The International Journal of GEOMATE, May, 2025 Vol.28, Issue 129, pp.113-120
ISSN: 2186-2982 (P), 2186-2990 (O), Japan, DOI: https://doi.org/10.21660/2025.129.g14294
Geotechnique, Construction Materials and Environment

ROLE OF CARBONATION AND RESIDUAL LIME CONTENT ON LONG TERM STRENGTH BEHAVIOR OF LIME TREATED SUBGRADE SOILS

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ABSTRACT: Lime stabilization of subgrade soils is an established practice to improve the engineering properties of poor-quality soils encountered in pavement construction. However, long term exposure to environmental conditions can cause diffusion of carbon dioxide (CO_2) into the stabilized matrix and can lead to carbonation of available lime which in turn can adversely affect the long-term strength gain in these treated layers. Limited studies are available on the role of residual lime on long term strength gain in lime stabilized mixes as there are no available techniques to quantify residual lime content after stabilization. The research proposes applying a thermogravimetric technique to quantify the residual amount of lime available in lime treated matrix at any given time. Manufactured soils with varying clay contents were treated with lime and used to identify the carbonation profile of the matrix. Mixes subjected to carbonation for up to 240 days were used to investigate the impact of changes in residual lime content on long term strength behavior. Observations of the study clearly show a reduction in long-term strength with carbonation of the matrix. Possible changes in Ca/Si ratio leading to transformation of calcium silicate hydrate phases to silicate gel and the precipitation of calcium carbonate phase can cause strength deterioration in mixes during carbonation. Residual lime content in the stabilized matrix was found to increase slightly during curing periods.

Keywords: Stabilization, Soil, Carbonation, Strength, Residual lime content

1. INTRODUCTION

Lime stabilization is a common practice in pavement construction to improve the engineering properties of clay rich subgrade soils. Presence of high amounts of expansive clays like montmorillonite in subgrade soils makes these soil unfit for pavement construction due to their intrinsic shrink swell behavior when subjected to moisture fluctuations [1-3]. Lime stabilization is a physio-chemical process that can address the detrimental effects of expansive soils by reducing their water holding capacity and swelling potential. The process also helps improve the textural properties and workability of these soils and provides the required strength properties to support the load bearing structures built over them [4-6]. Mechanism of lime stabilization primarily involves dissolution of silicate and aluminate phases in clays under high pH conditions to form cementing materials like calcium silicate hydrate (C-S-H) and calcium aluminate hydrate (C-A-H) that binds the clay particles together. Mechanisms/processes involved in this have been documented by various researchers from both engineering and geochemical standpoints [7-9]. The long-term strength gain in these mixes is dependent on factors like clay content, pH and curing conditions, carbonation etc. [10-14].

Recent studies have shown durability issues cementitious materials to occur primarily from carbonation or due to the removal of stabilizing agents by moisture intrusion and leaching [15-19]. Among these, carbonation has been cited as one of the major durability concerns for lime stabilized subgrade soils as it can interfere with the long term strength of these layers [20-21]. Since C-S-H is the major strength contributing factor in stabilized materials, their susceptibility to carbonation can inversely affect the long-term performance infrastructure build on top of them [22-23]. Carbonation studies thus far have focused on carbonation of stabilized materials due atmospheric exposure to identify mechanisms and kinetics of C-S-H carbonation [23-26]. Carbon dioxide (CO_2) intrusion into the stabilized matrix is known to be a slow process and the early age carbonation of C-S-H phases is diffusion-controlled process [27]. The end product of carbonation in these samples were identified to be calcium carbonate and calcium modified silica gel [23-24]. In addition to an increased silicate binding and rapid decalcification during carbonation, prior studies have suggested the formation of stable phases (polymorphs) of calcium carbonate ($CaCO_3$), i.e., calcite, vaterite, and aragonite in C-S-H samples with Ca/Si of 1.5 [25-26, 28]. Carbonation studies in cement concrete domain have shown that rate of carbonation varies directly with the square root of exposure time [29-30].

Not many studies have been performed on long term impact of carbonation in stabilized soil matrix due to challenges involved in quantification of carbonates and a lack of available techniques to quantify residual lime available in the matrix. Further, only limited studies are available that correlate long term strength of stabilized soil matrix to carbonate exposure and residual lime available within the matrix. Presently, there are no published studies available on quantification of residual lime or on the impact of changes in their concentrations during the service life on durability properties of stabilized materials. Even though some researchers have looked into residual concentration changes during lime interactions, most of these were in conducted agronomy and were focused on changes occurring in aqueous phases. The results of such studies are often contradictory and their impact were not translated to engineering properties of stabilized matrices [31-34]. However, the general consensus is that the strength loss due to carbonation is related to the associated volume changes incurred during the process and due to the impact of CO_2 on non-crystalline C-S-H gels and cementitious crystalline phases precipitated during stabilization process [15, 35-38]. The combined disruptive effect of the above can lead to micro-shattering of the structure of carbo-calcareous mixture along with shrinkage incurred in the due process [39-40].

Present study attempts to quantify the changes in concentrations of critical phases like calcium carbonate and residual lime as a result of long-term exposure to CO_2 in atmospheric conditions. Observations in carbonated C-S-H phases were compared against responses obtained using pure reference materials (lime and calcium carbonate) to estimate the concentration of respective phases in the matrix. Additional details relevant to quantification are included in the materials and method section. The subsequent sections also discusses the significance of present study, materials used and methods adopted in the research which is followed by observations and the salient outcomes of the study.

2. RESEARCH SIGNIFICANCE

The study attempts to quantity the amount of calcium carbonate formed in cementitious mixtures due to diffusion of carbon dioxide in ambient conditions. The study also attempts to estimate the amount of residual lime present in cementitious mixtures at various time periods in the service life of cementitious mixtures. Finally, the strength properties of mixes were also measured to evaluate the impact of carbonation on engineering properties of stabilized layers due to long term exposure to environmental conditions. The study introduces a novel approach based on thermogravimetric analysis in tandem with precipitation stoichiometry to quantify carbonate phases and residual lime in cementitious materials. The suggested approach can be extended to be an effective tool for practicing engineers and research community to access durability issues and long term behavior of stabilized materials.

3. MATERIALS AND METHODS

Manufactured soils with high plasticity index needed for the study were prepared by mixing commercial bentonite and grade III quartz sand confirming to standard in two different proportions of 40:60 and 60:40 [41]. Physical characteristics of the manufactured soil used in the study are detailed in Table 1. Deionized water was used in mixing and curing samples prepared for testing. The manufactured soils were characterized based on their physical properties and treated with 6% lime for stabilization. Mixtures used in study were compacted using static compaction and kept in sealed in airtight zip lock bags and cured at 41ºC for 28 days prior to CO_2 exposure [42-44].

Table 1 Physical characteristics of soil.

Terminology of soil	S1	S3
G-III std. sand: bentonite (%)	60:40	40:60
Plasticity Index	65.63	93.74
OMC (%)	12.9	17.9
MDD (gm/cc)	1.72	1.95
OLC (%) (pH)	6 (12.39)	6 (12.33)
Specific Gravity (without lime)	2.629	2.661

Cylindrical samples of two different sizes- 10 × 20 mm (50 nos.) and 38 × 76 mm (86 nos.) were used in the study. Compacted and accelerated cured samples were subjected to atmospheric carbon dioxide environment under controlled conditions. Among the 10 × 20 mm samples, cylindrical sides of 24 nos. samples (identified as ‘C1’ in the following sections) were wrapped with food grade wrapping plastic to ensure unidirectional diffusion of CO_2 into the samples. The remaining samples (24 nos.) were left unwrapped allowing CO_2 exposure from all directions (identified as ‘C2’ in the following sections). At the end of designated exposure time, the samples were removed from carbonation chamber and stored in a desiccator. Pure nitrogen gas was purged to remove available carbon dioxide inside the desiccator to arrest additional carbonation. Subsamples needed for the study were prepared by cutting the samples at 10 mm and 15 mm depths along the axis of the cylinder from both surfaces. Powder samples required for analysis were randomly sampled from the cut sections and labeled according to their depths and given in the following sections of this paper. Differential scanning calorimeter-Thermogravimetric analyzer (SDT Q600

DSC-TGA, TA instruments make) was used to quantify the residual lime and carbonate content in sub samples at different exposure durations. Samples used in thermogravimetric analysis were heated to 1000°C at a constant heating rate of 10°C/min. A constant nitrogen purge flow of 100 ml/min. was maintained during the heating process. Weight loss incurred by 99 percent pure calcium hydroxide ($Ca(OH)_2$) and 98 percent pure calcium carbonate ($CaCO_3$) at 382°-496°C and 550°-800°C respectively were used as reference for quantification purpose. TGA data of pure calcium carbonate and lime used are given in Fig. 1.

Observations agree well with data available in literature [45-46]. Weight loss peak observed between 382°-496°C for $Ca(OH)_2$ corresponds to loss of chemically bound water whereas the weight loss peak between 550°-800°C for $CaCO_3$ corresponds to removal of CO_2 from carbonate matrix. Quantification of these two in lime treated samples were done based on comparison of weight loss peaks at respective locations with the data obtained using reference materials. The DSC-TGA was initially calibrated as per manufacturer's recommendations (zinc for temperature, alumina for heat flow and calcium oxalate monohydrate for precision). Fig. 2 explains the workflow as a schematic.

Weight percentages of (a) water chemically bound to lime (Eq. 1) and (b) CO_2 chemically bound to $CaCO_3$ (mostly calcite) (Eq. 2) were estimated by comparing the thermograms of powder soil samples

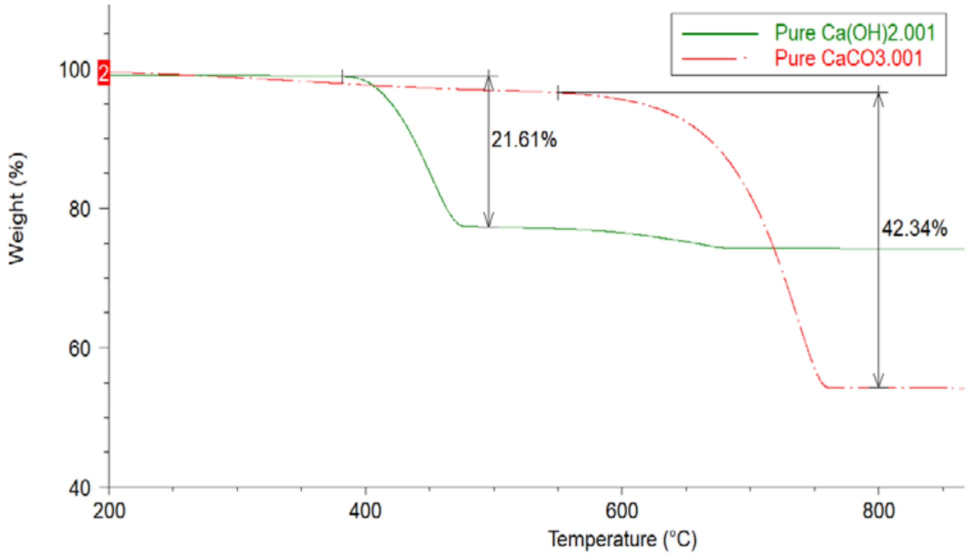

Fig. 1 Thermograms of pure $Ca(OH)_2$ (green, continuous line) and $CaCO_3$ (red, dash-dot line)

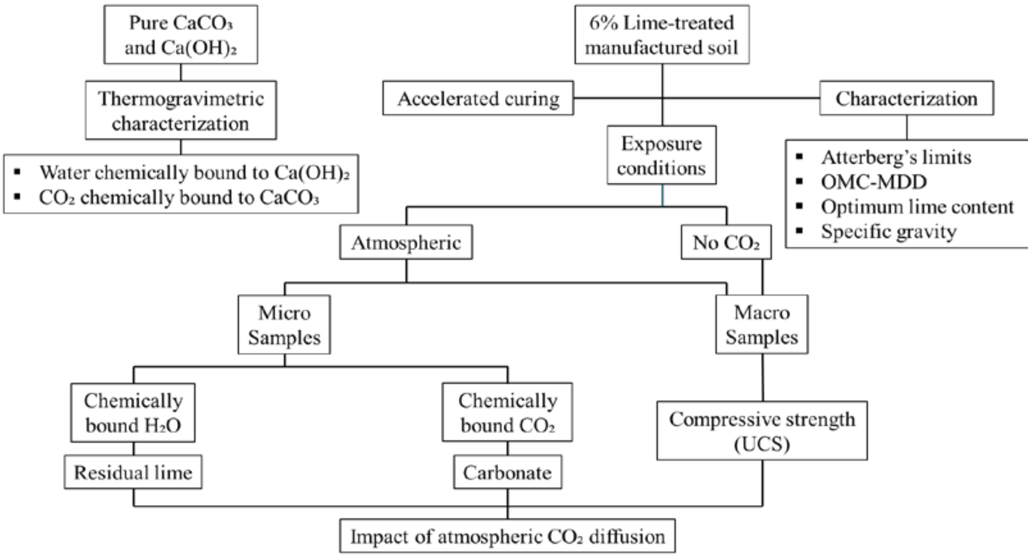

Fig. 2 Schematic of workflow

with respective peaks from reference materials listed above.

$$Ca(OH)_2 \triangleq CaO + H_2O \uparrow \quad (1)$$

$$CaCO_3 \triangleq CaO + CO_2 \uparrow \quad (2)$$

Residual lime and carbonate concentrations were estimated based on stoichiometric equations listed above and detailed in Eq. (3) and Eq. (5) respectively. Molecular weights of 74.092 gm, 18.015 gm, 60.008 gm and 40.01 gm were used for $Ca(OH)_2$, H_2O, CO_3^{2-} and CO_2 respectively.

$$[Ca(OH)_2](\%) = \text{Evaporated } H_2O \times \frac{74.092}{18.015} \quad (3)$$

$$CaCO_3(aq) = Ca^{2+}(aq) + CO_3^{2-}(aq) \quad (4)$$

$$[CO_3^{2-}](\%) = \text{Evolved } CO_2 \times \frac{60.008}{40.01} \quad (5)$$

38 mm diameter samples (86 nos.) were exposed to atmosphere and used to estimate compressive strength of mixes. These samples were exposed to atmospheric conditions in a controlled environment inside the laboratory. Strength testing was done using a uniaxial compression (UCS) testing machine and the UCS values were corrected to an aspect ratio of 2 [47]. In the subsequent section (section 4), i.e., results and discussion, all the legends in Fig. 3 to 6 denote the depth of the powder soil-subsample in millimeters (mm).

4. RESULTS AND DISCUSSIONS

Observations in Fig. 3 (a-b) and 4 (a-b) show the residual lime concentrations in stabilized matrix with curing period.

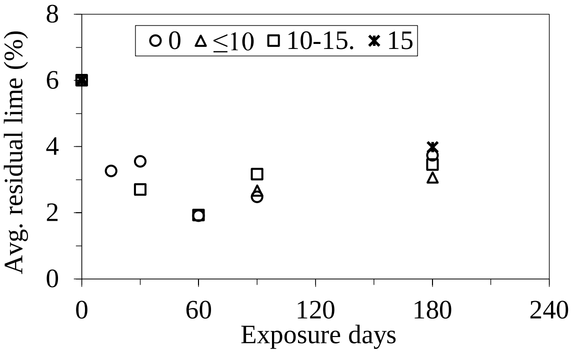

Fig. 3a Residual lime content in uni-directionally (C1) exposed S1 (40% bentonite) soil samples across depth

The observed concentrations were found to be less than optimum lime content (6 percent) used in the study. However, the residual lime concentrations in samples were found not to change or slightly increase with curing time. Lime concentrations in top layers were also found to be slightly higher than quantities estimated at depth of 15 mm. Observations in Fig. 3 (a-b) suggest the possibility of formation of lime through reduction in concentration of (HCO_3^-), as shown in Eq. (6).

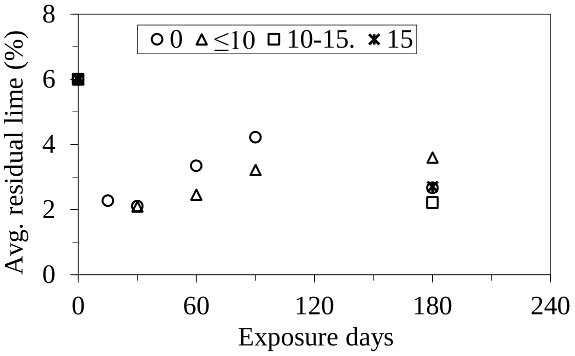

Fig. 3b Residual lime content in bi-directionally (C2) exposed S1 (40% bentonite) soil samples across depth

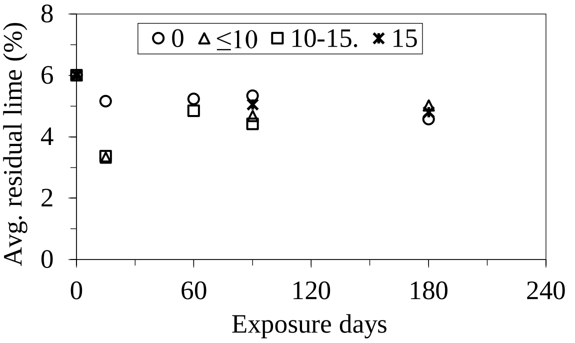

Fig. 4a Residual lime content in uni-directionally (C1) exposed S3 (60% bentonite) soil samples across depth

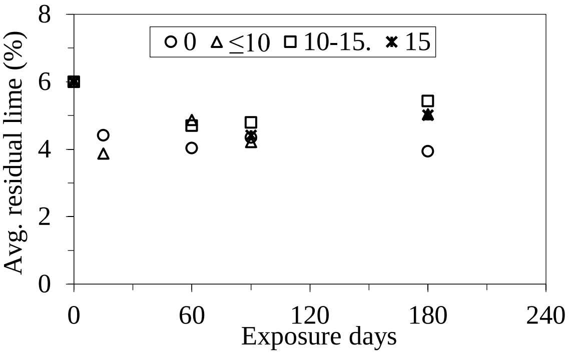

Fig. 4b Residual lime content in bi-directionally (C2) exposed S3 (60% bentonite) soil samples across depth

$$Ca(OH)_2\,(aq) + CO_2\,(aq) \leftrightarrow Ca^{2+}\,(aq) + 2(HCO_3^-)\,(aq) \quad (6)$$

Fig. 5 (a-b) and 6 (a-b) shows the variation of carbonate content across the depth of the exposed samples. Observations suggest an overall increase in carbonate concentration in all the samples. Concentrations in ‘C2’ samples were found to be higher than 'C1' which are in line with the exposure conditions used in the study (two directional vs unidirectional exposure respectively). Continuous dissociation of (HCO_3^-) in presence of CO_2 (g and/or aq.) as explained in Eq. (7) may be causing the observed increase in (CO_3^{2-}) concentrations, leading to increase in $CaCO_3$ concentrations.

$$[\mathbf{HCO_3^-}](\mathbf{aq}) \leftrightarrow \mathbf{H^+}(\mathbf{aq}) + [\mathbf{CO_3^{2-}}](\mathbf{aq}) \quad (7)$$

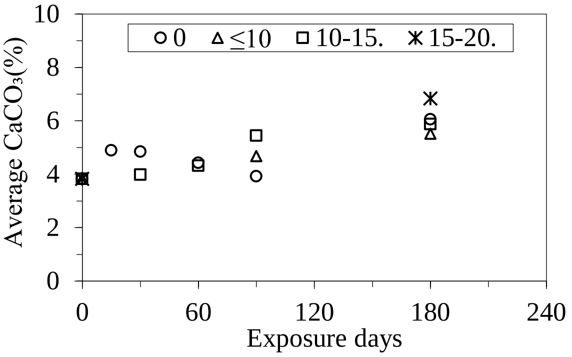

Fig. 5a Average $CaCO_3$ content in uni-directionally (C1) exposed S1 (40% bentonite) soil samples across depth

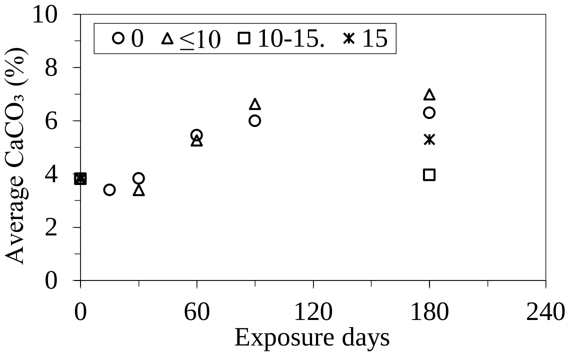

Fig. 5b Average $CaCO_3$ content in bi-directionally (C2) exposed S1 (40% bentonite) soil samples across depth

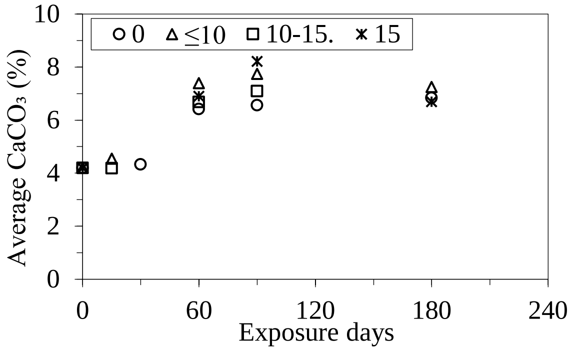

Fig. 6a Average $CaCO_3$ content in uni-directionally (C1) exposed S3 (60% bentonite) soil samples across depth

The coefficient of determination (R^2) values were observed to vary from 0.4446 to 1 and from 0.3824 to 1, for changes in average $CaCO_3$ and residual $Ca(OH)_2$ content respectively.

Unconfined compressive strength (UCS) changes used in the study are included in Fig. 7.

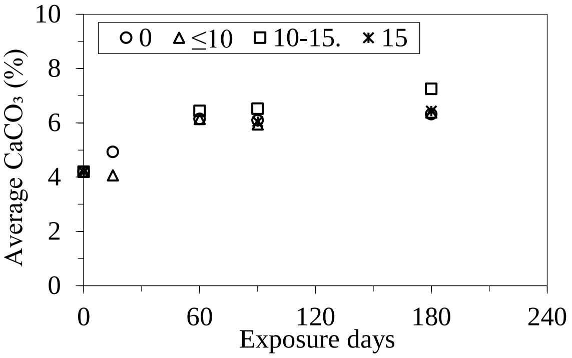

Fig. 6b Average $CaCO_3$ content in bi-directionally (C2) exposed S3 (60% bentonite) soil samples across depth

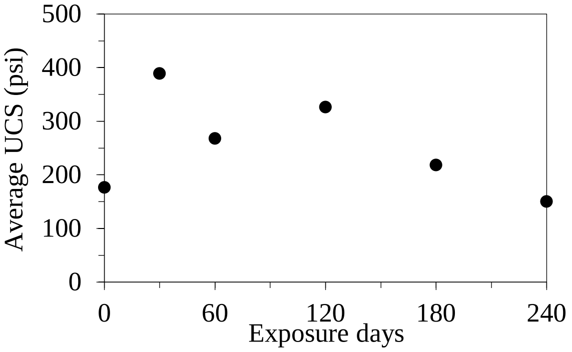

Fig. 7a Average unconfined compressive strength (UCS) of lime stabilized 40% bentonitic soil (S1) samples exposed to atmosphere

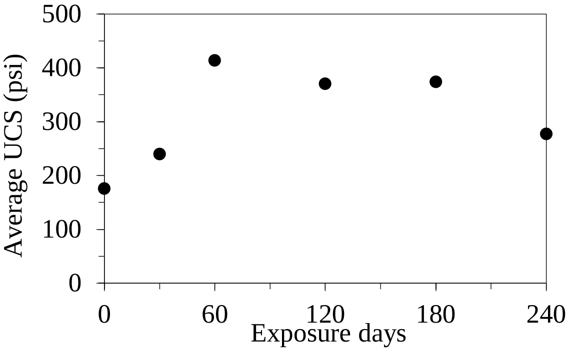

Fig. 7b Average unconfined compressive strength (UCS) of lime stabilized 60% bentonitic soil (S3) samples exposed to atmosphere

Observations also show that the strength properties of mixes deteriorate once exposed to atmospheric conditions, possibly due to formation of carbonate phases in the mix. The rate of deterioration was found to be more pronounced in mixes with a lower clay content (S1). Since the S1 fraction was found to have a lower concentration of $CaCO_3$, slower pozzolanic reactions in these mixes are causing a lower strength during initial curing period. The slower long-term strength gain observed in the mixes is possibly due to lower amounts of lime available for pozzolanic reactions in the matrix (Fig. 3-6). For compressive strength observations of lime stabilized soil, the values of the coefficient of determination (R^2) were found to be 0.6658 and 0.862, for S1 (40% bentonitic soil) and S3 (60% bentonitic soil) respectively.

Since the higher void fraction in these mixes (due to a larger sand fraction) can facilitate the diffusion of carbon dioxide into the matrix, a possible conversion of cementing phases (calcium silicate and aluminate hydrates) to lower strength silicious gel due to carbonation reactions can also be a possible reason for strength reduction in these mixes [48, 49]. Micromorphological impact of atmospheric conditions on lime stabilized soil with respect to changes in carbon content and Ca/Si ratio is shown in Figure 8.

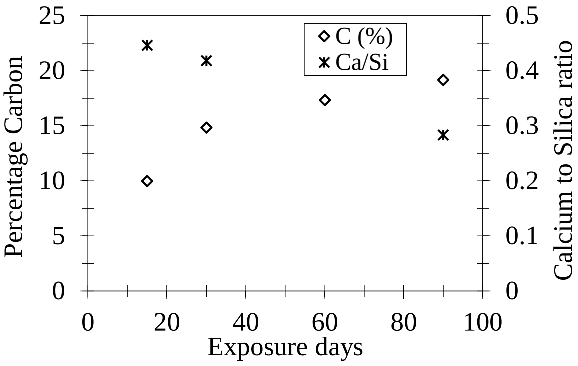

Fig. 8 Changes in carbon content and calcium to silica ratio in lime stabilized soil during atmospheric exposure

Observations in Fig. 7 suggest that exposure to carbon dioxide environment can be detrimental to the long-term strength of stabilized mixes. Decalcification in the matrix as observed in Fig. 8 is indicative of degradation of reaction products in the matrix which essentially results in deterioration of strength as observed in Fig. 7. Long term exposure to carbon dioxide environments can result in further reduction in Ca/Si ration and translates the cementing materials to amorphous and non-cementitious products like polymerized silica (Si-OH) gel. Since carbonation of residual lime can start during early ages of curing period, delays in construction of overlying hot mix asphalt (HMA) layers can result in extended interaction of stabilized layers with atmospheric CO_2 which in turn can affect the long-term strength/performance of the constructed pavement sections. The concentrations of various species identified in this research are based on weight percentages obtained using thermogravimetric technique. However, the engineering properties of stabilized mixes can vary with the polymorphs of these precipitated phases which cannot be estimated based on the current approach. Hence further research on identifying the concentrations of individual polymorphs is critical in developing a carbonation model for stabilized soil matrices. In addition, the observation in the study needs to be validated based on field studies.

5. CONCLUSION

Summarizing the observations, the conclusions are as follows:

I. Thermogravimetric technique can estimate the residual lime contents and the concentrations of carbonate phases in stabilized mixes.
II. Observations suggest that the rate of strength deterioration in stabilized mixes due to carbonation is more pronounced in mixes with lower clay contents.
III. In addition to carbonation reactions, breaking down of cementing phases in stabilized mixes due to carbon dioxide exposure may also be contributing to strength loss in cementitious mixes when mixes are exposed to environmental conditions.

6. ACKNOWLEDGMENTS

We sincerely thank Transportation Laboratory and Advanced Materials' Characterization Laboratory (Transportation Division) of IIT Kanpur for extending all the help and facilities for completion of this work.

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