Impact of Carbonation on Lime-Treated High Plasticity Index Clayey Soils

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Abstract—Lime stabilization is a sustainable and economically viable option to address strength deficiencies of subgrade soils. However, exposure of stabilized layers to environmental elements can lead to a reduction in post-stabilization strength gain expected in these layers. The current study investigates the impact of carbonation on the strength properties of lime-treated soils. Manufactured soils prepared using varying proportions of bentonite silica mixtures were used in the study. Lime-treated mixtures were exposed to different atmospheric conditions created by varying the concentrations of CO_2 in the testing chamber. The impact of CO_2 diffusion was identified based on changes in carbonate content and unconfined compressive strength (UCS) properties. Changes in soil morphology were also investigated as part of the study. The rate of carbonation was observed to vary polynomially (2nd order) with exposure time. The strength properties of the mixes were observed to decrease with exposure time.

Keywords—Manufactured soil, carbonation, morphology, unconfined compressive strength.

I. INTRODUCTION

INFRASTRUCTURALLY, minimization of construction cost and saving of natural resources always remain the prime goal of practising engineers, while ensuring the performance and durability of structures. These concerns favour use of additives to improve the chemo-mechanical performance of otherwise inferior quality soils. Lime has been used as an additive across the globe for more than 8 decades now, and is considered as a reference for subgrade improvement, to withstand loads in infrastructure like pavements, railways, buildings etc [1], [2]. Addition of lime has been observed to alleviate problems faced with expansive soil, both in short [1], [3]-[5] and long-term [1], [6] through different mechanisms [7]-[9], rendering huge economic relief as well [10]. Practical benefits of usage of lime stabilization have been explained by various researchers [11], [12] through these mechanisms. This paper is aimed at investigating a key durability concern, diffusion of carbon dioxide, known as carbonation, related to lime-treated expansive subgrade soil.

II. BACKGROUND AND LITERATURE REVIEW

Carbonation has emerged as one of the most key durability properties gaining attention in recent times. Long-term adverse impact of atmospheric diffusion of CO_2 in lime-treated pavement sections was reported by a few researchers [2], [13]-[15]; many have focused on Mg-O admixed reactive soils [16], showing notable strength-improvement when exposed to accelerated CO_2 pressures. It was observed that 37% of the applied lime was utilized to form carbonates, in a 34-year-old motorway embankment in Germany [17]. Almost similar quantitative observations were noted by some other researchers for varying location exposed to atmosphere for almost the same amount of time [18], [19]. Comparison of degrees of carbonation was done for a silty soil in case of varying exposure conditions [20]. There are also studies pertaining to diffusion of carbon dioxide, without quantification of degree of diffusion [21]-[24].

While the mechanism of carbonation of calcium silicate hydrate, the product primarily responsible for strength gain in all cementitious materials, has been explained by many, degree of carbonation has been rarely quantified. Various methodologies have been employed to synthesize the hydration product and unrealistic forced experimental schemes have been applied to the synthesized products, leading to outcomes which are not representative of prolonged natural exposure-impact. In most of these material-investigation cases, rate of carbonation has been expressed as a function of square root of exposure time, where the constant (k) is mentioned to be independent of material properties, which raises doubt as degree of confinement and compaction appears to directly impact the depth of diffusion, alongside the two primary factors-temperature and relative humidity [25]-[28]. The process of carbon dioxide diffusion has been explained by various researchers from solid-liquid-gas interaction perspectives [29]-[32].

This work focuses at measuring the impact of carbonation on expansive subgrade soil in terms of UCS, carbonate content, and morphology, in relation to impact on pure calcium silicate hydrate (C-S-H) through similar levels of varying degrees of exposure. Through this work, our main goal is to determine a realistic prediction of rate of carbonation of lime stabilized soil.

III. MATERIALS AND METHODS

Pure Calcium Silicate Hydrate (C-S-H) was synthesized using reagent grade nano-silica and calcium oxide, following works done by Greenberg [33] and his co-researchers [34]. Cylindrical samples were prepared replicating highly expansive soil using commercial bentonite and grade-III standard sand in three proportions, by applying static compaction, in two different dimensions: a. 38 mm diameter × 76 mm height using

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World Academy of Science, Engineering and Technology
International Journal of Urban and Civil Engineering
Vol:18, No:5, 2024

standard mould and ejector, for compressive strength (UCS) testing and b. 10 mm diameter × ~ 40 mm height using hydraulic dye-press and ejector for bound CO_2 content using thermogravimetry (TGA) and microstructure and morphology experiments using Field emission scanning electron microscope

- energy dispersive spectroscopy (FESEM-EDS). All the mixtures were stabilized by using optimum lime content. Tap water was used for all the sample preparation and their physical characterization whereas Millipore water was used for synthesis of pure C-S-H.
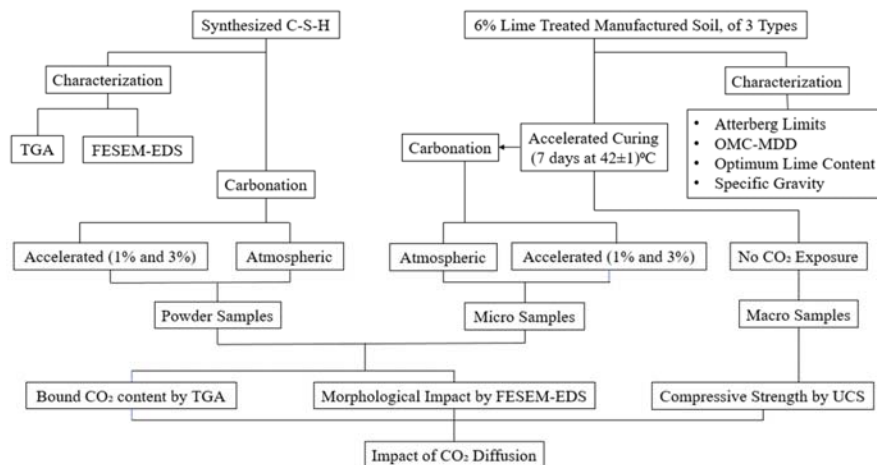

Fig. 1 Schematic of experimental methods

TABLE I
PHYSICAL CHARACTERISTICS OF MANUFACTURED SOILS

Terminology of soil	S1	S2	S3
Grade III standard sand (%)	60	50	40
Commercial bentonite (%)	40	50	60
Plasticity Index	65.63	70.83	93.74
OMC (%)	12.9	16.9	17.9
MDD (gm/cc)	1.72	1.85	1.95
OLC (%)	6	6	6
pH at OLC	12.39	12.33	12.33
Specific Gravity without lime	2.629	2.647	2.661
Specific Gravity with lime	2.671	2.673	2.714

TABLE II
EFFECTIVE DEPTH CALCULATION

| Actual depth (mm)	Consider as (mm)	Midpoint (mm)	Calculated depth (mm)	Effective depth (mm)
0	0		0	0
6			6	5
12		=33.5/2 =	12	10
18		16.75	=(33.5-18)=15.5	15
26			=(33.5-26)= 7.5	5 or 10
33.5	0		0	0

A. Experimental Methods

The experimental methods, as presented in Fig. 1, consist of compressive strength testing of lime treated soil samples, analysis using TGA and FESEM-EDS techniques, of synthesized pure C-S-H and lime-artificial soil mixtures, subjected to varying CO_2 exposure. For FESEM-EDS, all subsamples were placed on both sided copper tape to eliminate presence of other possible sources of carbon.

B. C-S-H Synthesis and Characterization

Type I and II C-S-H, following standard classifications were synthesized following method cited in Section III, covering Ca

to Si molar ratio of 1 to 2.25 [35]. Synthesized precipitated phases of C-S-H were characterized by interlayer water loss (ILWL), i.e., the weight loss at 110-160 ºC region, and the bound CO_2 content, i.e., the weight loss at 550-800 ºC temperature region, as shown in Fig. 2 [36]-[44]. The second temperature range is further validated from thermograms of pure lime $(Ca(OH)_2)$ and calcium carbonate $(CaCO_3)$ as well, referring to primary chemical transition of interest these chemicals undergo on heating (1):

$$CaCO_{3(s)} = CaO_{(s)} + CO_{2(g)} \qquad (1)$$
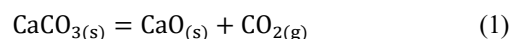
$Ca(OH)_2$ also contains small (< 5%) amounts of $CaCO_3$, which is plotted in Fig. 2 (b).

C. Preparation of Lime Treated Soil Mixtures

Varying proportions of commercial grade bentonite and grade III standard sand were used to prepare three manufactured soils. Also, conventional physical characterizations were carried out and details are mentioned in Table I. All results tabulated are average of 3 replicates' observations.
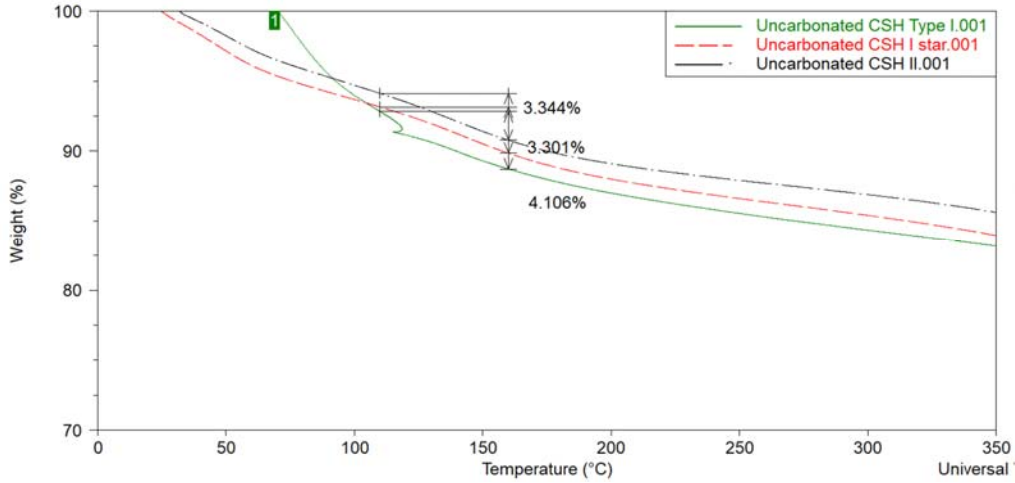
D. Exposure Conditions

After preparing cylindrical samples and extruding them out, all the samples are accelerated cured for 7 days at 42 ± 1 ºC [45]-[47] and then, half of the samples were wrapped on their edges by food-grade wrapping plastic and then, they were subjected to atmospheric and accelerated (1% and 3% CO_2) conditions for long and short durations, respectively. Once the exposure is complete, powder subsamples were taken from across the depth of exposed samples, as detailed in Table II for further analysis. Samples exposed all ways were termed C2 and ones wrapped in edges, were noted as C1.

World Academy of Science, Engineering and Technology
International Journal of Urban and Civil Engineering
Vol:18, No:5, 2024

For the limited scope of this paper, we are only showing observations pertaining to S1 and S3 type of soil, covering the upper and lower bounds of plasticity indices (PI).
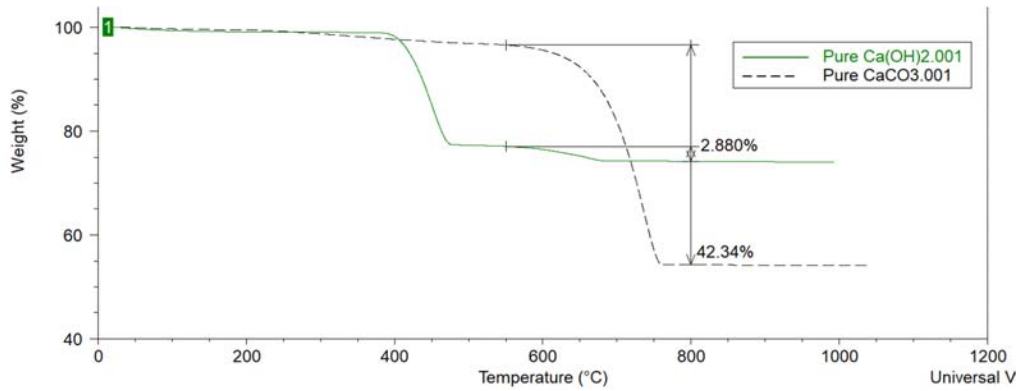
IV. Results and Discussions
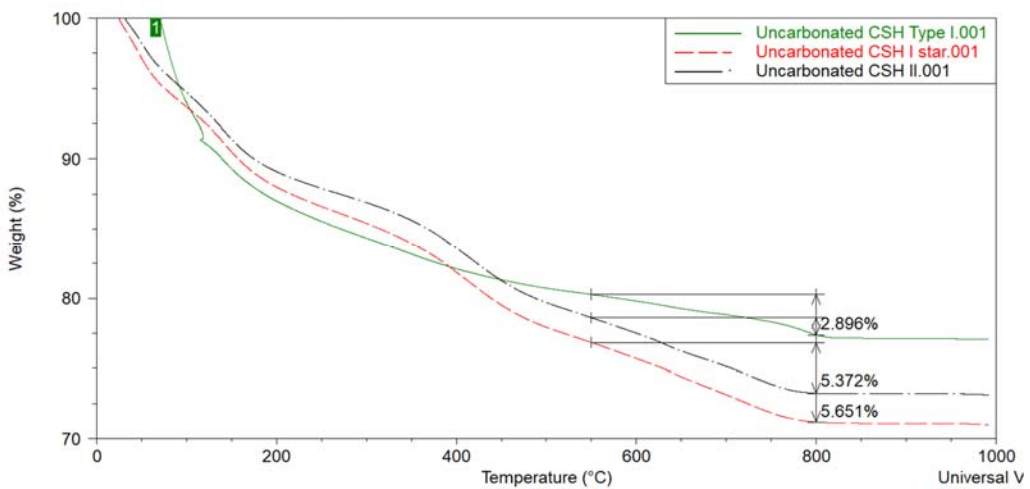
A. Bound CO_2 Content

The bound CO_2 content, obtained directly from percentage mass loss in the temperature range of 550-800 ºC from TGA thermograms, for soils subjected to different exposure conditions, are plotted in Fig. 3. Majority of the data points plotted are average of 3 replicates.
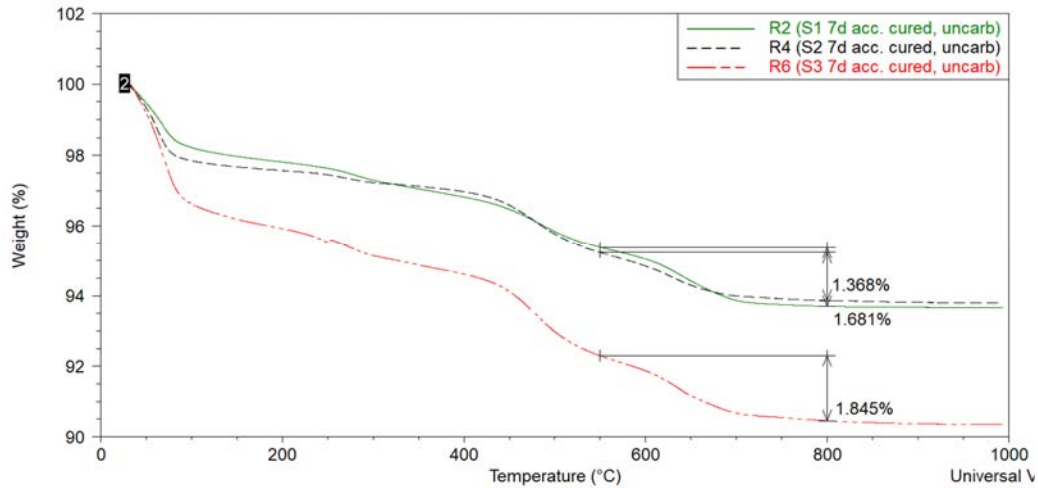
(a) ILWL of C-S-H

(b) Bound CO_2 content of Ca(OH)$_2$ and CaCO$_3$

(c) Bound CO_2 content of pure C-S-H

World Academy of Science, Engineering and Technology
International Journal of Urban and Civil Engineering
Vol:18, No:5, 2024

(d) Bound CO_2 content of lime soil mixtures

Fig. 2 Characterization of uncarbonated C-S-H, pure reference chemicals and lime soil mixtures through TGA
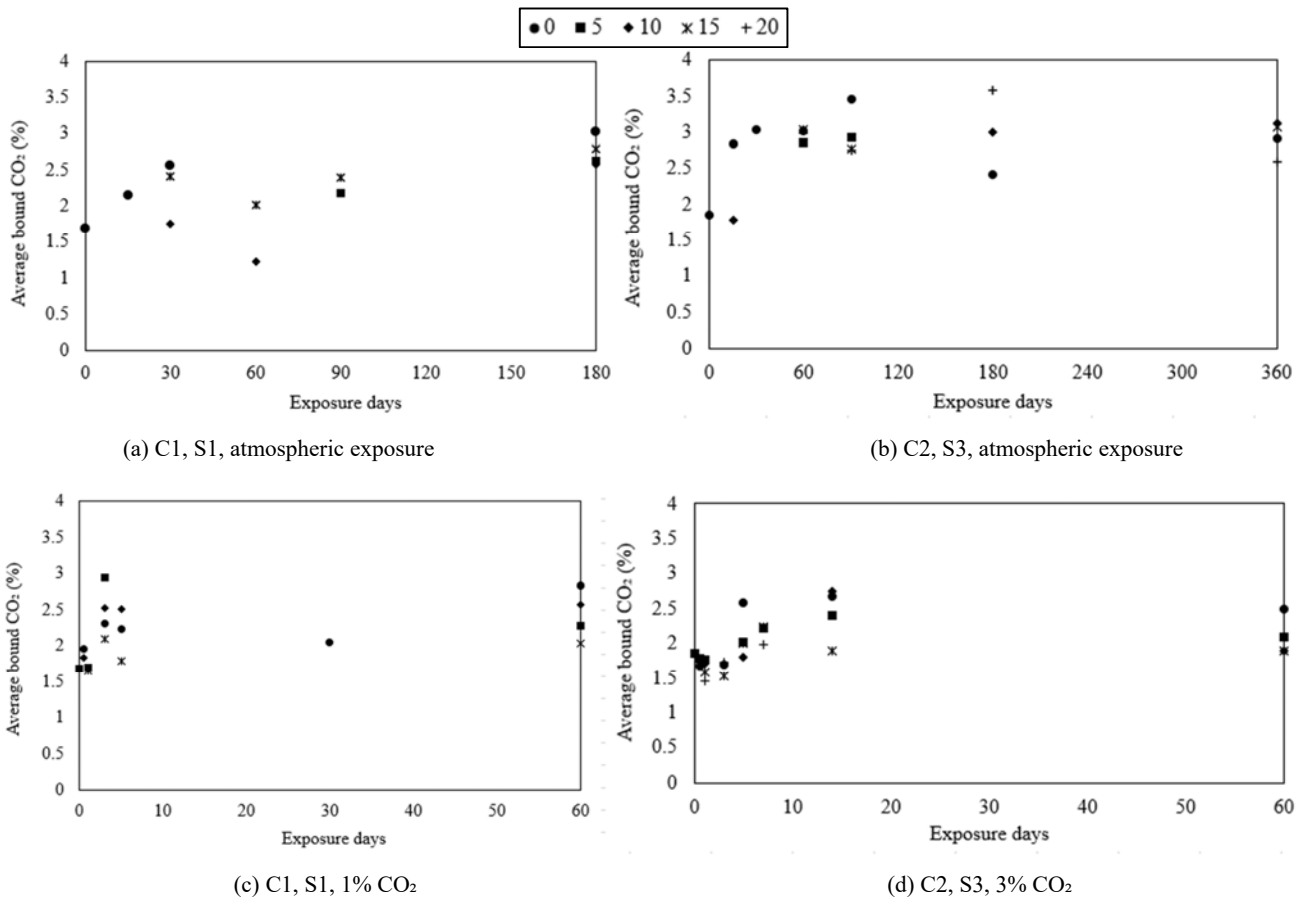

(a) C1, S1, atmospheric exposure

(b) C2, S3, atmospheric exposure

(c) C1, S1, 1% CO_2

(d) C2, S3, 3% CO_2

Fig. 3 Average bound CO_2 content of exposed soil samples

As it is evident from atmospheric exposure conditions, the bound CO_2 content directly varies with temperature and relative humidity conditions that are prevalent throughout the atmospheric exposure duration, which is in accordance with prior findings [48]-[51]. For accelerated exposure, the decrease in bound CO_2 content can happen for the following reasons:

- Possibility of slow leakage of the compressed gas from the low-cost customized setup.

- Balance gas (Argon) in the compressed gas mixture slowly replacing CO_2 inside the setup-environment.

- Bound CO_2 existing in forms other than $CO_{2(g)}$ which is not evaporated at the said temperature range.

World Academy of Science, Engineering and Technology
International Journal of Urban and Civil Engineering
Vol:18, No:5, 2024

B. Compressive Strength

For UCS observations, alongside the previously explained schemes, samples kept completely sealed i.e., out of any possible additional CO_2 exposure (marked as 0% in the graph), were also tested. The observations are plotted in Fig. 4. Data limited to 180 exposure days are presented in this paper to clearly detail the evolution in strength data of samples exposed to accelerated CO_2 partial pressure, as the exposure duration in accelerated schemes is limited to 60 days only. For UCS observations, only C2 (un-wrapped samples, for CO_2 diffusion to take place along the length and diameter) samples were used.
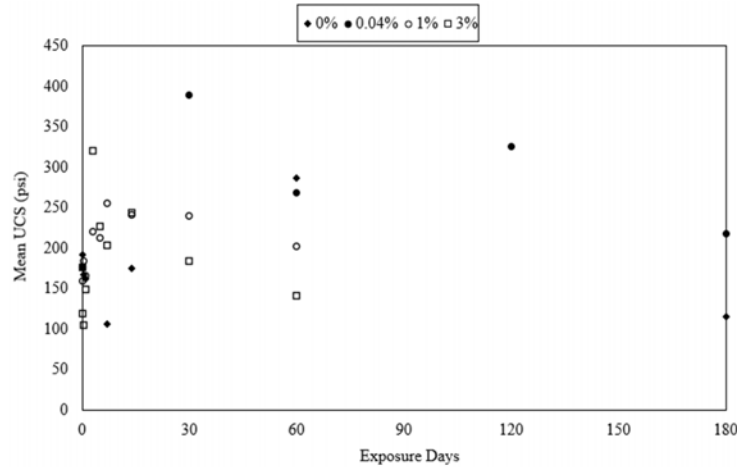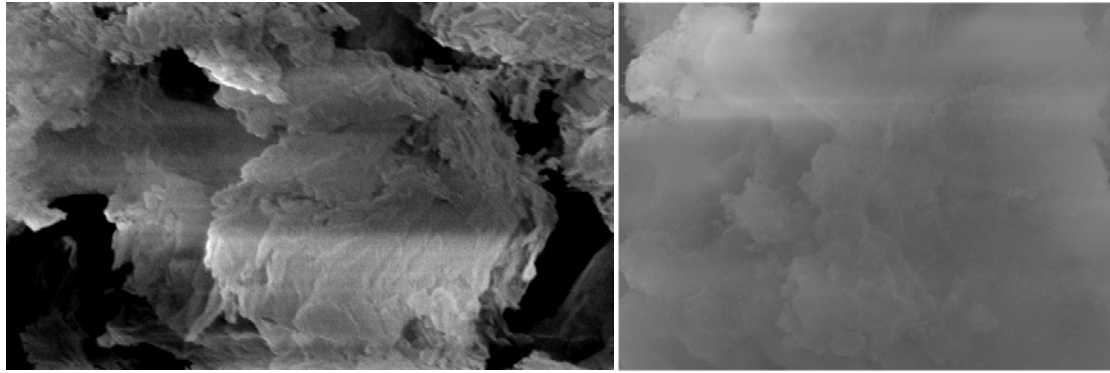

(a) S1
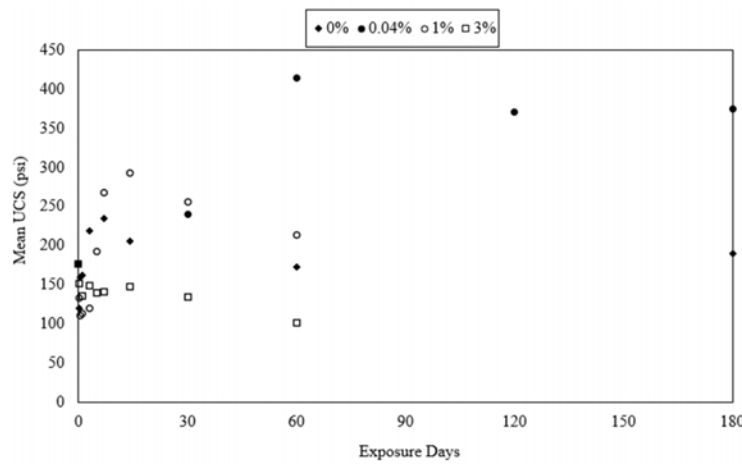
(b) S3

Fig. 4 Mean UCS of lime-treated soils exposed to varying CO_2 environments

What is evident also in the UCS observations of samples is, an initial sharp increase, due to lime-induced reactions imparting more strength by forming reaction-products like C-S-H, C-A-H etc. in the matrix, followed by a continuous reduction which can be attributed to carbonation and drying-both the factors. In addition to this, another observation here is, samples (of both the types of lime-treated bentonitic soil) which were not exposed to any form of CO_2 conditioning, end up with considerably higher long-term strength than the conditioned ones, which can either be due to the fact that amorphous $CaCO_3$ formed in the matrix are not exerting any swell pressure on the matrix or prolonged reactions of uncarbonated lime with remaining moisture within the mix.
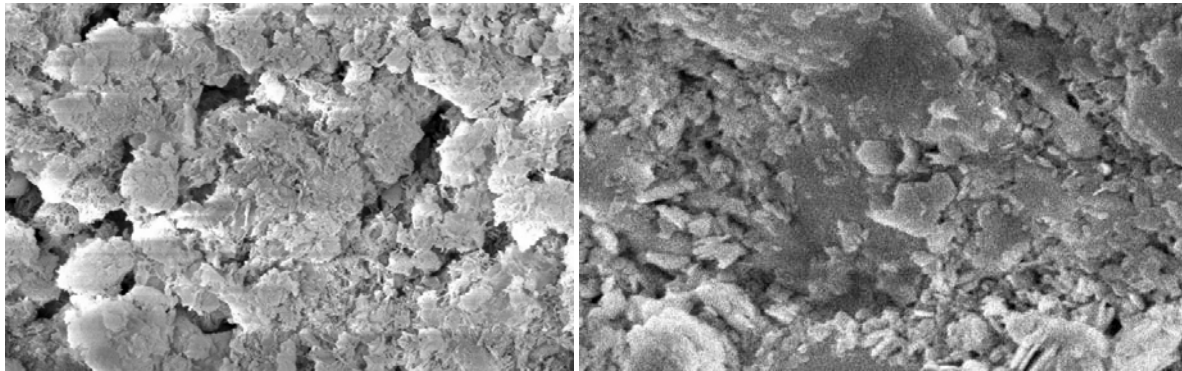
These observations point to the fact that carbonation has a profound negative impact on compressive strength of soil samples, despite being treated with calcium additives, which can be attributed to the formation of metastable bicarbonates initially leading to precipitation of calcium carbonate crystals. Also, to emphasize the long-term impact of carbonation, the reduction in long term strength of samples exposed to accelerated CO_2 pressures, experienced a faster and sharper drop in compressive strength. This can be attributed to the internal pressure exerted by formed or precipitated $CaCO_3$ crystals.

World Academy of Science, Engineering and Technology
International Journal of Urban and Civil Engineering
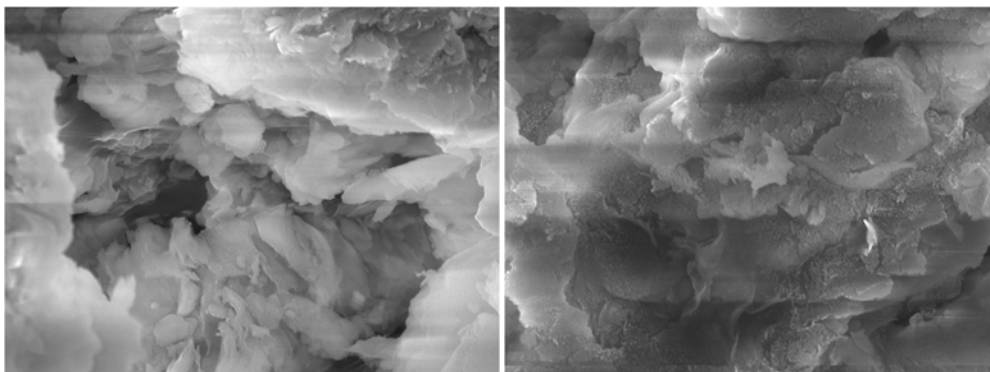Vol:18, No:5, 2024

(a) C-S-H I, uncarbonated

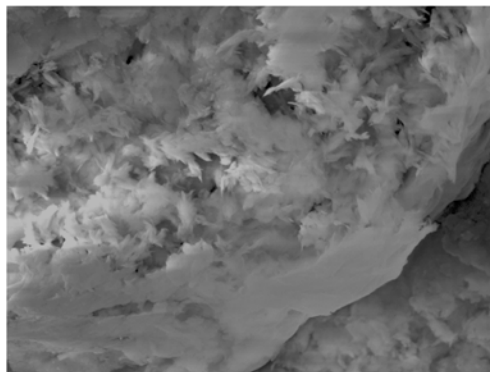(b) C-S-H I, exposed to atmosphere for 4 months

(c) C-S-H I, exposed to atmosphere for 8 months

(d) C1, S1-top surface, uncarbonated

(e) C1, S1-top surface, exposed to atmosphere for 1 year

(f) C1, S1-middle, exposed to atmosphere for 1 year

(g) C1, S1-top surface, exposed to 1% CO_2 for 60 days

Fig. 5 Morphological evolution of carbonated C-S-H and lime treated soil

World Academy of Science, Engineering and Technology
International Journal of Urban and Civil Engineering
Vol:18, No:5, 2024

C. Mineralogy

The powder subsamples obtained from across the depth of compacted soil samples and that of synthesized C-S-H, were also subjected to FESEM-EDS to obtain their morphology and elemental concentrations, for understanding the impact of diffusion of CO_2 into lime stabilized soil matrix and the reaction products that is primarily responsible for increase in strength. Here a comparison of morphological impact of carbonation on C-S-H and lime stabilized soil, exposed to varying levels of CO_2 partial pressure, is presented. The images obtained at 10-20K magnification level and 10-15kV accelerating voltage are given in Fig. 5.

From the attached images, it can be clearly seen that with the progress of carbonation, and increase in exposure duration, the matrix structure evolves from a stacked plate or foil like formation to that of a floating cloud-like connected network of loosely held fibrils, both in case of C-S-H (Figs. 5 (b) and (c)) and lime-treated soil, irrespective of depth of the subsample (Figs. 5 (e)-(g)). This evolution in the microstructure of both C-S-H and lime-treated soil, irrespective of depth of the collected subsample and direction or severity of exposure, emphasizes the fact that progressive diffusion of CO_2 into cementitious materials containing lime or lime-silica hydration reaction products, renders the structure weak (as also described in Section IV *B*) and porous, which, in turn can lead to reduced durability and decreased service life of the treated structure in the long run, leading to deterioration in engineering properties.

V. Conclusions and Inferences

Up to 3% of partial pressure of CO_2 exposure for around 2 months can mimic the field-impact of atmospheric CO_2 diffusion of one year or more, as observed from maximum bound CO_2 content, which is a direct measure of carbonation. Increase in CO_2 exposure-duration combined with suitable temperature and relative humidity conditions, enhances the possibility of formation of species of carbonate products, initially metastable bicarbonates which, then, following the dynamic equilibria [52]-[54], gets converted to different crystalline and amorphous forms of calcium carbonates, namely, calcite and aragonite and vaterite respectively, and other carbonate species. While reactions contribute to increase in strength of cementitious materials, as severity of CO_2 exposure increases, accompanied with natural cycles of drying and wetting, carbonation negatively impacts long-term strength gain. CO_2 primarily reacts with calcium present in the lime stabilized cementitious materials, both from $Ca(OH)_2$ and C-S-H, leaving the matrix as a network of slack needle-like fibrous formations, possibly of Si-OH, as calcium (Ca) gets detached and used up in the carbonation reaction, rendering the structure made up of silicate chains only, in agreement with some of the previous researchers [55]-[57]. This progressive decalcification of lime and C-S-H contributes to deterioration in engineering properties of the stabilized cementitious matrices [58], [59].

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World Academy of Science, Engineering and Technology
International Journal of Urban and Civil Engineering
Vol:18, No:5, 2024

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2167, 2 November 2018.