Optimum Science Journal

Journal homepage: https://optimumscience.org

Original Research Article

Mitigating Metastability of Calcareous Soil Using a Sodium Silicate Solution

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ARTICLE INFO

Received 26 February 2025

Accepted 14 April 2025

Available Online 17 April 2025

Keywords:

Metastable soils

Calcareous

Sodium silicate

ABSTRACT

Metastable soils have continued to be a bane to civil engineering causing infrastructural damages hosted on them and resulting in both economic and lives losses. To mitigate metastability or collapsibility of a calcareous clayey soil, a suitable sodium silicate solution was formulated under a rigorous process in a preliminary investigation to ascertain an optimal solution by altering silica/sodium ratios and concentrations. However, treatment was unsuccessful for samples containing greater than 12 % calcite content. The optimal solution was then applied in the main study, to treat samples by impregnation of pre-formed oedometer specimens. Impregnation (a non-destructive means of treatment) was preferred to benefit from the inherent structural bonding of samples. Oedometer specimens treated were basically heat-cured (45 to 50 ^{0}C) for twenty-four hours as informed by the preliminary study. Collapse potential was estimated by the percentage reduction in height of a specimen due to wetting at 300 kPa overburden stress. A 5% acidic solution and distilled water were used as wetting fluids to examine the effect of pore fluid pH on metastability. It was found that at 300 kPa wetting pressure, silicatization (silicate treatment) and heat-curing reduced collapse potential of samples by 70 % to 78 % under acidic solution wetting and 59% to 73% under distilled water wetting. Therefore, it is concluded that a well formulated and optimised sodium silicate solution can mitigate metastable calcareous soils without additives. However, setting of treated samples occurs only after heat-curing.

To cite this article: Opukumo, A. W. & Oki, A. O. (2025). Mitigating metastability of calcareous soil using a sodium silicate solution. *Optimum Science Journal*, Issue, pp-pp. http://doi.org/10.5281/zenodo.15225663

1. Introduction

Calcareous soils are defined by their calcium carbonate ($CaCO_3$) content (Thompson, 2007). In Kishchuk (2000), calcareous soils have been marked as any soil that contains a significant amount of $CaCO_3$ and grouping these into

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E-ISSN: 3023-817X •2025 HASON Publishing

weakly calcareous (< 5%), *moderately calcareous* (5–15%), *strongly calcareous* (15–25%), *very strongly calcareous* (25–40%) and *extremely calcareous* (> 40%). With this definition, a large range of soils widely distributed globally can be called calcareous, with arid and semi-arid climates supporting more of their production (Houston *et al.*, 2001). For instance, there are extensive occurrences of these soils in the many regions of the Middle East, Australia, France, Switzerland, Canada, U.S.A, U.K. (Mansour *et al.*, 2008; Milodowski *et al.*, 2015; Opukumo *et al.*, 2022; Opukumo *et al.*, 2024). Furthermore, carbonation of ordinary Portland cement or lime treated soils precipitates pedogenic $CaCO_3$ and increases the scope of calcareous soils (Washbourne *et al.*, 2012). In the arid part of Botswana, a study carried out on a road base treated with lime revealed conversion of lime to $CaCO_3$ of varying degrees (Bagoniza *et al.*, 1987). Additionally, loessial soils (most common natural collapsible soil) are also calcareous because they contain $CaCO_3$ contents rising to 30 % (Jefferson *et al.*, 2005; Li *et al.*, 2016).

Collapsible soils have honeycomb structure that is supported by $CaCO_3$ bonding that makes them strong and stiff in their in situ unsaturated state, but upon wetting, with or without extra loading, the honeycomb structure collapses rapidly and becomes densified (Ayeldeen *et al.*, 2017; Lutenegger & Saber, 1988). These soils are some of the most problematic geomaterials (Houston *et al.*, 2001; Vandanapu *et al.*, 2016) with a global presence, posing structural and geotechnical engineering challenges.

Colossal loses, both lives and economic, have ensued from collapsible soils (Derbyshire, 2001; Houston *et al.*, 2001; Watts & Charles, 2015). However, the expansion of cities and need for construction of new earthwork infrastructure globally, as well as the concept of sustainable construction has made it practically inevitable for collapsible soils being utilized as either host or fill materials (Sakr *et al.*, 2008; Watts & Charles, 2015) despite warnings, e.g., from *code of practice for foundations (BS 8004:1986)* (Watts & Charles, 2015). Because of the geotechnical engineering challenges and losses arising from these problematic soils, extensive research works on their mechanical behaviours and methods of mitigation have been carried out over many decades, although mainly traditional techniques (e.g., use of cement, lime, etc.), with identified environmental drawbacks ranging from large CO_2 emissions from the production of ordinary Portland cement to development of new deleterious minerals arising from reaction of lime and sulphate minerals for instance (Gadouri, 2023; Gadouri & Mezian, 2023; Iranpour, 2016; Jefferson *et al.*, 2005; Rafalko *et al.*, 2007). However, Sokolovich (1965) identified a technique called "silicatization" (Equation 1) that is environmentally friendly with the potential to improve some types of collapsible soil with the help of additives. This technique needed further research on, but little has been done. Equation 1 shows the resultant cementitious product of a chemical reaction of calcium ions (from calcium carrying soil) and a solution of sodium silicate.

$$\mathrm{AC}\left[\begin{matrix}Ca\\Ca\\Ca\end{matrix}\right. + 3\mathrm{Na_2SiO_3} + 3H_2\mathrm{O} \rightarrow \mathrm{AC}\left[\begin{matrix}Na\\Na\\Na\\Na\\Na\\Na\end{matrix}\right. + \left[\begin{matrix}Ca(OH)_2.\ SiO_2\\Ca(OH)_2.\ SiO_2\\Ca(OH)_2.\ SiO_2\end{matrix}\right. \tag{1}$$

Furthermore, there is a gap in the literature related with the comparative effect of wetting fluid pH on collapse of both pretreated soils and their post-treated counterparts as well as the potential of a sodium silicate solution without additives. Also, for geotechnical characterization of soils, focus has largely been on mechanical behaviour with a disregard for chemical properties, which could cause a misunderstanding or misinterpretation of the comprehensive engineering behaviour of some soils, e.g. calcareous soils whose $CaCO_3$ component may dissolve and impact their mechanical behaviour (Lamas *et al.*, 2002; 2011; Opukumo *et al.* 2024). This investigation explores the potential benefits of a well formulated Na_2SiO_3 solution, with no additive, for mitigating the collapse potential (CP) of silty clay calcareous soils. In addition, is investigating the effect of the pH of the collapse triggering fluid.

2. Material and Method

2.1 Materials

Soil materials investigated were artificial loose-structured calcareous clays manufactured as in Opukumo *et al.* (2022). Soils were manufactured into cylindrical test specimens and their CPs were measured and reported in Opukumo *et al.* (2024). They are regarded here as pre-treatment samples (see Table 1 below). Duplicates of those samples were treated with a formulated Na_2SiO_3 solution adjudged most suited, and CPs investigated post-treatment. This formulation was of a silica to sodium (Si/Na) ratio of 3, and a molarity, M of 4. A summary of the formulation and selection procedures are reported in the foregoing section.

Table 1. Pre-treatment samples composition and physical characteristics including collapse potential (adapted from Opukumo *et al.*, (2022); (2024)).

Sample ID	**% Quartz silt**	**% Kaolin clay**	**Clay/ silt ratio**	**% $CaCO_3$**	**Degree of calcareousness**	**Particle density, ρ_s**	**Specimen height *(mm)***	**Dry density *(Mg/m^3)***	**Collapse potential, CP at 300 kPa**	
									Distilled water wetted	**5% Acid solution wetted**
A1	45	45	1	~ 12	Moderately	2.66	19	0.93	20.2+0.33	25.2+0.19
B1	31	59	1.9	~ 12	Moderately	2.64	19	0.93	18.8+0.49	23.5+0.79
C1	18	72	4	~ 12	Moderately	2.63	19	0.93	17.0+0.19	21.9+0.53

2.2 Methods

2.2.1 Formulation of a suitable sodium silicate solution and implementation

The implication of Hurley and Thornburn (1971)'s research findings is that, based on soil composition, a Na_2SiO_3 solution with distinctive properties would need to be formulated to suite the treatment of each soil. Workability is one other factor to be considered. Thus, following Hurley and Thornburn (1971), solutions of differing concentrations and SiO_2:Na_2O (Si/Na) molar ratios were formulated, and examined at a pilot stage to ascertain the most suiting and effective to improve the different samples utilizing a non-destructive technique such as absorption.

A commercial Na_2SiO_3 solution with a Si/Na molar ratio of 1.71 supplied by *Ingessil S.r.l. Silicate Industry, Italy* was utilized alongside a colloidal silica supplied by *Fishers Scientific, UK*. Proportions of the colloidal silica were mixed with fractions of the Na_2SiO_3 solution to raise its silica component thereby increasing the solution's concentration and molar ratio to different levels. Table 2 presents three types of solutions (original and formulated) investigated at differing concentrations by trial-and-error approach. This was done as a preliminary study that adopted the impregnation method. Specimens were half-soaked in the solution and allowed to uptake solution by capillary rise in one-dimension to prevent air entrapment. Individual specimens (in oedometer rings) were placed in a small ceramic dish (see Figure 1) with a porous stainless disc sited at the base of specimen. The solution was afterward gently poured into the ceramic dish and specimen allowed time to progressively absorb from the bottom to the top until full saturation was observed as shown in Figure 1 (a, b and c).

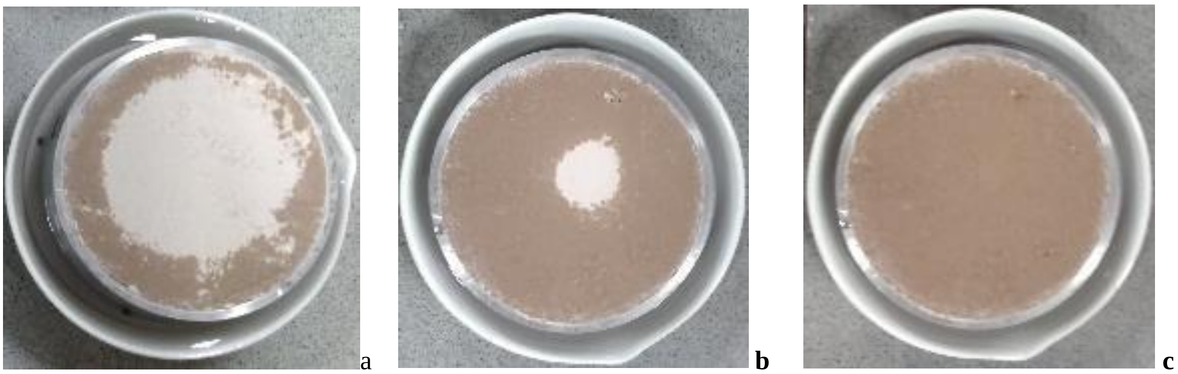


Figure 1. Solution uptake in oedometer specimens: a, b and c are different stages of the uptake process.

Table 2. Sodium silicate (Na_2SiO_3) solutions formulated and assessed for suitability at pilot scale (treatment by impregnation).

SiO_2:Na_2O (Si/Na) Ratio	**Concentration (*M*)**	**Density *(g/ml)***	**Remarks**
1.71*	5.52	1.54 (original)	Solution too viscous to be absorbed by any sample.
	3.20	1.32 (diluted)	A1 specimens treated. Setting has only occurred after ~ 48 hours. Significant shrinking and cracking observed.
	4.80	1.47 (diluted)	A1 and C1 specimens treated. Setting has only occurred after ~ 48 hours. No cracking, but significant shrinking observed.
3.01	5.20	1.47 (formulated)	Solution too viscous to be absorbed by any sample.
	2.00	1.18 (diluted)	C1 specimens treated. Setting has occurred at ~ 24 hours. Significant cracking observed.
	3.00	1.28 (diluted)	C1 specimens treated. Setting occurred earlier than 24 hours. Specimens significantly cracked and broke.
	3.50	1.32 (diluted)	Solution too viscous to be sufficiently absorbed.
	4.00	1.36 (diluted)	C1 specimens treated. Setting occurred earlier than 24 hours. No shrinking, but minor cracking only on the top face. No visible cracking where the top face has a direct contact with solution.
3.40	5.10	1.45 (formulated)	Solution too viscous to be absorbed by any sample.
	4.00	1.36 (diluted)	Solution too viscous to be absorbed by any sample.

The solution with Si/Na=3, M=4 was adjudged best suited following the preliminary studies. Physical observation of degree of shrinkage and crack occurrence post-drying was adopted to define most suitable solution type to be implemented in the main treatment regime (see few examples in Figure 2). This was essential because of the adverse effects of specimen shrinking (syneresis) and fracturing, which are common properties of soils stabilized with certain sodium silicate formulations.

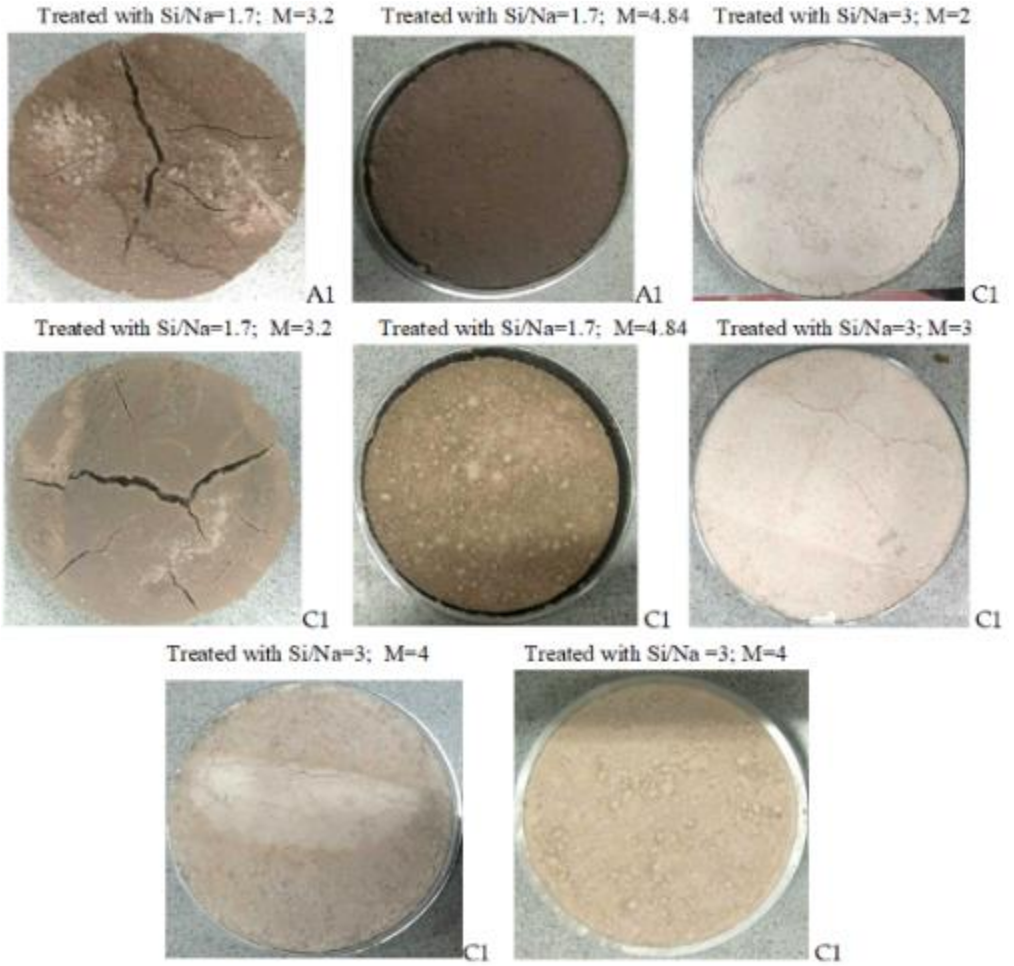


Figure 2. Observed specimens surface consistency post-treatment (impregnation by absorption), setting, and 2 days oven-drying at 45 -50 ^{0}C

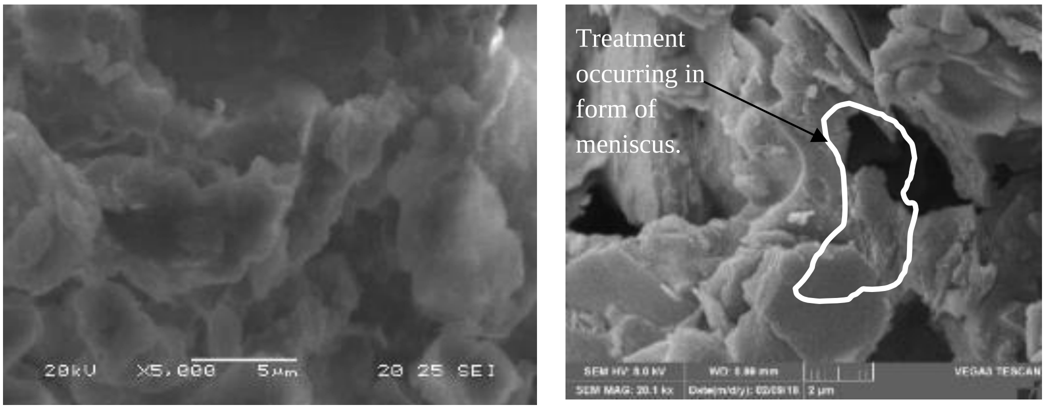
The major disadvantage with this technique was the difficulty to control both the distribution of absorbed solution and its quantity. It was presumed that a complete saturation may possibly mean an even distribution. However, there is the likelihood that the concentration of the solution could change along its absorption course. However, this was not investigated.

In all, as absorption progressed, more solution was provided until complete saturation was achieved. Thereafter, treated specimens were weighed and allowed to set under ambient conditions for about 24 hours before oven-drying for an additional 24 hours at temperatures between 45 to 50 ^{0}C. Setting was taken as the initial gel time, i.e., when the formation of a firm gel was achieved. This was assessed with a gentle finger press down.

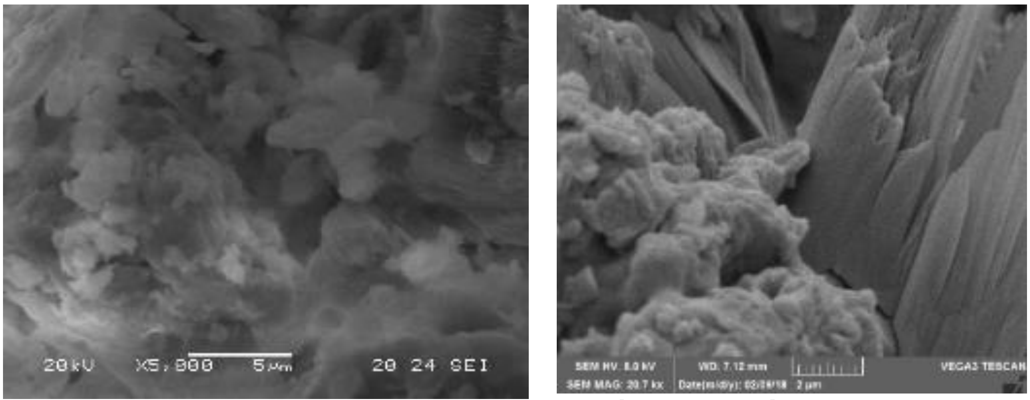
Whereas the literature reports Na_2SiO_3 solutions with densities of 1.10 to 1.15 g/ml as acceptable for treating specific collapsible loessial soils (Jefferson *et al.*, 2005; Sokolovich 1976), those treatments were merely successful when enhanced with certain additives like formamide or ammonia solutions and in some other cases, an alternating injection of carbon dioxide and a sodium silicate solution. The present treatment approach employs a single-step (non-additive) treatment method where only Na_2SiO_3 solution was utilised. Consequently, only solutions with greater densities (> 1.3 g/ml) were effective.

2.2.2 Microstructure of treated specimens

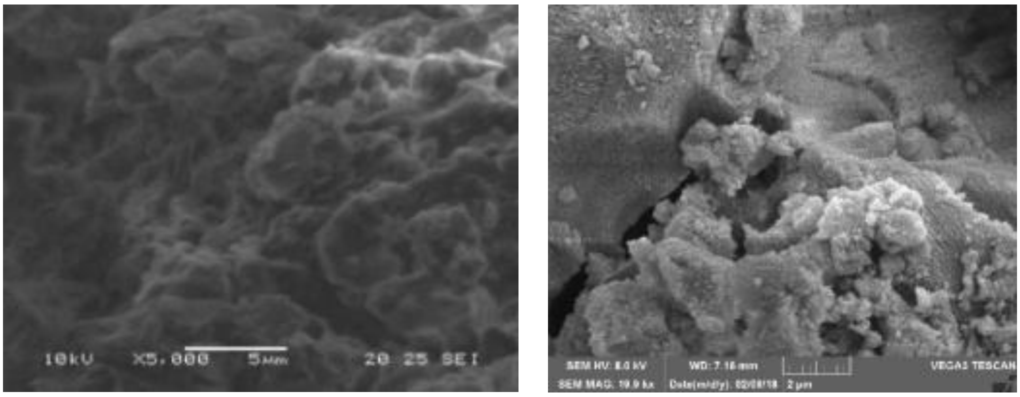
The microstructure of a few treated specimens has also been examined under the scanning electron microscope (SEM). See SEM images in Figure 3 below:



Sample A1 – untreated *Sample A1 – treated*



Sample B1 – untreated *Sample B1 – treated*



Sample C1 – untreated *Sample C1 – treated*

Figure 1. SEM Micrographs of samples treated with Na_2SiO_3 solution (Si/Na=3, M=4) and oven-dried compared with untreated counterpart.

As expected, the SEM micrographs in the Figure 1 above appear to suggest that treatment by impregnation could have reasonably preserved the original structural framework of specimens with the absorbed solution sticking to the surfaces of particles as well as filling micropores. Probably, the crystalline calcite structure, which provides the initial stability was preserved and might likely positively contribute to the specimen post-treatment strength properties. It could be observed from micrographs of samples A1 and B1 that huge voids were preserved post-treatment, remarkably in sample A1 with greater silt content. The solution could be seen creating menisci bonding at particles contacts, bridging them together. In specimens where original large voids were minimal, the menisci bonding material contributed to building a dense mass structure, as is obvious in samples C1. Clearly, the infilling of micropores by the silicate or its by-products will lead to a decreased porosity and permeability, thereby improving strength and reducing metastability.

2.3 Collapse Testing Methods

The single oedometer and double oedometer procedures of the one-dimensional oedometer system implemented in Opukumo *et al.* (2024) were replicated using both distilled water and a 5% acetic acid solution as wetting fluids. A wetting stress of 300 kPa was adopted and collapse potential (CP) estimated following Equation 2 (ASTM, 2003).

$$CP = \frac{\Delta H}{H_0} x\, 100 \qquad (2)$$

where CP is collapse potential, ΔH is change in specimen height resulting from wetting, H_0 is specimen height before wetting. The degrees of severity of collapse were distinguished in accordance with **Hata! Başvuru kaynağı bulunamadı.**.

Table 3. Collapse percentage as an indication of severity (Bell & Culshaw, 2001).

Collapse (%)	Severity of Problem	Abbreviation
0–1	No trouble	NT
1–5	Moderate trouble	MT
5–10	Trouble	T
10–20	Severe trouble	ST
> 20	Very severe trouble	VST

3. Results and Discussions

In general, in ground improvement of metastable or collapsible soils, efforts have largely been directed at increasing strength, e.g., Jefferson *et al.* (2005); Iranpour (2016). However, this study has rather targeted CP and results presented in one standard deviation of the mean values. Samples exhibiting worst-case collapse potential (*'very severe trouble (VST)'*) have been treated and tested, utilizing distilled water and the acid solution as wetting fluids in the single oedometer system while only the acid solution utilized in the double oedometer (presoaked) system. Wetting was applied at a vertical stress of 300 kPa in the single oedometer and CP values had estimated standard deviations of 0.1 to 0.3.

Due to the sticky nature of the Na_2SiO_3 solution, at the end of each testing, both oedometer ring and porous discs were observed to stick together with tested specimen and became difficult to disassemble these components. In many cases, efforts to detach them caused breaking of porous discs while in other cases porous discs had to be soaked for several days and sticking soil scratched hard to remove. This resulted to damage of equipment and therefore further testing was not possible. Sadly, this limited the number of treated/tested samples to simply those which exhibited *"very severe trouble"* CP. Their properties are given in Table 4 below:

Table 4. Properties of sodium silicate treated oedometer specimens (moderately calcareous)

Specimen ID	**Clay:Silt ratio**	**$CaCO_3$ content** *(%)*	**Mean dry mass of pretreated specimens** *(g)*	**Wet mass post treatment** *(g)*	**Dry mass post oven drying** *(g)*	**Dry density** *(Mg/m^3)*
A1	1	~12	81.37	153±0.7	130±3	1.51±0.02
B1	1.9	~12	81.52	151±1.6	124±3	1.45±0.02
C1	4	~12	80.95	152±1.4	125±2	1.46±0.02

After treatment and curing, specimens increased significantly in dry weight, ranges between 52% to 60% were recorded, with sample A1 recording highest. These dry weight improvements have followed the quantity of the Na_2SiO_3 solution absorbed by each specimen, which possibly is a function of the samples' moisture holding capability in terms of available pore spaces. According to Table 1 which shows properties of untreated samples, A1, B1, and C1 are in a decreasing order of porosity and void ratio as could also be seen from their open structure in the SEM images in Figure 3. However, a key setback of the treatment method was lack of effective control of excess solution adhering to the surfaces of specimens. As a result, there were indiscriminate quantities of treatment solution held by specimens, bringing about a lack of clear understanding of the control of the samples' physical properties on absorbability. With volume remaining constant and mass increasing, dry density increased from about 1.0 Mg/m^3 to around 1.5 Mg/m^3, i.e., nearly 50% increase in dry density was created. Mechanically, this might possibly improve strength and stability (Jefferson *et al.*, 2005). The stress-deformation relationships of these treated samples under the single and double oedometers comparing with those of their untreated counterparts are presented in Figure 4 to Figure 9. A summary of their CP values is shown in Table 5.

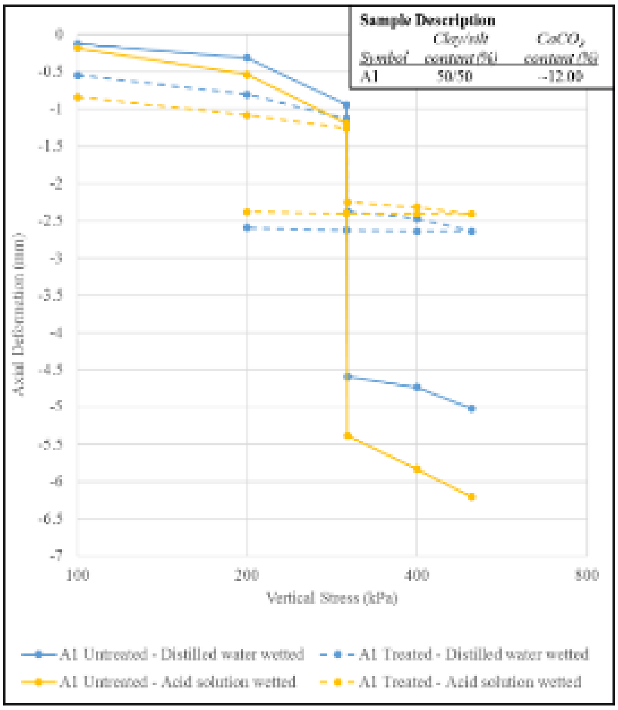


Figure 4. Single oedometer test, comparison of untreated and treated A1

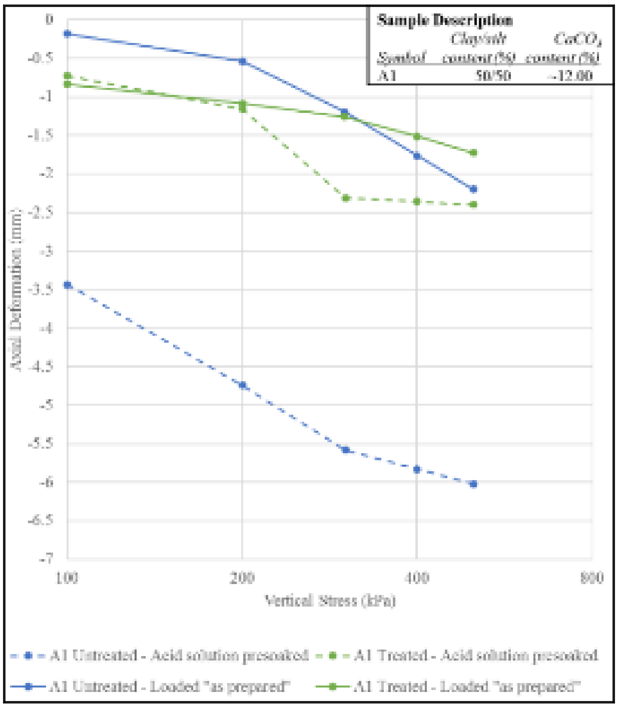


Figure 5. Double oedometer test, comparison of untreated and treated A1

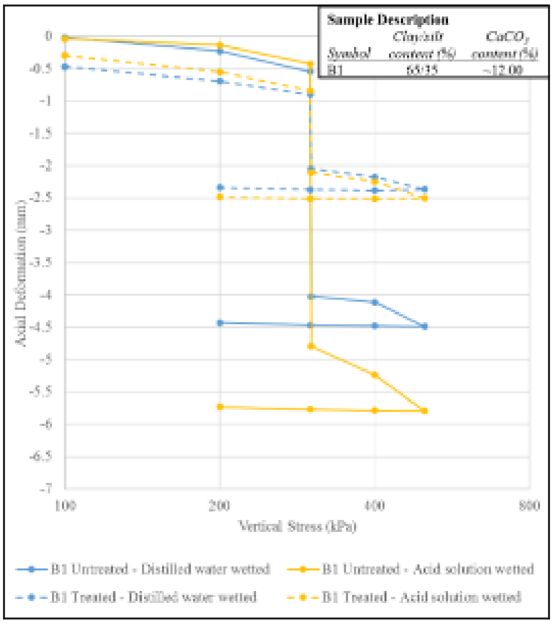


Figure 6. Single oedometer test, comparison of untreated and treated B1

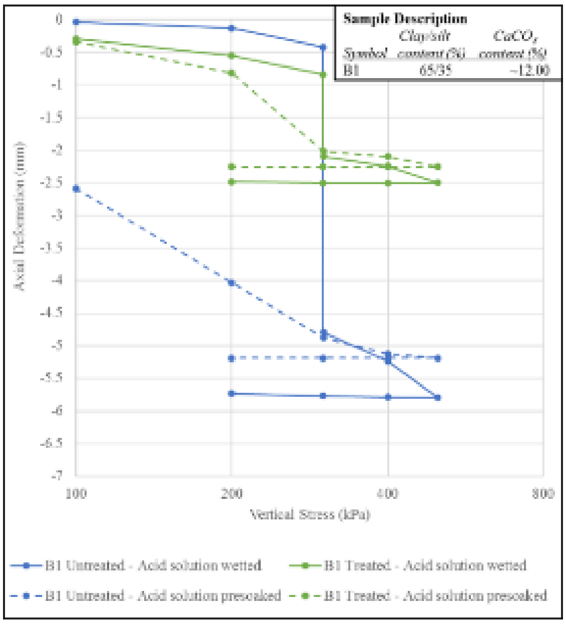


Figure 7. Double oedometer test, comparison of untreated and treated B1

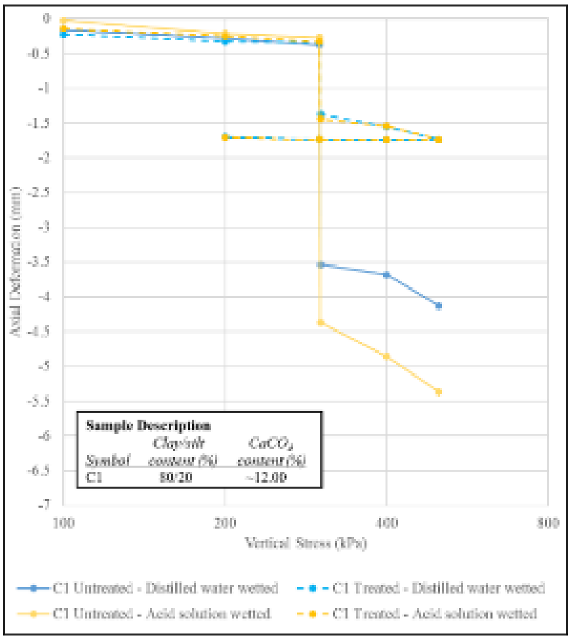


Figure 8. Single oedometer test, comparison of untreated and treated C1

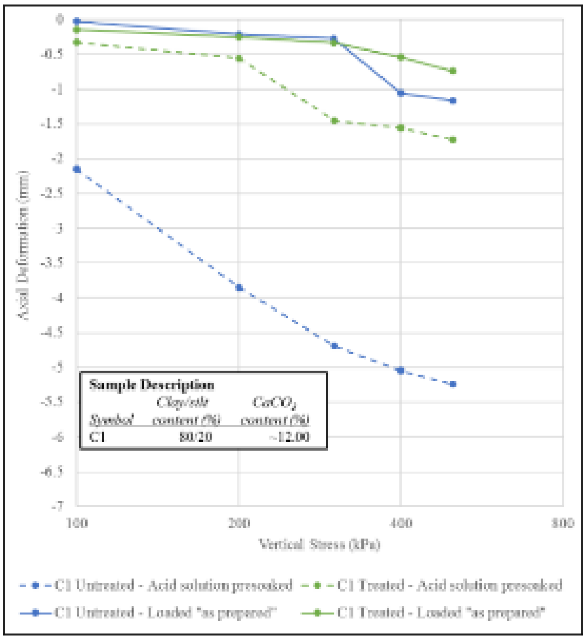


Figure 9. Double oedometer test, comparison of untreated and treated C1

Table 5. Treated specimens - deformability during loading, CPs and degree of severity.

Sample	Initial specimen height *(mm)*	**Distilled water wetted (single oedometer)**				**5% acid solution wetted (single oedometer)**			
		Height pre-wetting *(mm)* (at 300kPa stress)	Height at end of collapse *(mm)* (distilled water wetted)	CP *(%)* (distilled water wetted)	Degree of severity	Height pre-wetting *(mm)* (at 300kPa stress)	Height at end of collapse *(mm)* (acid solution wetted)	CP *(%)* (5% acid solution wetted)	Degree of severity
A1	19	17.9	16.6	7.0	T	17.7	16.7	5.6	T
B1	19	18.1	16.9	6.4	T	18.2	16.9	6.9	T
C1	19	18.7	17.6	5.5	T	18.7	17.6	5.9	T

	CP *(%)* at different vertical overburden stresses, from acid solution presoaked (double oedometer)					
	At 100kPa	*Degree of severity*	*At 200kPa*	*Degree of severity*	*At 300kPa*	*Degree of severity*
A1	-0.6	NT	0.4	NT	5.9	T
B1	0.3	NT	1.4	MT	6.5	T
C1	1.0	NT	1.6	MT	6.0	T

Treated specimens similarly became denser than their untreated counterparts and appearing stiffer under loading; only compressing marginally at pre-wetting loading stages. Although specimens with lower clay contents absorbed slightly higher amounts of the treatment solution, their pre-wetting deformations and CPs at 300kPa (SOT) stayed greater with A1 > B1 > C1, comparable behaviour to their untreated counterparts.

On the contrary, CPs assessed at different wetting stresses from the double oedometer testing proved otherwise. At both 100 kPa and 200 kPa wetting stresses, CP values were fairly higher with increasing clay content while at 300 kPa, CP values did not align with any trend. The pre-soaked curves have shown a noteworthy deformation pattern at every stress increment. A fairly steady amount of deformation is seen at each stress increment except for 300 kPa where an inconsistent deformation occurred. This is an indication of a probable stress threshold at which collapse is maximum for these specimens. Nevertheless, it cannot be confirmed as it was not examined further. The near zero gradient of the rebound curves proves a permanence in the deformations that occurred.

The control of wetting fluid type on the magnitude of collapse observed seemed to be similar. Apart from a potential increase in strength resulting from the treatment, treated specimens additionally exhibited moisture-repelling behaviour. At the end of the oedometer wetting period only a part of the specimen became saturated. In single oedometer tests, wetting lasted for around 26 hours while it was about 28 hours soaking in the double oedometer.

Results displayed in Figures 10 and 11 demonstrate significant positive impact of the treatment regime in improving collapse properties of the soils. In Figure 10, single oedometer (at 300 kPa wetting stress) results of both the distilled water and acidic solution wetting conditions generally showed mitigation levels approximating between 59% to 73% and 70% to 78%, respectively.

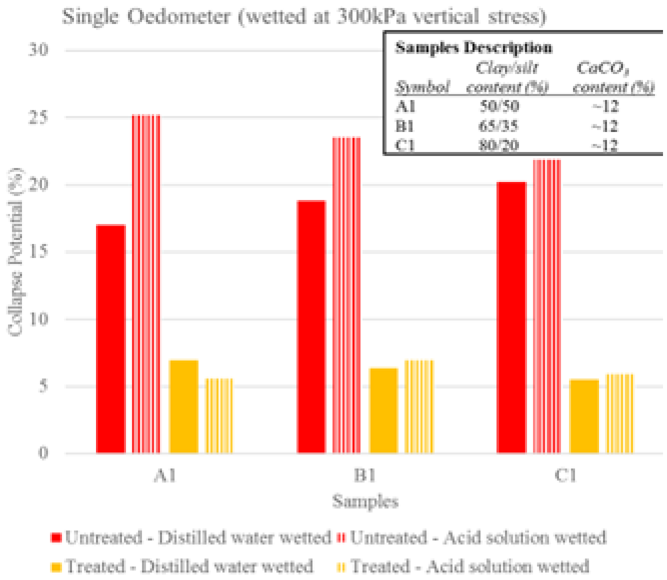


Figure 10. Calcareous silty clay collapses mitigating effect of a Na_2SiO_3 solution tested under distilled water and acidic solution wetting conditions of the single oedometer.

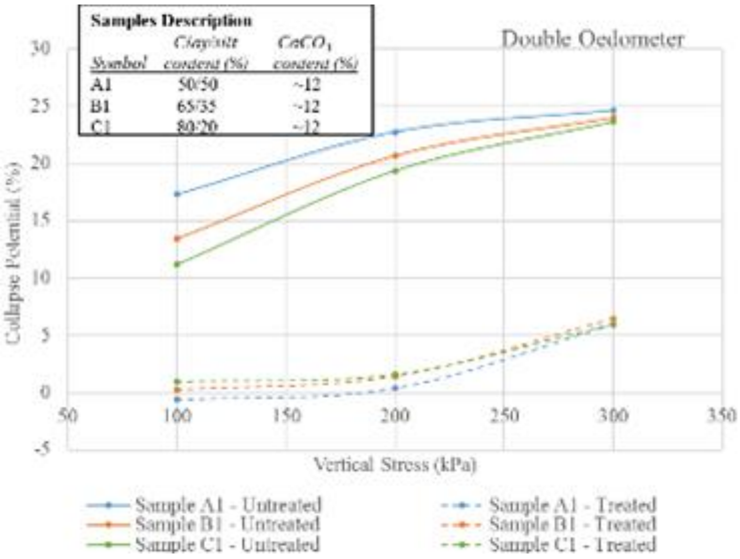


Figure 11. Calcareous silty clay collapses mitigating effect of a Na2SiO3 solution tested under distilled water presoaked (double oedometer).

The double oedometer results (distilled water pre-soaked) in Figure 11 revealed around 73% to 75% mitigation effect at a 300 kPa wetting stress but reaches to between 91% to 98% at 100 and 200 kPa stresses. In one case (sample A1 treated), some swelling was noted at a 100 kPa stress.

As mentioned earlier, the reaction of Na_2SiO_3 with Ca (calcium) is an ionic exchange reaction understood to precipitate water-resistant (insoluble) silicate sheaths that surround the soil particles, producing cementation effect (Hurley & Thornburn, 1971). While Na_2SiO_3 treated specimens remained collapsible, treatment has obviously reduced the degree of severity from '*very severe trouble*' to '*trouble*' under acidic solution wetting, and from *'severe* *trouble'* to *'trouble'* under distilled water wetting, both at 300 kPa wetting stress. Lower than 300 kPa, collapse reduction was from *'very severe trouble'* to *'moderate trouble'* at a 200 kPa and from '*severe trouble'* to '*no trouble'* (non-collapsible) at a 100 kPa.

Results in Figure 10 clearly showed a greater damaging impact of distilled water on silicatized (silicate treated) soils than acidic solution. It can also be explained that acidic solution has the potential to reduce the magnitude of collapse obtained of a silicatized soil compared to distilled water. This agrees with Iler (1979) argument that an acidic environment boosts the gelation or polymerization process of sodium silicate treatment. Therefore, perhaps the silicatized specimens showed some further improvement during the test by soaking in the acidic solution. Despite that heat-curing proved the potential to produce stronger bonds than room-curing, Welds & Lange (1969) have argued that it is not an adequate process to obtaining complete insolubility of silicatized soils. Welds Lange (1969) also reported that curing by polymerization better creates insolubility. This could explain why the treated specimens remained collapsible at some levels of overburden stresses. Nevertheless, it is also common knowledge that at higher shear stresses beyond the bonding strength of a soil, bond breaking happens, and densification becomes apparent upon wetting and/or loading.

4. Conclusions

From the results of this study, it is concluded that a well-designed formulation of a sodium silicate (Na_2SiO_3) solution (defined in terms of concentration and SiO_2:Na_2O ratio) mitigates the metastability of calcareous soils even in a 'single step' (non-additive) application. Nevertheless, there is an indication that the presence of an acid enhances its effectiveness as revealed by the acid solution wetted collapse testing results.

Sodium silicate treatment reduces calcareous soils' susceptibility to wetting by both distilled water and acidic solution. However, specimens still displayed some level of metastability, possibly due to their original low-dry density and ductility of the cured silicate solution. Collapse mitigation stood between 59% to 73% under distilled water wetting and 70% to 78% under AS wetting.

The effectiveness of sodium silicate solution in soil improvement is controlled by its concentration and SiO_2:Na_2O molar ratio. Other factors include curing time and method. These factors influence the rate and type of silicate bond formation and the specimen's syneresis (i.e., moisture loss and shrinking).

Lastly, it was concluded that for an effective improvement to take place with sodium silicate treatment, a quantity of solution exceeding that which satisfies the chemical equilibrium of reactants and products is required. Thus, it is recommended that a full-scale field application of sodium silicate should be preceded by a laboratory treatability investigation and a field trial.

Declaration of Competing Interest and Ethics

The authors declare no conflict of interest. This research study complies with research publishing ethics. The scientific and legal responsibility for manuscripts published in OPS Journal belongs to the authors.

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