Durability and Permanency Studies in Sulfate-Laden Soils Treated with Nano- and Crystalline Silica-Based Admixtures

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Abstract: A research study was performed to assess the durability and permanency of chemical treatment in sulfate-rich expansive soils with novel silica-based admixtures and calcium or Ca-based stabilizer. Crystalline Silica (CS) and Nanosilica (NS) admixtures were selected and used with a traditional dolomitic-hydrated lime to treat problematic high-sulfate expansive soils. The chemically treated soils were investigated for the changes in plasticity at different curing periods, and subsequently, strength tests were performed before and after subjecting the soil specimens to moisture conditioning through capillary soaking. The values of strength, volumetric swell strains, and weight changes after moisture conditioning were compared with those of control specimens. To study the permanency of treatment method, leaching studies were performed using internal flushing on the chemically treated soils to simulate the moisture ingress/digress into subsoils. The effects of different silica-based admixtures with lime were analyzed after 7 leaching cycles. Test results indicated that the normalized specific surface area (SSA_N) of 1.05 with CS phases has better moisture susceptible durability and long-term permanency as compared to NS treatment (SSA_N of 0.70) or traditional treatment methods. Overall, the paper provides a comprehensive understanding of the durability and permanency aspects of the novel treatment techniques, which may be of enormous benefit to geotechnical and transportation practitioners in enhancing the durability of chemical treatments. **DOI: 10.1061/JMCEE7.MTENG-16456.**

Introduction and Background

Calcium or Ca-based stabilizers have been successfully used over the past several decades to improve the performance of expansive soils for supporting transportation infrastructures (Akula et al. 2021; Al-Mukhtar et al. 2010; Biswas et al. 2021a; Little et al. 2000; Nelson and Miller 1992; Puppala and Pedarla 2017; Samridh et al. 2023). The presence of sulfate-bearing phases, however, subjects the treated soil prone to sulfate-induced heaving due to the formation of the calcium-alumino-sulfate-hydrate mineral known as ettringite (Consoli et al. 2019; Herbert et al. 2009; Hunter 1988; Jang et al. 2021, 2022b; Puppala et al. 2005). The precipitation and growth of ettringite mineral results in major infrastructure distress and subsequently amount to millions of dollars of annual infrastructure maintenance and rehabilitation costs (Jang et al. 2022a; Puppala et al. 2003b). Several studies that have attempted to effectively treat such problematic sulfate-rich soils suggested that cost-effectiveness and durability are the key components that still need to be addressed for using Ca-based treatments (Biswas et al. 2021b; Kota et al. 1996; Nair and Little 2009).

The strength and hydraulic properties of treated soils are affected by moisture intrusion, leaching, presence of organic matter, and carbonation (Aldaood et al. 2015; Harris et al. 2009; Le Runigo et al. 2011; Vitale et al. 2021). Past research studies have indicated a strength loss of up to 40% or higher in stabilized soils when exposed to moisture conditioning during early curing stages (Little 1996; Little and Nair 2009; Thompson and Dempsey 1969). An increase in the saturation levels ($S_r \approx 90\%$) due to soaking could be attributed as a major factor for the deterioration of the engineering performance (Dumbleton 1962; Thompson 1970). The depreciation in engineering performance reduces appreciably with a longer curing period due to the formation of a strong binding matrix and a corresponding reduction in the moisture affinity (Chakraborty and Nair 2018; Thompson 1970). However, the hydrophilic nature of the precipitated cementitious compounds could often be responsible for the strength loss incurred in the treated soils (Chakraborty and Nair 2020; Mindess et al. 2003). Therefore, several studies concurred that the extent of strength loss depends on the nature of soils, initial compaction conditions, design dosage, and the amount of residual stabilizer after exposure to continuous moisture circulation (Bell 1996; Chittoori et al. 2011; McCallister and Petry 1992).

The permanency of the chemical stabilizers, when subjected to continuous water intrusion, affects the durability and long-term efficacy of treatments (Barenberg 1970; De Bel et al. 2005). The leaching of the stabilizers is responsible for changes in key geomaterial properties, including residual pH, cationic ratios, soil permeabilities, and unconfined strengths (Ghosh and Subbarao 2006; Hara et al. 2008; Thompson 1966). The depreciation in the engineering properties is partially attributed to the decalcification

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Note. This manuscript was submitted on February 9, 2023; approved on May 23, 2023; published online on September 28, 2023. Discussion period open until February 28, 2024; separate discussions must be submitted for individual papers. This paper is part of the ***Journal of Materials in Civil Engineering***, © ASCE, ISSN 0899-1561.

of the cementitious products during leaching (De Bel et al. 2005; Revertegat et al. 1992; Le Runigo et al. 2009). Problems associated with the dissolution of ingredients (Ca^{2+} or Si^{4+}) from the cementitious phases during water circulation have also been reported by a few researchers (Baston et al. 2012; Harris et al. 2002). The leaching of ions has been attributed as a function of the quantity of water in circulation rather than the flow duration (Moghal et al. 2020; Le Runigo et al. 2011). Consequently, the permeability of treated soil is an important component advocating the process and was observed to be maximum for specimens treated at optimum lime dosages (McCallister 1990; McCallister and Petry 1992). Therefore, few researchers advocated designing with a higher lime dosage, such that the loss of stabilizers could be compensated with additional availability of stabilizers (Chittoori et al. 2013; Lequiller et al. 2008). But, several past studies have provided evidence that lime dosage beyond certain limits is often counterproductive due to the absence of appreciable cohesion or friction in the material itself and could cause a significant reduction in long-term strength (Dash and Hussain 2011; Kumar et al. 2007). Therefore, the application of admixtures with Ca-based stabilizers may be considered as a potential alternative for mitigating the problems associated with moisture-susceptible durability and leaching of stabilizers.

Different industrial waste products such as fly ash, kiln dust and blast furnace slags have been traditionally used to improve the performance of chemically treated soils as sources of additional pozzolanic phases of silica and alumina (Behnood 2018; Kogbara and Al-Tabbaa 2011; Moghal 2017; Puppala et al. 2003a; Sargent et al. 2020; Yi et al. 2015). Past studies have provided certain evidence of the improvement in moisture susceptible durability and reduction in leachability in the presence of admixtures due to an enhancement in the precipitation of less soluble cementitious phases (Ghosh and Subbarao 1998; Moghal and Sivapullaiah 2012). Yet, the research community still strives to identify new materials that could potentially improve the moisture-susceptible durability as well as long-term permanency of chemical treatments in high-plastic sulfate-bearing soils.

Quarry fines are crystalline silica-rich waste products generated during the crushing operation of stone blocks in a quarry (Kumar and Hudson 1992; Satvati et al. 2021). Limited use of quarry fines has been reported over the last few decades in different industries (Galetakis and Soultana 2016). The geoenvironmental impacts from the stockpile and disposal of the dust are a considerable cause of concern for the stone industry [Ingalkar and Harle 2017; Lamb 2005; J. T. McKennon, "Method for Producing Enhanced Soil Stabilization Reactions between Lime and Clay Soils due to the Effect of Silica Addition," US Patent No. 5,336,022 (1994)]. The quarry byproducts have been recently gaining traction for application in different engineering applications (Qamhia et al. 2019; Zhang et al. 2019). Some research studies have also shown that silica-rich quarry dust has the potential to improve chemical treatments in plastic soils (Biswas et al. 2021b, 2023; Chakraborty et al. 2022). In recent years, a major research impetus has been focused on the application of nanomaterials for improving the performance of cementitious reactions (Behnood 2018; Correia and Rasteiro 2016; Niroumand et al. 2013). Application of nanomaterials, such as nanosilica, is motivated by the inherent material characteristics, including large specific surface area and high surface energy and cation exchange potential. Nanosilica has been used extensively used to improve the microstructure of the treated composite and enhance the rate of pozzolanic reactions (Givi et al. 2013; Sobolev et al. 2009). Several researchers have reported that the use of nanosilica with traditional stabilizers has significantly improved the strength and hydraulic properties in treated geomaterials (Bahmani et al. 2014; Changizi and Haddad 2017; Taha and Taha 2012). However, the authors could not identify any significant research progress on the durability and long-term permanency of treatments when the silica-based admixtures have been used with traditional Ca-based stabilizers. The use of silica-based admixtures with traditional stabilizers has enormous potential for the future and needs to be properly investigated. The next section outlines the research objective and primary variables investigated in this work.

Research Objectives and Variables

The principal objective of this research study was to understand the influence of silica-based admixture with dolomitic hydrated lime on the durability and permanency of chemical treatments of sulfate-rich soil. Two different silica-based admixtures, including crystalline silica (CS) fines obtained from quarry dust and laboratory-grade nanosilica (NS) were selected for this study. For comparison purposes, tests were also performed on untreated and lime-treated soil specimens.

The effects of adding admixtures with traditional Ca-based stabilizers on soil plasticity and corresponding moisture susceptibility were first determined from Atterberg's limit test at different curing periods of 0, 3, 7, 14, and 28 days. Subsequently, unconfined compressive strength (UCS) tests were performed on the treated specimens at the end of the five curing periods after subjecting to moisture intrusion through capillary soaking, and the strength loss was determined relative to the unsoaked strength. The long-term permanency of the treatments was also determined using moisture ingress/digress through treated specimens by an internal flushing test. Treated specimens after two curing periods, 7 and 28 days, were subjected to 7 leaching cycles. x-ray fluorescence (XRF) studies were performed on the collected leachate to identify the ionic concentrations. The concentrations of calcium (Ca^{2+}) and sodium (Na^{+}) ions in the collected leachate and pH of the leachate were used to predict the permanency of different treatment methods. Finally, a simplified approach was developed using the normalized specific surface area of silica-based co-additives to understand the effects of additional reactive phases on the overall durability and long-term permanency of treatment.

The following sections present the materials used for the research and the different testing methods used to determine the optimum dosage, moisture susceptibility tests, and leaching studies. Subsequently, a comprehensive analysis and discussion of the observed test results are presented.

Materials and Testing Methods

Materials Used and Selection of Optimum Dosages

Geomaterial and Calcium-Based Stabilizer

The soil for this research study was collected from a pavement construction site near Dallas-Fort Worth, Texas. The basic soil characterization studies were performed considering different American Society for Testing and Materials (ASTM) and Texas Department of Transportation (TxDOT) codes of practice, as presented in Table 1 (ASTM 2014, 2020, 2021a, b, c; Texas DOT 1999). The soil was classified as high-plastic clay (CH) in accordance with Unified Soil Classification System (USCS) standards. The sulfate concentration in the soil was determined as 14,000 parts per million (ppm) using colorimetric method [Tex-145-E (Texas DOT 1999)]. The high sulfate concentration, compounded by plasticity index (PI) value and considerable free swell and shrinkage

Table 1. Basic characterization test results of natural soil

Property	Standard	Magnitude
Specific gravity (G_s)	ASTM D854	2.71
Liquid limit (LL)	ASTM D4318	66.0
Plastic limit (PL)	ASTM D4318	29.5
Plasticity index (PI)	ASTM D4318	36.5
Silt (%)	ASTM D7928	28.8
Clay (%)	ASTM D7928	70.2
USCS soil classification	ASTM D2487	CH
Maximum dry unit weight (MDUW) (kN/m^3)	ASTM D698	15.5
Optimum moisture content (OMC) (%)	ASTM D698	19.0
Vertical free swell strain (%)	ASTM D4546	23.0
Linear shrinkage (%)	Tex-107-E	16.5

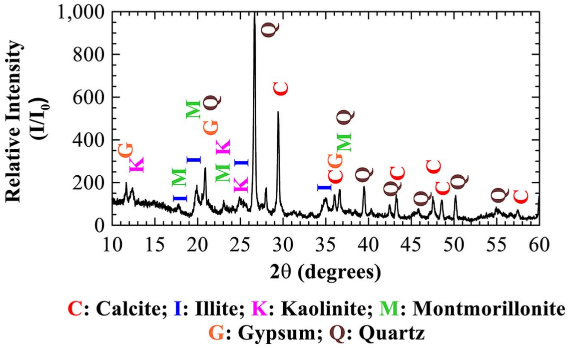


Fig. 1. X-ray diffractogram for untreated high-sulfate soils.

Table 2. Chemical composition of tested materials from X-ray florescence studies

	Mass (%)	
Parameters	Natural soil	Ca-based stabilizer
Si as Silicon oxide (SiO_2)	39.69	4.43
Al as Aluminum oxide (Al_2O_3)	35.99	9.45
Fe as Ferric oxide (Fe_2O_3)	14.36	0.33
Ca as Calcium oxide (CaO)	2.18	51.49
Mg as Magnesium oxide (MgO)	1.80	28.29
S as Sulphur trioxide (SO_3)	1.57	0.01
Na as Sodium oxide(Na_2O)	0.17	0.11
K as Potassium oxide (K_2O)	1.52	0.01
Loss on ignition	2.71	5.85

strain percentages, made this soil ideal for treatment in this research study. The high-sulfate (HS) soil was also analyzed for mineralogical composition using XRD and XRF studies (Fig. 1 and Table 2). The natural soil was composed of calcite, quartz, gypsum and different clay minerals, including montmorillonite, illite, and kaolinite. Dolomitic hydrated lime conforming to ASTM C977 (ASTM 2018) was used as the primary Ca-based stabilizer. The mineralogical composition of the lime indicated the presence of more than 50% CaO, 28% of MgO and the remaining concentration of other oxides (Table 2).

Nano- and Crystalline Silica Admixtures

Two different silica-based admixtures were selected to be used with the traditional Ca-based stabilizer in this study. The crystalline silica (CS) admixture was obtained by crushing locally available sand and collecting particles passing #200 US sieve. Specific surface area of the CS admixture was determined as 2.45 m^2/g using the methylene blue spot test. Laboratory-grade nanosilica (NS) with 50% weight in suspension in H_2O and specific surface area of 140 m^2/g was used as the second admixture.

Optimum Dosage Selection

The optimum dosage of lime by dry weight of the soil was determined as 7% in accordance with Eades and Grim pH testing method [ASTM D6276 (ASTM 2019a)]. 30% CS by the dry weight of soil was selected as the target dosage with 7% lime, based on some preliminary studies by the authors (Biswas 2022; Biswas et al. 2021b; Chakraborty et al. 2022). The target dosage of NS with lime was selected based on an unconfined compressive strength test in accordance with ASTM D5102 (ASTM 2019b) after 7 days of curing. The strength of a subgrade is considered a key criterion for the design of a pavement infrastructure and therefore was selected as the governing condition for stabilizer dosage optimization. Based on the strength test, 1% NS with 7% lime was determined and used for this study as the most suitable dosage for engineering property enhancement. The next section presents a discussion of the different testing methods used for this research study.

Laboratory Testing Methods

Preparation of Treated Soils

The soil-lime powder mixture was prepared by dry blending 7% lime by dry weight with the natural soil. Thereafter, the lime-treated soil (HS-7L) was prepared by adding target molding water (20% by the dry weight of soil solids) to the dry mixture. For combined CS and lime-treated soils, CS in the air-dried state was uniformly mixed with dry soil and lime to their target proportion and subsequently, the molding water was added to prepare the homogeneous mixture (HS-7L-30CS). The laboratory-grade NS admixture was available commercially in a colloidal state. Therefore, mixing the colloid with dry soil and lime could often lead to nonuniformly mixed geomaterial. To address the issues, 1% NS (percentage weight of dry soil) was first mixed with the target molding water and stirred uniformly using a magnetic stirrer. Next, the combination of NS and water was added to the dry soil–lime blend and uniformly mixed to develop a homogenous lime and NS-treated soil (HS-7L-1NS).

Atterberg's Limits Tests

The treated soils were cured as powdered form in hermetically sealed chambers (relative humidity, RH ≈ 100%) at room temperature of 23°C ± 2°C. The treated soils were tested for liquid and plastic limits in accordance with ASTM D4318 (ASTM 2017) at the end of each curing period. The application of Ca-based stabilizers to high-plastic clay is expected to alter the soil-water reaction mechanism in the treated matrix. Furthermore, the addition of silica-based admixture would likely influence the interaction between the clay particles and interparticle water. The changes in soil plasticity, when subjected to the novel treatment, would help us to develop a preliminary understanding of water-related distress on such soils.

Durability and Volumetric Swell Strain Studies

Cylindrical specimens of diameter 1.33 in. (33 mm), with an aspect ratio of 2:1 [Height(H): Diameter(D)] were prepared using static compaction, demolded and cured in hermetically sealed chambers (RH ≈ 100% and 23°C ± 2°C). The treated specimens were

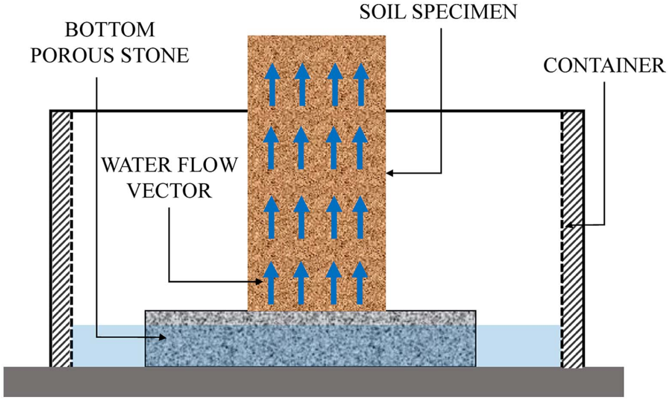


Fig. 2. Capillary soaking for 48 h before strength testing.

compacted at a target dry unit weight of 14.67 kN/m^3 and a moisture content of 20% [ASTM D558 (ASTM 2019). All treated soil groups were compacted at same target dry unit weight and moisture content to ensure a similar zero-day strength condition. Unconfined compressive strength tests were performed after all five curing periods of 0, 3, 7, 14, and 28 days. A total of 6 specimens were prepared for each curing period, of which at the end of each curing period, 3 specimens were subjected to capillary soaking for 48 h at room temperature (23°C ± 2°C) before testing for unconfined strength values in accordance with ASTM D5102 (Fig. 2). The dimension changes in the capillary soaked specimens were measured in radial and vertical directions using π-tape and caliper, respectively. The change in the volume of the specimens relative to the original volume was reported as the volumetric swell percentage (V_s) after capillary soaking. In addition to the soaked strengths, the remaining three specimens were subjected to unsoaked strength tests at the end of each curing period. The combined assessment of the unsoaked strengths and the retained strengths with volumetric swell were used to study the durability of the novel admixture treatments as compared to only lime-treated soils.

Leaching Studies

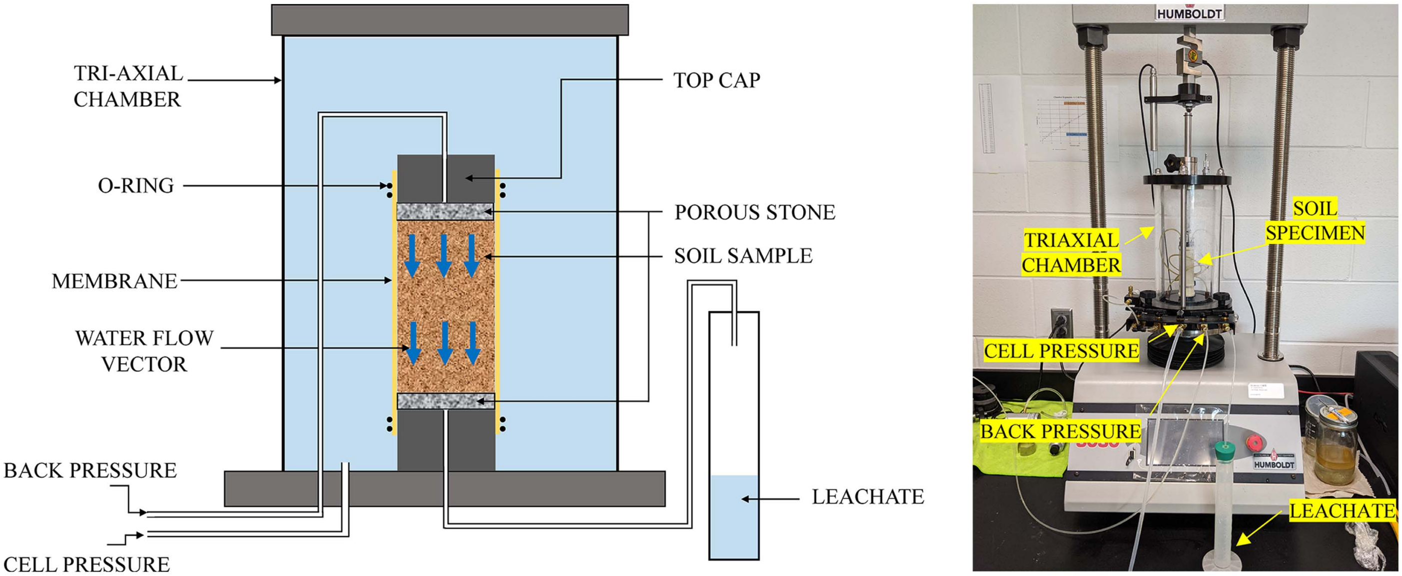
The long-term permanency of the chemical treatments was determined using a redesigned leaching test protocol based on the guidelines of some previous research studies by McCallister (1990) and Chittoori et al. (2013). Cylindrical specimens of diameter 1.41 in. (36 mm) with an aspect ratio of 2:1 (H:D) were prepared by static compaction (dry unit weight = 14.67 kN/m^3 and moisture content = 20%), demolded and cured following a similar procedure as the strength test specimens. Leaching studies were performed on duplicate specimens of HS-7L, HS-7L-30CS and HS-7L-1NS, cured for 7 and 28 days. The two curing periods were selected to understand the short-term (7 days) and long-term (28 days) benefits of silica-based admixtures over traditional techniques.

The leaching test was performed in a modified triaxial chamber, as illustrated in Fig. 3(a). One leaching cycle was defined as the amount of leaching volume equivalent to the total pore volume of the specimen (Chittoori et al. 2013). The specimens were subjected to a constant moisture flow from the top (ingress zone) using a backpressure ranging between 5 and 25 kPa. The back pressure was adjusted regularly to ensure that one leaching volume of fluid was collected over a period of 24 h (1 day). The low inlet pressure was selected to ensure a minimum disturbance in the pore structure of the treated soils during the course of the test. The specimens were saturated prior to the leaching test and precautionary measures were taken to prevent water flow between the membrane and the soil. An effective stress of 5 kPa was ensured through confining pressure ranging between 10 and 30 kPa to enable the flow of fluids only through the cross section of the tested specimen.

The laboratory leaching cycles were selected to closely simulate the field infiltration conditions during the early years of pavements in service. The number of months simulated in this research study was calculated as presented in the following.

Field Infiltration.

- The average annual rainfall in Texas $(\text{AAR}) = 27.25'' = 0.692$ m (PRISM Climate Group 2020).
- Approximate percentage of infiltration $(I\%) = 35\%$ (Chittoori et al. 2013; McCallister 1990).
- Infiltrated rainwater per year $(I_{field}) = \text{AAR} \times \text{I}\%$.



(a) (b)

Fig. 3. Leaching test to determine the permanency of treatment (a) schematic diagram; and (b) test setup.

Laboratory Infiltration.

- Diameter of tested specimen in laboratory $(d) = 1.41\text{ in.} = 0.0358\text{ m}$.
- Flow area in the specimen $(A) = \pi d^2/4 = 0.001007\text{ m}^2$.
- Volume of water infiltration in 1 cycle $(I_1) = 32\text{ ml}$.
- Volume of water infiltration in 7 cycles $(I_7) = 7 \times I_1 = 224\text{ ml} = 0.000224\text{ m}^3$.
- Water head of infiltration $(I_{lab}) = I_7/A = 0.223\text{ m}$.
- Number of years replicated in the laboratory $= n = I_{lab}/I_{field} = 0.92\text{ years} \approx 11\text{ months}$.

It should be noted that the total time period simulated in the laboratory environment is significantly lower than the general design life of any chemically treated layers for transportation infrastructures. However, the leaching cycles in the laboratory represent aggressive moisture flow regimes. Field sections are expected to be less likely prone to similar direct moisture ingress and therefore, the results could be conveniently used to predict the long-term permanency of the stabilizers and admixtures used for treatment.

The leachate was collected from the bottom of the soil specimen (egress zone) in a burette and tested for ionic concentrations [Fig. 3(b)]. The collected leachate for each cycle were analyzed for ionic concentrations of calcium (Ca^{2+}) and sodium (Na^+) ions using x-ray fluorescence (XRF) studies. The relative change in the concentration of both cations for different treatment techniques was used to understand the pore chemistry as well as the permanency of treatment. In addition to ionic concentrations, the pH of the collected leachate was also determined for a comprehensive characterization of the treated soils. The pore fluid of the natural untreated specimens was also extracted using 1 leaching cycle and analyzed using XRF studies to determine the natural cationic concentration (Ca^{2+} and Na^+) and pH for comparative purposes. The next section presents the results and discussion of the engineering and chemical studies performed in this research program. The analyses of the test results are expected to provide a comprehensive understanding of the durability and leachability behavior of treatments with novel silica-based admixtures.

Results and Discussions

Consistency Limits Studies

The water content values corresponding to Atterberg's limits at different curing periods are presented in Fig. 4. The application of a Ca-based stabilizer immediately reduced the liquid limit and partially increased the plastic limits. The immediate reduction in soil plasticity could be attributed to the modification reactions, including cation exchange, reduction in the thickness of double diffused layer and flocculation-agglomeration of the clay particles making the soil more friable and workable. The presence of silica admixtures notably improved the soil plasticity as compared to only Ca-based treatment. The partial replacement of expansive soils by cohesionless CS fines for the same dry unit weight resulted in an improvement in soil PI value. In NS-treated soils, the immediate precipitation of cementitious products coupled with the modification reactions increased the cohesion and averted the moisture absorption on clay surface, respectively.

Over the longer curing periods in HS-7L group, a marginal reduction in LL and improvement in PL was observed after initial curing period. In a high alkaline environment, Ca-based stabilizers react with dissolved soil silicates and aluminates to precipitate Calcium-Silicate-Hydrate (C-S-H) and Calcium-Aluminum-Hydrate (C-A-H). These cementitious products reduce moisture affinity and consequently help to bind the soil matrix resulting in the improvement of plasticity. The CS-treated soils (HS-7L-30CS) underwent an appreciable reduction in moisture affinity (or, LL) as compared to only Ca-treated soils. Presence of CS fines and the reduction in the precipitation alumina-sulfate hydrate phases reduces the moisture affinity (Chakraborty et al. 2022). A stark difference in the behavior of NS-treated soils (HS-7L-1NS) was observed as compared to only lime-treated or lime-CS-treated soils. Both LL and PL results were observed to increase for longer curing periods, but the increase in PL was higher as compared to LL, resulting in an overall decrease in the PI. Prolonged curing and the presence of amorphous silica particles favored precipitation of cementitious phases that coated and bound the clay particles (Biswas et al. 2023). Subsequently, the coarser particles developed random or flocculated

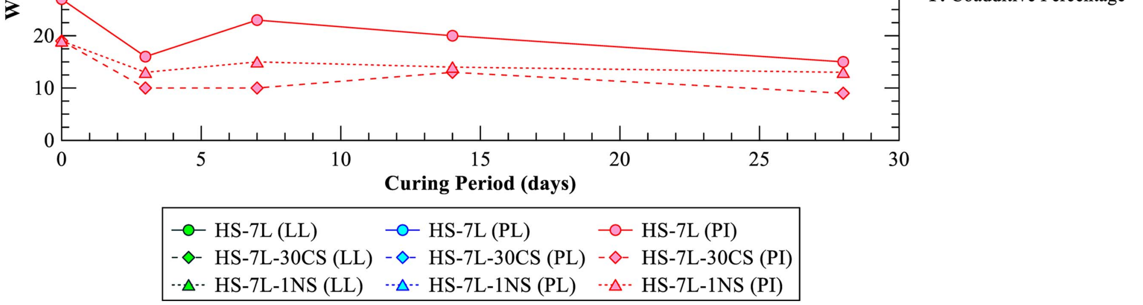


Fig. 4. Variation of Atterberg's limits over different curing periods for HS soil.

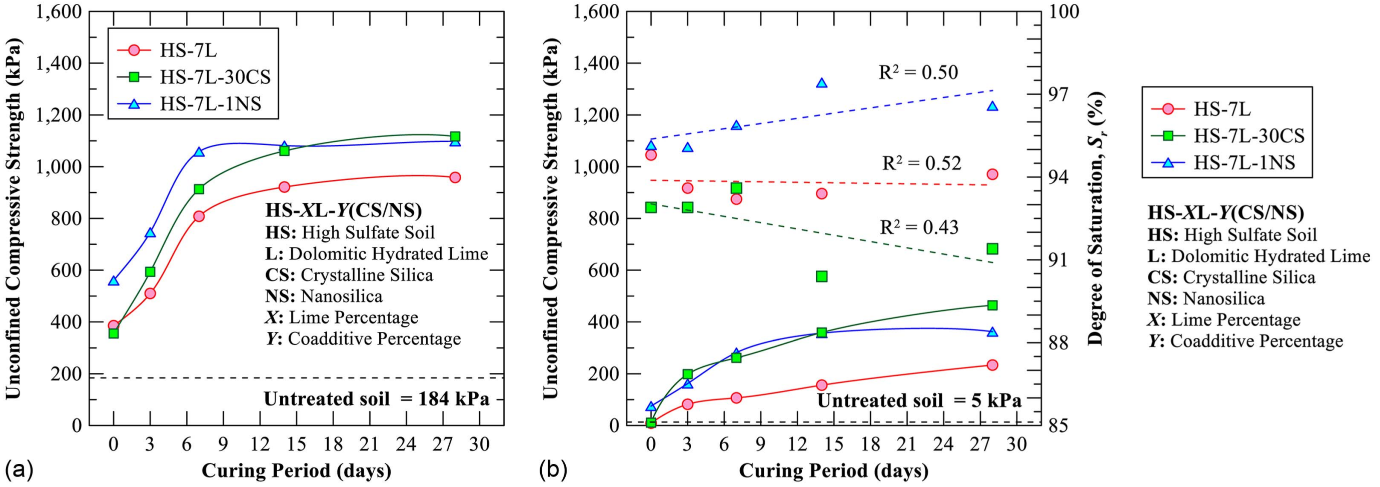


Fig. 5. Unconfined strength values of treated soils: (a) before soaking; and (b) after capillary soaking for 48 h and degree of saturation, S_r (%).

arrangements, which entrapped water in the void spaces of the soil fabric and increased the LL on curing. Parallelly, the cementitious compounds improved the binding between the soil particles and subsequently reduced the soil plasticity.

Overall, both admixtures improved the tendency of treated soils to reduce water absorption on clay surfaces. But the influence of CS was partially higher as compared to NS. The next section discusses the effects of changes in moisture affinity on the engineering strength property of the treated soils.

Durability and Volumetric Swell Strain Analyses

The unconfined compressive strength (UCS) values of HS-7L, HS-7L-30CS, and HS-7L-1NS soil groups at different curing periods, before and after subjecting the specimens to capillary soaking are illustrated in Fig. 5. The UCS results of untreated soil before and after capillary soaking was obtained as 184 and 5 kPa. These values clearly indicated that the soils performed poorly in durability and needed chemical stabilization. Application of Ca-based stabilizers immediately improved the strength properties in treated soils due to soil modification reactions [Fig. 5(a)]. The improvement in strength was notably high for HS-7L-1NS soil groups due to the immediate precipitation of C-S-H phases due to the reaction between amorphous silica particles and Ca^{2+} ions from the stabilizer. No appreciable improvement was observed from CS treatment on initial strength as the cementitious products were expected to form only at the broken edges of silica fines. Over a longer curing period, the silica-based admixtures developed approximately 1.25 times higher strength as compared to HS-7L soil group [Fig. 5(a)]. This clearly indicates that the presence of silica-based admixtures precipitated additional cementitious binding gels with traditional pozzolanic products. These reaction products cemented the soil matrix and helped to develop higher strengths over the longer curing periods.

Fig. 5(b) and Table 3 present the values of soaked strengths for treated soil groups over different curing periods. Table 3 also presents the values of percentage retained strengths in capillary-soaked soil specimens as compared to unsoaked soil specimens. The moisture intrusion during the early curing stages caused significant loss to the original unconfined strength value of the treated soils. Strength loss of more than 90% was observed for HS-7L and HS-7L-30CS groups immediately after treatment (0-day cured). Absence of cementitious binding phases from pozzolanic reactions and the loss of apparent cohesion from suction forces resulted in the loss. In HS-7L-1NS group, the strength retained was marginally higher as compared to other groups, likely from the immediate precipitation of C-S-H phases due to reaction between amorphous NS and Ca-oxides.

After a longer curing period of 28 days, the strength loss in HS-7L specimens was observed to be more than 80%. Curing of treated soils precipitates cementitious phases of C-S-H and C-A-H which binds the soil matrix and reduces moisture affinity. However, in this present research the detrimental effects of high soluble sulfate concentrations were evident. The Ca-based stabilizers react with soluble sulfates and soil aluminates to precipitate the trisulfate aluminate hydrate mineral ettringite (Chakraborty et al. 2022). The growth of ettringite crystals when subjected to external water sources results in a reduction in soil bulk density and the development of repulsive forces from water molecules on cementitious binding gels. The nucleation and growth of ettringite resulted in an appreciable reduction in strength after capillary soaking. Application of silica-based admixtures improved the performance of the treated soils after curing. The presence of additional silica sources likely reduced the preferential dissolution of soil aluminates to react

Table 3. Comparison of retained strength after capillary soaking for HS-7L, HS-7L-30CS, and HS-7L-1NS specimens

Curing period (days)	Before capillary soaking (kPa)			After capillary soaking (kPa)			Retained strength (%)		
	L-HS	L-HS-30CS	L-HS-1NS	L-HS	L-HS-30CS	L-HS-1NS	L-HS	L-HS-30CS	L-HS-1NS
0	385.4	355.1	560.2	9.1	10.8	75.1	2.4	3.1	13.4
3	510.0	593.7	746.7	81.9	198.4	162.2	16.1	33.4	21.7
7	808.2	913.3	1,058.3	106.6	261.6	281.9	13.2	28.6	26.6
14	921.4	1,060.9	1,081.7	155.9	359.1	356.6	16.9	33.8	32.9
28	958.4	1,116.9	1,098.2	233.6	464.1	362.3	24.4	41.6	32.9

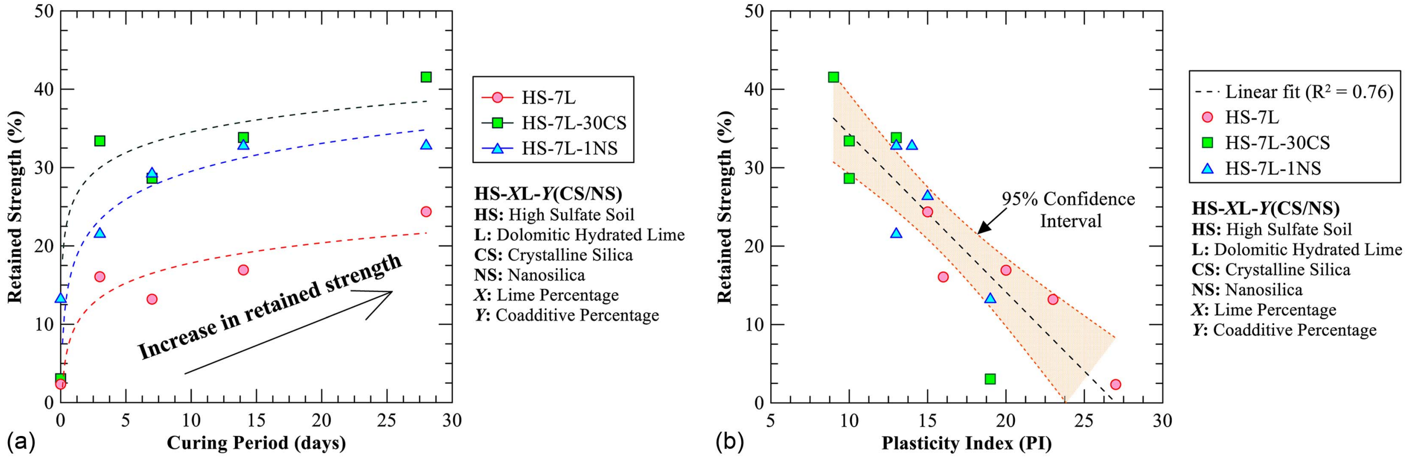


Fig. 6. Percentage retained strength for HS-7L, HS-7L-30CS, and HS-7L-1NS specimens (a) at different curing periods (b) function of plasticity index (PI).

with soluble sulfate and parallelly developed additional phases of C-S-H with the Ca^{2+} ions from Ca-based stabilizers. The reduction in the precipitation of deleterious ettringite crystals helped to improve the engineering performance.

Several past studies recommended an optimum strength of 350 kPa after moisture conditioning for soils supporting pavement infrastructures, considering long-term serviceability performance (Chakraborty et al. 2022; Little and Nair 2009; Thompson 1968). HS-7L-30CS and HS-7L-1NS test groups retained the required strength after 28 days of curing. Fig. 6(a) presents the percentage of retained strengths over different curing periods, and it could be noted that the retained strength gradually increases with time. Precipitation of cementitious reaction products from pozzolanic reactions helped to retain higher strength with the progress of the curing period. However, even though the unsoaked strengths of silica-based treatments were comparable after 28 days of curing, the percentage strength retained after soaking in NS-treated soils were considerably low as compared to CS treatment [Fig. 6(a)]. A correlation was developed between the PI values of the treated soils and the retained strength after capillary soaking for 48 h [Fig. 6(b)]. The results illustrate that the soil plasticity has a direct influence on the ability of treated soils to retain strength after capillary soaking.

The application of NS with lime partially improved the soil plasticity by reducing the affinity for moisture. Furthermore, the presence of a silica source deterred preferential dissolution of alumina, restricting the precipitation of ettringite. However, the inherent property of nanoparticles enhanced flocculation of clay particles, which resulted in an increase in microporosity and reduced bulk density which likely countered the bonding from cementitious phases. The increase in micropores resulted in absorption of more water in the pore structure during moisture conditioning and increased the degree of saturation and subsequently reduced the post-soaked strength [Figs. 5(b) and 6(a)]. For CS-treated soils, partial replacement of high-plastic sulfate-rich soil with cohesionless CS admixture reduced the moisture affinity and improved the plasticity [Figs. 4 and 5(b)]. Additionally, the presence of reactive silica fines developed additional regions for forming cementitious phases that anchored the soil matrix.

The volumetric swell strain values and the corresponding weight of water absorbed in each treated specimen are illustrated in Fig. 7. At 0 days curing, HS-7L and HS-7L-30CS treated soils recorded significant volumetric strains (43% and 35%, respectively) and corresponding water absorption (35% and 31%, respectively). The slow pozzolanic reactions and absence of bonding gels resulted in distress. Over longer curing periods, the volumetric strains in lime-CS-treated soils were significantly less as compared to only lime-treated soils. The advantages of using CS fines to mitigate ettringite-induced heave are evident from the swell values. The volumetric strain and water absorption in HS-7L-1NS soils were immediately reduced to 11% and 15%, respectively, after treatment. Marginal reduction in swell strains and water absorption was observed over longer curing periods. The correlation between the volumetric swell strain and the PI before subjecting the specimens to capillary soaking was developed as presented in Fig. 7(c). Similar to the retained strength values, a direct impact of soil PI was observed on the final swell strains of the treated soils.

From the previous discussions on moisture susceptible related durability studies, it is evident that the ability of the silica-based admixtures to improve the soil plasticity has a significant influence on the post-soaking engineering performance. The following section discusses the influence of these admixtures on the permanency of the Ca-based treatments to develop a comprehensive idea of the long-term performance of these novel admixture treatments.

Leaching Studies to Assess Permanency of Chemical Treatment

The analyses of the leachate collected after each cycle from the treated soils provide a partial understanding of the soil pore chemistry as well as the factors responsible for durability of the treated soils. The leachate for the tested specimens was collected after every cycle and analyzed for cationic concentrations (expressed as ppm) of divalent Ca^{2+} and monovalent Na^{+} ions. The results of the tests on HS-7L, HS-7L-30CS, and HS-7L-1NS specimens cured for 7 and 28 days are summarized in Figs. 8 and 9. The pH of the pore fluids extracted after each cycle was determined from the leachate and illustrated in Fig. 10.

The pore fluid collected from untreated specimens had an average concentration of 490 ppm and 950 ppm of Ca^{2+} and Na^{+} cations, respectively. Chemical treatment using Ca-based stabilizer and silica-based admixtures affected the soil fabric structure and consequently affected the cation retaining ability in macro and micropores of treated soil. A marginal reduction in leaching of

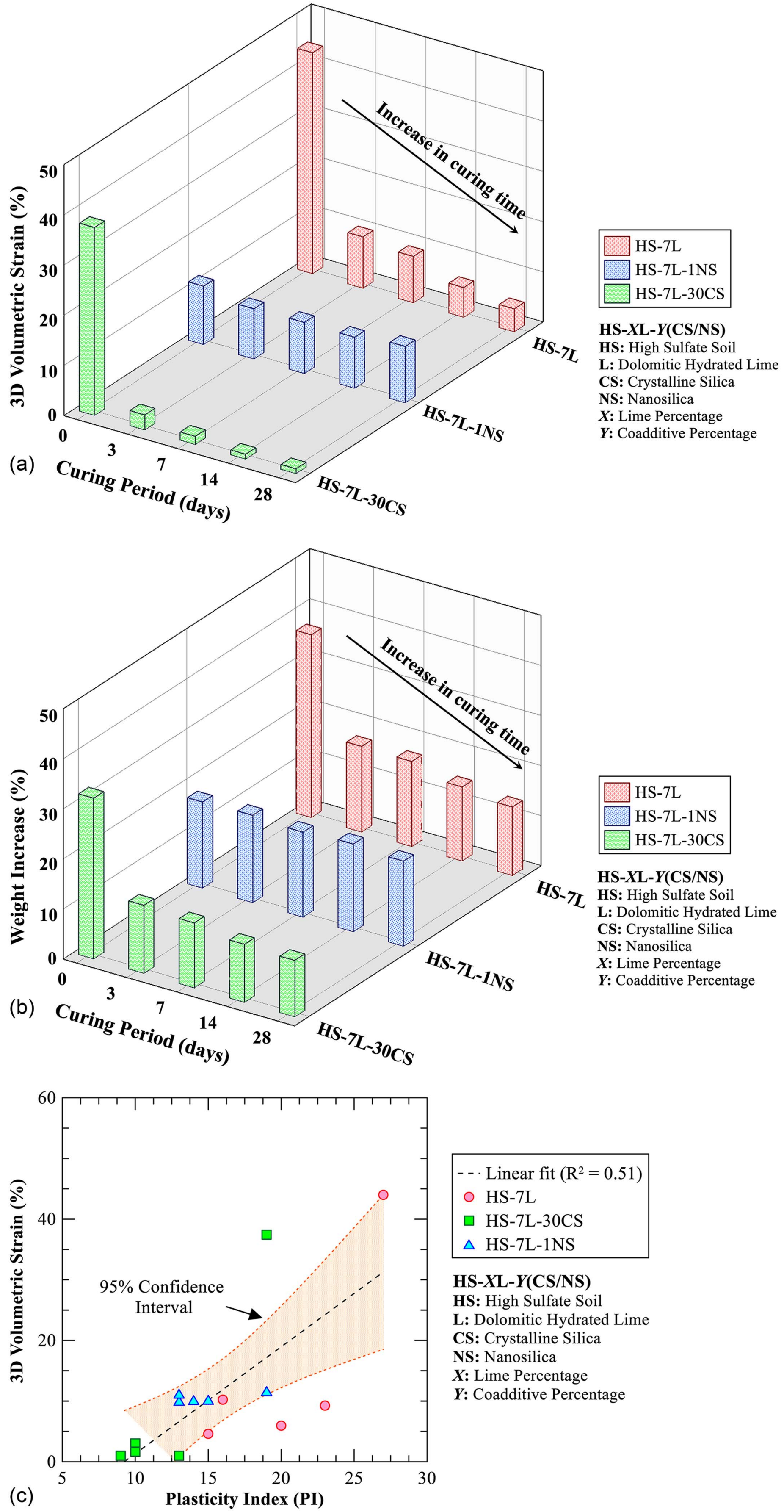


Fig. 7. Soaked results for HS-7L, HS-7L-30CS, and HS-7L-1NS specimens: (a) 3D volumetric strain; (b) percentage weight increase; and (c) relation between soil plasticity (PI) and 3D volumetric strains.

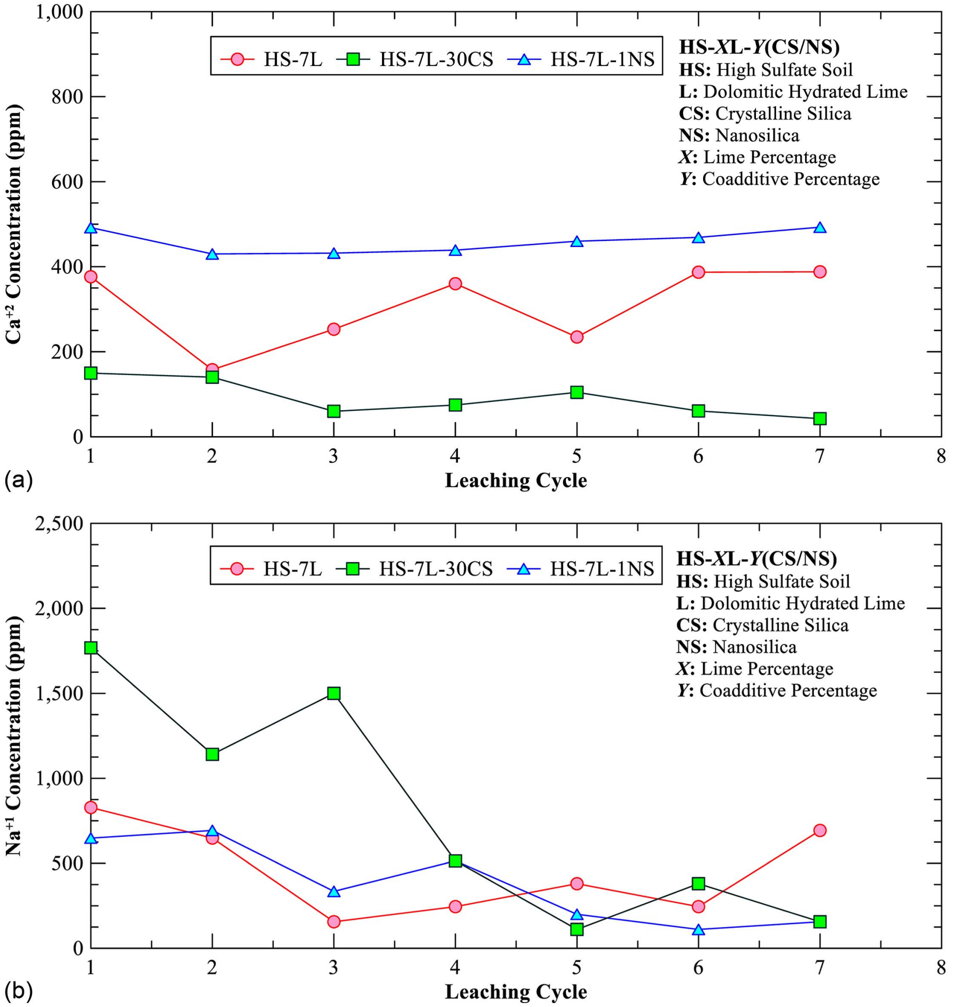


Fig. 8. Cation concentration in pore fluid after different leaching cycles at 7 days curing: (a) Ca^{+2} concentration; and (b) Na^{+} concentration.

Ca^{2+} ions during the early curing period indicated a utilization of the available cations in precipitation of reaction products. In HS-7L group, the average Ca^{2+} ions leached over 7 cycles were observed as 308 ppm and 493 ppm, after 7 and 28 days of curing, respectively [Figs. 8(a) and 9(a)]. The concentration of Ca^{2+} leached was uniform and no significant reduction was observed over different leaching cycles. Application of CS admixture reduced the leaching of Ca^{2+} ions notably as compared to HS-7L group. A reduction in leaching of the Ca^{2+} cations was observed with the progress of leaching cycles, especially after 28 days of curing. In the HS-7L-1NS soil group, application of nanosilica resulted in an increase in leaching of Ca^{2+} cations as compared to both HS-7L and HS-7L-30CS groups. Similar to only lime-treated high-sulfate soil (HS-7L), the average concentration of Ca^{2+} ions leached for both silica-based admixtures increased with longer curing periods.

In addition to the Ca^{2+} ions, the concentration of Na^{+} ions over 7 leaching cycles was also determined for all treated soil groups [Figs. 8(b) and 9(b)]. The concentration of leached of Na^{+} ions was comparable for HS-7L and HS-7L-1NS soil groups. However, for HS-7L-30CS soils, the concentration of leached Na^{+} ions was significantly higher than the remaining tested groups. The concentration of leached cations decreased over longer leaching cycles for both 7 and 28-day cured specimens. Furthermore, the average concentration of Na^{+} ions leached marginally reduced with curing for all treated soil groups. A $pH > 11.5$ was noted in the pore fluids extracted from HS-7L and HS-7L-30CS specimens for all leaching cycles and after 7 and 28 days of curing. However, in the HS-7L-1NS specimens, a low value ($pH \sim 8.5$) was observed for all leaching cycles and after both curing periods.

Application of Ca-based stabilizers to plastic clays results in the precipitation of cementitious phases of C-S-H and C-A-H due to pozzolanic reactions that help to bind the soil matrix and provide long-term durability. However, when the Ca-based stabilizers are added at optimum dosage, several research studies have indicated an increase in soil porosity. Furthermore, in sulfate-rich soil, the precipitation and growth of ettringite is expected to increase the soil void ratio and subsequently affect the size of soil micro- and macropores. These simultaneous factors were responsible for an increase in the concentration of Ca^{2+} leaching over longer curing periods. Application of CS admixtures significantly reduced the cation leaching primarily due to partial replacement of plastic clay by CS fines as well as precipitation of additional C-S-H phases and reduction in the crystallization and growth of ettringite.

In HS-7L-1NS soils, the presence of nanoparticles was observed to be marginally detrimental, considering the permanency aspect of treatments. NS admixtures provide additional reactive surfaces for the precipitation of cementitious phases and also prevent the preferential dissolution of alumina to form alumino-sulfate hydrate phases. However, the NS admixtures tend to form agglomerates

J. Mater. Civ. Eng., 2023, 35(12): 04023469

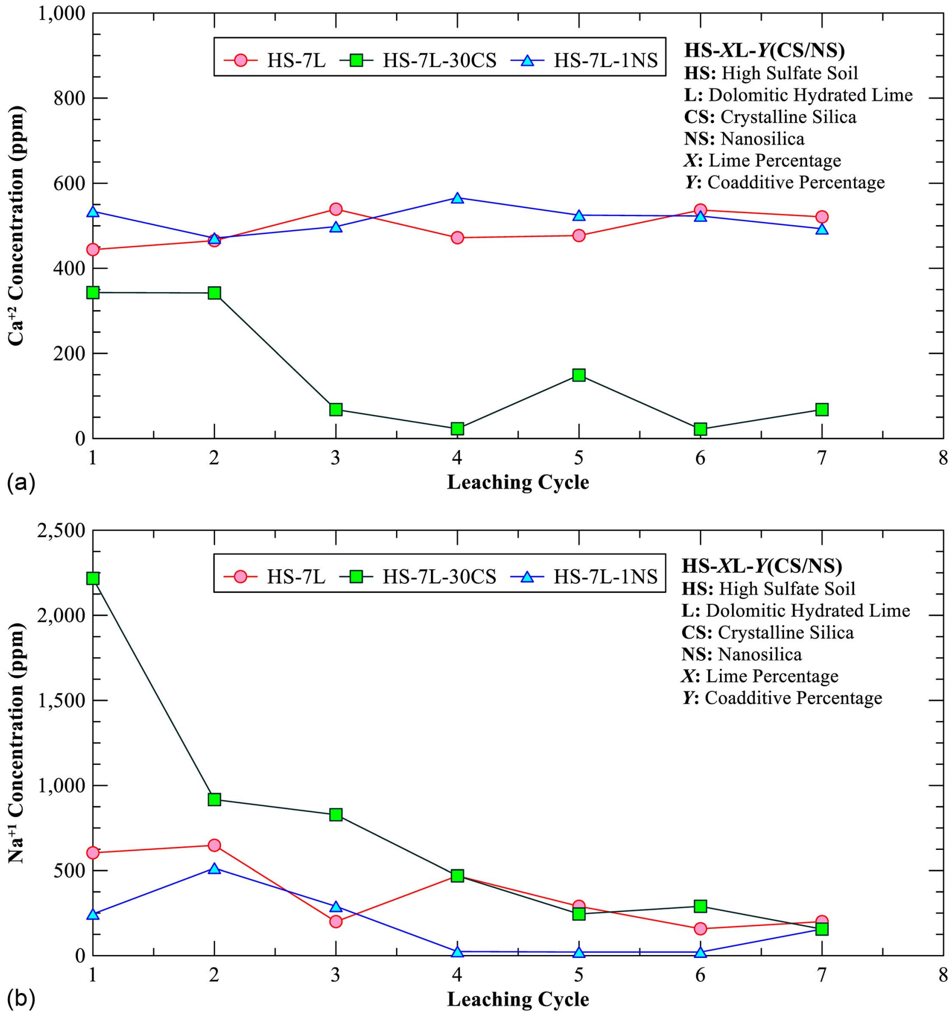


Fig. 9. Cation concentration in pore fluid after different leaching cycles at 28 days curing: (a) Ca^{+2} concentration; and (b) Na^{+} concentration.

under an alkaline environment that consequently affects the reactivity of the nanoparticles with stabilizers and clay minerals. Moreover, nanoparticles can induce flocculation, leading to the development of micro and macropores that affect the soil matrix's void ratio. Therefore, the unreacted nanosilica agglomerates likely formed a metastable state in the available soil pores which, when subjected to a continuous water flow leached with unreacted Ca^{2+} ions resulting in a high calcium cation concentration and low pH ($pH < 8.5$, which is similar to the pH of pure nanosilica) in the collected leachate extract for all cycles (Fig. 10).

The concentration of Na^{+} ions leached is an indirect indication of the amount of cation exchanged during the chemical reaction stages. The application of CS with lime yielded maximum cation exchange indicating high concentration of Na^{+} ions in the leachate for both curing periods. For HS-7L and HS-7L-1NS, the leaching of Na+ ions was similar. The application of CS with high-plastic soil improved workability and resulted in a more uniform distribution of the stabilizer during mixing, which in turn led to a higher exchange of monovalent cations by divalent calcium ions. The NS admixture in alkaline environment likely formed agglomerates, which subsequently reduced its reactivity with soil minerals and thus its capacity for cation exchange. A further discussion of the durability aspects of the silica-based treatment is presented in the next section, using a simplified approach based on the specific surface area of the admixtures.

Effects of Silica-Based Admixtures on Durability and Permanency of Treatments

In this section, a simplified approach was developed to understand the effects of silica-based treatments with traditional Ca-based stabilizers on durability and permanency of treatment on sulfate-bearing soils. The addition of silica-based admixtures with traditional Ca-based stabilizer was based on the hypothesis that the presence of additional reactive phases of silica was expected to influence the kinetics of the chemical treatment. In CS fines, the presence of broken bonds due to crushing the larger particles provided reactive edges for the precipitation of cementitious phases. The NS particles had an inherent high specific surface area of amorphous silica, which could develop binding gels in addition to traditional pozzolanic reaction products.

The specific surface area (*SSA*) of CS aggregates was determined as $2.45\ m^2/g$ by methylene blue spot test. The SSA of NS was obtained from the product standard sheet as $140\ m^2/g$. The calculation steps for developing the simplified approach of analyses are presented as follows:

- Specific Surface Area (*SSA*) of admixture added to the geomaterial $= \chi\ m^2/g$.
- Percentage of admixture added to solid by the dry unit weight of geomaterials $= \gamma\%$.
- Weight of admixture per 100 gm of dry soil $(W) = \gamma\% \times 100\ g = \gamma\ g$.

J. Mater. Civ. Eng., 2023, 35(12): 04023469

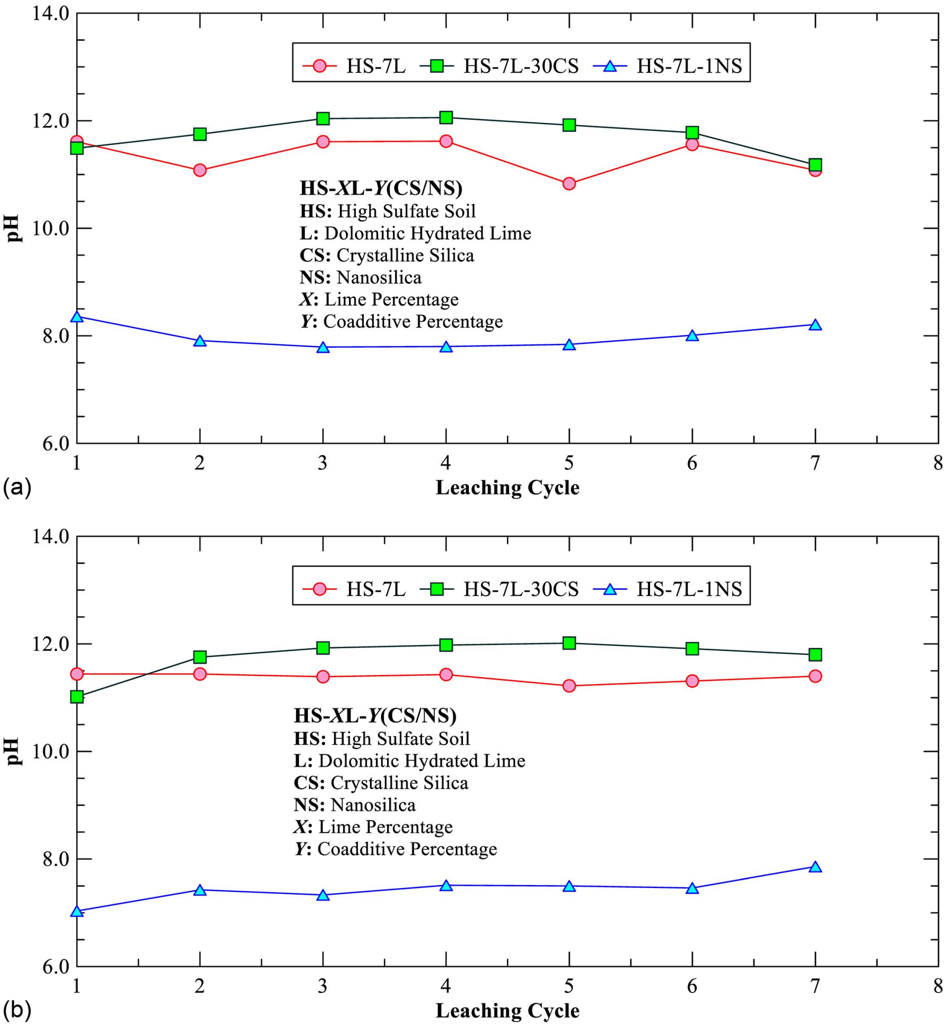


Fig. 10. pH of pore fluid after different leaching cycles (a) 7-day cured; and (b) 28-day cured specimens.

- Total SSA of the admixture in dry solids $(SSA_T) = \chi\,(\mathrm{m}^2/\mathrm{g}) \times \gamma$ g.
- Normalized SSA of admixtures with respect to dry soil $(SSA_N) = SSA_T/(100-\gamma)\ \mathrm{m}^2/\mathrm{g}$.

The normalized SSA values of both CS and NS admixtures used for this research study are presented in Table 4. The NS admixture is available in a colloidal state with 50% by weight suspension in water. Therefore, the actual weight of NS admixture in 100 g of soil solids was calculated as 0.5 g. The HS-7L specimens were considered as the control group since no admixtures were added and used for comparative analyses.

The effects of additional reactive phases were analyzed based on three target parameters of treated soils, including soil plasticity or PI value, the moisture susceptible durability or the retained strength after capillary soaking and the permanency of treatment or leaching of Ca^{2+} ions after curing for 7 and 28 days. Table 5 and Fig. 11 present the effects of normalized SSA of silica-based admixtures on the three target parameters. The application of additional reaction phases affects the soil plasticity after curing the specimens. The silica phases promoted the precipitation of additional cementitious phases of C-S-H compounds, in addition to the traditional pozzolanic reaction products, which helps to reduce soil plasticity [Fig. 11(a)].

Table 4. Specific surface area (*SSA*) of silica-based admixtures in treated soils

Admixture	*SSA* $(\mathrm{m}^2/\mathrm{g})$	% of admixture in soil solids	*W* (g)	SSA_T (m^2)	SSA_N $(\mathrm{m}^2/\mathrm{g})$
CS	2.45	30	30	73.5	1.05
NS	140	1	0.5[a]	70.0	0.70

[a] The NS admixture was commercially available as colloidal solute with 50% by weight in suspension with water.

Table 5. Effects of specific surface area of admixtures on durability and permanency of treatments

SSA_N	Curing period (days)	Plasticity index	Retained strength (%)	Avg. Ca^{2+} ions leached in 7 cycles (ppm)
Control (HS-7L)	7	23	13.2	308.1
	28	15	24.4	493.6
1.05 (HS-7L-30CS)	7	10	28.6	90.5
	28	9	41.6	145.0
0.70 (HS-7L-1NS)	7	15	26.6	459.3
	28	13	33.0	515.7

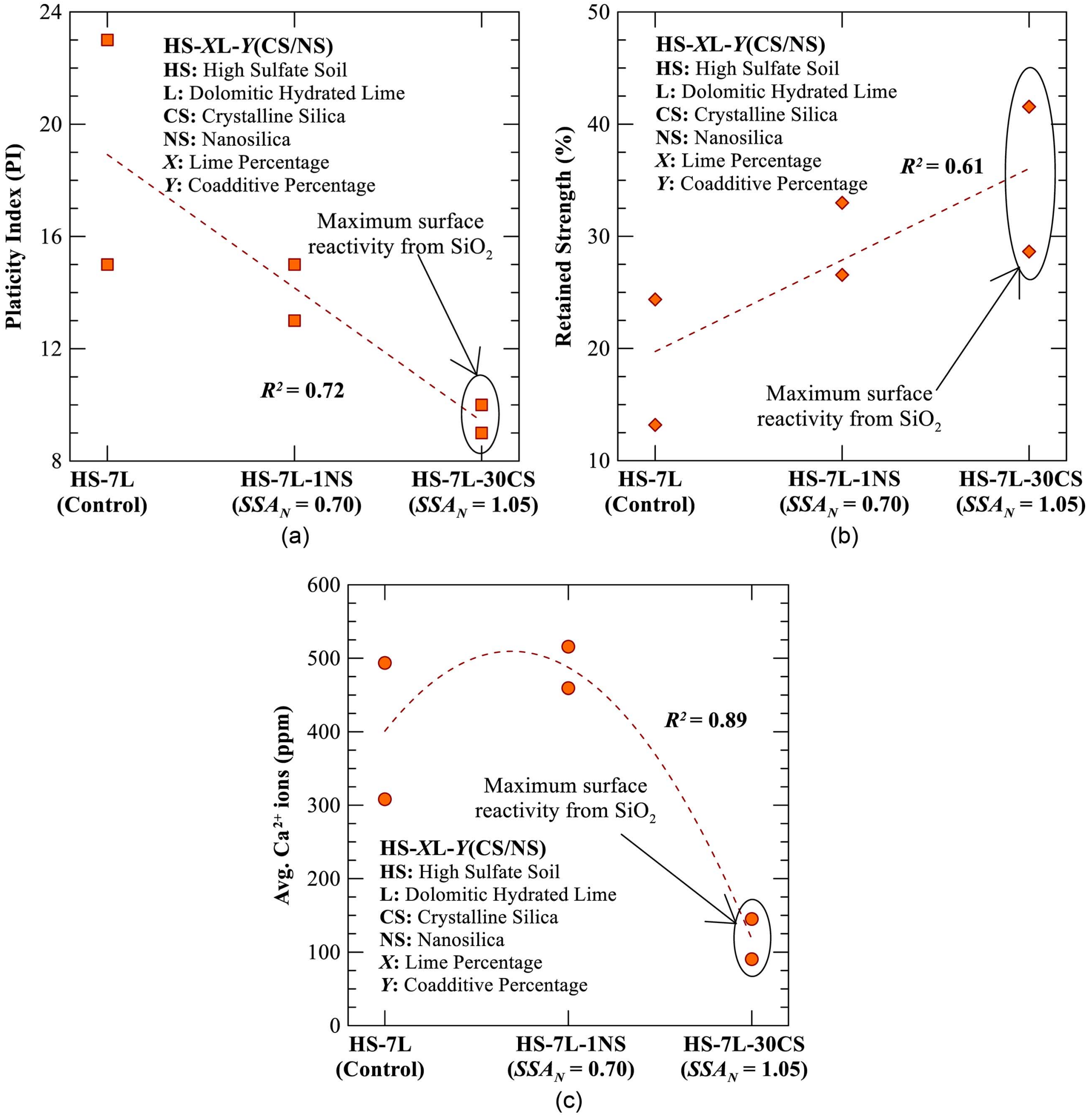


Fig. 11. Effects of silica-based admixture on (a) geomaterial plasticity; (b) moisture susceptible durability; and (c) leaching of calcium affecting the permanency of treatment.

In the previous sections, it was observed that engineering properties were correlated to soil plasticity. Therefore, using silica-based admixtures could significantly contribute in modifying certain engineering properties like strength and durability. The additional phases of C-S-H also helped to bind the soil matrix and helped to prevent strength loss when subjected to moisture intrusion through capillary soaking [Fig. 11(b)]. In addition to plasticity and durability, the presence of silica-based coadditives also marginally influences the permanency of treatment when significant concentration of silica dosage is added [Fig. 11(c)]. However, it should also be noted that the reaction mechanism of amorphous nanosilica and crystalline silica should be considerably different from each other and the effect of the reaction mechanism was not considered in this section and could be considered for future studies.

Conclusions and Recommendations

The application of calcium-based stabilizers is often found to be counterproductive when treating soils rich in sulfate-bearing phases. To overcome the problems associated with traditional treatment techniques, silica-based admixtures, including crystalline silica (CS) fines from quarry dust and laboratory-grade amorphous nanosilica (NS) were used with dolomitic hydrated lime to treat such soils. However, the design of infrastructures using novel admixtures and subsequent application in infrastructure construction requires the treatment to be durable and sustainable for the long-term serviceability of the pavement. The research study was undertaken to identify and address both durability and permanency-related issue associated with silica-based treatments.

The application of silica-based admixtures significantly affected the moisture susceptibility of the treated soil as compared to only lime treatment. Both CS and NS amendments helped to retain higher strength as compared to only lime-treated soils when specimens were subjected to moisture susceptible durability through capillary soaking. The ability to retain higher strengths was found to depend on both the plasticity value after treatment and the curing period. The lime-CS treated soils retained maximum strength as compared to all other treated soil groups for longer curing periods. The reduction in the precipitation of trisulfate alumino-hydrate phases and the precipitation of additional binding C-S-H phases were identified as contributing factors. The lime-NS treated specimens performed better than only lime-treated group; however, the soils absorbed more water due to hydrophilic nature of binding phases as well as the development of micropores after treatment.

The long-term permanency of the treatments was tested using leaching studies, where specimens were subjected to 7 cycles of internal flushing by controlled backpressure system in a triaxial chamber. Each cycle involved flushing the specimens with one pore volume of water, and the resulting leachate was analyzed for cation concentrations. The application of CS with lime illustrated minimum leaching of calcium ions while maintaining a high pH value, indicating the presence of CS could be suitable for long-term permanency of treatment. The application of NS was found to have a similar effect to that of lime treatment alone due to the development of micropores and potential agglomeration of nanoparticles in an alkaline environment.

Finally, a simplified analysis was performed where the target engineering performance including soil plasticity, retained strength, and calcium leachability, was determined based on the normalized specific surface area of silica-based admixture. Overall, the research study provided evidence that silica-based admixtures could have a significant impact on the treatment of sulfate-rich soils. Among the two admixtures, the CS admixture was found to be more suitable for achieving long-term benefits than the NS admixture. Therefore, the outcome of this research study will be immensely helpful for transportation practitioners and engineers. However, full-scale prototype studies are recommended in the future to develop a comprehensive understanding of the in-situ performance. In addition, a future analysis of the sustainability and resiliency of the treatment over the entire life cycle of the infrastructure will help practitioners develop long-lasting and durable subgrade layers on sulfate-rich soils using this novel chemical treatment.

Data Availability Statement

Some or all data, models, or code that support the findings of this study are available from the corresponding author upon reasonable request.

Acknowledgments

This research was funded NSF Industry-University Cooperative Research Center (I/UCRC) program funded ‘Center for Integration of Composites into Infrastructure (CICI)’ site at Texas A&M University (NSF PD: Dr. Prakash Balan; Award # 2017796). The authors would also like to thank Dr. Sayantan Chakraborty for his input during the research program.

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