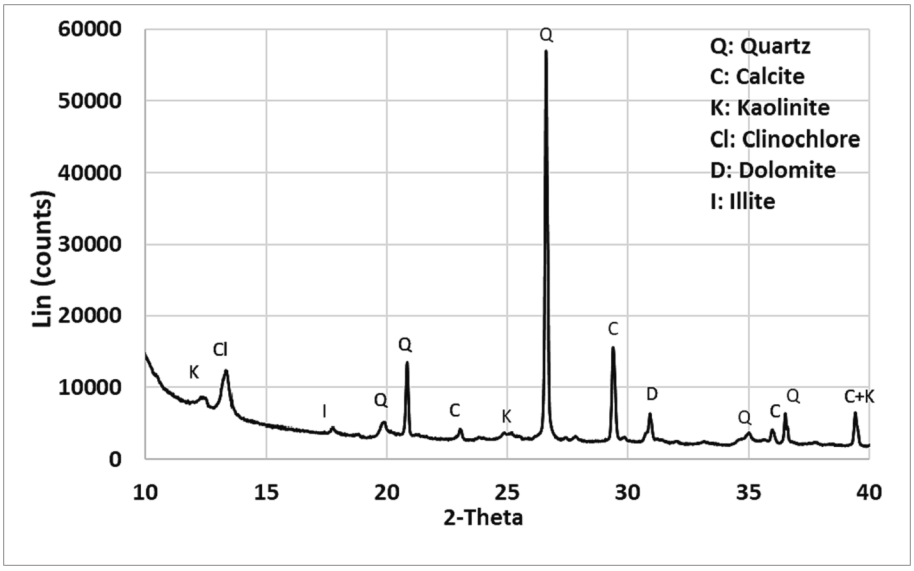


Fig. 1. X-ray pattern of the silty soil.

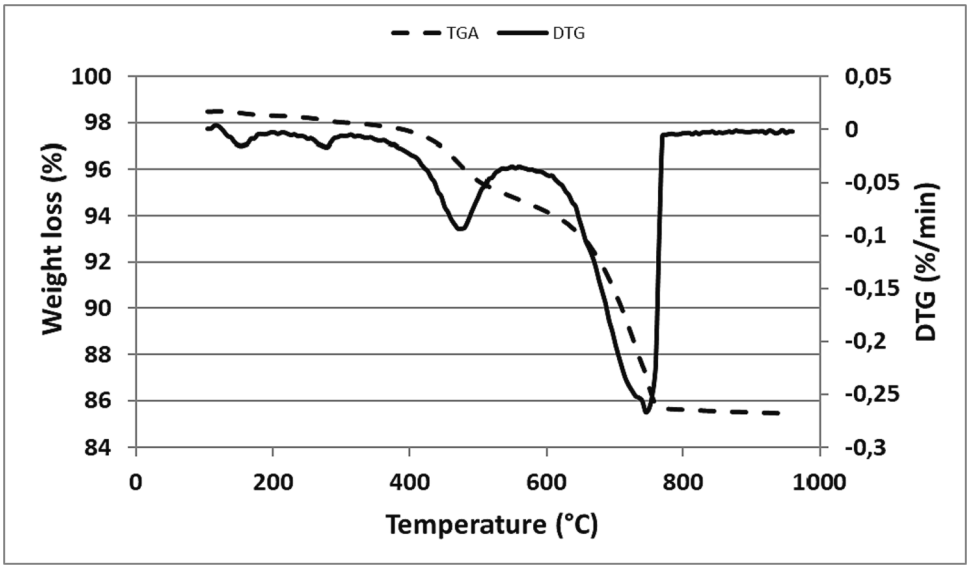


Fig. 2. TGA/DTG analysis of the soil.

with a standard deviation of 0.3 % (absolute value).

The quicklime used for the soil treatment was a CL 90Q according to NF EN 459–1 [26]. This lime contained 91.5 % of available CaO and has a reactivity t_{60} (which represent the time (in minutes) needed to reach 60 °C when mixing quicklime with water according to the standard NF EN 459–2: 2010 [12]) of 1.4 min. The lime fixation point (LFP) of the soil was determined according to the Eades and Grim test [8] and gave a result of 1 %.

The carbonation study was done on four mix designs:

- L0 corresponding to the reference soil (without lime treatment);
- L1 corresponding to the soil treated with 1 % lime addition corresponding to LFP, which is the typical lime content used for soil improvement;
- L2 corresponding to the soil treated with 2 % lime addition corresponding to the typical onsite application for stabilization works (LFP + 1);
- L4 corresponding to the soil treated with 4 % lime addition. Although this lime content might seem somewhat high compared to what is generally done on the worksite for this type of soil, it was chosen to maximize the potential amount of neo-formed calcium carbonates and make it hopefully easier to differentiate vs native calcium carbonates from the soil.

The proctor test was performed according to the European standard NF EN 13286–2 [27]. The proctor curves of the 4 mix designs are represented in Fig. 3 and the corresponding optimum dry density and moisture data are given in Table 3. The optimum was defined at the intersection with the 85 % saturation curve. The results showed that the higher the lime dosage, the higher the optimum water content and the lower the dry density. As expected, this behaviour is consistent with the effects of the lime treatment and soil stabilization already reported elsewhere [123428].

Table 3
Optimum water content and dry density (defined at the intersection with 85% saturation curve) for compaction of the specimens according to their lime content.

Lime content (%)	Water content (mass %)	Dry density (g/cm^3)
0	12.1	1.97
1	16.2	1.79
2	19.2	1.68
4	20.4	1.64

Preparation of the samples and exposure conditions

This study was conducted on samples compacted from the top to 85 % at optimum proctor dry density (OPN) by static axial compression in cylindrical moulds of a height of 100 mm and a diameter of 50 mm. Before compacting, the soil of each mix design was mixed with water using a soil cutter machine in order to reach the desired moisture content, and kept in sealed bags for 2 days for moisture content homogenization. Then, the moisture content was verified and adjusted if needed before treatment and compacting. For the treated samples, lime was added (1, 2 and 4 %) to the soil and the treated soil was put in a bucket with a height of 40 cm. The mix was exposed to air for a mellowing period of 4 h before homogenization and compaction.

Table 3 shows the optimum moisture content and the dry density of the 4 mixes. Three curing times (28, 90 and 180 days) were chosen to follow the carbonation of the soils. Therefore, 3 samples were prepared for each testing time, each exposure condition and each mix design. A total of 36 samples were thus compacted for each mix design.

After compaction, the samples were exposed to three curing conditions:

- C1: in a carbonation chamber at 20 °C, 70 % Relative Humidity (RH), and 1 % carbon dioxide.
- C2: in a climatic chamber at 20 °C, 70 % RH, and atmospheric carbon dioxide concentration.

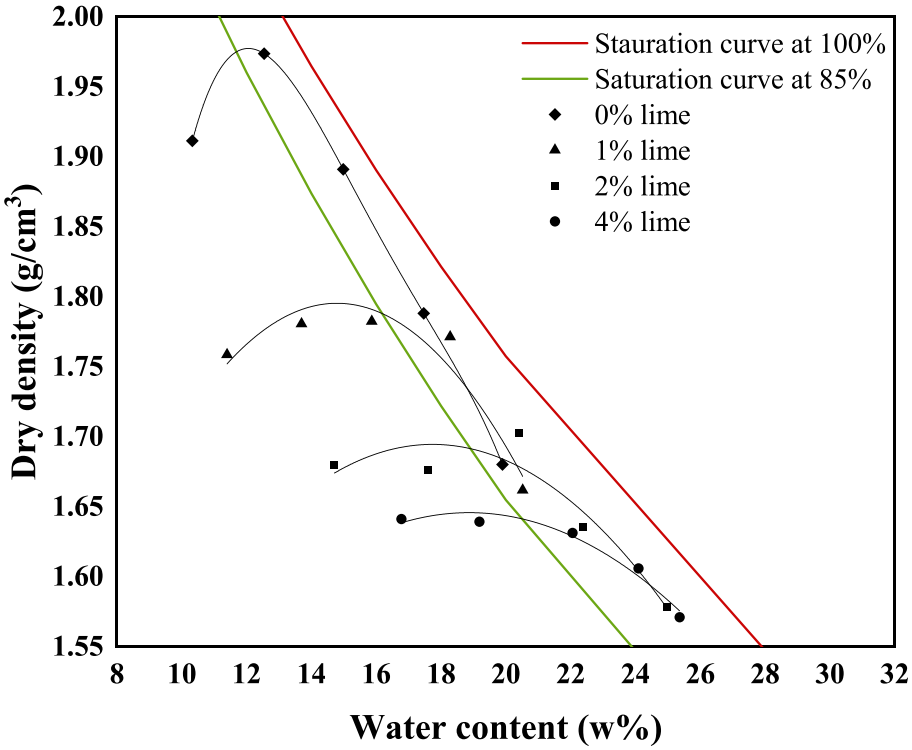


Fig. 3. Proctor curves for the 4 mix designs.

- C3: in sealed bags at 20 °C (0 % carbon dioxide) as described in previous studies [15293031].

The exposure to the different conditions was unidirectional from the top of the sample. To do this, the lateral surfaces and the bottom of the samples were covered with a plastic film to protect them from the carbonation reaction. Due to the fact that there are very limited studies done on the carbonation of lime-treated soils, RH was chosen according to what is known for Portland-cement based concrete. In such studies, it was reported that concrete carbonates faster at 20 °C when RH is between 40 and 80 % [32]. So, the 70 % RH was chosen in this study in order to maximize carbonation. The sample preparation process and the curing are illustrated in the Fig. 4 below.

Methods

At each curing time different analyses were performed to follow the carbonation process of the samples and to study the effects of the different exposure conditions and of the lime treatment on the carbonation reactions (Fig. 5a). Therefore, the following analyses were realized:

- Unconfined Compressive Strength tests (UCS) were performed at 28, 90 and 180 days. 3 samples were tested for each condition and mix design to calculate the average for the three values and assess repeatability. The tests were performed at a constant displacement rate of 1 mm/min using an INSTRON press of 150 KN.
- Phenolphthalein test consists in spraying a phenolphthalein solution on the sample from the surface to the bottom to determine the carbonation depth as is commonly done for concrete [3334] and less commonly for lime-treated soils [15]. The carbonation depth was evaluated according to the colour deviation of the phenolphthalein; the colour of the solution changes to purple for pH $>$ 9 indicating an uncarbonated zone (hydrated lime gives a pH of 12.4), while for pH $<$ 9 the solution remains colourless indicating a carbonated zone for the lime treated specimens [15 3334]. This test was performed for all samples after 28, 90 and 180 days using a 1 % phenolphthalein ethanol solution from Merck.
- The mass loss at 105 °C was measured for all samples at each curing time to compare the measured water content to the initial moisture content.
- Thermogravimetric analysis TGA/DTG was carried out on samples treated with 1, 2 and 4 % of lime addition at all curing times (28, 90 and 180 days). Two TGA analysis were performed for each sample, one specimen was taken from the surface slice of the sample using a spatula to collect the material, and the second one from the bottom slice as shown in Fig. 5b.

Results

Unconfined compressive strength

Fig. 6 presents the results of the evolution of the UCS in function of time (28, 90 and 180 days) for the 3 tested exposure conditions (C1, C2 and C3) and the different lime addition (0, 1, 2 and 4 % labelled as L0, L1, L2 and L4 in the following).

For the reference untreated samples L0 (Fig. 6), the C1 and C2 conditions gave a similarly higher compressive strength at all curing times when compared to C3. This can be linked to the evolution of the water content inside the samples (Fig. 7).

For the reference formulation L0, the water content before compaction was the proctor optimum of 12.1 % (Table 3). For the C3 exposure condition the water content at 28 days, remained stable at approximately 12.1 % (Fig. 7); the samples were stored in sealed bags so water evaporation was limited. Still, a slight decrease in water content after 90 and 180 days was observed. This decrease induced a slight increase in the compressive strength (Fig. 6). Whereas, for the C1 and the C2 conditions, the water contents decreased significantly to approximately ~ 4–5 % after 28 days (Fig. 7), as a consequence of the exposure to 20 °C and 70 % RH. Then the water content stabilized at a value around 3.2 % slightly after, suggesting an equilibrium time somewhere between 28 and 90 days.

For the treated samples (L1, L2 and L4) at all curing times, the UCS for the C2 condition was always higher than for samples exposed to C1 condition (Fig. 6). In addition, for the C1 condition, UCS remained essentially constant for all treated samples from 90 days on.

In parallel, for the C1 and the C2 conditions, a decrease in the water content from initial proctor optimum (Table 3) to almost the same value of water content (around 6 % after 28 days and around 4 % after 90 days) regardless of the lime content (Fig. 7).

Concerning the C3 condition for treated soils, the UCS increased significantly with increasing lime addition (Fig. 6). The strength reached a higher value than the C1 condition for the L2 treatment at 180 days and even exceeded the strength of the C2 condition for the L4 treatment. In parallel the water content did not change considerably between 28 and 180 days (Fig. 7).

Phenolphthalein test and carbonation depth

The carbonation depth of samples was determined after 28, 90 and 180 days of exposure by spraying the phenolphthalein solution on the sample from the top to the bottom. Fig. 8 shows the pictures of the samples after spraying the phenolphthalein solution, at 28 days and for the different exposure conditions and the different lime additions. The carbonation depth was defined as the distance measured from the top down to the carbonation front where the phenolphthalein changed colour.

As expected, the samples for the untreated reference (L0) didn't produce a colour change to violet, indicating that they always presented a pH below 9, regardless of the exposure conditions (Fig. 8).

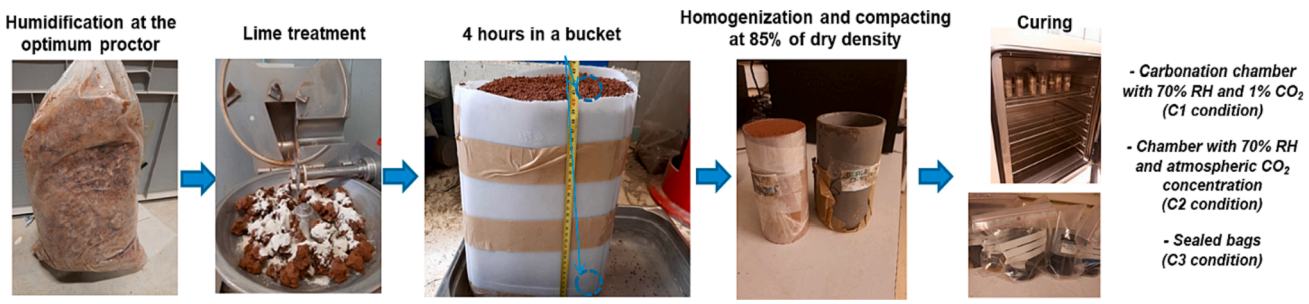


Fig. 4. The sample preparation process and curing conditions.

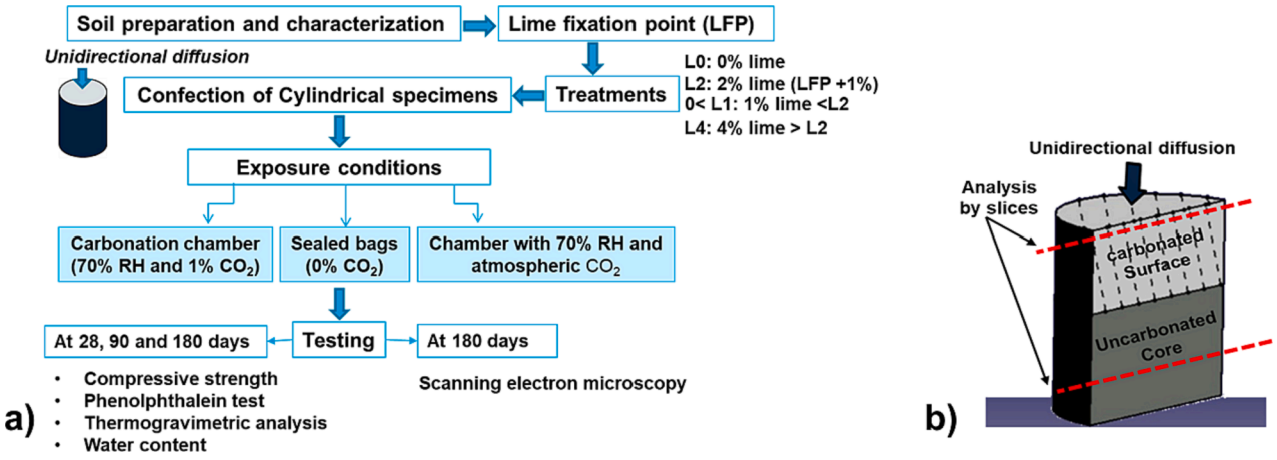


Fig. 5. A) graphical experimental procedure; b) graphical representation of a vertical cross-section of a cylindrical soil sample.

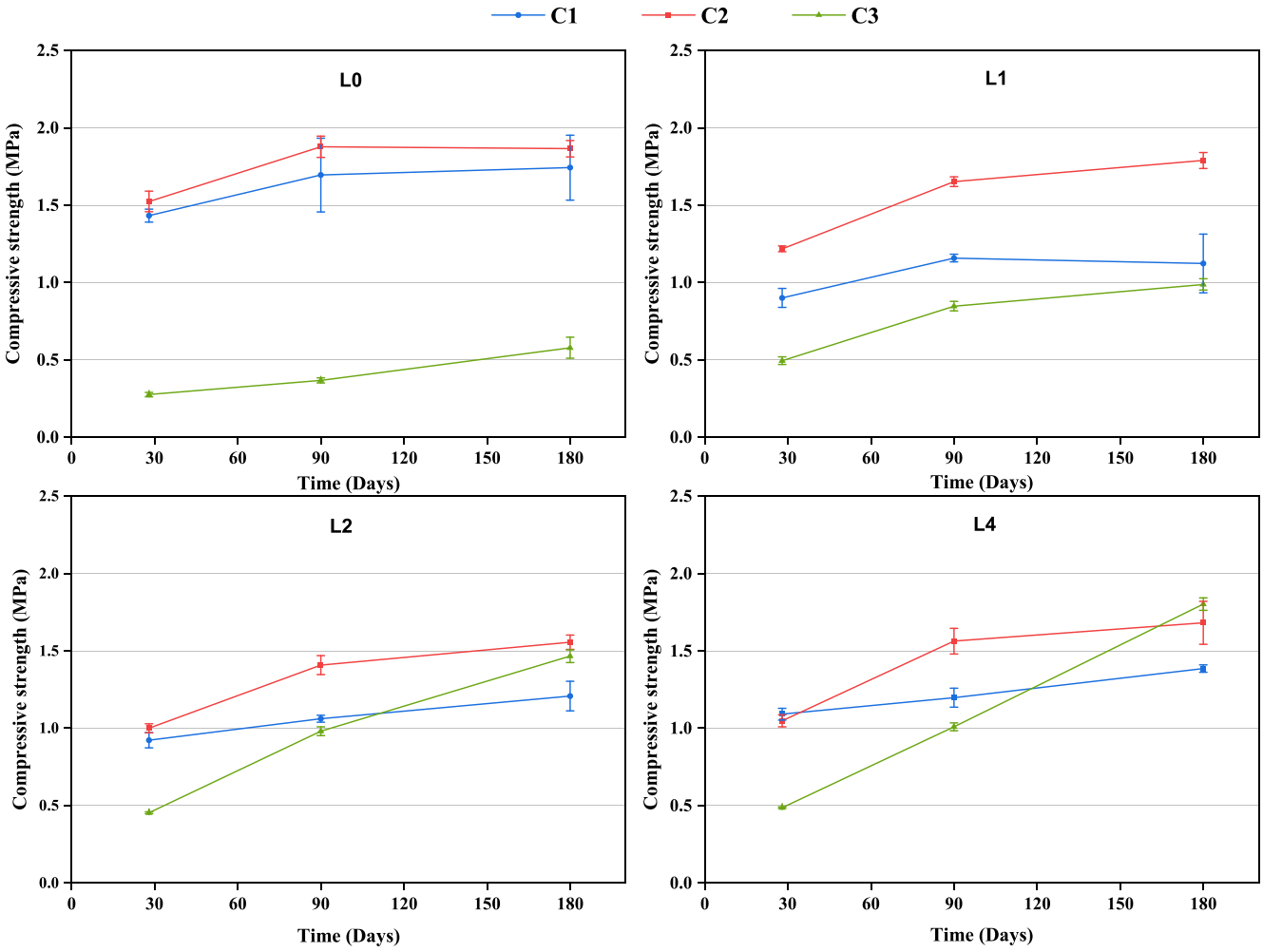


Fig. 6. Evolution of the unconfined compressive strength (UCS) for each lime treatment (L0, L1, L2 and L4) as a function of time for the three exposure conditions (C1, C2 and C3).

After 28 days, the samples treated with 1 % of lime showed carbonation depths of 7.9 cm after exposure to C1 condition (sample L1C1), ~0.4 cm when exposed to C2 condition (sample L1C2) and negligible for the C3 condition (sample L1C3). Similar results could be observed for the samples with 2 and 4 % of lime addition in the three different exposure conditions, except that the carbonation depth decreased with increasing lime addition (Fig. 8).

Fig. 9 shows the evolution of the carbonation depth over time for the different mix designs and exposure conditions. For the treated samples under C1 exposure condition (blue curves), and according to the phenolphthalein test, the carbonation was complete after 90 days for all lime contents (Fig. 9). For the C2 condition the carbonation increased

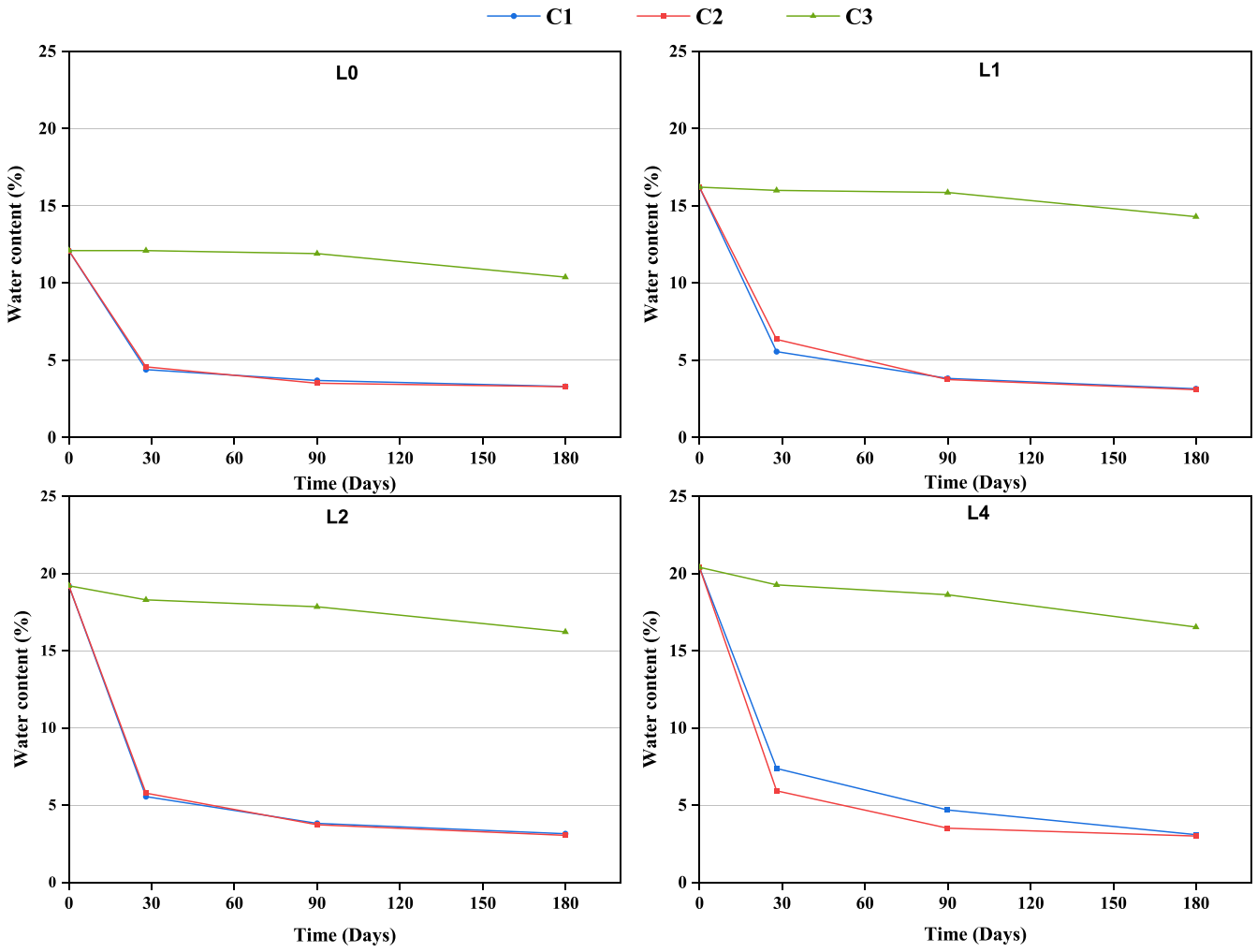


Fig. 7. Water content as a function of time for the different exposure conditions (C1, C2 and C3) for each lime treatment (L0, L1, L2 and L4).

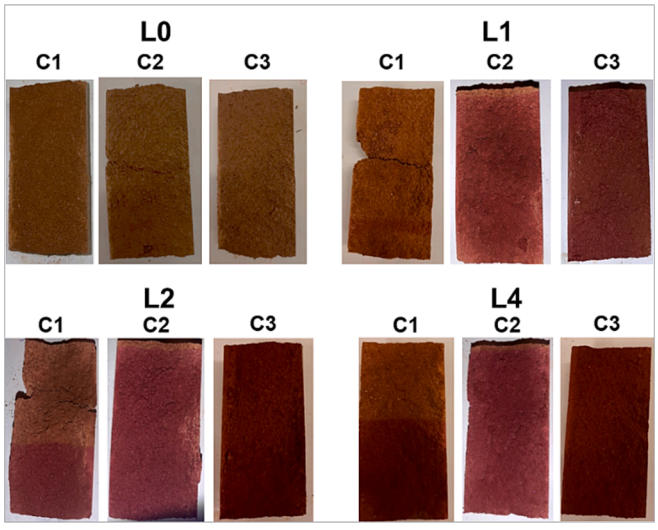


Fig. 8. Phenolphthalein test after 28 days for each lime treatment (L0, L1, L2 and L4) and exposure conditions (C1, C2 and C3).

slowly between 28 and 180 days. For the C3 exposure condition, almost no carbonation was visible after 90 days (except maybe for L1 treatment). After 180 days, significant carbonation was always observed but remained smaller than in the C2 condition. In addition, as mentioned before, the carbonation depth decreased with increasing lime content (Fig. 9).

Thermogravimetric analysis

The methodology followed for the TGA analysis consisted in taking specimens from the top and the bottom slices of the samples for each treatment and each exposure condition (Fig. 5). Using the phenolphthalein test, the carbonated zone was distinguished from the non-carbonated zone and sampling for TGA analysis could be done accordingly. For each treated sample, two TGA specimens were thus collected, one from the top and the second from the bottom of the sample.

TGA analysis of the samples measured the mass loss of the specimens. The mass loss between 750 and 800 °C was again attributed to carbon dioxide release as a consequence of the decarbonation of calcium carbonates. From this, the mass fraction of the total calcium carbonates in the soil at curing time t $x_{CaCO3}(t)$ can be calculated using equation (4):

$$x_{CaCO_3}(t) = \frac{\Delta m}{m(t)} \cdot \frac{M_{CaCO_3}}{M_{CO_2}} \quad (4)$$

Where m(t) is the treated-soil dry mass at curing time t. All other symbols are defined as before (equation (3)

The total carbonates now come from two sources: the native ones with the already determined mass fraction $x_{CaCO3,0}$ (equation (3), and the neo-formed carbonates coming from the carbonation reaction of the

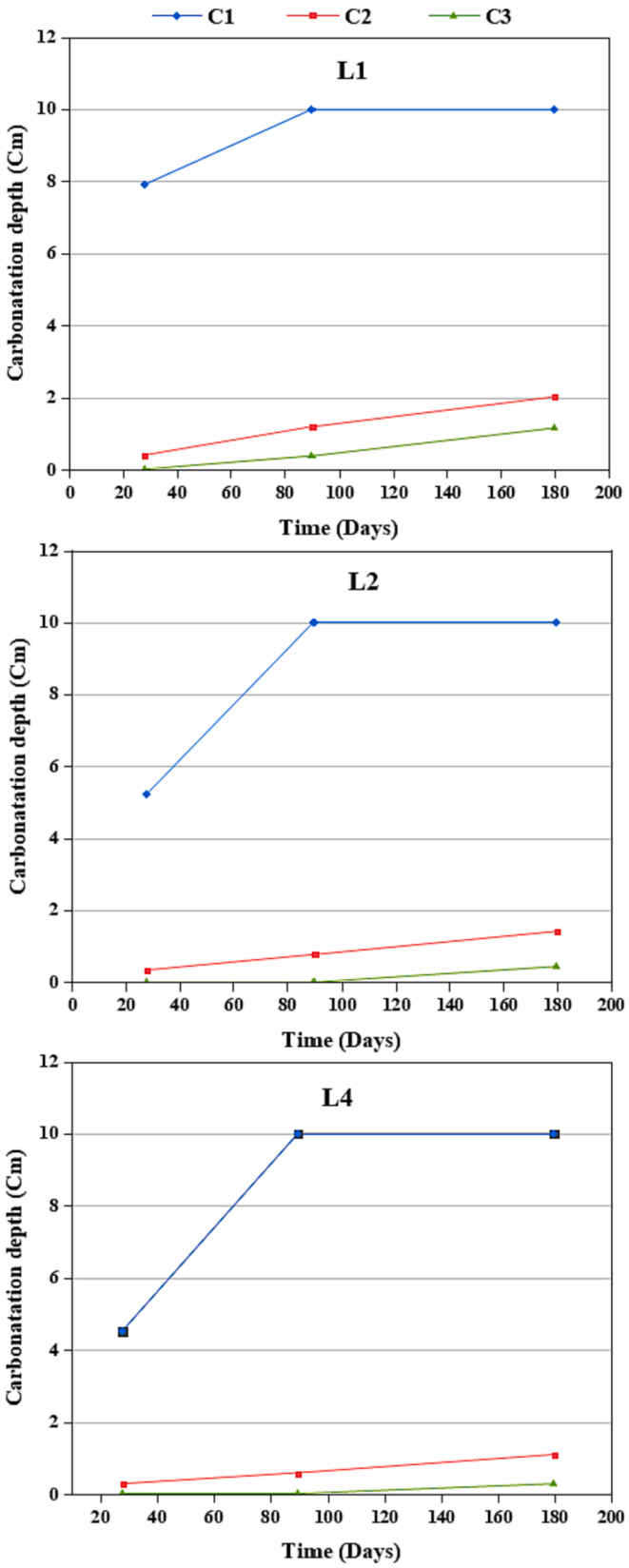


Fig. 9. Carbonation depth for all samples for each lime treatment (L1, L2 and L4) and exposure conditions (C1, C2 and C3) as a function of time.

added lime under carbon dioxide exposure (equation 2). The mass fraction of neo-formed carbonates in the treated soil at time t, $x_{CaCO3,nf}(t)$ can be calculated as follows:

$$x_{CaCO_3,nf}(t) = x_{CaCO_3}(t) - f.x_{CaCO_3,0} \quad (5)$$

where f = 1/(1 + q) is a factor taking into account the fact that the soil now consists of the initial soil plus a fraction q of quicklime (based on soil mass). f is therefore 0.99, 0.98 and 0.96 respectively for 1, 2 and 4 % lime addition.

The mass fraction k(t) of the initial quicklime (also later called "extent of carbonation of lime"), that is now present in the form of calcium carbonate at curing time t can then be calculated as:

$$k(t) = \frac{x_{CaCO_3,nf}(t)}{q}.\frac{M_{CaO}}{M_{CaCO_3}} \quad (6)$$

Where M_{CaO} is the molar mass (56 g/mol) of quicklime.

Using equation (6), it was thus possible to express the TGA results in terms of extent of carbonation of the added lime.

Extent of carbonation of lime versus carbonation according to the phenolphthalein test

As just described, TGA specimens were collected from the top and the bottom slices of treated samples (Fig. 5) and according to the phenolphthalein test the zones were identified as carbonated or non-carbonated. In the rest of the article they will be called respectively PPT-carbonated and PPT-non-carbonated. TGA made it possible to study whether the lime was really fully carbonated in the samples collected from the PPT-carbonated zones.

Fig. 10 presents the extent of carbonation of the lime as a function of time, when considering only samples taken from PPT-carbonated specimens, regardless of lime dosage, conditioning and curing time. Clearly, the TGA results show that a very limited number of specimens reached 100 % carbonation of lime even when the phenolphthalein test suggested full carbonation. This situation was essentially observed for the top of the L1 and L2 samples under the C1 and C2 conditions (Fig. 10). The extent of carbonation of lime varied indeed from 50 to 100 % (Fig. 10). Samples treated with 4 % of lime addition had the lowest percentage of carbonated lime compared to the two other treatments (1 and 2 % of lime addition). The average extent of carbonation and corresponding standard deviation were calculated for each treatment regardless of the exposure condition (Fig. 11). The results show that for the L1 samples, regardless of exposure condition, an average of 95 % of the lime was carbonated in the PPT-carbonated zones. Whereas, for 4 % lime addition, even if PPT-carbonated, only 75 % of lime was truly carbonated. The extent of carbonated lime decreases linearly with the lime dosage used for the soil treatment (Fig. 11).

Extent of carbonation of lime for the no carbon dioxide exposures (C3)

The C3 condition (sealed bags) represents the less favourable condition for carbonation and none of the treated samples showed signs of carbonation in their bottom zone according to the phenolphthalein test. The previous section showed that even if samples were PPT-carbonated, not all of the added lime was always fully carbonated. In a symmetrical way, this part now investigates the carbonation extent in the bottom of samples exposed to the C3 condition. Fig. 12 shows that for the 3 treatments exposed to the C3 condition, a significant part of the added lime was indeed carbonated at all testing times (28, 90 and 180 days). The extent of carbonation varied from 30 to 66 %. The L4C3 bottom samples recorded the lowest percentage of carbonated lime at all testing times, compared to the two other samples (L1C3 bottom and L2C3 bottom – Fig. 12). Therefore, although the samples were PPT-non-carbonated, and amid the very limited carbon dioxide presence in the C3 conditions, a part of the lime used for the soil treatment could carbonate. The average extent of carbonation of lime in the bottom of the specimens was calculated for the three different treatments exposed to

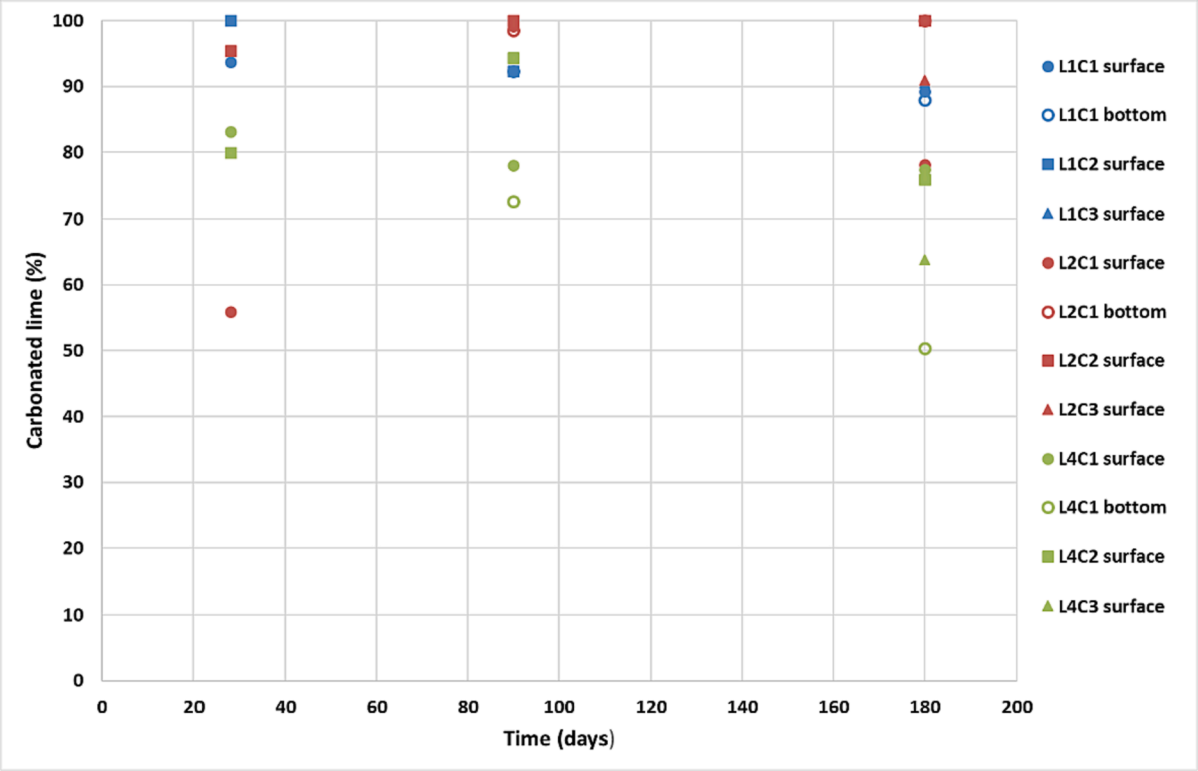


Fig. 10. Extent of carbonation of lime as a function of time for PPT-carbonated specimens (i.e. carbonated according to phenolphthalein test).

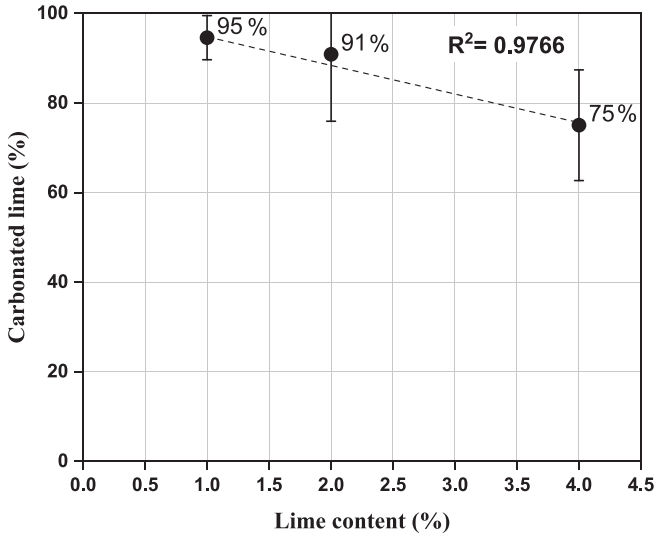


Fig. 11. Average extent of carbonation of lime for specimens from the PPT-carbonated zones for each lime treatment (1, 2 and 4%) and corresponding standard deviation.

the C3 condition (Fig. 13). The results show that the extent of carbonation decreased with increasing lime content. Those averages calculated in a condition where almost no carbon dioxide was present, represent the minimum carbonation that the soil could experience. Given the low exposure to carbon dioxide during the C3 curing conditions, the carbonation could only proceed from the initial preparation, most likely during the 4-hours mellowing period before compaction. This point will be further discussed in section 4.3.

Microscopic observations

The treated soils were observed after 180 days for the three different exposure conditions (C1, C2 and C3) by scanning electron microscopy (SEM) to investigate the effect of the exposure condition on the microstructure and on the formation of calcium carbonate. The study was conducted on untreated samples (L0) and the treated one with 2 % lime addition (L2C1, L2C2 and L2C3) using a SEM type Hitachi S-4300SE/N. The sample for SEM analysis was taken from the middle part of the cylindrical specimen.

Fig. 14 shows the SEM images for the L0 (Fig. 14a, c) and L2 (Fig. 14b, d) under C1 condition with the most important carbonation rate; the presence of large calcium carbonate crystals was found in the L2C1 samples, when none were found in sample L0C1. The shape was well defined suggesting that they could easily grow at the particle surface. Small interconnecting carbonate crystals couldn't be found, unlike former observations on lime mortars [35] or treated soils[15]. This probably explains why no binding effect was found according to UCS results (section 3.1). Fig. 15 shows the SEM images for the L2 samples exposed to the three different exposure conditions (C1, C2 and C3); for the L2C2 and L2C3 very seldom new carbonate crystals could be found in the SEM images. They always seem to develop on top of existing soil particles.

Discussions

Evolution of the mechanical properties

The data presented in section 3.1 and illustrated in Fig. 6, show that the strength of the materials significantly evolved during the different conditionings. Still, the origin of this evolution must be carefully studied to try to relate compositional changes to final properties.

As a first contributor, changes in moisture content have a strong impact on the mechanical properties.

The untreated soil presents a high strength of ~ 1.5 MPa after 28

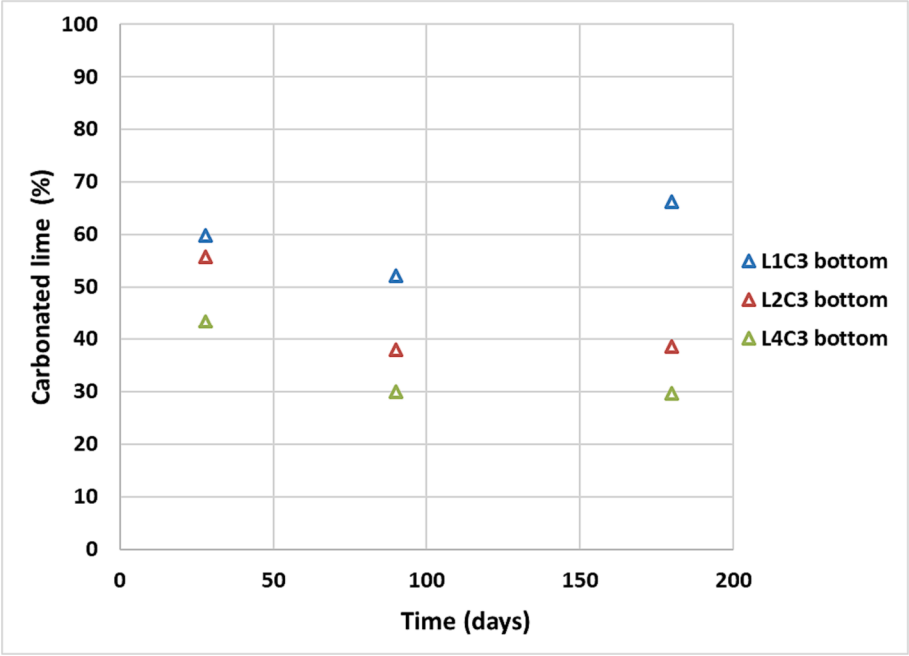


Fig. 12. Extent of carbonation of lime in the bottom of samples exposed to C3 condition as a function of time.

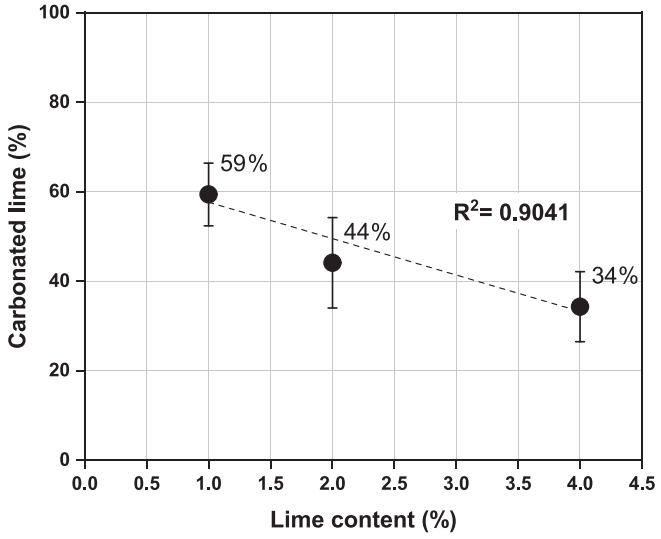


Fig. 13. Average extent of carbonation of lime in the bottom of the specimens exposed to C3 condition for the three different lime treatment, and corresponding standard deviation.

days of exposure to the C1 and C2 conditions (Fig. 6) compared to that obtained with C3 condition (0.3 MPa). As already noticed, this is consistent with the fact that the samples dried from an initial 12.1 % moisture to ~ 4–5 % moisture after 28 days in the C1 and C2 conditions (Fig. 7). The drying intensity and kinetics is consistent with compacted silts exposed to 70 %RH [3637]. As a consequence, the level of saturation in the soil decreased which in turn increased the water suction. It is then well documented that the compressive strength significantly increases with the suction [38394041]. Thus, the changes in moisture content, as a consequence of the 70 % RH, induce a five-fold increase in unconfined compressive strength and are the main contributor of the strength increased in the case of the untreated specimens under the C1 and C2 conditions. It has also to be noted that the final equilibrium moisture content under these conditions (70 % RH / 20 °C) is close to 3.2 % moisture and was reached somewhere between 28 and 90 days (Fig. 7).

Second, pozzolanic reactions are believed to be the main contributor of the strength increase observed for lime-treated specimens exposed to the C3 conditions [124243]. Here, the progressive increase from initially 0.5 MPa up to 1, 1.5 and 1.8 MPa after 180 days with respectively 1, 2 and 4 % lime addition is very similar to data reported for soils having a similar plasticity [19424344].

Then, it is interesting to compare the strength data after 28 days for untreated and lime-treated materials. The data in Fig. 6 clearly show that drying (in the untreated soils under the C1 and C2 conditions) has a stronger impact on compressive strength than early pozzolanic set (treated soils under C3 conditions). It is interesting to note that, for the tested material, it takes 180 days of the C3 curing and 4 % lime addition for the strength gain due to pozzolanic reactions to surpass the strength gain due to drying of untreated specimens (Fig. 6).

Also, a close look at results under the C1 conditions for the lime-treated specimens give very interesting findings (Fig. 6). As a matter of fact, the compressive strength with the C1 conditions and all lime contents, experienced a very similar behaviour as the one found for the untreated samples under the C1 and C2 conditions: As a result, the compressive strength after 180 days is not systematically higher than that after 90 days, and only marginally higher than that after 28 days. This indicates a stabilization of the strength gain somewhere between 28 and 90 days (Fig. 6). In parallel, for example, the moisture content for L4 specimens experienced a sharp decrease from 20.4 % (Table 3) down to 6 % in 28 days and then stabilizes at a value close to 4 % moisture content (Fig. 7). This strongly suggests that compressive strength evolution was essentially governed by the moisture changes, just like the untreated soil. Since significant early carbonation was observed under these conditions for all of the lime additions (Figs. 8-9), we conclude that the neo-formed calcium carbonates had almost no binding effect and didn't participate in the strength gain. This is further evidenced by SEM observations that showed that neo-formed crystals were essentially observed on the surface of mineral particles and not at the edge of them, with no visible links created between neighbouring particles (Fig. 15).

Finally, all treated materials under the C2 conditions kept gaining strength after 180 days, with a UCS systematically higher than that after 90 days (Fig. 6). This is to be compared with C1 conditions where almost

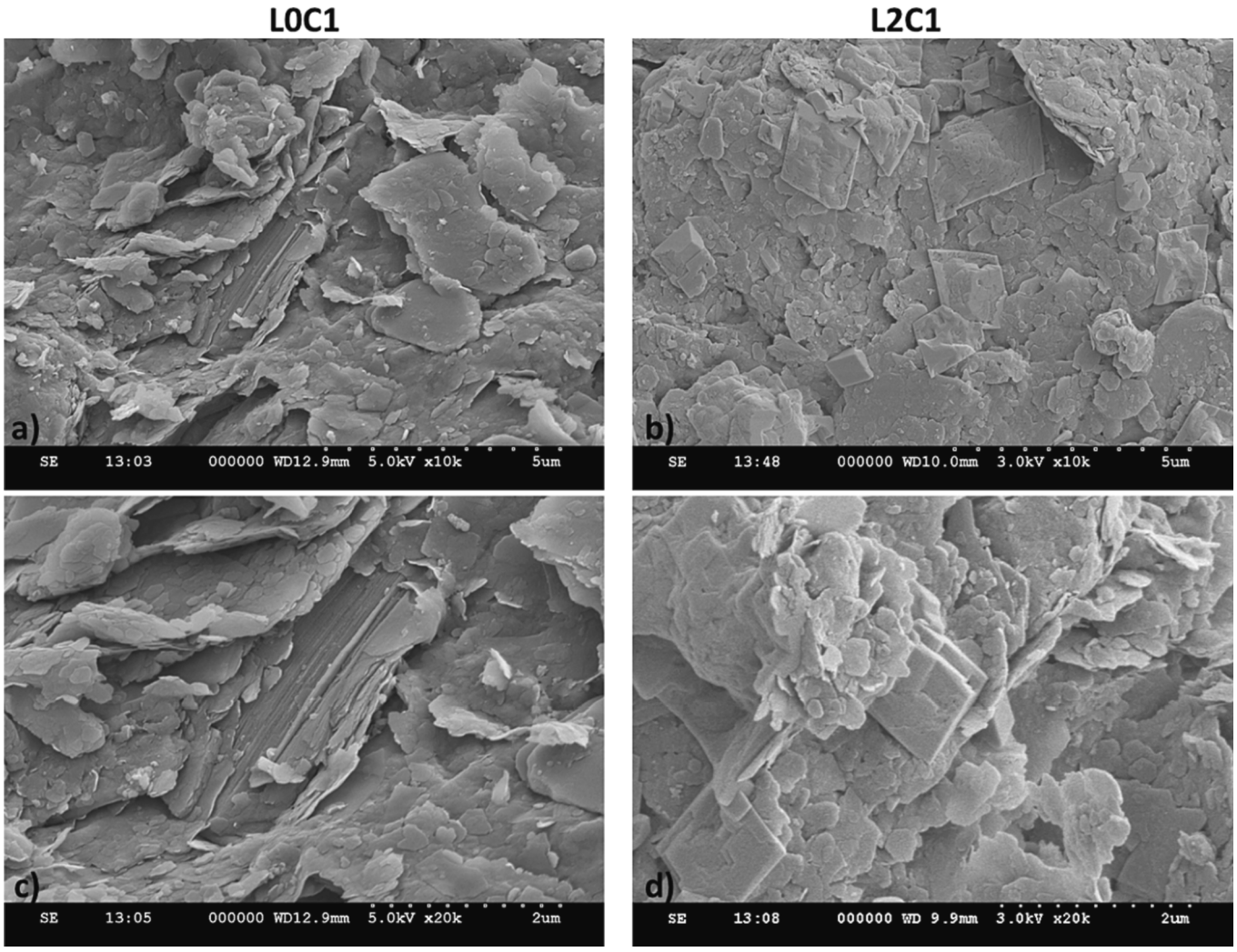


Fig. 14. SEM images 180 days for non-treated sample (L0) (a, c) and treated samples with 2 % lime (L2) (b, d) exposed to C1 condition.

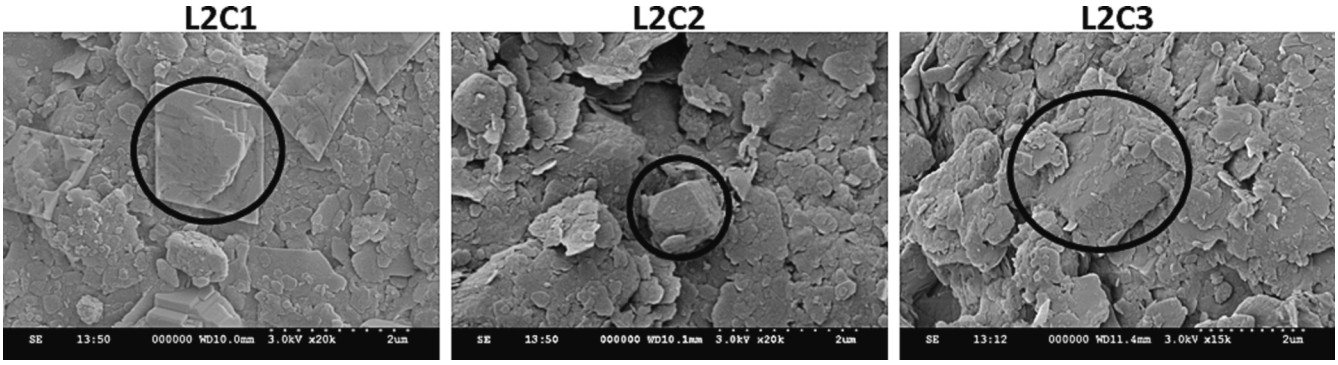


Fig. 15. SEM images of samples at 180 days after treatment with 2 % lime and exposure to the three different conditions C1, C2 and C3.

a plateau was instead reached at long curing times. Clearly, this means that drying alone was not responsible for the strength gain and it is therefore very likely that the unreacted lime could induce slow pozzolanic reactions as seen for the C3 conditions. However, the sharp decrease in moisture content probably limits the precipitation points for the newly formed hydrates to small pores with a limited effect on overall material strength.

Thus, the evolution in compressive strength vs time, condition and lime-content gives very valuable information on the mechanisms behind the observed strength gains:

- The untreated samples could gain significant strength due to drying when cured at 70 % RH (C1 and C2 conditions), reaching 1.5 MPa after only 28 days,
- The lime-treated specimens showed the expected slow strength gain due to the pozzolanic reactions when cured in sealed bags (C3 conditions), reaching 1.8 MPa after 180 days for the highest lime addition (4 %),

- The highly carbonated lime-treated specimens (C1 conditions) only gained strength as a consequence of drying and no binding effect from the neo-formed carbonates was observed,
- The lime-treated specimens under the C2 conditions (atmospheric carbon dioxide and 70 % RH) showed a mixed behaviour, with part of the strength gain coming from drying combined with a slow development of pozzolanic reactions consuming the remaining available lime

Carbonation kinetics

The data presented in section 3.2 and illustrated in Figs. 8 and 9, show that, according to the phenolphthalein test, the carbonation proceeds very rapidly for the C1 conditions (1 % carbon dioxide). After 28 days, the carbonation front had already reached 45, 52 and 79 mm for, respectively, 4, 2 and 1 % lime addition. Clearly, the lower the lime content, the higher the carbonation rate. This corresponded to carbonation speeds of 1.6, 1.9 and 2.8 mm/day. At this carbonation speed, all lime-treated 100-mm high specimens were fully PPT-carbonated after 90 days (Fig. 8).

Under the C2 conditions (atmospheric carbon dioxide), carbonation occurred much more slowly: the front increased linearly until reaching 11, 14 and 20 mm after 180 days for respectively 4, 2 and 1 % lime additions (Fig. 9). This corresponded to carbonation speeds of respectively 0.061, 0.078 and 0.11 mm/day.

Carbonation occurred even slower under the C3 conditions and was barely measurable after 90 days and reached respectively 3, 4 and 11 mm for respectively 4, 2 and 1 % lime addition after 180 days (Fig. 8), corresponding to carbonation speeds of respectively 0.017, 0.022 and 0.061 mm/day.

Note that the extent of carbonation as measured by phenolphthalein was indeed incomplete and on average, only 75, 91, 95 % of the lime was really in the form of calcium carbonate for respectively 4, 2 and 1 % lime addition samples (Fig. 11).

Only few studies on the kinetics of carbonation of lime-treated soil are available; making comparison with our data difficult. Still, Deneele et al. (2021) [15] observed values of 0.35 and 0.70 mm/days for the carbonation of a silt (PI 5–11) treated with respectively 5 and 2 % quicklime addition. Compacted specimens were stored outside for up to 28 days near Paris (France) during the spring with temperatures ranging from 12 to 18 °C and an average humidity of 65 % RH [15]. This was 4 to 7 times faster than what was observed in the present study considering the more similar exposure condition (i.e. the C2 conditions). Nevertheless, it was confirmed that the higher the lime content, the lower the carbonation rate.

Détry et al. (2005) [45] reported on a 30-years-old lime-treated experimental embankment built at the Belgian Road Research Center (BRRC) near Brussels. The silt (PI ~ 8) was treated with a very high amount of quicklime addition (7 %). Due to a compactor failure during the jobsite, the target compaction was not obtained and the lime-treated silt only achieved 80 % of Proctor optimum [45]. Consequently, a small damaged zone ("crust") on top of the embankment was observed; the crust measured 10 mm after the first year, 20 mm after 5 years and 100 mm after 30 years. The damage mostly consisted in loss of cohesion and allowed for the development of vegetal growth. Since plants growth is often restricted in high pH environments, their presence was attributed to the combination of carbonation and freeze–thaw damages (decohesion initially appeared after the first winter). Assuming that the crust corresponded to a carbonated zone, the carbonation speed was therefore of the order of 0.027 mm/day after 1 year, 0.0068 mm/day for years 2 to 5 and 0.0088 mm/day for years 5 to 30.

A recent study on a 7-years old experimental embankment in Rouen (France) [46] showed that a silt (PI 5–11) treated with 2.5 % quicklime addition had a similar carbonated crust, as identified by a lower pH than in the core of the structure, measuring at least 12 cm. This would correspond to an average carbonation speed of the order of 0.047 mm/day. The same embankment was further investigated on 7-years old lime-treated zones that were initially in the core of the embankment (and therefore not directly exposed to atmospheric carbon dioxide until then), one year after being put in direct contact with the surrounding air [16]. Based on pH and TGA, they observed that a carbonated layer of 40 mm had formed, corresponding to a carbonation speed of 0.11 mm/day.

The data from the studies above mentioned are compared in Fig. 16. Even if the tested soils, lime contents and curing conditions are different, this limited data set can represent an overview of the carbonation speeds of silts exposed to atmospheric carbon dioxide. This suggests that the carbonation kinetics slows down with increasing measurement times and that carbonation speed can vary from 0.006 to 0.7 mm/days.

Thus, assuming a linear kinetics for the carbonation, as done herein based on the data in Fig. 9, is most likely a simplification due to the fact that the set of data presented in this study is limited to only 6 months at most. It makes it difficult to consider more complex kinetics models. For example, it would be interesting to test whether the 2-steps modelling proposed by Van Balen and Van Gemert (1994) [47] for lime mortars, applies to lime-treated soils. This might not be the case given that the lime contents are much lower for soils as compared to mortars and the competition with pozzolanic reactions was not considered by Van Balen and Van Gemert [47] and is generally not considered in the carbonation of mortars [4849].

The carbonation of concrete might be closer to the case at stakes, and was found to follow a power law vs time with exponent between 0 and −0.2 [32]. The data in Fig. 16 suggests that a power law of exponent −1 might be more realistic for lime-treated silts. Clearly more data are needed to verify that the kinetics of carbonation in lime treated soil proceeds in the same way as in mortars [47] or concrete [32].

Initial carbonation

Additional tests were performed to calculate the initial (early) carbonation of the soil treated before compaction and during air exposure in the container of a height of 40 cm. The previously described TGA method (sections 2.3 and 3.3) could not be applied; it needs such a small amount of matter (a few mg) that it was impossible to take specimens that would be visually homogeneous in terms of lime content, given that lime nodules are clearly visible at this early sample preparation stage. So, a different methodology was applied on the uncompacted soil using larger amounts of material to reduce the variability induced by the distribution of lime grains in the treated soil. The samples were taken from the top of the container and from the bottom after 1 and 4 h from the treatment. Then, the weight losses at 550 and 950 °C were measured. The initial carbonation was still calculated according to equations (4) to (6), except that the mass loss Δm in equation (4) was instead determined by the loss on ignition at 950 °C (LOI_{950}) instead of TGA analysis.

The procedure was applied for the three uncompacted soils (L1, L2 and L4) after mixing with the three lime additions and after 1 h and 4 h mellowing period. The results showed that the variability induced by the sampling method does not make it possible to measure reliably the rate of carbonated lime for the L1 and L2 samples, this is why we will just present the results of the L4 samples. 7 samples were collected at each testing time (at 1 h and 4 h mellowing) and an average value was calculated. The results in Table 4 and Fig. 17 show that after 1 h the extent of carbonation of the lime in the soil was around 18.7 % and it increased to 35.4 % after 4 h mellowing period. This result compares very well with the results in section 3.3.2 where the L4 samples presented an extent of carbonation of lime for the C3 condition of 34 % (Fig. 13).

Conclusion

This work presented a study of the carbonation of a silt treated with quicklime contents of 1, 2 and 4 % and cured under different carbon dioxide exposures for up to 180 days.

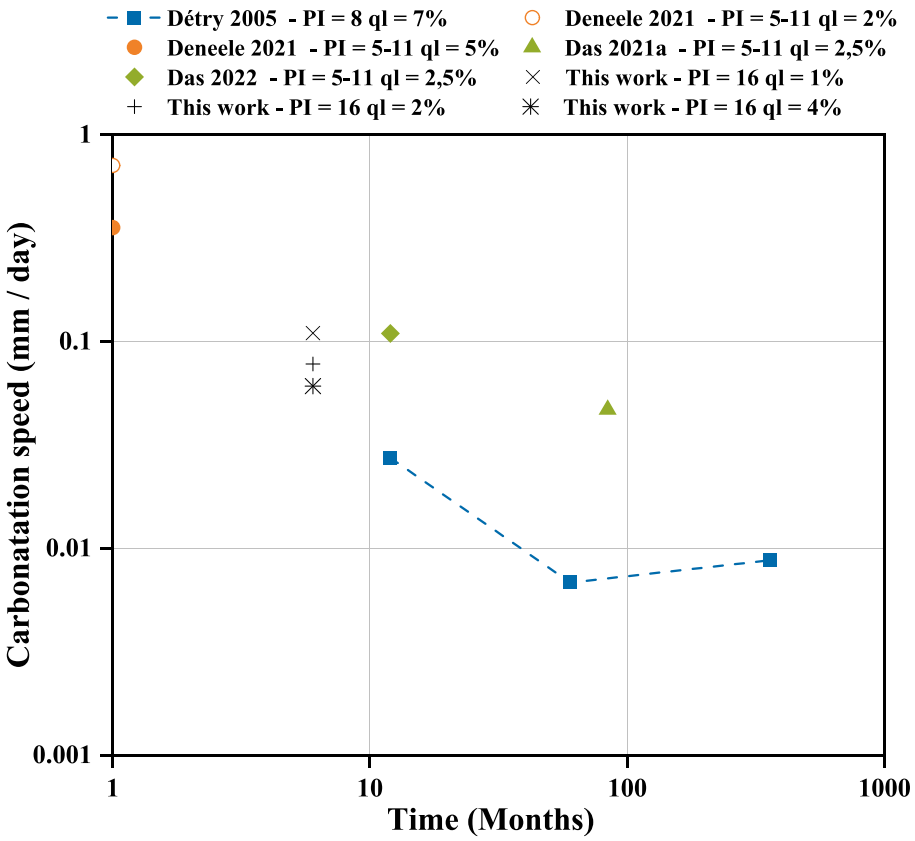


Fig. 16. Carbonation speed of different lime-treated silts as a function of time: comparison between literature data and the data presented in this study. PI stands for the plasticity index of the tested soil and ql for the quicklime content used in the study. Carbonation conditions were: Détry et al. 2005 [45] studied an experimental site near Brussels (Belgium), Deneele et al. 2021 [15] monitored cylindrical lab compacted specimens stored outside in the spring near Paris (France), Das et al. 2021a [46]and 2022 [16] followed an experimental embankment built in Rouen (France).

Table 4
Data used to estimate the carbonation extent of lime k(t) during the initial mellowing period for the soil treated with 4% lime addition (L4) before compaction.

	$\Delta m/m(t)$ (based on LOI_{950})	$x_{CaCO3}(t)$	$x_{CaCO3,nf}(t)$	$k(t)$
	%	%	%	%
Natural soil (L0)	10.14	23.1	–	–
L4 1 h	10.33	23.5	1.3	18.7
L4 4 h	10.85	24.7	2.5	35.4

When the samples were stored in sealed bags (C3 conditions with no carbon dioxide), the evolution of their mechanical properties followed the expected progressive increase attributed to pozzolanic reactions leading to compressive strength higher than 1 MPa after 180 days for all the lime contents.

When stored in a high carbon dioxide environment (C1 conditions with 1 % carbon dioxide), carbonation occurred rapidly and the samples were fully carbonated after 90 days according to the phenolphthalein test, regardless of the added lime content. The higher the lime content, the slower the carbonation speed.

When stored under atmospheric carbon dioxide conditions (C2 conditions), a similar trend as for C1 regarding the effect of the lime content on carbonation was found and the carbonation front progressed linearly at 0.055, 0.083 and 0.11 mm/days for respectively 4, 2 and 1 % of lime addition. These values are consistent with published data for similar soils, lime-treatments, and environment that cover a wide range of carbonation speeds from 0.006 to 0.7 mm/days. Even if linear kinetics for the propagation of the carbonation front were observed in this work based on a limited data set between 28 and 180 days, comparison with published data suggest that the kinetics decreases with time and might be indeed inversely proportional to time when longer periods are considered.

Clearly, more work is needed in order to confirm the kinetics of lime carbonation, especially at long times. Also, the influence of soil parameters (plasticity, porosity, moisture….) on the carbonation kinetics and overall carbonation process (including carbonation extent) must also be assessed by repeating this type of study on different soils and treatment conditions. On a more fundamental standpoint, it would also be very interesting to understand how it compares with the carbonation of mortars or concrete.

The carbonated zones in the samples according to the phenolphthalein test, generally contained significant amounts of unreacted lime. On average the 1, 2 and 4 % lime addition specimens showed carbonation extents of respectively 95, 91 and 75 % in these PPT-carbonated zones. This is consistent with the data already published by Deneele et al (2021)[15].

In parallel, the uncarbonated zones (according to the phenolphthalein test) could still hold a significant amount of carbonated lime, found to be on average 59, 44 and 34 % for respectively 1, 2 and 4 % lime addition.

As a result, the samples having experienced the lowest exposure to carbon dioxide, corresponding to the bottom parts of the C3 conditioning (sealed bags) at all times and some of the top portions at early times, always showed an average conversion rate of lime into calcium carbonate of 59, 44 and 34 % for respectively 1, 2 and 4 % lime addition. Interestingly, these values are close to the ones documented in the literature regarding the conversion of old lime-treated embankments; Haas and Ritter (2019) [11] reported a value of 37 % after 34 years for a silt treated with 2 % quicklime and Akula et al. (2020)[13], a value of

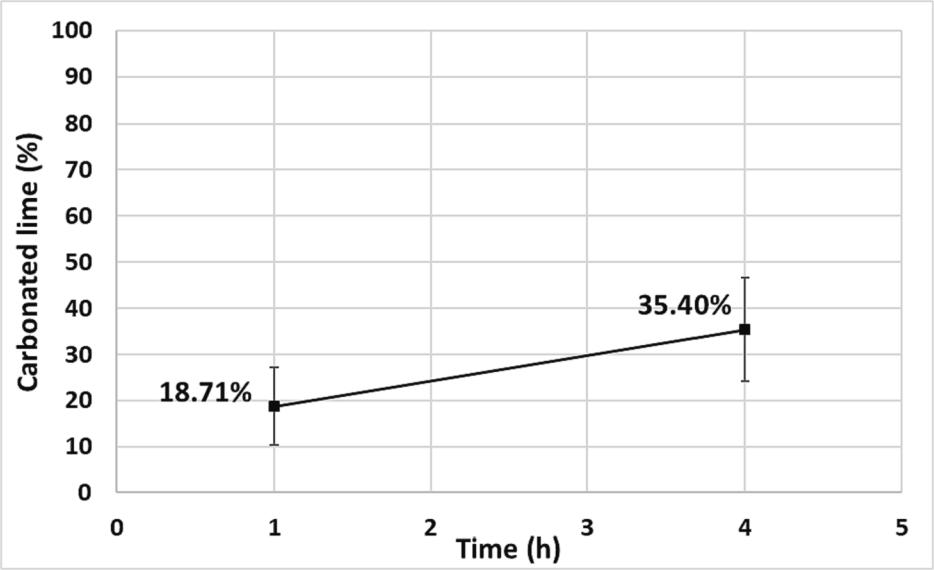


Fig. 17. Extent of carbonation of the lime for the 4% treated soil after 1 and 4 h mellowing.

34 % after more than 40 years for a clay treated with 3–4 % quicklime.

The results of this study strongly suggest that most of the carbonation extent occurs essentially during the early mixing phase of the soil with lime, where the heat, the high moisture content, and the mixing all favour the carbonation of lime. Therefore, it seems reasonable to assume that most jobsites currently using lime-treatment in Europe, usually made with 1–2 % high purity quicklime probably reach 30–40 % of lime carbonation when the time between mixing and compaction is significantly higher than 1 h. More investigations are needed to confirm this hypothesis.

The methodology proposed in this article was applied for the first time to a fine soil containing significant amounts of native calcium carbonate (~20 %). It was still possible to make reasonable measurements of neo-formed calcium carbonates even with the studied quicklime contents of 1, 2 and 4 %. Uncertainty was lower with the highest lime dosage, but reliable data could still be acquired at the lowest lime content for compacted specimens. This was not true anymore when working with uncompacted specimens and only the data with 4 % lime were reliable. Therefore, the variability of native calcium carbonate is key in order to replicate the methodology to fine soils containing significant amounts of native calcium carbonate. Data on soils containing other amounts of native calcium carbonates will be needed before concluding on the capability of the method to be used for any soil.

CRediT authorship contribution statement

J. Kleib: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Conceptualization. **D. Lesueur:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **W. Maherzi:** Writing – review & editing, Validation, Methodology, Investigation, Conceptualization. **M. Benzerzour:** Writing – review & editing, Validation, Resources, Project administration, Methodology, Investigation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgment

This work was financially supported by the European Lime Association (EuLA). The EuLA Civil Engineering Task Force is thanked for its technical and scientific support.

References

[1] Little DN. Stabilization of pavement subgrades and base courses with lime. Dubuque, Iowa: Kendall/Hunt; 1995.
[2] Belgian Road Research Center. Soil treatment with lime – European experiences for soil improvement and soil stabilization - State of the art. Brussels: 2021.
[3] National Lime Association. Lime-treated Soil Construction Manual: Lime Stabilization and Lime Modification. The Association; 2004.
[4] Little DN, Nair S. Recommended Practice for Stabilization of Subgrade Soils and Base Materials. Washington, DC: 2009. 10.17226/22999.
[5] AFNOR NF EN 16907-4. Earthworks - Part 4: Soil treatment with lime and/or hydraulic binders, CEN, 2018.
[6] Guide for Soil Treatment (French "Guide Traitement des Sols" - GTS) : Setra (2008) "Treatment of soils with lime and/or hydraulic binders - Application to the construction of pavement base layers", Service d'Etudes Technique des Routes et Autoroutes (SETR. n.d.
[7] Nguyen TTH, Cui YJ, Ferber V, Herrier G, Ozturk T, Plier F, et al. Effect of freeze-thaw cycles on mechanical strength of lime-treated fine-grained soils. Transp Geotech 2019;21:100281. https://doi.org/10.1016/j.trgeo.2019.100281.
[8] ASTM D6276-99a. Standard Test Method For Using PH To Estimate The Soil-Lime Proportion Requirement For Soil Stabilization, 2006.
[9] Bell FG. Lime stabilization of clay minerals and soils. Eng Geol 1996;42:223–37. https://doi.org/10.1016/0013-7952(96)00028-2.
[10] Ciancio D, Beckett CTS, Carraro JAH. Optimum lime content identification for lime-stabilised rammed earth. Constr Build Mater 2014;53:59–65. https://doi.org/10.1016/j.conbuildmat.2013.11.077.
[11] Haas S, Ritter HJ. Soil improvement with quicklime–long-time behaviour and carbonation. Road Mater Pavement Des 2019;20:1941–51. https://doi.org/10.1080/14680629.2018.1474793.
[12] AFNOR. NF EN 459-2, Chaux de Construction — Partie 2 : Méthodes d'essais., 2021.
[13] Akula P, Hariharan N, Little DN, Lesueur D, Herrier G. Evaluating the long-term durability of lime treatment in hydraulic structures: Case study on the friant-kern canal. Transp Res Rec 2020;2674:431–43. https://doi.org/10.1177/0361198120919404.
[14] Herrier G, Berger R, Bonelli S. The Friant-Kern canal: a forgotten example of lime-treated structure in hydraulic conditions. 6th Int Conf scour Eros 2012:p. 8-p.
[15] Deneele D, Dony A, Colin J, Herrier G, Lesueur D. The carbonation of a lime-treated soil: experimental approach. Mater Struct 2021;54:1–12. https://doi.org/10.1617/s11527-021-01617-w.

[16] Das G, Razakamanantsoa A, Saussaye L, Losma F, Deneele D. Carbonation investigation on atmospherically exposed lime-treated silty soil. Case Stud Constr Mater 2022;17:e01222.
[17] Vitale E, Deneele D, Russo G. Effects of carbonation on chemo-mechanical behaviour of lime-treated soils. Bull Eng Geol Environ 2021;80:2687–700. https://doi.org/10.1007/s10064-020-02042-z.
[18] Xu L, Zha F, Liu C, Kang B, Liu J, Yu C. Experimental Investigation on Carbonation Behavior in Lime-Stabilized Expansive Soil 2020;2020:22–4.
[19] Fan W, Chen W, Zhang Q, Wu G. Effects of sticky rice on the carbonation reaction of lime-treated soil in earthen sites. Constr Build Mater 2023;378.
[20] Denayer C, Shtiza A, Schlegel T, Haas S. Geotechnical know-how that supports decision making towards sustainable infrastructure. XVII Eur. Conf Soil Mech. Geotehc Eng. Geotech. Eng. Found. Futur., 2019. 10.32075/17ECSMGE-2019-0898.
[21] Pietro CF, Tua C, Biganzoli L, Pantini S, Grosso M. Natural and enhanced carbonation of lime in its different applications: a review. Environ. Technol Rev 2021;10:224–37. https://doi.org/10.1080/21622515.2021.1982023.
[22] AFNOR. NF P94-068, Sols : reconnaissance et essais - Mesure de la capacité d'adsorption de bleu de méthylène d'un sol ou d'un matériau rocheux - Détermination de la valeur de bleu de méthylène d'un sol ou d'un matériau rocheux par l'essai à la tache., 1998.
[23] AFNOR. NF EN ISO 17892-12, Reconnaissance et essais géotechniques - Essais de laboratoire sur les sols - Partie 12 : détermination des limites de liquidité et de plasticité, 2018.
[24] AFNOR. NF EN 16907-2, Terrassement - Partie 2 : Classification des matériaux, 2018.
[25] LCPC-SETRA. Guide des Terrassements Routiers, Réalisation des remblais et des couches de forme (GTR), Fascicules I et II. 1992.
[26] AFNOR. NF EN 459-1, Chaux de construction - Partie 1 : définitions, spécifications et critères de conformité, 2015.
[27] AFNOR NF P94-093. Sols : reconnaissance et essais - Détermination des références de compactage d'un matériau - Essai Proctor Normal - Essai Proctor modifié, 2014.
[28] Moretti L, Natali S, Tiberi A, D'Andrea A. Proposal for a methodology based on xrd and sem-eds to monitor effects of lime-treatment on clayey soils. Appl Sci 2020:10. https://doi.org/10.3390/app10072569.
[29] Trung ND, Sakaguchi A, Hayano K, Yamauchi H. Effects of several factors in accelerated carbonation on neutralization and carbon dioxide capture of alkaline sludges treated with paper sludge ash-based stabilizer. Case Stud Constr Mater 2023;19:e02215.
[30] Yue Y, Wang JJ, Basheer PAM, Bai Y. Establishing the carbonation profile with raman spectroscopy: Effects of fly ash and ground granulated blast furnace slag. Materials (Basel) 2021:14. https://doi.org/10.3390/ma14071798.
[31] Araújo MT De, Ferrazzo T, Jordi G, Bruschi J, Consoli NC. Transportation Geotechnics Mechanical and Environmental Performance of Eggshell Lime for Expansive Soils Improvement 2021;31. 10.1016/j.trgeo.2021.100681.
[32] Von Greve-Dierfeld, S. Lothenbach B, Vollpracht A, Wu B, Huet B, Andrade C. . ., De Belie N. Understanding the carbonation of concrete with supplementary cementitious materials: a critical review by RILEM TC 281-CCC. Mater Struct 2020;53:136.
[33] Bui H, Delattre F, Levacher D. Experimental methods to evaluate the carbonation degree in concrete—state of the art review. Appl Sci 2023;13. https://doi.org/10.3390/app13042533.
[34] Chang CF, Chen JW. The experimental investigation of concrete carbonation depth. Cem Concr Res 2006;36:1760–7. https://doi.org/10.1016/j.cemconres.2004.07.025.
[35] Cizer O, Van Balen K, Elsen J, Gemert D Van. Crystal Morphology of Precipitated Calcite Crystals From Accelerated Carbonation of Lime Binders. 2nd Int Conf Accel Carbonation Environ Mater Eng Rome, Italy,1-3 Oct 2008 2008:1–3.
[36] Mpawenayo R, Debaste F, Douzane M, Gerard P. On the mechanisms and kinetics of evaporation of a compacted silt. E3S Web Conf., vol. 9, 2016, p. 12001. 10.1051/e3sconf/20160912001.
[37] Azil A, Touati K, Sebaibi N, Le Guern M, Streiff F, Goodhew S, et al. Monitoring of drying kinetics evolution and hygrothermal properties of new earth-based materials using climatic chamber simulation. Case Stud Constr Mater 2023;18:e01798.
[38] Kato S, Kawai K, Yoshimura Y, Sunden W. Effect of suction on unconfined compressive strength and undrained shear strength of a compacted silty clay. 3rd Int. Conf Unsaturated Soils 2002:513–9.
[39] Pineda JA, Colmenares JE. Stress-strain-suction behaviour of two clayey materials under unconfined conditions. Fourth Int Conf Unsaturated Soils 2006:1109–20.
[40] Bardanis M, Grifiza S. Soil-water characteristic curves and evolution of the tensile and unconfined compression strength of drying slurried soils. Unsaturated Soils Res. Appl.. Berlin Heidelberg: Springer; 2012. p. 249–56.
[41] Chitimbo T, Prime N, Plé O, Abdulsamad F. Drying experiment on rammed earth structure. Eur J Environ Civ Eng 2022:1–17.
[42] Al-Mukhtar M, Khattab S, Alcover JF. Microstructure and geotechnical properties of lime-treated expansive clayey soil. Eng Geol 2012;139–140:17–27. https://doi.org/10.1016/j.enggeo.2012.04.004.
[43] Rosone M, Celauro C, Ferrari A. Microstructure and shear strength evolution of a lime-treated clay for use in road construction. Int J Pavement Eng 2020;21:1147–58.
[44] Das G, Razakamanantsoa A, Herrier G, Deneele D. Compressive strength and microstructure evolution of lime-treated silty soil subjected to kneading action. Transp Geotech n.d.;29:100568. 10.1016/j.trgeo.2021.100568.
[45] Détry J, Van den Bergh H, Bollens Q. Study of a 30 years old lime treated area. 3rd Int Symp Treat Recycl Mater Transp Infrastruct 2005.
[46] Das G, Razakamanantsoa A, Herrier G, Saussaye L, Lesueur D, Deneele D. Evaluation of the long-term effect of lime treatment on a silty soil embankment after seven years of atmospheric exposure: Mechanical, physicochemical, and microstructural studies. Eng Geol n.d.;281:105986. 10.1016/j.enggeo.2020.105986.
[47] Van Balen K, Van Gemert D. Modelling lime mortar carbonation. Mater Struct 1994;27:393–8.
[48] Galan I, Glasser FP, Baza D, Andrade C. Assessment of the protective effect of carbonation on portlandite crystals. Cem Concr Res 2015;74:68–77. https://doi.org/10.1016/j.cemconres.2015.04.001.
[49] Van Balen K. Carbonation reaction of lime, kinetics at ambient temperature. Cem Concr Res 2005;35:647–57. https://doi.org/10.1016/j.cemconres.2004.06.020.