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# Effects of carbonation on chemo-mechanical behaviour of lime-treated soils

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## Abstract

In the paper, a multi-scale experimental investigation on the occurrence of carbonation and its effect on chemo-mechanical behaviour of lime-treated soil has been presented. Carbonation affects chemo-mineralogical evolution of lime-treated soils depending on the time scale at which reaction mechanism takes place. In the short term, a progressive carbonation of portlandite is responsible for the formation of calcium carbonate with consumption of available lime for pozzolanic reactions (*lime carbonation*). In the long term, carbonation of the secondary phases resulting from pozzolanic reactions weakens the bonding effects induced by hydrated compounds (*carbonation of secondary reaction products*). Mineralogical and microstructural features of lime-treated and carbonated samples have been monitored at increasing curing times by means of microstructural analyses. Triaxial drained compression tests have been performed on treated samples cured in different conditions for short and long term. Mineralogical investigations showed precipitation of calcium carbonate for lime-treated samples exposed to atmospheric CO<sub>2</sub> since the very short term. Exposure of lime-treated sample to CO<sub>2</sub> after precipitation of hydrated phases favoured decalcification of cementitious compounds and formation of calcium carbonate. In both cases, precipitation of calcite relevantly affects the mechanical behaviour of lime-treated samples. From the observed behaviours, it will be possible to take into account relevant factors for performing the best practice finalized to efficient and durable soil treatment.

**Keywords** Lime-treated soil · Carbonation · Chemo-physical evolution · Microstructure · Mechanical behaviour

## Introduction

Minimizing costs in construction of civil infrastructures and saving natural resources favour the use of stabilising agents (such as lime, cement or alkali-activated binders) to improve engineering properties of soils not suitable as construction materials for earthworks. Lime treatment is a soil improvement technique widely used for geotechnical engineering applications (Sherwood 1993; Metcalf 1977; Little 1995; Russo 2016). Addition of lime has a strong impact on physical and mechanical properties of soils in the short and long term after

treatment (Glenn and Handy 1963; Brandl 1981; Bell 1996; Rogers and Glendinning 1996; Locat et al. 1996; Sivapullaiah et al. 2000; Tremblay et al. 2001; Rao and Shivananda 2005; Vitale et al. 2020). Several studies on the basic lime-clay-reaction mechanisms have been widely reported in literature (Eades and Grim 1960; Diamond and Kinter 1965; Boardman et al. 2001; Maubec et al. 2017; Pomakhina et al. 2012; Dewindt et al. 2014; Chemedda et al. 2015), allowing a better understanding of the chemo-physical evolution of the system and the interpretation of the mechanical improvement induced after the addition of lime (Russo et al. 2007; Metelková et al. 2012; Russo and Modoni 2013; Russo et al. 2015; Russo 2019; Vitale et al. 2016a; Vitale et al. 2016b; Vitale et al. 2016c; Vitale et al. 2016d; Vitale et al. 2017; Guidobaldi et al. 2017; Guidobaldi et al. 2018; Cecconi et al. 2020).

When lime is added to the soil-water system, either in the form of quicklime (CaO(s)) or as hydrated lime (Ca(OH)<sub>2</sub>), a change in the pore water chemistry occurs. Dissociation reaction of lime leads to an increase of pH and to a high concentration of calcium ions in the pore water. The increase of pH

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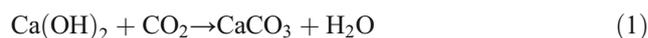
alters the distribution of charge on clay platelets, whereas the calcium ions released from lime replace the exchangeable cations from soil, leading to a reduction of thickness of double-layer surrounding clay particles (Rogers and Glendinning 1996). This process, referred to as cation exchange reaction, induces flocculation and agglomeration of clay particles, with changes in plasticity, workability and particle size distribution of treated soil. The high pH environment induced by lime enables dissolution of the alumino-silicate constituents of clay minerals promoting development of pozzolanic reactions. Silica and alumina released from clay minerals react with lime leading to precipitation of calcium silicate hydrate (C-S-H), calcium aluminate hydrate (C-A-H) or calcium aluminate silicate hydrate (C-A-S-H), cementitious compounds bonding soil particles together (Ingles and Metcalf 1972; Little 1995; Bell 1996). Pozzolanic reactions are time-dependent, whose kinetic is ruled by clay mineralogy, amount of unconsumed portlandite ( $\text{Ca}(\text{OH})_2$ ) in the system, curing time and temperature (Rao and Shivananda 2005; Rojas and Cabrera 2002; Al-Mukhtar et al. 2010; Vitale et al. 2019).

Durability of physical and mechanical improvement induced by lime is referred to as the maintenance of designed performance above target during service life of construction. A wide range of environmental factors may affect durability of treated soils. Several studies have been conducted on detrimental impact of freezing/thawing and wetting/drying cycles on durability (Ismeik and Shaqour 2020; Zhang et al. 2019). Thompson and Dempsey (1969) showed that freezing and thawing cycles induced significant volume changes on lime-treated clayey soils accompanied by reduction of shear strength. Many authors observed the increase of swelling behaviour (Guney et al. 2007; Cuisinier and Deneele 2008; Stoltz et al. 2014) or soil stiffness reduction (Tang et al. 2011) after cycles of wetting and drying on different lime-treated soils. The detrimental impact of leaching on durability of lime-treated soils has been evidenced by Mc Callister and Petry (1992), De Bel et al. (2005), Le Runigo et al. (2009), Rosone et al. (2020), Chakraborty and Nair (2018), Chakraborty and Nair (2020) and Moghal et al. (2020).

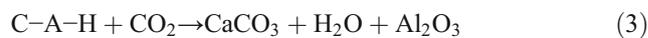
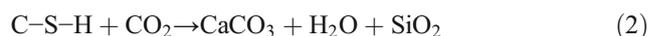
Less attention has been paid in literature to the occurrence of carbonation and its effect on durability of lime-treated soils. Netterberg and Paige-Green (1984) focused on carbonation of lime and cement stabilised layers in road pavements. Carbonation occurring before sealing causes lime transformation into limestone and makes lime no longer available to form cementing compounds. Paige-Green et al. (1990) provided recommendations on prevention and rehabilitation of carbonation-induced damages on road pavements. Bagonza et al. (1987) investigated the occurrence of carbonation as consequence of different curing conditions on cement and lime-treated soils (i.e. humidity, temperature and environmental carbon dioxide). Samples cured in a high carbon dioxide environment showed a fast carbonation process and a limited

increase of strength due to low pozzolanic activity. Deneele et al. (2013) discussed carbonation effects on lime-treated silt mechanical properties. Experimental investigations showed the rapid occurrence of carbonation for  $\text{CO}_2$ -exposed samples and a related increase of unconfined compressive strength (UCS) due to carbonation of portlandite, whereas a slower but higher increase of UCS for samples stored in controlled environment was observed over curing time due to pozzolanic reactions without occurrence of carbonation.

The effects of carbonation on chemo-mechanical behaviour of lime-treated soil depend on time scale at which reaction mechanism takes place. These aspects have not been specifically considered in previous studies on carbonation of treated soils. *Lime carbonation* is a short-term chemical reaction, which involves atmospheric  $\text{CO}_2$  (carbon dioxide) and lime available in the system after the treatment. It is accompanied by a decrease of pore water pH and can be synthesized as follows (Moorehead 1986):



The precipitation of  $\text{CaCO}_3$  (calcite) occurs due to combination of  $\text{CO}_2$  in water and calcium ions  $\text{Ca}^{2+}$ , due to dissolution of  $\text{Ca}(\text{OH})_2$  (portlandite). In the long term, if hydrated phases formed due to pozzolanic reactions are exposed to  $\text{CO}_2$ , carbonation of hydrated compounds takes place as follows (Richardson 1998; Morandeau et al. 2014):



The acidic environment induced by dissolution of  $\text{CO}_2$  in water promotes the progressive decalcification of hydrated phases (Alexander et al. 2013), converted into  $\text{CaCO}_3$  by reaction with  $\text{CO}_2$ , and release of amorphous silica and alumina gel.

Physical states (i.e. void index, initial water content), as well as microstructural features of treated soil, play an important role in the kinetic of carbonation process affecting rate of  $\text{CO}_2$  diffusion, reactant ( $\text{CO}_2$ ,  $\text{Ca}(\text{OH})_2$ ) dissolution and concentration, and therefore the formation rate of  $\text{CaCO}_3$  (Van Balen and Van Gemert 1994; Fernández-Bertos et al. 2004; Van Balen 2005).

In the present study, a multi-scale analysis on the occurrence of carbonation and its effects on chemo-mechanical behaviour of lime-treated soils has been performed. Influence of *lime carbonation* and *carbonation of secondary reaction products* has been investigated by comparing different environmental curing conditions for treated samples (namely *indoor*, *outdoor* and *indoor + outdoor curing* as defined in the following), adding novel information about the impact of carbonation on mechanical performances of lime-treated soils. Chemical, mineralogical and microstructural changes induced

by lime addition have been monitored at increasing curing time by means of phenolphthalein indicator tests, X-ray diffraction (XRD), thermo-gravimetric analysis (TGA), mercury intrusion porosimetry (MIP) and scanning electron microscopy (SEM). Mechanical behaviour of lime-treated and carbonated samples has been analysed by means of triaxial drained compression tests on saturated lime-treated samples. Experimental results allow a deeper insight into the impact of carbonation processes on effectiveness and durability of lime treatment.

## Materials and experimental methods

### Materials

A highly refined kaolin (Speswhite kaolinite, Imerys Minerals) from deposits in the South West of England has been used. The specific gravity is  $G_s = 2.6$ , and the surface area determined by nitrogen adsorption (BET) is  $14 \text{ m}^2/\text{g}$ . According to ASTM D 4972 2001, the pH value of the clay is about 4.6. The liquid and plastic limits are 70% and 32% respectively, with a plastic index IP of 38%. The sample is mainly formed by kaolinite clay minerals with a small amount of quartz ( $\text{SiO}_2$ ) and muscovite ( $\text{KAl}_3\text{Si}_3\text{O}_{10}(\text{OH})_2$ ). No swelling clay phases were detected after the ethylene glycol treatment. The element composition of kaolin has been reported in Table 1.

Quicklime used for treatment of the studied soil contained more than 90% of free calcium oxide, 1%  $\text{Ca}(\text{OH})_2$  and 4% calcite.

### Sample preparation

Lime-treated samples were prepared by hand mixing dry soil with 3% of CaO, corresponding to the minimum amount of quicklime required for stabilisation as determined by means of pH measurements (according to ASTM D 4972 2001). Dry mixes were humidified with distilled water, allowing the quicklime to hydrate for 24 h. Untreated and treated samples for mechanical testing were then compacted at optimum water content following the standard Proctor procedure (ASTM D

698-91e1 2000). Initial water content of untreated samples was  $w = 30\%$ , dry unit weight  $\gamma_d = 14.02 \text{ kN/m}^3$ , void ratio  $e_0 = 0.81$  and degree of saturation  $S_r = 96\%$ , whereas initial water content of lime-treated samples was  $w = 33\%$ , dry unit weight  $\gamma_d = 12.9 \text{ kN/m}^3$ , void ratio  $e_0 = 0.96$  and degree of saturation  $S_r = 89\%$ . In Fig. 1, standard Proctor compaction curves of untreated and lime-treated samples are presented.

Three different curing conditions were considered for lime-treated samples, namely *indoor curing (IN)*, *outdoor curing (OUT)* and *indoor + outdoor curing (IN + OUT)*. *Indoor curing* refers to a storage without exposure of samples to carbon dioxide ( $\text{CO}_2$ ). Specimens were sealed in hermetic plastic bags and were cured for increasing time intervals, namely 24 h, 7, 28 and 90 days. Outdoor curing of treated samples was performed by exposing samples to air (i.e. standard concentration of  $\text{CO}_2$  in air) since the very short term (24 h after mixing) up to 90 days. During curing stages, temperature varied in the range of  $22 \pm 2 \text{ }^\circ\text{C}$  and relative humidity ranged between  $65 \pm 5\%$ . Finally, curing indoor for 90 days followed by a 30-day exposure to environmental  $\text{CO}_2$  has been referred to as indoor + outdoor curing (IN + OUT).

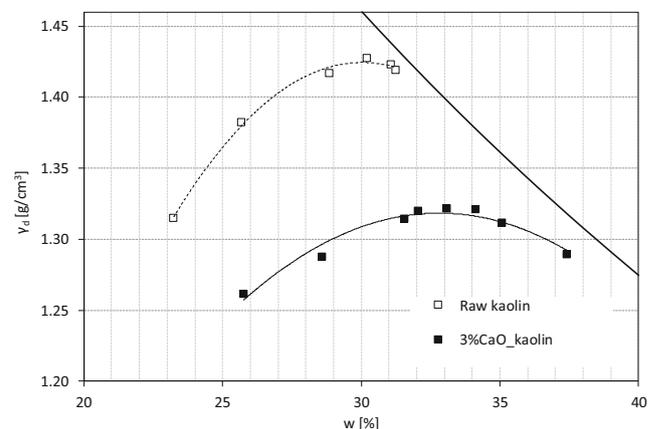
Samples for performing chemo-physical analyses (XRD and TGA) were selected by cutting slices of the entire compacted specimen along horizontal cross sections. This method allowed to consider the progressive development of carbonation and the consequent not homogeneity of the specimens. The samples were then dehydrated and grinded before performing the tests. Samples for microstructure analyses (MIP and SEM) were similarly retrieved from the compacted specimen carefully preserving the structure induced by the compaction stage.

### Chemical tests—phenolphthalein indicator

According to UNI:EN 13295 2005, time evolution of the carbonation process was monitored by spraying phenolphthalein on cut sections of treated samples cured at increasing curing

**Table 1** Elemental composition of Speswhite kaolin

Element	Percentage (%)
$\text{SiO}_2$	53.80
$\text{Al}_2\text{O}_3$	43.75
CaO	0.02
$\text{K}_2\text{O}$	1.45
$\text{Na}_2\text{O}$	0.93
$\text{TiO}_2$	0.05



**Fig. 1** Standard Proctor compaction curves of untreated and 3%CaO-treated kaolin

times (i.e. 24 h, 7, 12 and 28 days). The carbonation front was then determined by observing the turning of phenolphthalein pH indicator from pink-coloured zone ( $\text{pH} > 9$ ) to not-coloured zone ( $\text{pH} < 9$ ), being the latter carbonated.

## Microstructure analyses

### X-ray diffraction

Mineralogical composition of untreated soil and mineralogical composition of treated samples have been investigated by X-ray analyses, performed on randomly oriented powder using a step size of  $0.021^\circ$ . The mineralogical evolution over curing time of treated samples has been monitored at 24 h, 7, 28 and 90 days.

### Thermo-gravimetric analysis

Thermo-gravimetric analysis has been performed. Thermo-gravimetric analysis has been performed on finely ground soil heated at a rate of  $10^\circ\text{C min}^{-1}$ , under argon atmosphere, from ambient temperature to  $1000^\circ\text{C}$ , increasing curing times (namely 24 h, 7, 28 and 90 days) have been considered for treated samples.

### Scanning electron microscopy

Surface state modifications and fabric changes of soil particles due to lime treatment have been examined through scanning electron microscopy (SEM). Untreated and lime-treated samples have been dehydrated by freeze-drying technique (Delage and Pellerin 1984). A gold coating pre-treatment has been performed for SEM observations.

### Mercury intrusion porosimetry

MIP tests were performed by a double chamber Micromeritics Autopore III apparatus. In the filling apparatus (dilatometer), samples have been outgassed under vacuum and then filled by mercury allowing the increase of absolute pressure up to ambient. Using the same unit, the intrusion pressure has been raised up to approximately 200 kPa by means of compressed air. The detected entrance pore diameters range between 134 and  $7.3\ \mu\text{m}$  (approximately 0.01–0.2 MPa for a mercury contact angle of  $139^\circ$ ). After depressurisation to ambient pressure, samples have been transferred to high-pressure unit, where mercury pressure has been increased up to 205 MPa following a previously set intrusion program. Corrections to pore size distribution due to compressibility of intrusion system have been applied performing a blank test. MIP tests have been performed on both untreated and treated samples cured in three different environmental conditions at increasing time intervals (namely 28 and 90 days).

## Triaxial tests

Drained triaxial compression tests have been performed on saturated samples by means of a controlled stress-path triaxial cell (Aversa and Vinale 1995). Saturated samples have been isotropically compressed up to a mean effective stress of  $p' = 300\ \text{kPa}$ . Deviatoric stages in drained conditions have been then performed at deformation rate of  $0.5\%/h$  up to failure. Treated samples cured under different conditions (namely IN, OUT and IN + OUT) have been tested at increasing curing times (namely 24 h, 28 and 90 days).

## Results

### Phenolphthalein indicator tests

Carbonation of samples was evidenced by means of phenolphthalein indicator test. Sections of indoor cured treated samples at increasing time intervals (namely 24 h, 7, 28 days) are reported in Fig. 2a after spraying phenolphthalein pH indicator. Presence and permanence over time of a highly alkaline environment induced by lime are evidenced by pink surface of indoor cured samples. For outdoor cured samples, the progressive reduction of pink section over time highlights the evolution of carbonation front (Fig. 2b). Average carbonation radius ( $R_c$ ) normalised by radius of sample section ( $r$ ) is plotted as function of time of exposure to atmospheric  $\text{CO}_2$ , as shown in Fig. 3. Complete carbonated surface of treated sample has been observed after 21 days of curing.

### XRD

X-ray diffraction patterns of untreated and lime-treated sample at increasing curing time are shown in Fig. 4a. For treated samples, new reflections attributed to unconsumed portlandite are detected since the very short term (24 h of curing). Intensities of these peaks decrease with increasing curing time, until their disappearance after 28 days of curing. Precipitation of calcium aluminate hydrate (C-A-H) is detected by new peak at  $11^\circ$  2-theta after 28 days of curing. Time-dependent mineralogical changes induced by immediate exposure of lime-treated samples to atmospheric  $\text{CO}_2$  (outdoor curing) are shown in Fig. 4b. XRD patterns of outdoor cured samples evidence unconsumed portlandite in the system since the very short term, whose reflections progressively reduce over time until their disappearance after 7 days of curing. New peaks corresponding to calcite are also detected starting from 24 h of curing, whose intensity increases with curing time. In the long term, no new reflections attributed to hydrates are observed for outdoor cured samples.

Comparison between XRD patterns of treated samples cured for long time intervals (i.e.  $> 90$  days) in three different curing conditions is shown in Fig. 4c. The consequence of exposure of

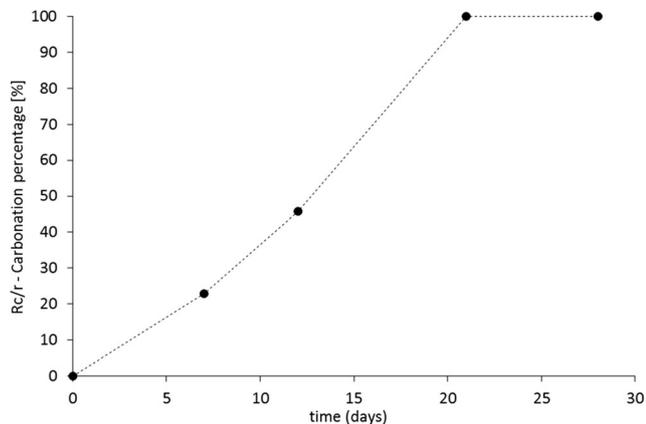
**Fig. 2** Phenolphthalein tests: evolution of carbonation front over time. **a** Indoor cured samples. **b** Outdoor cured samples



indoor cured samples to environment  $\text{CO}_2$  (i.e. IN + OUT samples) is the dissolution of hydrated phases, as evidenced by disappearance of their characteristic peaks and precipitation of calcite.

### TGA

The results of thermo-gravimetric analyses (TGA) on indoor and outdoor lime-treated samples are shown in Fig. 5. Quantitative interpretation of TGA is represented in Fig. 6. Mass losses of indoor cured samples as function of curing time are shown in Fig. 6a with reference to the range 390–460 °C, characteristic of portlandite, and 110–350 °C, characteristic of hydrates. Portlandite consumption is detected before precipitation of new mineralogical hydrated phases, whereas pozzolanic compounds form after long curing time (i.e. longer than 28 days). Outdoor cured samples show (Fig. 6b) a similar consumption of portlandite as observed for indoor cured samples, with immediate precipitation of calcite, as highlighted by the increase of mass loss in its characteristic range of temperature (650°–800 °C).



**Fig. 3** Evolution of carbonation front of the outdoor cured samples as function of exposure time

### MIP

Cumulative intruded void ratios and pore size distributions of untreated and indoor cured samples at increasing curing times are shown in Fig. 7. Increase of cumulative intruded void ratio is observed after lime addition at 28 days of curing time. Reduction of porosity of treated sample is evidenced in the long term. Pore size distributions are characterized by modal diameter pore size ranging between 0.1 and 0.3  $\mu\text{m}$ , with negligible evolution over time; in the smallest pore range (i.e. entrance pore diameter < 0.1  $\mu\text{m}$ ), an increase of frequency with curing time is observed.

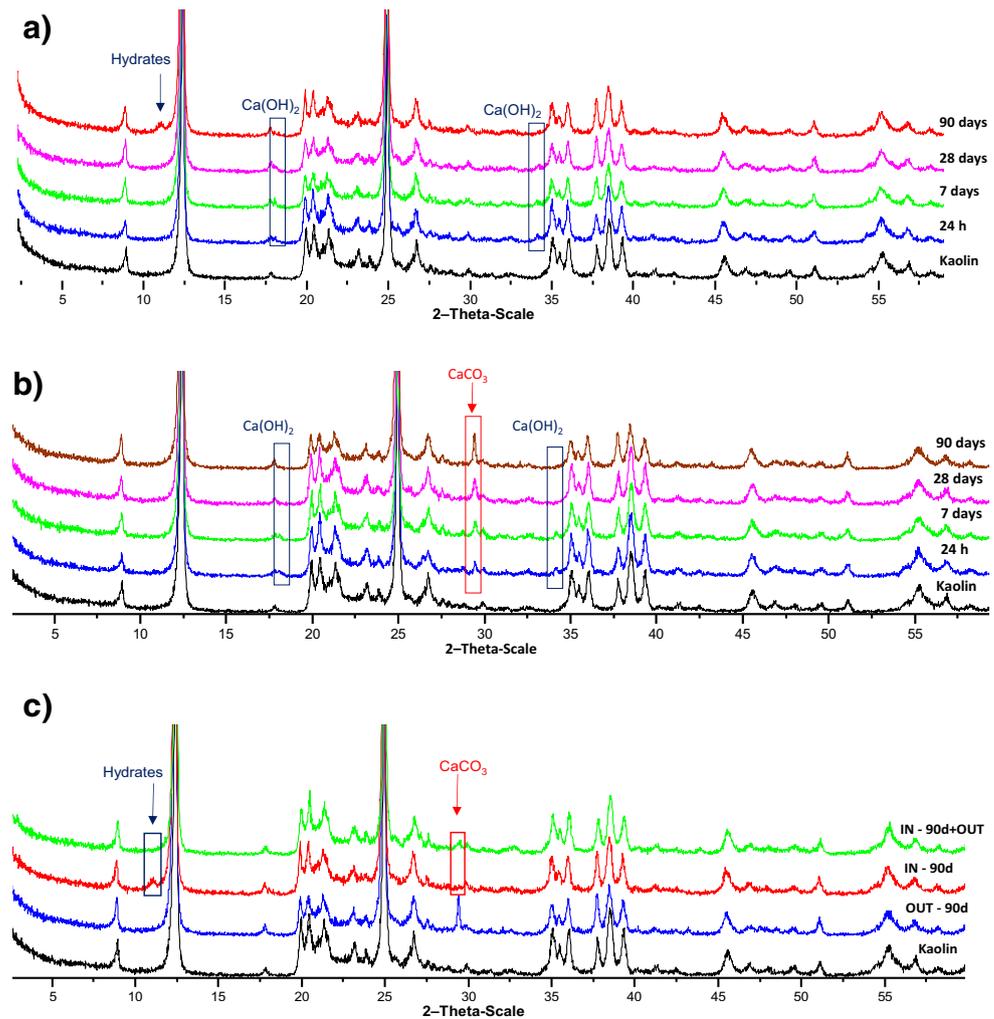
MIP results on lime-treated samples exposed to environment  $\text{CO}_2$  (outdoor curing) are shown in Fig. 8 as function of curing time. Cumulative intruded void ratio decreases as curing time increases, highlighting a gradual overall reduction of porosity over time (Fig. 8a). Pore size distributions show a reduction of frequency of pores between 0.3 and 1  $\mu\text{m}$ . Frequency of small pores (i.e. pore entrance diameter 0.04  $\mu\text{m}$ ) and the modal pore size are not affected by curing conditions (Fig. 8b).

Cumulative intruded void ratios of treated samples cured for long time intervals in three different curing conditions are reported in Fig. 9. Curing outdoor after the indoor stage induces a reduction of porosity of the sample, as highlighted by the cumulative intruded void ratio of the sample, as well as a frequency reduction of pores in the small diameter range, compared to indoor cured sample.

### SEM

SEM observations of untreated and lime-treated compacted samples cured for long time intervals (i.e. > 90 days) in three different curing conditions are reported in Fig. 10. SEM images of untreated kaolin sample clearly show particle

**Fig. 4** X-ray diffraction patterns as function of curing times. **a** Indoor cured samples. **b** Outdoor cured samples. **c** Comparison between different curing conditions in the long term (IN vs. OUT vs. IN + OUT curing)



morphology, as well as aggregated fabric induced by compaction (Fig. 10a-b). Figure 10 c and d show treated samples cured outdoor for more than 90 days. Calcite crystals of irregular morphology resulting from carbonation of portlandite in the system and forming a continuous chain-like structure (Cizer et al. 2006) are clearly identified. Figure 10 e and f show lime-treated sample after long-term indoor curing. Precipitation of hydrated phases covering the surface of clay particle aggregates is observed. As shown in Fig. 10g-h for samples cured IN + OUT, subsequent stage of exposure to environmental carbon dioxide triggers in situ transformation of hydrated phases, with relevant modification of gel surface into carbonates.

### Triaxial tests

Details about triaxial tests have been provided in Table 2. In Fig. 11, results of triaxial shearing on untreated and treated saturated samples cured for 24 h under different conditions (namely indoor and outdoor) are reported in terms of

deviatoric stress  $q$  and volumetric strain  $\varepsilon_v$  ( $\varepsilon_v = \varepsilon_a + 2\varepsilon_r$ ) as functions of shear strain  $\varepsilon_s$  ( $\varepsilon_s = (\varepsilon_a - \varepsilon_r)/2$ ). Treated samples show a similar behaviour regardless of curing conditions, with a brittle behaviour, with a peak strength and the subsequent softening stage. Compared to untreated sample behaviour, treated samples show an increase of stiffness and shear strength. The volumetric behaviour is contractive for both untreated and treated samples, with larger volumetric strains for treated samples. The observed behaviour is consistent with the higher initial void ratio of treated samples.

Further insights about mechanical behaviour of treated samples up to critical state have been pursued by plotting triaxial test data in terms of effective stress obliquity ratio  $\eta$  ( $\eta = q/p'$ ) as a function of both deviatoric strain  $\varepsilon_s$  and dilatancy  $d$  ( $d = d\varepsilon_v/d\varepsilon_s$ ). In the  $\{\varepsilon_s, \eta\}$  plane, stress-strain curves representative of each test should converge towards a horizontal line, corresponding to critical value  $M$  of the stress obliquity ratio, as the deviatoric strain increases. The same tests in the plane  $\{d, \eta\}$  should tend to a single point lying on the  $\eta$  axis corresponding to the critical state condition  $d = 0$ . In

**Fig. 5** TGA analyses at increasing curing times. **a** Indoor cured samples. **b** Outdoor cured samples

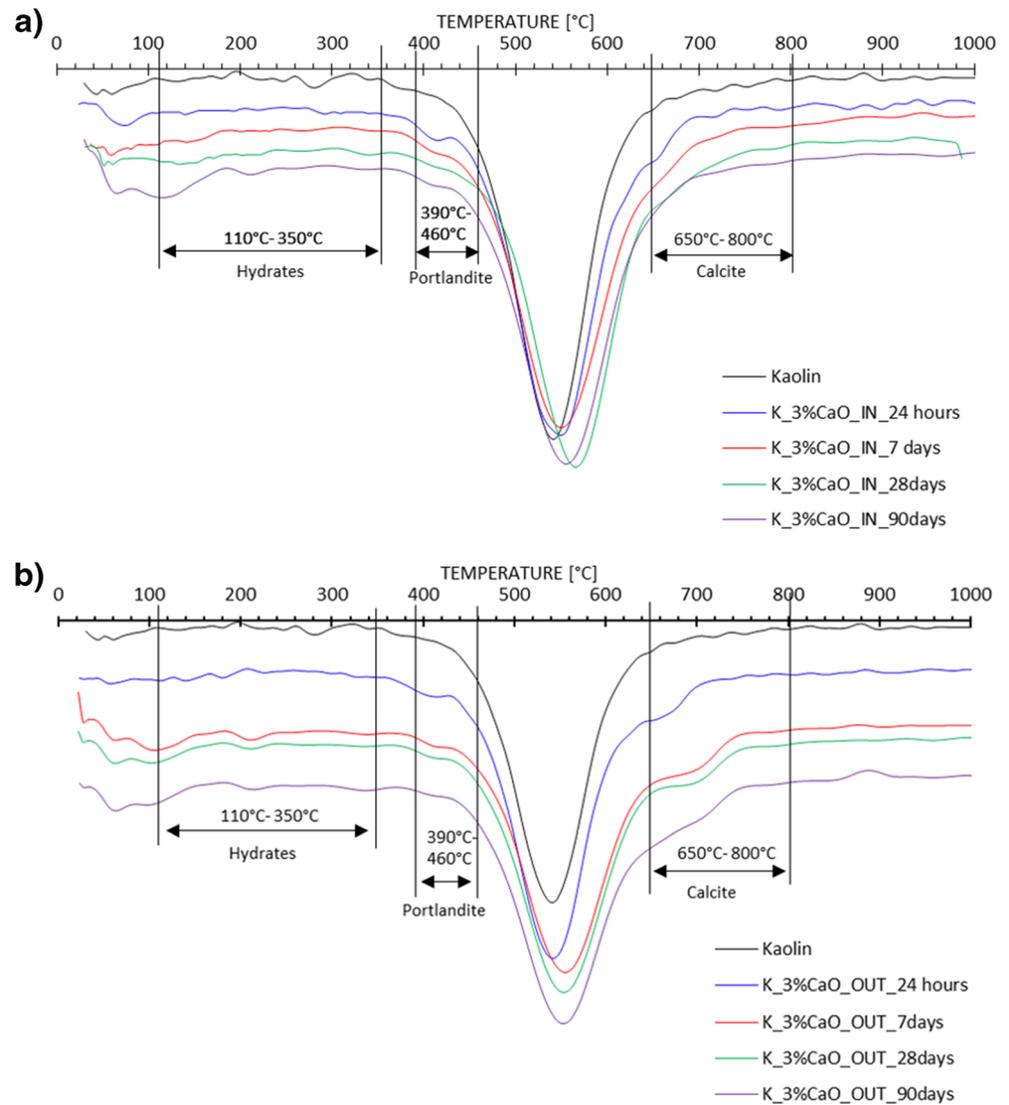


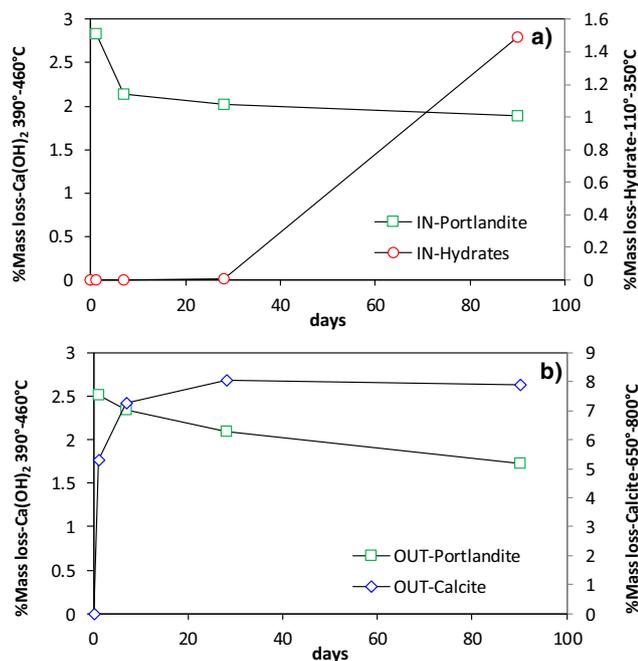
Fig. 12, results of triaxial tests on indoor cured samples at increasing curing times (i.e. 24 h, 28 and 90 days) evidence a similar behaviour for samples up to 28 days of curing, with an initial hardening and a subsequent softening stage, whereas longer curing times result in a relevant increase of stiffness, a marked fragile behaviour of the sample with a peak shear strength and a softening stage. For the strain level reached, no strain localization was detected for tested samples.

Critical state parameter  $M$ , determined following the aforementioned procedure, evolves over time as a consequence of chemo-physical evolution and microstructure reorganization of lime-clay-water system, starting from values equal to  $M = 0.88$  (corresponding to a constant value friction angle  $\varphi' = 22.5^\circ$ ) for short curing time (i.e. 24 h of curing), up to  $M = 0.99$  ( $\varphi' = 25.1^\circ$ ) for 28 days of curing, and finally  $M = 1.13$  ( $\varphi' = 28.4^\circ$ ) reached by samples cured indoor for 90 days. Dilatancy before peak shear strength is positive (Fig. 11b), with a trend progressively approaching the null value

characterizing the end of the test, as a confirmation of the reached critical state of the sample. All the samples show an overall contractive behaviour as shown in Fig. 11c. Larger volumetric strains were detected for 28 days cured samples.

In Fig. 13, results of triaxial tests on outdoor cured samples at increasing curing times (i.e. 24 h, 28 and 90 days) are shown. Outdoor curing slightly affects the observed behaviour of samples cured for 24 h and 28 days, whereas is relevantly impacting on 90 days cured samples, which do not show any further shear strength increase (as shown for indoor cured samples).

Moreover, the 90 days cured samples are characterized by a marked softening stage after the maximum shear strength. Critical state is reached with a contractile behaviour (Fig. 13c) at similar  $M$  values for each outdoor cured samples ( $M = 0.85$  ( $\varphi' = 21.8^\circ$ ) for 24 h of curing and  $M = 0.83$  ( $\varphi' = 21.4^\circ$ ) for 28 and 90 days of curing), as shown also by stress-dilatancy plots in Fig. 13b.



**Fig. 6** Quantitative interpretation of TGA analyses. **a** Indoor cured samples—evolution of portlandite and hydrates with curing time. **b** Outdoor cured samples—evolution of portlandite and calcite with curing time

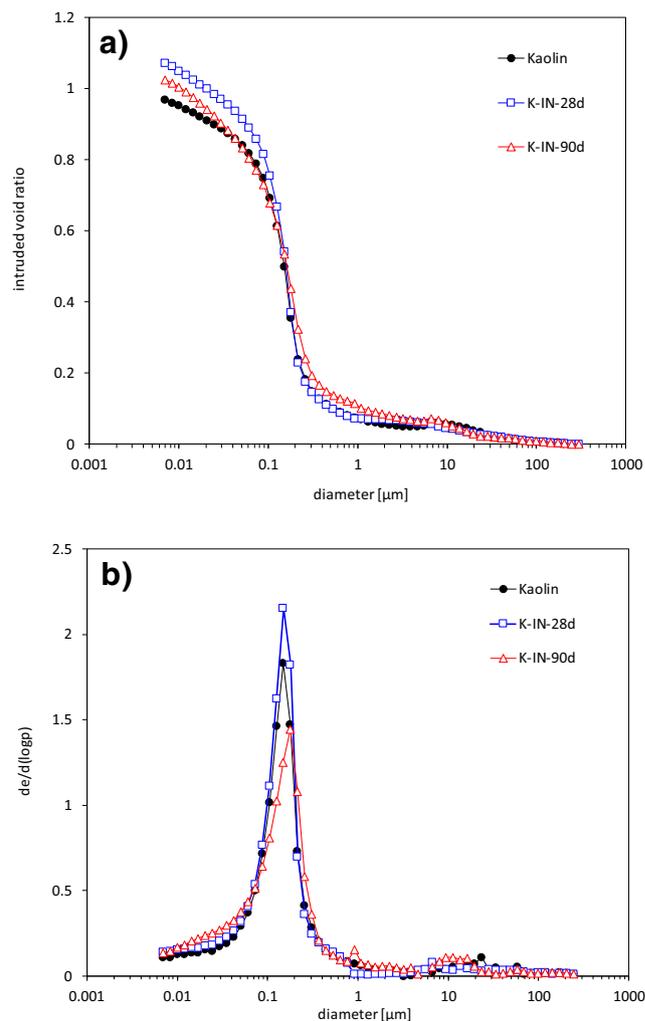
Results of triaxial tests on treated samples cured for long time intervals in three different curing conditions (namely IN, OUT and IN + OUT) are reported in Fig. 14. Curing outdoor after the indoor stage induces a decrease of shear strength and a reduction of the critical state parameter  $M$  ( $M = 0.89 - \varphi' = 22.8^\circ$ ) compared to indoor curing conditions. This behaviour is intermediate between those showed by indoor cured samples and outdoor cured samples.

## Discussion

Mineralogical and microstructural investigations highlighted the role of curing conditions on the chemo-physical evolution of clay-lime water systems, and their effect on mechanical behaviour of treated samples.

For lime-treated kaolin samples cured indoor, results of XRD showed a low reactivity of kaolin in promoting development of pozzolanic reactions, as highlighted by the delay of portlandite consumption and precipitation of new mineralogical phases (i.e. C-A-H) (Vitale et al. 2017). Pozzolanic compounds form only after long curing times (i.e. 90 days). In the short term, lime addition induces flocculation of particles into larger aggregates (Russo et al. 2007; Vitale et al. 2016a), which leads to an increase of the porosity of treated sample, as detected by MIP analyses.

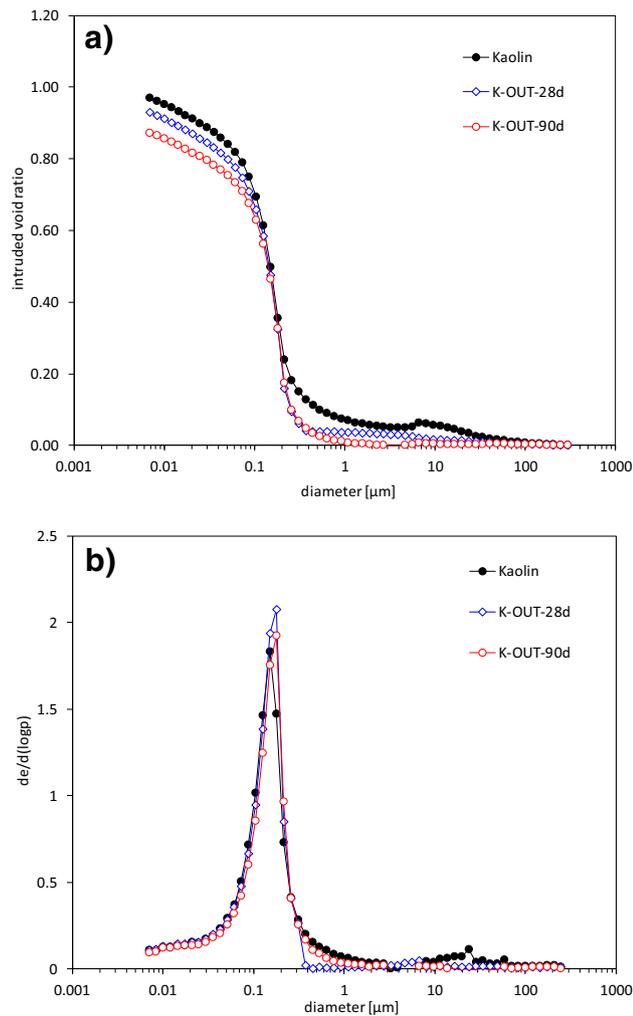
Development of pozzolanic reactions in the long term favours precipitation of calcium aluminate hydrate gel which



**Fig. 7** **a** Cumulative intruded volumes and **b** pore size distributions of indoor cured samples as function of curing time

covers clay particle aggregates, as evidenced by SEM observations. As a consequence, a reduction of porosity of treated samples and an increase of pore frequency in the small diameter range are detected by MIP results.

A different mineralogical evolution was induced in the clay-lime-water system by immediate CO<sub>2</sub> exposure of treated sample (outdoor curing). Dissolution in water of atmospheric carbon dioxide neutralizes the alkalinity of the pore water, as evidenced by the phenolphthalein indicator tests, and favours the gradual transformation of portlandite to calcium carbonate, whose amount increases over time as a result of the ongoing carbonation reaction (*lime carbonation*). In the long term, *lime carbonation* inhibited precipitation of cementitious compounds, as shown by XRD analyses. On one side, the reduction of pH induced by dissolution of CO<sub>2</sub> in water prevented silica and alumina dissolution. On the other side, unavailability of calcium phases due to carbonation reactions inhibited the development of pozzolanic reactions. Precipitation of calcite crystals and formation of chain-like calcite aggregates, as

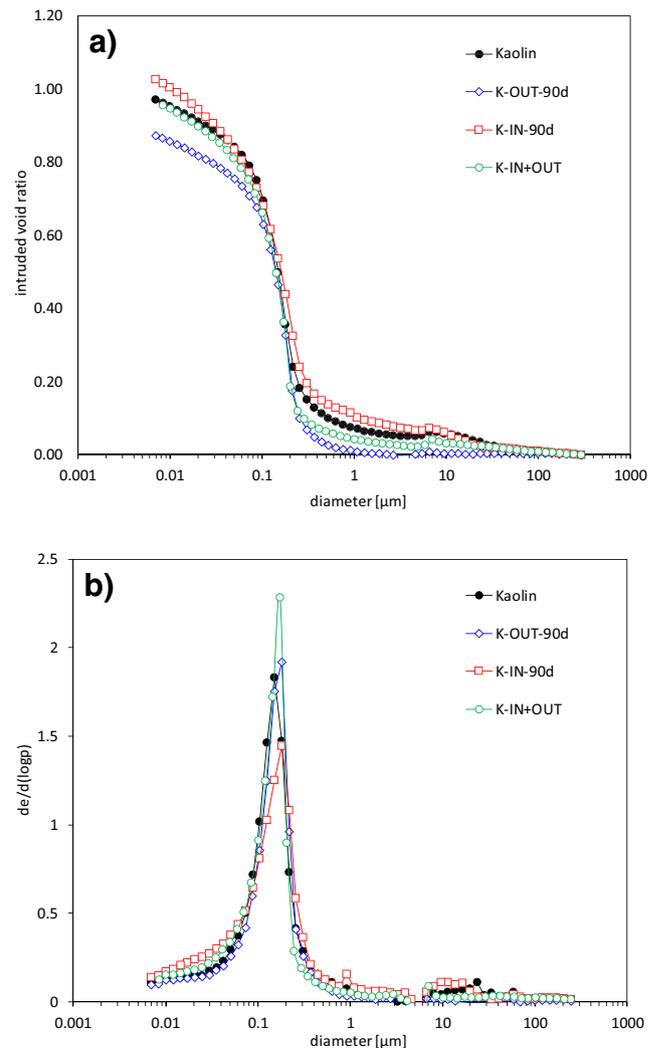


**Fig. 8** **a** Cumulative intruded volumes and **b** pore size distributions of outdoor cured samples as function of curing time

shown by SEM, seem to play a key role into the modification of pore structure of carbonated samples. Molar volume of portlandite is smaller than the molar volume of  $\text{CaCO}_3$  phases (Moorehead 1986; Cazalla et al. 2001; Pesce 2014; Houst 1996; Arandigoyen et al. 2006). As a consequence, the progressive formation of calcite due to in situ transformation of portlandite seems to be consistent with reduction of frequency of larger pores as detected by MIP results.

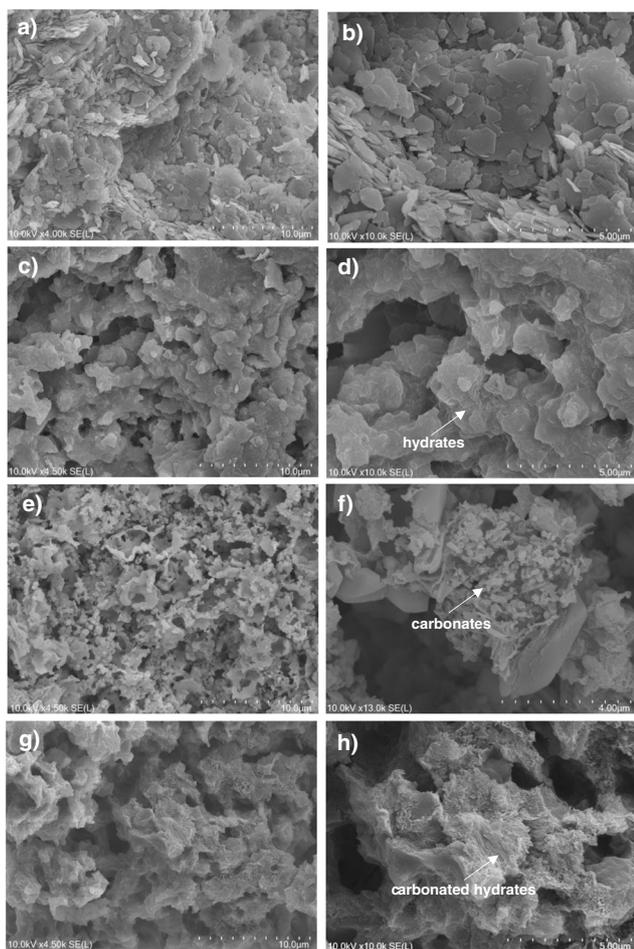
In the short term, the detected modification induced by different curing conditions does not produce different mechanical responses of treated samples. An increase of shear resistance with reference to compacted untreated kaolin is detected for both indoor and outdoor cured samples (Fig. 11) mainly as a consequence of microstructural reorganization induced at very early stage by addition of lime, since the low reactivity of the clay mineral.

At increasing curing time, differences between the mechanical responses have been detected as consequence of curing conditions and therefore chemo-physical evolution of the



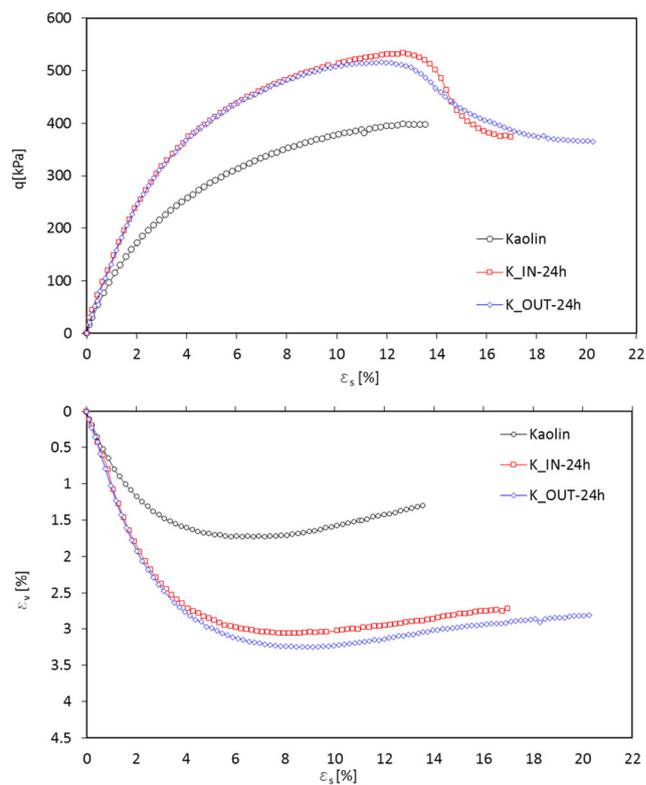
**Fig. 9** **a** Cumulative intruded volumes and **b** pore size distributions of indoor cured sample (90 days), outdoor cured sample (90 days) and indoor + outdoor cured sample (more than 90 days)

systems. For indoor cured samples, mechanical response of treated samples is relevantly improved after long curing times, due to the low reactivity of kaolin in promoting pozzolanic reactions. A combined effect of fabric and bonding determines the mechanical improvement of treated samples, which is consistent with the evidence of new cementitious phases formed after longer curing times, as evidenced by the relevant increase of stiffness and shear strength, and an increase of critical state parameter  $M$ . For outdoor cured samples, improvement of mechanical behaviour can be attributed to cementation caused by the precipitation of calcium carbonate (Fukue et al. 1999; Nagaraj 1964). Nevertheless, a limited evolution of the mechanical behaviour can be detected over curing time, as evidenced by results reported in Fig. 13, because occurrence of lime carbonation prevents the ongoing of pozzolanic reactions inhibiting potential mechanical improvement induced by lime treatment.



**Fig. 10** SEM observations. **a, b** Untreated compacted kaolin. **c, d** Carbonated kaolin after 90 days of outdoor curing. **e, f** Treated kaolin after 90 days of indoor curing. **g** and **h** Treated kaolin cured indoor 90 days and outdoor > 30 days

The occurrence of carbonation after long curing time (*carbonation of secondary reaction products*) affects mineralogical features of lime-treated samples. Exposure of lime-treated sample to CO<sub>2</sub> after precipitation of hydrated



**Fig. 11** Triaxial results: stress-strain curves of untreated and treated kaolin after 24 h of curing indoor and outdoor

phases, condition simulated by IN + OUT curing, leads to decalcification of cementitious compounds, which chemically react with carbon dioxide forming calcite, as shown by XRD analyses. In situ transformation of gel hydrates into calcite seems to be consistent with reduction of porosity and decrease of the frequency of pores in the small diameters compared to indoor cured samples. As showed by triaxial test results on IN + OUT cured samples, carbonation of secondary reaction products weakens the structure induced by lime during the previous indoor curing stage. As a consequence, the relevant mechanical

**Table 2** Summary of stress-path controlled drained triaxial compression tests on saturated samples

ID samples	End-of-curing conditions		Isotropic compression		Deviatoric stage		
	Curing conditions	$e_i$	$e$	$p'$ (kPa)	$e$	$q_{cs}$ (kPa)	$p'$ (kPa)
Untreated kaolin	-	1.01	0.974	300	0.968	398.06	435.72
K_IN-24 h	Indoor	1.29	1.263		1.252	373.83	424.64
K_IN-28 days		1.32	1.286		1.267	443.06	449.57
K_IN-90 days		1.05	1.045		1.041	538.52	477.76
K_OUT-24 h	Outdoor	1.40	1.371		1.359	365.27	427.58
K_OUT-28 days		1.21	1.181		1.169	329.56	398.61
K_OUT-90 days		1.05	1.030		1.021	345.54	416.31
K_IN + OUT	Indoor (90 days) + outdoor (30 days)	1.03	1.013		1.004	437.41	489.88

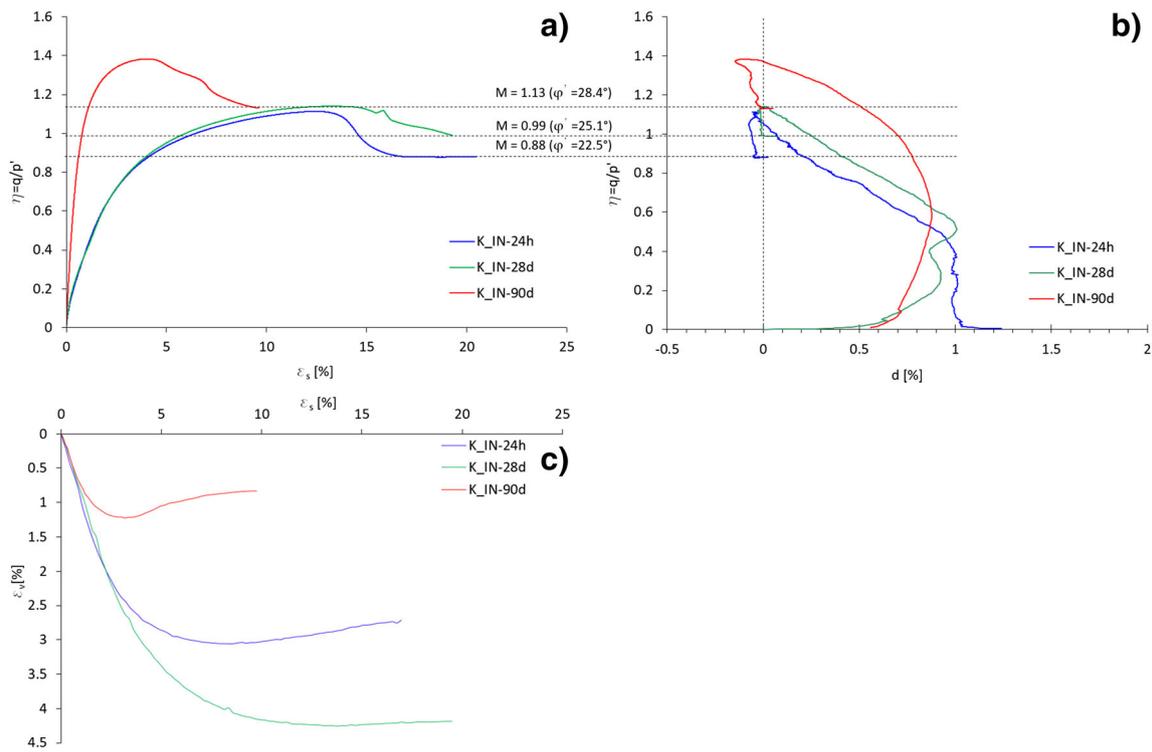


Fig. 12 Triaxial test results. **a** Stress-strain curves, **b** strain behaviour and **c** stress-dilatancy curves of lime-treated kaolin after 24 h, 28 days and 90 days of curing indoor

parameters (stiffness, shear strength, critical state parameter), improved during the previous indoor curing stage,

are subsequently lowered by weathering effects induced by  $\text{CO}_2$  exposure (Fig. 14).

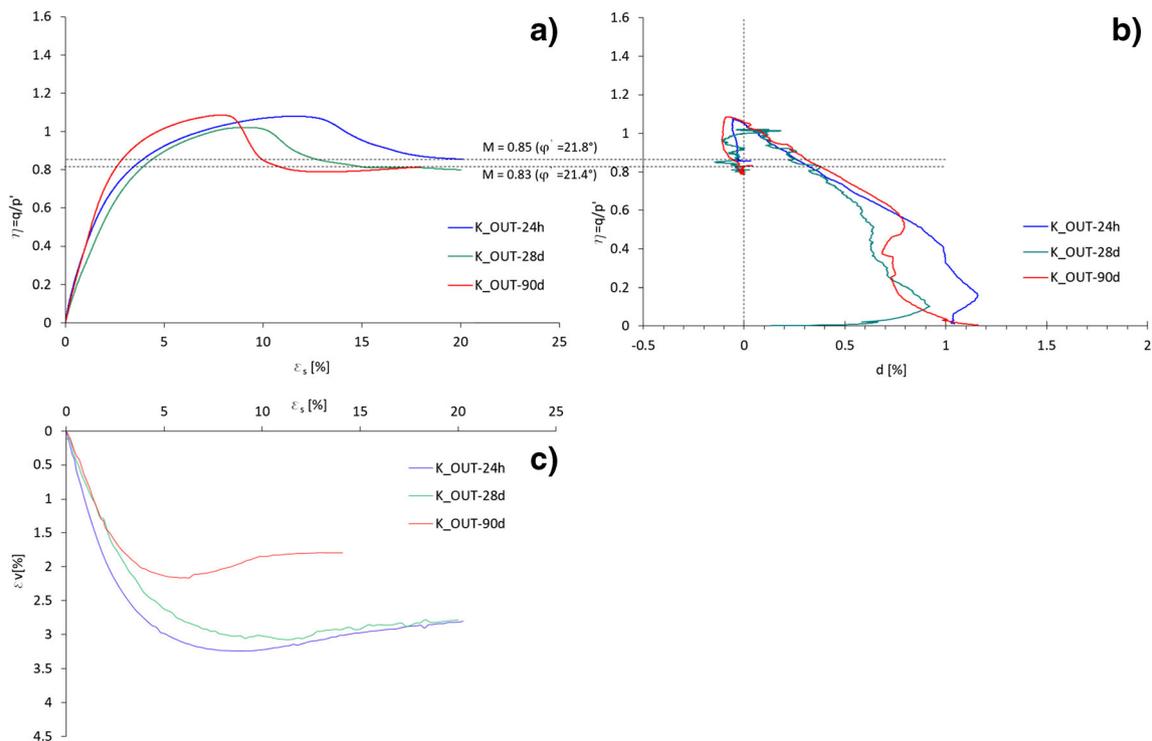
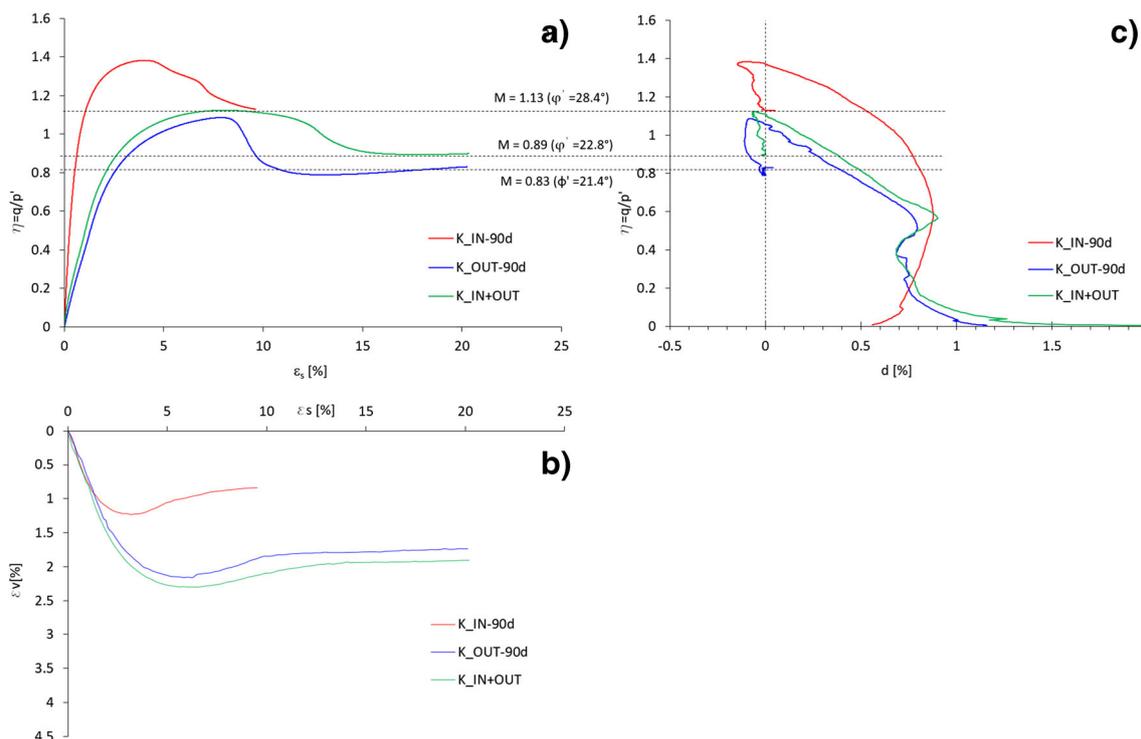


Fig. 13 Triaxial test results. **a** Stress-strain curves, **b** strain behaviour and **c** stress-dilatancy curves of lime-treated kaolin after 24 h, 28 days and 90 days of curing outdoor



**Fig. 14** Triaxial test results. **a** Stress-strain curves, **b** strain behaviour and **c** stress-dilatancy curves of lime-treated kaolin after 90 days of curing indoor (IN), 90 days of curing outdoor (OUT) and more than 90 days of curing indoor + outdoor (IN + OUT)

## Conclusions

In the paper, the results of an experimental multi-scale analysis on the effects of carbonation on chemo-mechanical behaviour of lime-treated soils have been reported. Chemo-physical evolution and microstructural features of lime-treated and carbonated samples cured under different environmental conditions have been investigated and interpreted by means of several experimental techniques. The results of micro-scale investigations have been directly linked to the experimental evidences at the volume scale of the sample, allowing an interpretation of the mechanical improvement of treated and carbonated samples in the short and long term as a function of the chemo-physical evolution of the system.

The main findings allow the following conclusions to be drawn:

- For lime-treated samples cured indoor, a relevant mechanical improvement is observed over time, as a result of the formation of hydrated compounds due to ongoing pozzolanic reactions on the long term.
- The mentioned mechanical improvement can be altered by occurrence of carbonation, depending on the time scale at which it takes place (*lime carbonation* and *carbonation of secondary reaction products*).
- *Lime carbonation* due to immediate exposure of lime-treated samples to atmospheric  $\text{CO}_2$  induces consumption of portlandite and precipitation of calcite, inhibiting the

onset of pozzolanic reactions in the system; as a consequence, mechanical improvement remains limited and does not evolve over time; potential benefits induced by lime treatment are limited as well.

- *Carbonation of secondary reaction products* occurring after exposure to  $\text{CO}_2$  of lime-treated samples, cured indoor for long term, favours decalcification of pozzolanic compounds and precipitation of calcium carbonate crystals, weakening the structure and the consequent improvement induced by lime treatment; this weathering effect can be potentially detrimental for use of lime treatment.

The study on the influence of *lime carbonation* and *carbonation of secondary reaction products* on the behaviour of lime-treated soils adds novel information about the impact of carbonation on mechanical performances of lime-treated soils, highlighting relevant factors for best practice finalized to efficient and durable soil treatment.

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