Tran-SET

Transportation Consortium of South-Central States

*Solving Emerging Transportation Resiliency, Sustainability, and Economic Challenges through the Use of Innovative Materials and Construction Methods: From Research to Implementation*

# Eco-Friendly Stabilization of Sulfate-Rich Expansive Soils Using Geopolymers for Transportation Infrastructure

**Project No. 19GTUTA01**

**Lead University: University of Texas at Arlington**

**Collaborative University: Texas A&M University**

**Progress Report**
**December 2020**

## Disclaimer

## Acknowledgements

The discussions and feedback by the Louisiana Department of Transportation and Development and Texas Department of Transportation are greatly appreciated.

# TECHNICAL DOCUMENTATION PAGE

| | | |
|---|---|---|
| **1. Project No.**<br>19GTUTA01 | **2. Government Accession No.** | **3. Recipient's Catalog No.** |

| | |
|---|---|
| **4. Title and Subtitle**<br>Eco-friendly stabilization of sulfate-rich expansive soils using Geopolymers for transportation infrastructure | **5. Report Date**<br>Dec 2020<br>**6. Performing Organization Code** |
| **7. Author(s)**<br>PI: Xinbao Yu https://orcid.org/0000-0002-5681-0390<br>Co-PI: Anand J. Puppala https://orcid.org/0000-0003-0435-6285<br>Co-PI: Miladin Radovic https://orcid.org/0000-0003-4571-2848<br>Post-doc: Sayantan Chakraborty https://orcid.org/0000-0002-6809-5953<br>GAR: Jungyeon Jang https://orcid.org/0000-0002-3739-6657<br>GAR: Oscar Huang https://orcid.org/0000-0001-7004-3506 | **8. Performing Organization Report No.** |
| **9. Performing Organization Name and Address**<br>Transportation Consortium of South-Central States (Tran-SET)<br>University Transportation Center for Region 6<br>3319 Patrick F. Taylor Hall, Louisiana State University, Baton Rouge, LA 70803 | **10. Work Unit No. (TRAIS)**<br>**11. Contract or Grant No.**<br>69A3551747106 |
| **12. Sponsoring Agency Name and Address**<br>United States of America<br>Department of Transportation<br>Research and Innovative Technology Administration | **13. Type of Report and Period Covered**<br>Technical Phase Research Report<br>Aug. 2019 – Nov. 2020<br>**14. Sponsoring Agency Code** |

**15. Supplementary Notes**

Report uploaded and accessible at [Tran-SET's website (http://transet.lsu.edu/)](http://transet.lsu.edu/).

**16. Abstract**

Traditional calcium-based stabilizers, such as lime, to stabilize sulfate-rich expansive soils, are lasting-challenges in geotechnical engineering. The biggest problem with using calcium-based stabilizers is that when calcium and water are exposed to sulfate-rich expansive soils, they form ettringite as an expansive mineral, which causes sulfate-induced heaving. The study aimed to investigate the feasibility of stabilizing sulfate-rich expansive soils using geopolymers for transportation infrastructure. Literature review on sulfate-induced heaving and sulfate-rich soils stabilized with geopolymers suggests a direction for how to stabilize sulfate soils using geopolymers. The engineering tests were conducted to determine the swell, shrinkage, and strength behavior of natural, lime stabilized- and geopolymer-stabilized soils. Scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS) and X-ray diffractometer (XRD) tests were performed to compare the microstructural changes of geopolymer-stabilized soils with natural and lime-stabilized soils. Laboratory tests were conducted considering the influence of the geopolymer composition, dosage, and curing period to investigate the effect of the geopolymer in stabilizing the sulfate-rich expansive soils.

| | |
|---|---|
| **17. Key Words**<br>Geopolymer, sulfate-rich soils, pavements, sustainability, stabilization, infrastructure | **18. Distribution Statement**<br>No restrictions. This document is available through the National Technical Information Service, Springfield, VA 22161. |

| **19. Security Classif. (of this report)** | **20. Security Classif. (of this page)** | **21. No. of Pages** | **22. Price** |
|---|---|---|---|
| Unclassified | Unclassified | 35 | |

**Form DOT F 1700.7 (8-72)** **Reproduction of completed page authorized.**

# SI* (MODERN METRIC) CONVERSION FACTORS

## APPROXIMATE CONVERSIONS TO SI UNITS

| Symbol | When You Know | Multiply By | To Find | Symbol |
|---|---|---|---|---|
| | | **LENGTH** | | |
| in | inches | 25.4 | millimeters | mm |
| ft | feet | 0.305 | meters | m |
| yd | yards | 0.914 | meters | m |
| mi | miles | 1.61 | kilometers | km |
| | | **AREA** | | |
| in$^2$ | square inches | 645.2 | square millimeters | mm$^2$ |
| ft$^2$ | square feet | 0.093 | square meters | m$^2$ |
| yd$^2$ | square yard | 0.836 | square meters | m$^2$ |
| ac | acres | 0.405 | hectares | ha |
| mi$^2$ | square miles | 2.59 | square kilometers | km$^2$ |
| | | **VOLUME** | | |
| fl oz | fluid ounces | 29.57 | milliliters | mL |
| gal | gallons | 3.785 | liters | L |
| ft$^3$ | cubic feet | 0.028 | cubic meters | m$^3$ |
| yd$^3$ | cubic yards | 0.765 | cubic meters | m$^3$ |
| | | NOTE: volumes greater than 1000 L shall be shown in m$^3$ | | |
| | | **MASS** | | |
| oz | ounces | 28.35 | grams | g |
| lb | pounds | 0.454 | kilograms | kg |
| T | short tons (2000 lb) | 0.907 | megagrams (or "metric ton") | Mg (or "t") |
| | | **TEMPERATURE (exact degrees)** | | |
| °F | Fahrenheit | 5 (F-32)/9<br>or (F-32)/1.8 | Celsius | °C |
| | | **ILLUMINATION** | | |
| fc | foot-candles | 10.76 | lux | lx |
| fl | foot-Lamberts | 3.426 | candela/m$^2$ | cd/m$^2$ |
| | | **FORCE and PRESSURE or STRESS** | | |
| lbf | poundforce | 4.45 | newtons | N |
| lbf/in$^2$ | poundforce per square inch | 6.89 | kilopascals | kPa |

## APPROXIMATE CONVERSIONS FROM SI UNITS

| Symbol | When You Know | Multiply By | To Find | Symbol |
|---|---|---|---|---|
| | | **LENGTH** | | |
| mm | millimeters | 0.039 | inches | in |
| m | meters | 3.28 | feet | ft |
| m | meters | 1.09 | yards | yd |
| km | kilometers | 0.621 | miles | mi |
| | | **AREA** | | |
| mm$^2$ | square millimeters | 0.0016 | square inches | in$^2$ |
| m$^2$ | square meters | 10.764 | square feet | ft$^2$ |
| m$^2$ | square meters | 1.195 | square yards | yd$^2$ |
| ha | hectares | 2.47 | acres | ac |
| km$^2$ | square kilometers | 0.386 | square miles | mi$^2$ |
| | | **VOLUME** | | |
| mL | milliliters | 0.034 | fluid ounces | fl oz |
| L | liters | 0.264 | gallons | gal |
| m$^3$ | cubic meters | 35.314 | cubic feet | ft$^3$ |
| m$^3$ | cubic meters | 1.307 | cubic yards | yd$^3$ |
| | | **MASS** | | |
| g | grams | 0.035 | ounces | oz |
| kg | kilograms | 2.202 | pounds | lb |
| Mg (or "t") | megagrams (or "metric ton") | 1.103 | short tons (2000 lb) | T |
| | | **TEMPERATURE (exact degrees)** | | |
| °C | Celsius | 1.8C+32 | Fahrenheit | °F |
| | | **ILLUMINATION** | | |
| lx | lux | 0.0929 | foot-candles | fc |
| cd/m$^2$ | candela/m$^2$ | 0.2919 | foot-Lamberts | fl |
| | | **FORCE and PRESSURE or STRESS** | | |
| N | newtons | 0.225 | poundforce | lbf |
| kPa | kilopascals | 0.145 | poundforce per square inch | lbf/in$^2$ |

# TABLE OF CONTENTS

TECHNICAL DOCUMENTATION PAGE ........ ii

TABLE OF CONTENTS ........ iv

LIST OF FIGURES ........ v

LIST OF TABLES ........ vi

ACRONYMS, ABBREVIATIONS, AND SYMBOLS ........ vii

EXECUTIVE SUMMARY ........ 8

1. INTRODUCTION ........ 9

2. OBJECTIVES ........ 10

3. LITERATURE REVIEW ........ 11

4. MATERIALS AND METHODS ........ 15

  4.1. Material Characterization ........ 15

  4.2. Geopolymer Synthesis and Selection ........ 17

  4.3. Chemical Stabilization of Expansive Soils ........ 17

    4.3.1. Engineering Tests ........ 17

    4.3.2. One-Dimensional Swell Test ........ 18

    4.3.3. Linear Shrinkage Bar Test ........ 19

    4.3.4. Unconfined Compressive Strength (UCS) Test ........ 20

    4.3.5. Micro-Materials Characterization ........ 21

5. ANALYSIS AND DISCUSSON OF RESULTS ........ 22

  5.1. Preliminary Test for Narrowing Down GP Composition ........ 22

  5.2. One-Dimensional Free Swell Test ........ 22

  5.3. Linear Shrinkage Bar Test ........ 23

  5.4. Unconfined Compression Strength Test ........ 24

  5.5. Micro-Materials Characterization ........ 26

6. SUMMARY AND CONCLUSIONS ........ 30

7. RECOMMENDATIONS ........ 31

REFERENCES ........ 32

## LIST OF FIGURES

Figure 1. Location of collecting soil samples. .... 16

Figure 2. Particle size distribution of natural soil. .... 16

Figure 3. 1-D swell test setup. .... 19

Figure 4. Experimental procedures for 1-D vertical swell tests: (a) Soil specimen preparation, (b) the specimen is placed in a consolidation ring, (c) applying a seating pressure, and (d) measure the swelling strain using a dial gauge. .... 19

Figure 5. Experimental procedures of the linear shrinkage bar test. .... 20

Figure 6. UCS tests: Natural soil specimens (a) before and (b) after testing and geopolymer-treated soil specimens (c) before and (d) after testing. .... 21

Figure 7. Stress-strain curve of preliminary GP composition as a stabilizer. .... 22

Figure 8. Swell test results for 3-days cured specimens of different soil groups. .... 23

Figure 9. Linear shrinkage bar test results for (a) low-sulfate (LS), (b) moderate-sulfate (MS), and (c) high-sulfate (HS) soil groups treated with lime and geopolymer. .... 24

Figure 10. Unconfined compressive strength test results for (a) low-sulfate (LS), (b) moderate-sulfate (MS), and (c) high-sulfate (HS) soil groups treated with lime and geopolymer .... 25

Figure 11. SEM of (a) native soil under low magnification, (b) native soil under high magnification, (c) GP under soil, (d) GP chunks throughout soil. .... 26

Figure 12. SEM-EDS result on area with GP. .... 27

Figure 13. SEM-EDS Elemental Mapping of GP treated Soil .... 28

Figure 14. XRD Spectra of Untreated and GP-treated Soil. .... 29

# LIST OF TABLES

Table 1. Basic soil characterization test results. .......... 15

Table 2. Compaction test results for the different soil groups. .......... 17

Table 3. Test variables considered in this study. .......... 18

# ACRONYMS, ABBREVIATIONS, AND SYMBOLS

| | |
|---|---|
| AASHTO | American Association of State Highway and Transportation Officials |
| ASTM | American Society for Testing and Materials |
| C-A-H | Calcium Aluminate Hydrate |
| C-S-H | Calcium Silicate Hydrate |
| EDS | Energy-Dispersive Analysis |
| FA | Fly Ash |
| FE-SEM | Field Emission Scanning Electron Microscopy |
| GGBS | Ground Granulated Blast furnace Slag |
| GP | Geopolymer |
| HS | High-Sulfate Expansive Soil |
| LS | Low-Sulfate Expansive Soil |
| MDD | Maximum Dry Density |
| MK | Metakaolin |
| MS | Moderate-Sulfate Expansive Soil |
| NCHRP | National Cooperative Highway Research Program |
| NLA | National Lime Association |
| OMC | Optimum Moisture Content |
| OPC | Ordinary Portland Cement |
| RPM | Revolutions Per Minute |
| SEM | Scanning Electron Microscopy |
| TxDOT | Texas Department of Transportation |
| UCS | Unconfined Compressive Strength |
| USACE | United States Army Corps of Engineers |
| USCS | Unified Soil Classification System |
| UTA | University of Texas at Arlington |
| XRD | X-ray powder Diffraction |

# EXECUTIVE SUMMARY

Traditional calcium-based stabilizers such as lime and cement are often used to improve the engineering properties of problematic soils. However, these stabilizers are ineffective in stabilizing sulfate-rich soils due to the formation of expansive minerals ettringite and thaumasite. Since sulfate-rich soils are predominantly obtained in the South and the Western United States and are widely used in these regions to construct pavements, alternative forms of stabilizing techniques are being sought after. In recent years, geopolymer has received much attention as an alternative to ordinary Portland cement (OPC) and lime for soil stabilization, applications for pavements, bridges, and other transportation infrastructures. Geopolymers exhibit comparable mechanical properties to traditional stabilizers. Moreover, geopolymers are eco-friendly and sustainable materials, since geopolymers can be processed at room temperatures from aqueous solutions of waste materials (e.g., fly ash) or abounded natural sources (e.g., clay). Although significant progress has been achieved over the last couple of decades on developing geopolymers with desired properties, their applicability for stabilizing sulfate-rich expansive soils is relatively unknown.

A collaborative research study was performed by research teams from University of Texas at Arlington and Texas A&M University to investigate the feasibility of stabilizing sulfate-rich expansive soils using metakaolin (MK)-based geopolymers. Effects of geopolymer dosage and curing period on overall performance and engineering properties of geopolymer-stabilized subgrade soils were studied in order to optimize the use of Geopolymer derived from local waste and natural materials for transportation infrastructure in Region 6. Extensive laboratory tests, including free swell tests, linear shrinkage bar tests, and unconfined compressive strength tests, were performed on low-sulfate (<3,000 ppm), moderate-sulfate (3,000-8,000 ppm), and high-sulfate (>8,000 ppm) expansive soils treated with lime and geopolymer. Both material characterization studies related to micro to macro behavioral changes of native soils and treated soils were performed as a part of this research.

The swell test results indicated that lime treatment was ineffective for stabilizing sulfate-rich soils due to ettringite-induced heaving after prolonged submergence in water. Geopolymer treatment effectively mitigated the swelling characteristics of expansive soils with low to high sulfate contents. The shrinkage characteristics of the geopolymer-treated soils were also less than lime-treated soils. Besides exhibiting excellent performance in mitigating swell-shrink potential, geopolymer treatment also improved the shear strength properties of the soil. The beneficial effect of geopolymer treatment increased with dosage and curing period. Overall, the findings of this research study affirm that MK-based geopolymer has a strong potential for utilization as a sustainable soil stabilizer.

# 1. INTRODUCTION

The establishment of transportation infrastructure using native materials is often the most desirable solution. However, the presence of soft or highly compressible soils in Region 6 often hinders the use of native soils in their original form. Stabilization of these soils with traditional calcium-based stabilizers generally results in enhanced mechanical properties due to the formation of pozzolanic compounds, which may satisfy the design requirements (*1*). When sulfate-rich native soils are encountered, traditional calcium-based stabilizers often become counter-productive due to the formation of highly expansive minerals like ettringite and thaumasite. Sulfate-rich soils are predominantly obtained in the South and the Western United States, including Texas, Oklahoma, New Mexico, Nevada, and California (*2*). Multiple cases of severe pavement heaving and distress were reported when sulfate-rich expansive subgrade soils were treated with traditional calcium-based stabilizers (*2–11*). This phenomenon is referred to as 'sulfate-induced heave,' and the cost of repair of such pavements exceeds the cost of stabilization (*10*).

Recently, a new class of alumino-silicate polymers, commonly referred to as geopolymers (GPs), has received much attention for its eco-friendly and sustainable nature and its cementitious properties. Unlike traditional stabilizers that have a high-carbon emission, geopolymers have low carbon footprint and impart comparable engineering properties as traditional stabilizers. The improvements in engineering properties of problematic expansive soils treated with geopolymer were studied in Tran-SET project number 17GTTAM02 and 18CTAM04. The geopolymer-stabilized soils exhibited significant improvement in mechanical properties in terms of strength, stiffness, and volumetric changes. Based on the findings of the previous project, an attempt was made to synthesize similar geopolymer and explore the suitability of such sustainable stabilizers in mitigating the sulfate-induced heave in soils native to Region 6. The following section presents the project objectives along with the tasks performed to attain the objectives.

## 2. OBJECTIVES

The overall objective of this study is to investigate the feasibility of stabilizing sulfate-rich expansive soils using geopolymers. The following four tasks were conducted to achieve the objectives:

***Task 1*** involved an extensive review of literature on sulfate-induced heaving and geopolymer stabilization of expansive soils. The geopolymer composition, dosage, the viscosity of the activated solution, curing time, and target mechanical properties of stabilized soils were initially selected based on information gathered from the available literature.

***Task 2*** consisted of selecting expansive soils from Region 6. A high plasticity clayey soil (CH) was selected for this study, and this soil was collected from a road construction site in north Texas. The sulfate content of the soil was modified using gypsum to represent Region 6 expansive soils with low (<3,000 ppm), moderate (3,000 ppm to 8,000 ppm), and high (>8,000 ppm) soluble sulfate contents.

***Task 3*** encompassed the development and characterization of geopolymers of different compositions. Mechanical properties of different geopolymers were studied to select two trial compositions that were most suitable for stabilizing the sulfate-rich soils selected in Task 2. The microstructural characteristics of the different geopolymer types were investigated using a scanning electron microscope (SEM) imaging, energy-dispersive spectroscopy (EDS), and X-ray diffraction (XRD).

***Task 4*** focused on characterizing the materials and evaluating the engineering properties of geopolymer-stabilized sulfate-rich soils. The geopolymer compositions selected from Task 3 were used to stabilize the soil groups of a low, moderate, and high sulfate levels. Engineering tests, such as one-dimensional swell tests, linear bar shrinkage tests, and unconfined compressive strength (UCS) tests were conducted to study the effectiveness of geopolymers for stabilizing sulfate-rich expansive soils. The results of engineering tests on geopolymer-treated soils were compared with natural soil and lime-treated soils, and these results were analyzed to gauge the effectiveness of geopolymer stabilization. SEM-EDS and XRD studies were performed on geopolymer and lime-treated soil specimens to understand the microstructural characteristics of the treated soils and their influence on the macro-scale behavior.

# 3. LITERATURE REVIEW

Natural sulfate-rich soils are prevalent in the southern states of the United States (*12*). Sulfate-induced (also known as ettringite-induced) heaving occurs when problematic sulfate soils are stabilized with calcium-based additives, such as lime and cement (*13*). Ettringite is an expansive mineral formed due to the chemical reaction of calcium ions from lime, sulfate ions from soluble sulfate salts, and alumina available from the dissolution of clay minerals in a high pH environment (>10.5) (*4*, *13–15*). Stoichiometrically, ettringite formation and hydration can result in a volume increase of 137%, and this expansive characteristic causes severe damages to transportation infrastructure.

Millions of dollars are spent each year for repairs and rehabilitation works associated with sulfate heaving (*15–18*). Many case studies showed that the sulfate-induced heaving resulted in distresses or structural damage (*2*, *17*, *19*). Between the late 1980s and the late 2000s, many studies were conducted in Nevada, Texas, and Georgia on observing much sulfate-induced damage in sulfate-rich soils stabilized with calcium-based stabilizers such as lime or cement (*2*, *4*, *5*). Subsequently, further studies have been conducted to deal with sulfate-induced heaving when treating sulfate-rich subgrade soils with the stabilizers in the field of transportation geotechnics (*1*, *13*).

Researchers in transportation geotechnics have increased interest in new technologies for the stabilization of sulfate-rich soils. Therefore, research from various viewpoints on this phenomenon has been continuously conducted. Various methods for measuring the concentration of sulfate in soils, which have the most critical factor in sulfate-induced heaving, have been proposed and developed (*20*). Suggested methods for measuring sulfates include AASHTO T-290 (1995), a modified UTA method (2003), and Texas Department of Transportation (TxDOT) Tex-145-E (2005) (*21–23*). AASHTO method and modified UTA method follow the gravimetric measurement principle, whereas the TxDOT method follows the turbidity measurement principle. Unfortunately, these methods for measuring sulfate concentrations in soil often do not provide reliable and repeatable results.

Nevertheless, these three methods are cheaper than the method based on the ion chromatography principle and do not generate erroneous values that can be provided from other salt sources in soils than the method based on the conductivity principle (*11*). Using the known sulfate and measured sulfate concentration, the most reliable sulfate concentration measurement method among AASHTO method, modified UTA method, and TxDOT method was determined. In conclusion, it was confirmed that the modified UTA method among the three methods provides the most repeatable and accurate sulfate concentration value (*11*).

The sulfate concentration measured using the proposed measurement methods can define the threshold level of sulfate that causes swelling and distress to the structures (*11*). The threshold sulfate levels for calcium-based stabilization depend on soil type, site conditions, and variability in the sulfate sources. Various researchers provided a variety of threshold sulfate levels. Hunter (1988) stated 10,000 ppm of sulfate was the threshold level for calcium-based stabilization, and Petry and Little (1992) were 2000 ppm, and Mitchell and Dermatas (1992) were 3000 ppm (*4*, *24*, *25*). In later studies, the degree of risk was differentiated when a calcium-based stabilizer was used according to sulfate concentration. Berger et al. (2001) classified threshold sulfate levels into low risk (<3,000 ppm), moderate risk (3,000 ppm to 5,000 ppm), moderate to high risk (5,000 ppm to 8,000 ppm), and serious risk (>8,000 ppm) (*26*). Harris et al. (2004) classified threshold sulfate levels into no risk (<3,000 ppm) and high risk (>7,000 ppm). Calcium-based stabilization of sulfate concentrations between 3000-7000 ppm is possible with mellowing (*27*). Also, Little and Nair (2009) classified the level of risk associated with lime stabilization in sulfate-rich clays: low risk (<3,000 ppm), moderate risk (3,000 ppm to 5,000 ppm), moderate to high risk (5,000 ppm to 8,000 ppm), high to an unacceptable risk (>8,000 ppm), and an unacceptable risk (>10,000 ppm) (*28*). This suggestion of threshold sulfate levels is difficult to reach consensus with most researchers, as the damage caused by sulfate-induced heaving occurs over a wide range of sulfate levels (325 ppm – 43,500 ppm) (*17*). Moreover, another reason is that the period of sulfate-induced heaving in chemically stabilized sulfate-rich soils occurs over a short period of several days to several months or longer (*2*, *4*, *7*, *29*). The dissolution properties of the minerals from various sources of sulfates in sulfate-rich soils, sulfate measurement techniques, soil types, and several other factors will affect the wide range of sulfate levels and the duration of chemical stabilization.

Despite these wide ranges and variability mentioned above, several research institutes, including the National Cooperative Highway Research Program (NCHRP), have suggested guidelines for the lime/modified treatment of sulfate soils with sulfate concentrations below 8,000 ppm (*28*). First, the sulfate content in natural soil is measured because the treatment methods depend on the sulfate level. If the sulfate concentration is less than 3,000 ppm, lime treatment with 1-day mellowing will not cause a serious problem because it can sufficiently control the expansion caused by ettringite or thaumasite formation (*10*). If the sulfate levels of natural soils are between 3,000 ppm and 8,000 ppm, the modified treatment methods can be applied. Although lime treatment will possibly treat these soils, two recommend guidelines by the Texas Department of Transportation (TxDOT) and National Lime Association (NLA) are the use of an extended mellowing period and the application of a minimum three percent above optimum moisture content (OMC) during mixing (*26*, *30*). Pre-compaction mellowing is typically recommended for sulfate contents ranging from 3,000 ppm to 8,000 ppm (*30*). Mellowing facilitates the formation of ettringite minerals and reduces the soluble sulfate level before the stabilized soil is compacted. This procedure requires additional water and calcium-based stabilizers over the optimum to allow the formation and swelling of ettringite crystals before compaction (*8*). Even though mellowing can reduce the chances of sulfate heaving, the mellowing time can potentially delay the construction work and might not effectively prevent future sulfate heaving induced by the migration of soluble sulfates with groundwater (*18*). The current state of practice recommends avoiding calcium-based stabilizers for sulfate contents greater than 8,000 ppm (*10*). Instead of the use of calcium-based stabilizers, TxDOT and NLA suggested several alternative treatments for treating sulfate-rich soils: 1) remove and replace them, 2) blend in low sulfate materials, and 3) alternative additives (*26*, *30*). Among these alternative treatments, many studies have been conducted on a method of mitigating sulfate-induced heaving by using several co-additives (e.g., ground granulated blast furnace slag (GGBS), fly ash, and quarry dust) with calcium-based stabilizers (*8*, *11*, *13*, *18*). Even though these admixtures have been effective in reducing sulfate heaving, researchers have been striving to identify non-calcium-based eco-friendly stabilizers with low carbon footprints, and geopolymer is one such promising soil stabilizer.

Geopolymers (GPs) have recently gained prominence as soil stabilizers, and these non-traditional stabilizers are effective in enhancing the engineering properties of different problematic soils (*31*–*35*). Fly ash, GGBS, and MK are commonly used aluminosilicate precursor sources activated using alkali-activated solutions of sodium hydroxide, or potassium hydroxide to form GPs (*35*–*38*). Geopolymer treatment usually increases the engineering properties of problematic soils, including unconfined compressive strength, stiffness, tensile strength, California bearing ratio, and durability

(*35–39*). The extent of improvement depends on the GP composition, dosage, viscosity of the activator solution, water content, curing time and temperature, and soil type.

The characteristics of the aluminosilicate precursor and alkaline activator solution primarily govern the dissolution of the aluminosilicates to form monomeric or oligomeric aluminosilicate species required for the geopolymerization process (*36*). Therefore, GPs synthesized from a precursor with higher aluminosilicate content and larger specific surface area typically result in better mechanical properties since the precursor will have a complete dissolution-precipitation reaction (*38*). An increase in activator to precursor ratio typically facilitates the geopolymerization process and subsequently enhances the mechanical properties of the treated soil since it is required for the aluminum to be charge-balanced by the cations (*40*). However, an increase in the molarity of the activator solution beyond optimum or, in other words, an excess of cations results in an unstable solution that crystallizes quickly, reduces the initial setting time and results in low workability (*40*). The utilization of potassium hydroxide instead of sodium hydroxide results in enhanced workability at the same water molar amount since sodium has a smaller cation radii; therefore it would have a more substantial ionic potential (*41*). Another study also showed that sodium-based GP retains more capillary water than potassium-based GP after 28 days of curing consistently throughout numerous compositions (*42*). This would imply that potassium-based GP would be preferred over sodium-based GP as a soil stabilizer since potassium-based GP would attract less water throughout wet/dry cycles therefore leading to a smaller magnitude of volumetric fluctuation. The water content or water to solid ratio also influences the mechanical and fresh properties of GP similar to other cementitious materials (*43*); therefore, it is vital to find a balance between mechanical properties and workability when implementing GP as a soil stabilizer, so that GP can be properly dispersed into soil while having sufficient strength for pavement. Excessive water results in incomplete chemical reactions, whereas too high water content can break the geopolymer-soil bonds *(24)*. GP stabilization results in the enhancement of engineering properties of the treated material that depends on the curing time and temperature (*37*). The time-dependent strength gain has been observed to continue for over one year, and the long-term strength has been reported to be as high as three times the strength gained in a month. Even though the geopolymerization process is accelerated at elevated temperature conditions, the use of high-temperature curing is energy-intensive and is not feasible for most field applications (*44*). The improvements in engineering properties are soil-specific and depend on the soil mineralogy. Hence, different research studies reported wide ranges in strength, stiffness, and durability improvements. However, all the research studies unanimously reported that improvements in engineering properties after geopolymer treatment are comparable, if not better, than lime or cement stabilized soils. The remaining of this section will do an in-depth review on the available literature investigated the usage of GP to stabilize sulfate-rich soil.

Zhang et al. (2015) are one of the earliest studies that investigated the feasibility of an MK-based GP as a stabilizer in sulfate-rich soils (*45*). In the study, the authors prepared their own synthetics soil from local sandy soil, a kaolin clay, and reagent grade gypsum. The synthetic soils have gypsum concentrations of 1000, 5000, and 10000 ppm and are all classified as lean clay or low plasticity clay. The GP used in the study is processed from MK and activated with a blend of sodium hydroxide and sodium silicate solution with Si/Al and Na/Al molar ratios of 1.7 and 1.0. From the results, 8% GP demonstrates comparable mechanical properties with 4% lime, but is much weaker than 5% cement. Their swelling test results demonstrated that GP can reduce vertical expansion significantly when compared to untreated soil if it is able to cure properly; on the other hand, the usage of lime causes significant vertical expansion. Overall, the study showed the effectiveness of MK-based GP over lime soil treatment; however, more could have been done to compare between GP and Portland cement.

Khadka et al. (2020) investigated the feasibility of using MK-based and class C fly ash-based GP as a soil stabilizer for highly expansive soils from Dallas, TX and Atlanta, GA that are both classified as high plasticity clay (*46*). For this study, both soils had gypsum added to achieve 15000 ppm of sulfate content since the purpose is only to study the effect of the stabilizer on the vertical expansion from the swell test. The GPs are synthesized with the mentioned aluminosilicate sources and activated with a blend of sodium hydroxide and sodium silicate. The study has demonstrated that GP outperforms lime significantly and class C fly ash is better than MK as the aluminosilicate source. Overall, the study has positive results, but more comprehensive testing would be needed. An example would be mechanical properties since it is essential in pavement design. Their finding that class C fly ash would perform better than MK is also counter-intuitive since the calcium content in the fly ash should react with sulfate and form more ettringite and therefore result in more shrinkage than MK; however, that is not the case. An explanation would be that the MK-based GP is not well cured, therefore, the clayey nature of MK resulted in more shrinkage than the non-absorbent fly ash.

The findings from previous studies suggest that geopolymer has a tremendous potential to be used as a sustainable soil stabilizer. Most of the studies did not consider geopolymer stabilization of sulfate-rich soils, and hence this research study aims at evaluating the suitability of geopolymer stabilization of sulfate-rich expansive soils that are prevalent in Region 6. The next sections elucidate the methodology adopted to synthesize geopolymer, the engineering tests performed on the geopolymer-treated soils and highlight the salient findings of this research study.

# 4. MATERIALS AND METHODS

This research study aims to evaluate the effectiveness of stabilizing sulfate-rich expansive soils with MK-based geopolymer. Natural expansive soil was collected from a road construction site in Texas, and the sulfate content of this soil was modified to represent different sulfate levels representative of problematic soils in Region 6. Extensive engineering and microstructural tests were conducted to characterize the soil and synthesized geopolymer and study the behavior of the soil treated with lime and geopolymer. This section elucidates the methodology adopted for material characterization and geopolymer synthesis. The details of the engineering tests and microstructural analysis performed on the chemically treated soils are also presented here.

## 4.1. Material Characterization

The natural soil was collected from a road construction site in Denison, Texas (Figure 1). The basic soil characterization tests were performed in accordance with the ASTM standards, and these results are presented in Table 1. Figure 2 depicts the particle size distribution of natural soil. The natural soil has a clay fraction of 58%, plasticity index of 33%, a free swell strain of 12.2%, and is classified as clay of high plasticity (CH) as per USCS classification. Based on these characteristics, this problematic natural soil requires chemical treatment and is ideal for this research study.

**Table 1. Basic soil characterization test results.**

| Property | Value |
|---|---|
| Specific Gravity (ASTM D854) | 2.72 |
| Liquid limit (LL) (ASTM D4318) | 60 |
| Plastic limit (PL) (ASTM D4318) | 27 |
| Plasticity index (PI) (ASTM D4318) | 33 |
| Gravel (%) (ASTM D6913) | 0.0 |
| Sand (%) (ASTM D6913) | 8.3 |
| Silt (%) (ASTM D7928) | 34.2 |
| Clay (%) (ASTM D7928) | 57.5 |
| USCS soil classification (ASTM D2487) | CH |
| Maximum dry density (MDD) (g/cm$^3$) (ASTM D698) | 1.66 |
| Optimum moisture content (OMC) (%) (ASTM D698) | 20.0 |
| Soluble sulfate content (ppm) (TEX-145-E) | 336 |
| Free swell strain (%) (ASTM D4546) | 12.2 |
| Linear shrinkage strain (%) (TEX-107-E) | 15.1 |

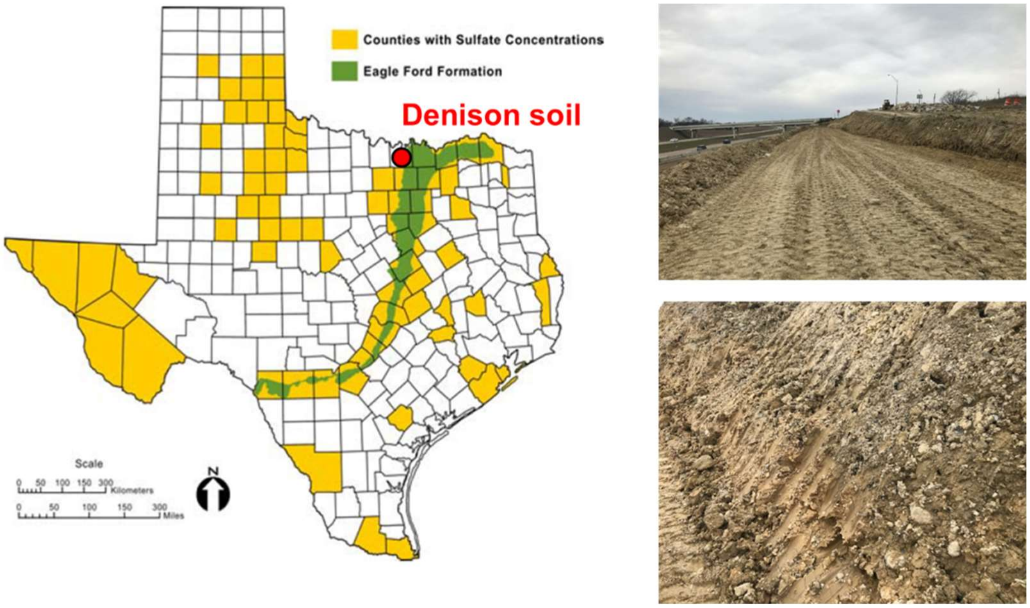


**Figure 1. Location of collecting soil samples.**

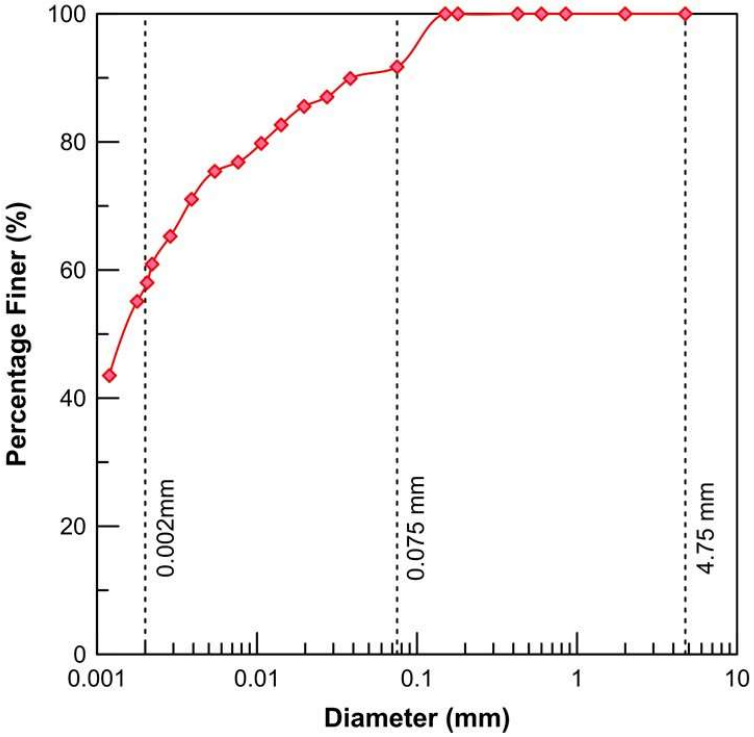


**Figure 2. Particle size distribution of natural soil.**

Artificial sulfate-rich expansive soils were prepared by adding gypsum to the natural soil. The soluble sulfate content of the natural soil was representative of low-sulfate contents (<3,000 ppm), and gypsum was added to increase the sulfate contents to moderate (3,000 ppm to 8,000 ppm) and high-sulfate contents (>8,000 ppm). The three soil groups are denoted as LS (low-sulfate), MS (moderate-sulfate), and HS (high-sulfate) throughout this report. Commercially available hydrated lime conforming to ASTM C977 and geopolymer synthesized in-house were used for stabilizing the problematic soil.

## 4.2. Geopolymer Synthesis and Selection

The GPs used in this research were synthesized by researchers using sodium or potassium hydroxide (Noah Technologies, TX), amorphous fumed silicon (IV) oxide (Alfa Aesar, MA) with 350- 410 $m^2/g$ specific surface area, MetaMax® (BASF Catalysts LLC, NJ) metakaolin, and deionized water. Metakaolin is a purer aluminosilicate source than the more commonly used fly ash with higher impurities and was therefore used as a precursor for GP synthesis in this research.

The sodium or potassium hydroxide was dissolved in deionized water to create a highly alkaline solution to process the alkali metal cations. The amorphous fumed silicon oxide was then added to adjust the $SiO_2/Al_2O_3$ ratio of the final product as desired, to create the activating solution for the synthesis of geopolymer. The activating solution was then mixed with metakaolin, which is a high-purity activating aluminosilicate source in a high-sheared mixer for 6 minutes at 400 revolutions per minute (RPM) to create a homogenized mixture, known as GP.

Since the number of possible GP compositions is endless, it is necessary to narrow down the compositions using data from previous works for the scope of this work. Based on the work that was done in Tran-SET projects #18CTAM04 and #19CLSU04, 2 preliminary compositions were chosen, namely K331 and K431 due to their high strength and suitable workability. Note that GP compositions are labeled as KXYZ where the first letter denote potassium (K) while XYZ numbers denote $SiO_2/Al_2O_3$ ratio, water to solid ratio used to prepare GP, and Na/Al or K/Al ratio respectively. For example, GP sample K431 is sample prepared with K-activator, and $SiO_2/Al_2O_3=4$, water/solid ratio=3, and K/Al=1.

## 4.3. Chemical Stabilization of Expansive Soils

### *4.3.1. Engineering Tests*

Expansive soil groups of samples with different sulfate contents (LS, MS, and HS) were treated with lime and MK-based geopolymer. The swell-shrink behavior and shear strength properties were studied using the free swell strain tests, linear shrinkage bar tests, and UCS tests. The different soil groups were treated with 6% lime, and 8% and 30% geopolymer. Dry soil, stabilizer (lime or geopolymer), and water were mixed in desired proportions, and the homogenized mixtures were used to prepare specimens for the different engineering tests. The compaction test results for the different specimen groups are provided in Table 2.

**Table 2. Compaction test results for the different soil groups.**

| Soil Group | Stabilizer type and dosage | Specimen group | OMC (%) | MDD ($g/cm^3$) |
|---|---|---|---|---|
| Low-sulfate soil (LS) | 6% Lime (L) | L-LS | 30.0 | 1.47 |
| | 8% Geopolymer (8GP) | 8GP-LS | 28.1 | 1.45 |
| | 30% Geopolymer (30GP) | 30GP-LS | 24.6 | 1.56 |
| Moderate-sulfate soil (MS) | 6% Lime (L) | L-MS | 30.0 | 1.47 |
| | 8% Geopolymer (8GP) | 8GP-MS | 28.1 | 1.45 |
| | 30% Geopolymer (30GP) | 30GP-MS | 24.6 | 1.56 |
| High-sulfate soil (HS) | 6% Lime (L) | L-HS | 30.0 | 1.47 |
| | 8% Geopolymer (8GP) | 8GP-HS | 28.1 | 1.45 |
| | 30% Geopolymer (30GP) | 30GP-HS | 24.6 | 1.56 |

The specimens for different tests were cured for 6 hours (0.25 day), 3 days, and 14 days at 25±1°C in hermetically sealed plastic bags (100% relative humidity environment). Table 3 presents the test variables considered in this study, along with the specimen designations. The specimen specifications and test protocols for the 1D free swell tests, linear shrinkage bar tests, and unconfined compressive strength tests are provided below.

**Table 3. Test variables considered in this study.**

| Test (Standard) | Stabilizer type and dosage | Number of specimens |
|---|---|---|
| 1-D Free swell test<br>(ASTM D4546) | Untreated | 2 replicates |
| | 6% Lime | 2 replicates x 3 curing periods |
| | 8% GP | 2 replicates x 3 curing periods |
| | 30% GP | 2 replicates x 3 curing periods |
| Linear shrinkage bar test<br>(TEX-107-E) | Untreated | 3 triplicates |
| | 6% Lime | 3 triplicates x 3 curing periods |
| | 8% GP | 3 triplicates x 3 curing periods |
| | 30% GP | 3 triplicates x 3 curing periods |
| Unconfined compressive strength (UCS) test<br>(ASTM D2166) | Untreated | 3 triplicates |
| | 6% Lime | 3 triplicates x 3 curing periods |
| | 8% GP | 3 triplicates x 3 curing periods |
| | 30% GP | 3 triplicates x 3 curing periods |

### *4.3.2. One-Dimensional Swell Test*

The one-dimensional (1-D) swell tests were performed according to ASTM D4546 Test Method A (Figures 3 and 4). The swell test results were analyzed to study the post-treatment swelling characteristics of the lime-treated and geopolymer-treated LS, MS, and HS soils. All soil samples were compacted at 98% of MDD and 2% wet of OMC, respectively (Table 2). The treated soil mixtures were statically compacted in 63.5 mm (2.5 in) diameter and 25.4 mm (1 in) height molds. After each curing period, duplicate specimens of each group were subjected to one-dimensional (1-D) free swell tests under 1 kPa (0.145 psi) vertical stress. The tests were continued till the dial gauge readings did not change for three consecutive days.

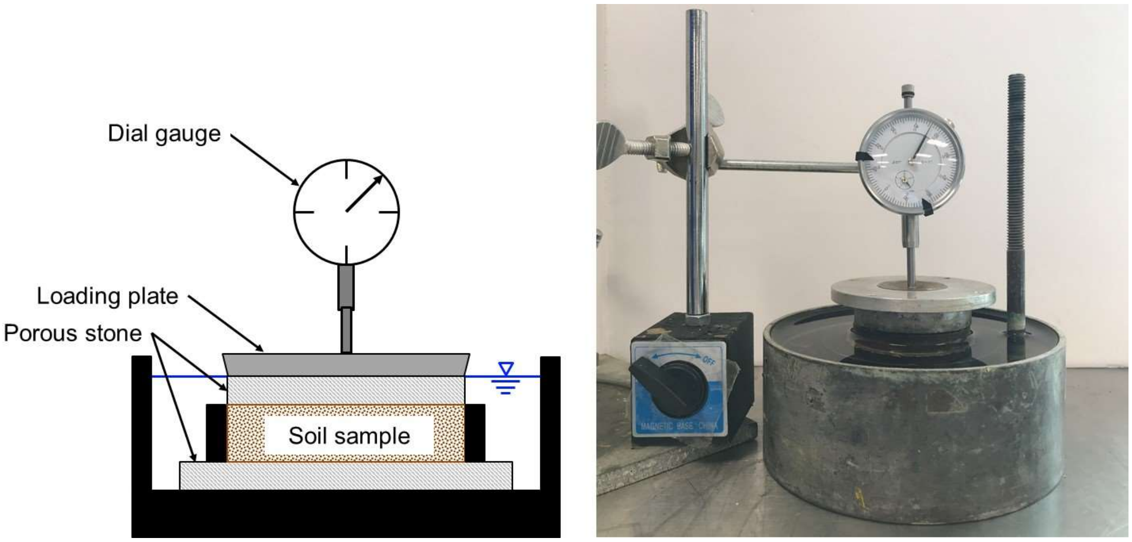


**Figure 3. 1-D swell test setup.**

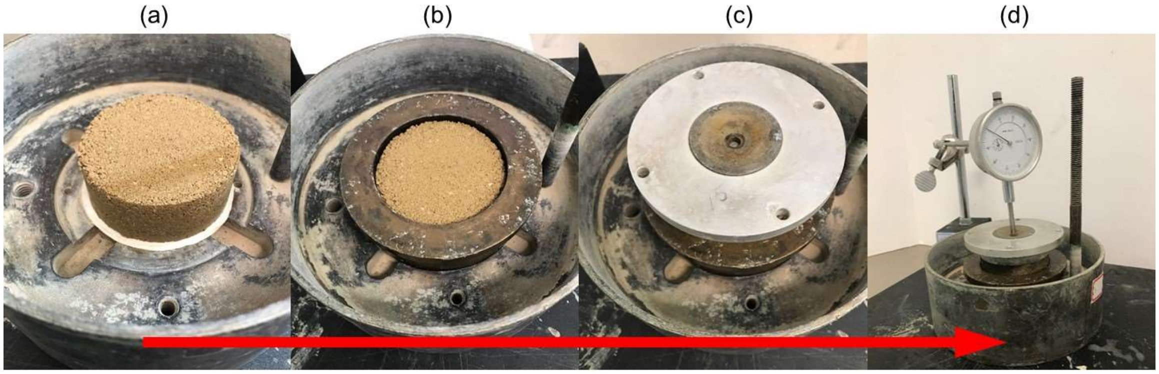


**Figure 4. Experimental procedures for 1-D vertical swell tests: (a) Soil specimen preparation, (b) the specimen is placed in a consolidation ring, (c) applying a seating pressure, and (d) measure the swelling strain using a dial gauge.**

### *4.3.3. Linear Shrinkage Bar Test*

The linear shrinkage bar tests were conducted in accordance with TEX-107-E to study the shrinkage behavior of natural and treated soils (Figure 5). Soil fraction passing the 425 µm (No. 40) sieve was mixed with water until it reached the required consistency. The inside walls of the 19mm x 19mm x 127mm linear shrinkage bar mold were greased with petroleum jelly to prevent adhesion of the soil to the mold. The wet soil was evenly placed in the mold, and the mold was gently shaken to remove any entrapped air bubbles. The top surface of the soil was trimmed with a straightedge, and the mold with the wet soil was placed in an oven at 110 ± 5°C after a slight change in soil color. After 24 hours of oven-drying, the length of the dried soil was measured to determine the linear shrinkage. For each specimen group and curing period combinations, the linear shrinkage bar tests were performed on triplicate specimens to ensure the reliability of the test results.

1. Prepare soil sample

2. Grease the inner wall of the mold with petroleum jelly

3. Fill the wet soil into the mold

4. Measure the length of the dry soil after oven-drying

**Figure 5. Experimental procedures of the linear shrinkage bar test.**

### *4.3.4. Unconfined Compressive Strength (UCS) Test*

The UCS tests were performed per ASTM D2166 to evaluate the improvements in the strength properties of the treated soil (Figure 6). The untreated soil and treated soil mixtures from different sample groups were statically compacted in 67 mm height and 33 mm diameter cylindrical molds to reach the same target dry densities and water contents as that considered for the 1-D free swell test specimens. After the stipulated curing periods, triplicate specimens of each group were subjected to UCS testing at an axial strain rate of 1%/min.

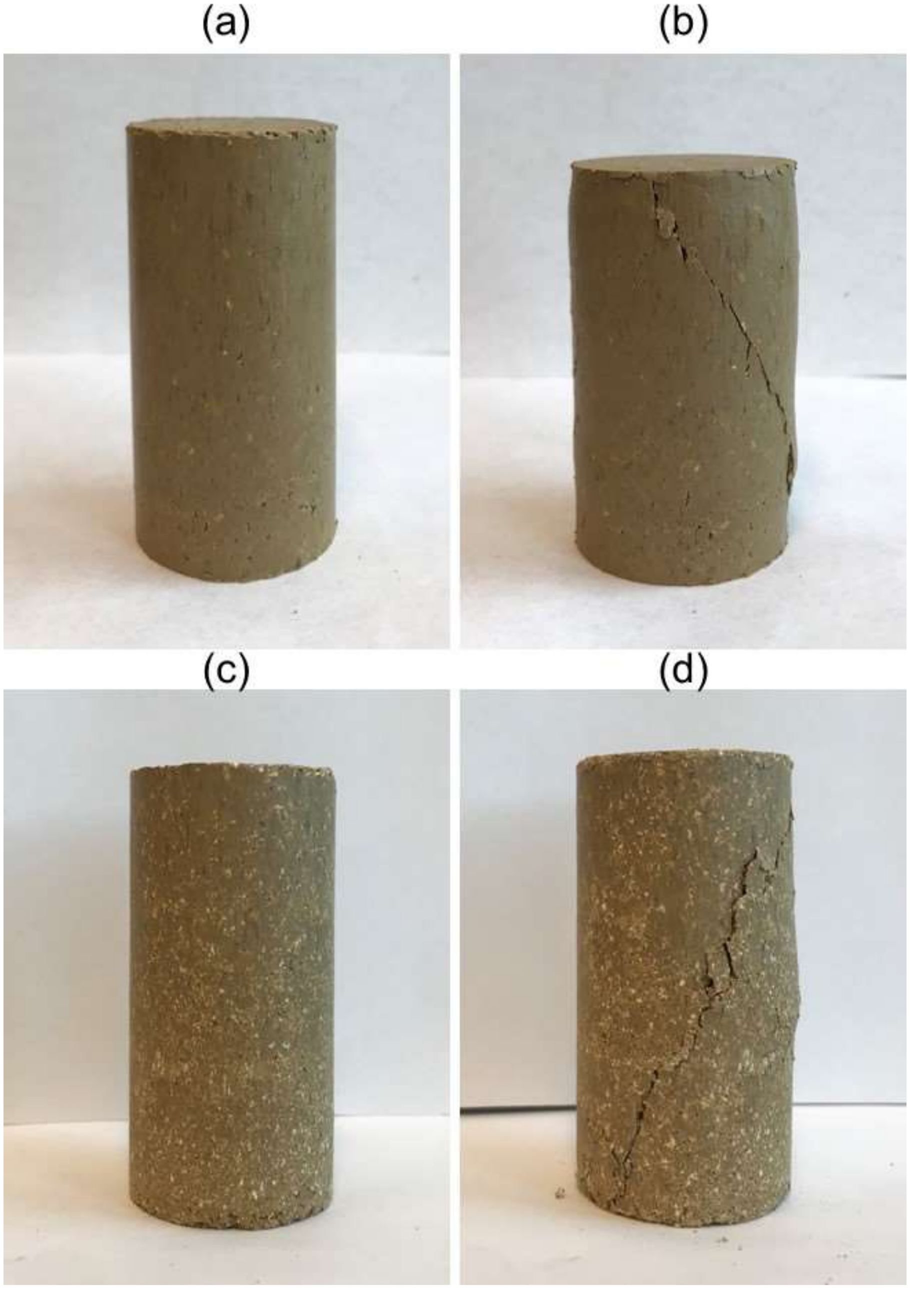


**Figure 6. UCS tests: Natural soil specimens (a) before and (b) after testing and geopolymer-treated soil specimens (c) before and (d) after testing.**

### *4.3.5. Micro-Materials Characterization*

Micro-materials characterization was carried out using SEM-EDS and XRD on GP-stabilized soil samples to observe the change in morphology and any phase changes. SEM and EDS were performed using the JEOL JSM-7500F (JEOL USA Inc, MA) Field emission scanning electron microscopes (FE-SEM), and the SEM images were taken with the JEOL SEM software while EDS were taken and analyzed using the INCA software. XRD is performed with the Bruker D8 X-ray (Bruker AXS LLC, WI) using Cu-Kα radiation.

# 5. ANALYSIS AND DISCUSSON OF RESULTS

## 5.1. Preliminary Test for Narrowing Down GP Composition

K331 and K431 were chosen based previous work as mention in methods. Therefore, a preliminary test using 30% GP dosage and 3-day curing is conducted to determine the effectiveness of each of the compositions. To make sure the result in reproducible, three replicates are made for each set, and the result can be found in Figure 7. Even though K431 significantly improves the mechanical property of the soil, K331 improves the mechanical properties even more. Therefore, K331 is chosen as the GP composition for the rest of the study, and all reference of GP implies the uses of K331 unless specified otherwise.

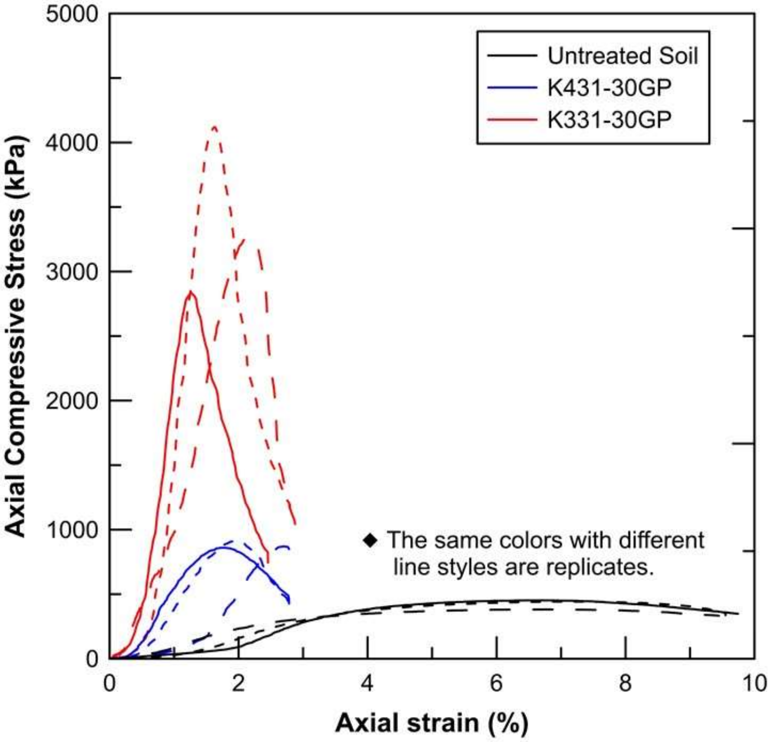


**Figure 7. Stress-strain curve of preliminary GP composition as a stabilizer.**

## 5.2. One-Dimensional Free Swell Test

The 1-D free swell test results of the untreated and 3-day cured chemically treated soil specimens are presented in Figure 8. . The untreated soil experienced a swell strain of 12.2% due to clay mineral swelling, and a major part of the swell strain was accumulated within one day of moisture exposure. Lime treatment of the LS, MS, and HS soil groups resulted in an immediate reduction in the clay mineral-induced swelling due to modification of the soil by cation exchange, flocculation-agglomeration, and reduction in the thickness of the double diffuse layer around the clay minerals. However, beyond one day of submergence in water, the L-HS specimens started swelling due to ettringite formation, crystal growth, and hydration. The ettringite-induced heaving was not prominent in the L-MS and L-LS specimens due to the lower concentration of soluble sulfates as compared to the L-HS specimens. Overall, the swell test results of the lime-treated specimens indicate that lime stabilization was ineffective in stabilizing sulfate soils. The swell strain accumulated in 30GP-HS specimens were much less than the lime-treated specimens, and this indicates that geopolymer is effective in treating sulfate-rich expansive soils. The 30GP-MS and 30GP-LS specimens exhibited negligible swell strain as compared to the other specimen groups. Overall, the swell test results affirm that MK-based geopolymer has a strong potential for utilization as a sustainable soil stabilizer.

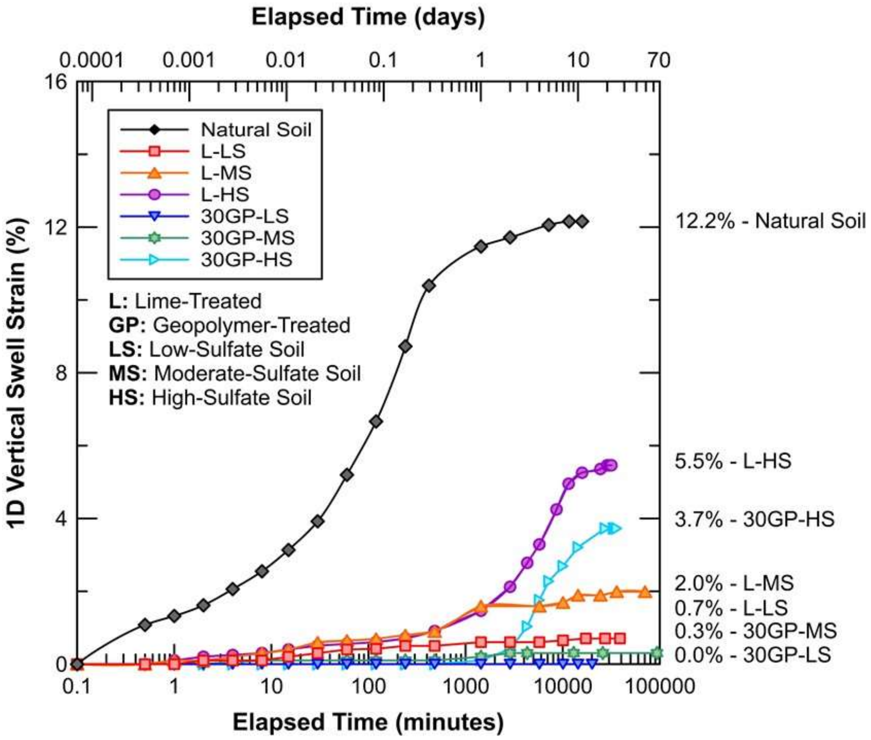


**Figure 8. Swell test results for 3-days cured specimens of different soil groups.**

## 5.3. Linear Shrinkage Bar Test

Linear shrinkage bar tests were conducted on untreated, lime-treated, and geopolymer-treated soil specimens to study the effect of the stabilizers in mitigating shrinkage characteristics of the soil. The results are presented in Figure 9. The natural soil had a shrinkage strain of 15.1%, which shows its high shrinkage potential. The high shrinkage potential is consistent with the high plasticity index and appreciable clay fraction present in the soil. The linear shrinkage strains decreased immediately after lime treatment of soils belonging to all three sulfate levels. The changes in texture, plasticity, and reduction in the water holding characteristics reduced the shrinkage strain in L-HS, L-MS, and L-LS specimen groups. Treatment with 8% geopolymer resulted in shrinkage strain reduction comparable with lime treatment. Increasing the geopolymer dosage to 30% further reduced the shrinkage strains due to the formation of a stabilized matrix with low plasticity characteristics. HS, MS, and LS soils stabilized with 30% geopolymer and cured for 14 days exhibited granular soil like characteristics, and it was not possible to measure the linear shrinkage strain using the linear shrinkage bar test. Generally, the beneficial effect of geopolymer treatment increased with an increase in geopolymer dosage and curing period. The results of free swell test and linear shrinkage bar test indicate that geopolymer is very effective in reducing swell-shrink characteristics of expansive soils with low to high soluble sulfate concentration.

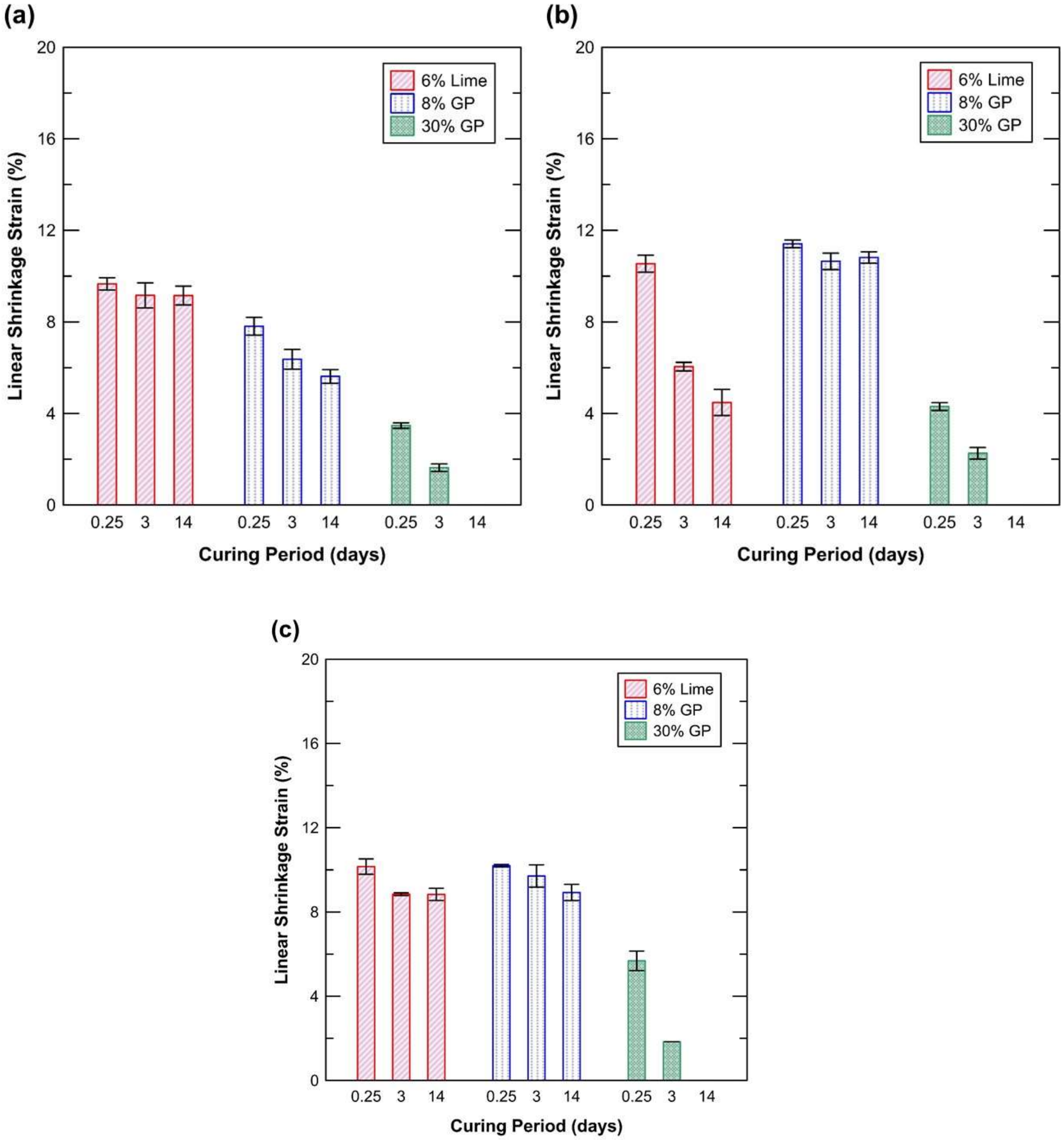


**Figure 9. Linear shrinkage bar test results for (a) low-sulfate (LS), (b) moderate-sulfate (MS), and (c) high-sulfate (HS) soil groups treated with lime and geopolymer.**

## 5.4. Unconfined Compression Strength Test

The UCS results of untreated, lime-treated, and GP-treated are presented in Figure 10. The natural soil has UCS of 145 kPa, which is not sufficient to serve as pavement subgrade. Lime treatment of the LS, MS, and HS soils increased the strength properties with curing period due to the formation of cementitious phases like calcium silicate (C-S-H) and calcium aluminate hydrate (C-A-H). The UCS values of the L-MS specimens were higher than the L-LS specimens, possibly due to the formation of ettringite crystals in moderate quantity. The ettringite crystals have needle-like structures and can contribute toward strength of the stabilized material. However, UCS of the L-HS specimens was less than the L-MS specimens due to the higher sulfate content. This resulted in higher quantity of ettringite crystal formation and potentially had a deleterious effect on the treated soil matrix. Even though lime treatment increased the strength properties, the UCS values of the lime-treated soils did not satisfy the minimum strength requirements recommended by different agencies (*47*, *48*).

Geopolymer treatment of the soil improved its strength properties, and the extent of strength improvements was strongly dependent on the geopolymer dosage. In the initial curing periods, 8% geopolymer treatment imparted comparable strength properties as lime. However, the time-dependent progressive strength gain was not appreciable in the 8% geopolymer-treated specimens. This may be attributed to the inadequate geopolymer network that could not effectively form a strong stabilized matrix. With an increase in geopolymer content to 30%, the UCS values increased drastically and satisfied the UCS requirements recommended by the United States Army Corps of Engineers (USACE) for use as subgrade or subbase material. The UCS values indicate that geopolymer dosage can result in strength improvements similar to or even better than traditional calcium-based stabilizers like lime.

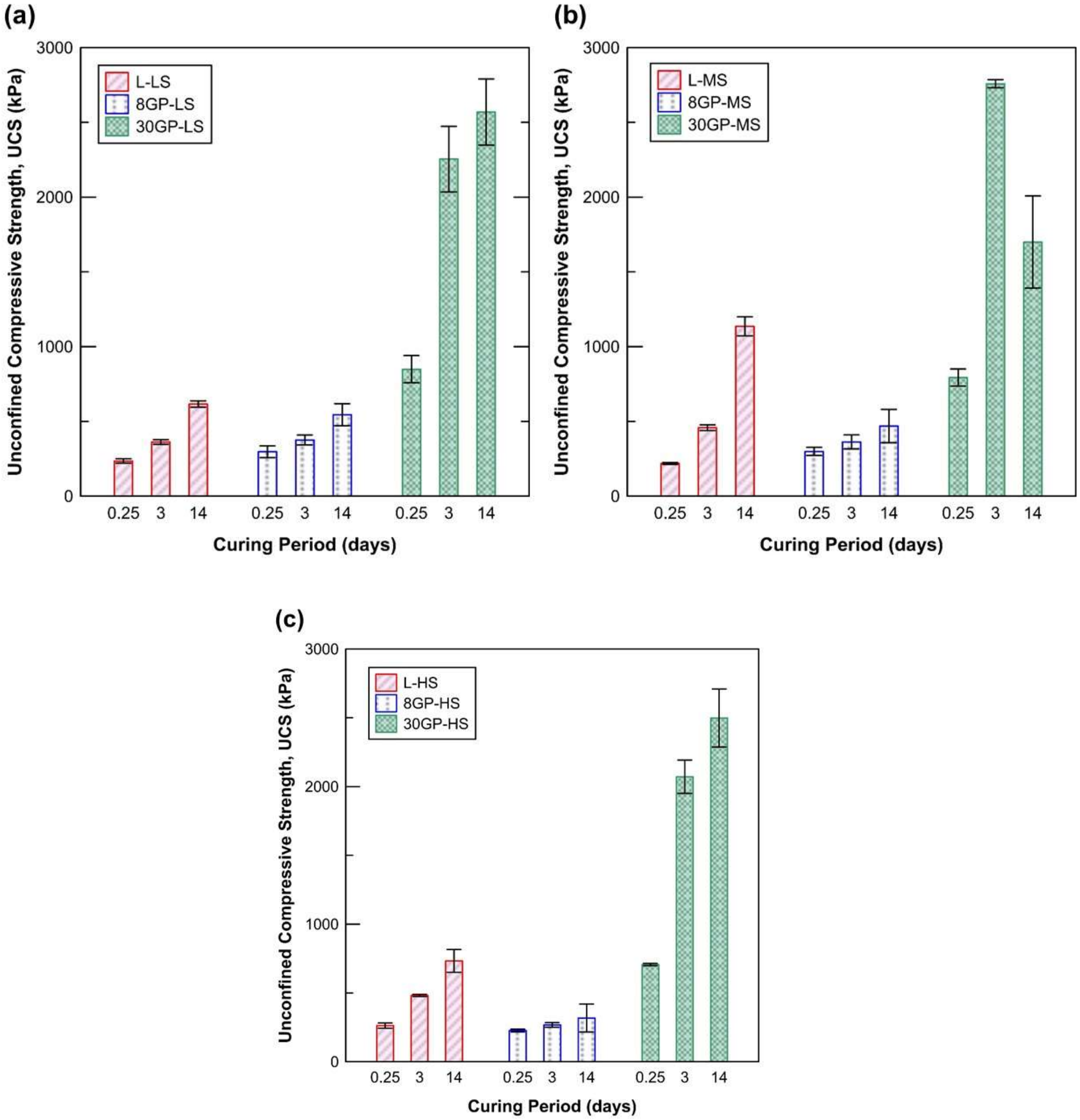


**Figure 10. Unconfined compressive strength test results for (a) low-sulfate (LS), (b) moderate-sulfate (MS), and (c) high-sulfate (HS) soil groups treated with lime and geopolymer.**

## 5.5. Micro-Materials Characterization

The result of SEM-EDS analysis is presented in Figure 11. Figure 11 illustrates the morphological difference in the soil once GP is added in as a stabilizer. Under SEM, native soil has typical morphology that one would expect for the soil with a high in clay mineral content, where it's somewhat loosely packed while having a variety of different morphology from all the clay minerals. When GP is added, it can be identified as the features with smooth surface, this is mainly because GP is an amorphous gel.

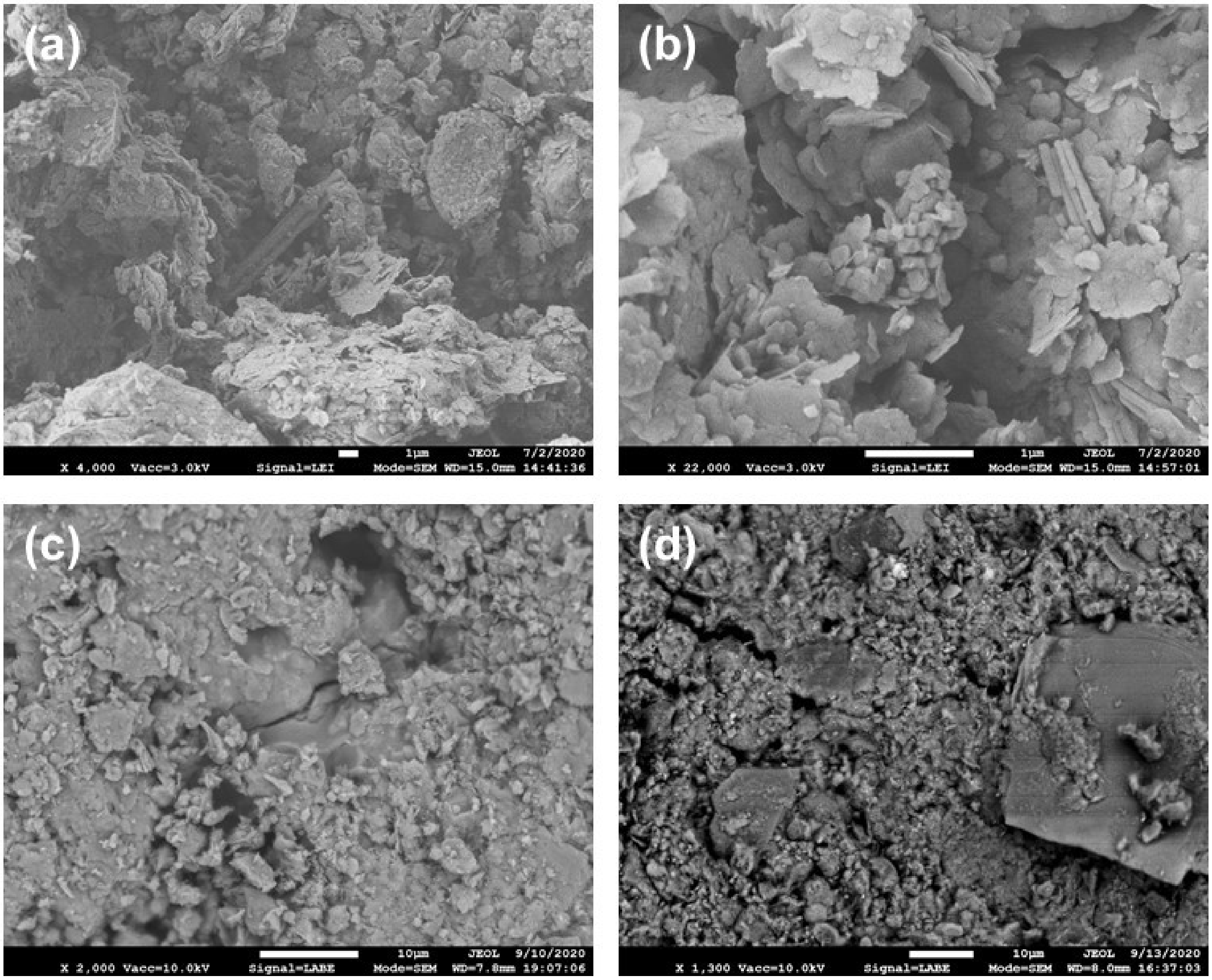
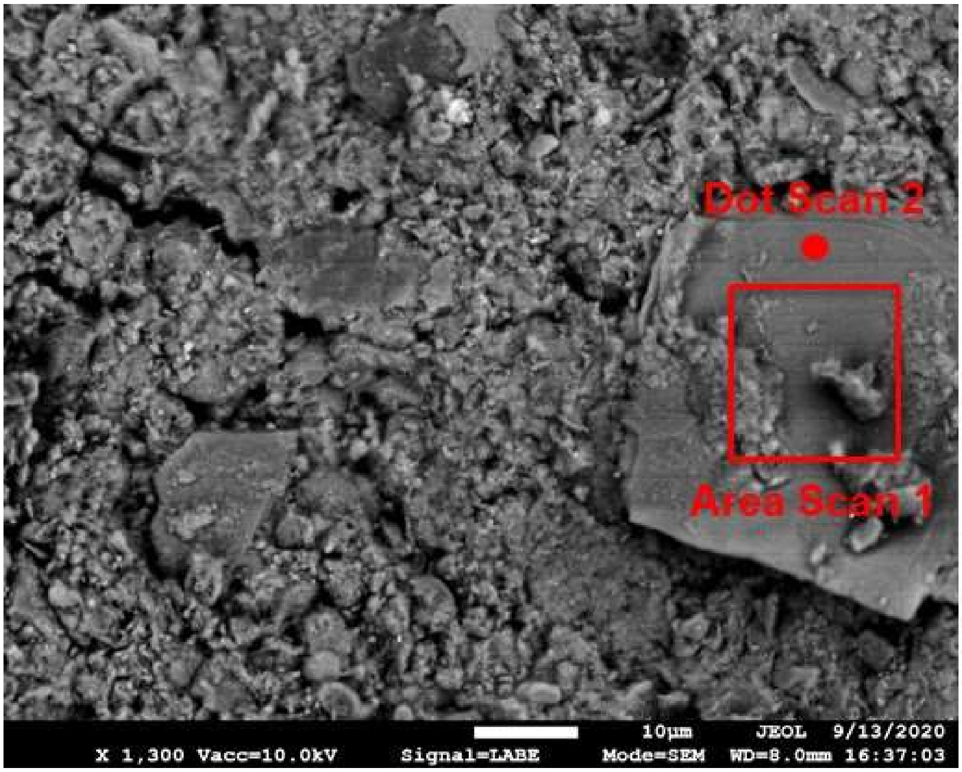


**Figure 11. SEM of (a) native soil under low magnification, (b) native soil under high magnification, (c) GP under soil, (d) GP chunks throughout soil.**

To confirm that those smooth features are GP, several EDS analyses were done on the largest chunk of GP presented in the SEM. From both the area and dot scanned done in Figure 12, the molar ratio of Si/Al are both roughly 1.3-1.45, which is very close to the theoretical Si/Al of the GP used in this study ($SiO_2/Al_2O_3 = 3$ or Si/Al = 1.5). However, the potassium content is not at the ratio that we are expecting. For K331, K/Al = 1, but in both the area and dot scans, K/Al = 0.30-0.35. This most likely means that potassium leached out from the GP into the soil. This is also confirmed EDS mapping result in Figure 13. Looking at the elemental map for potassium, it was expected that it would be concentrated at where the GP is, however, the signal is actually distributed evenly throughout the image. On the other hand, it can be observed that the lack of Ca is a good indicator for where the GP is supposed to be since the composition of clay minerals contains a lot of silicon and aluminum.



| | Area 1 | Dot 2 |
|---|---|---|
| Element | Atomic % | Atomic % |
| O K | 76.22 | 76.50 |
| Mg K | 0.51 | 0.43 |
| Al K | 8.02 | 8.39 |
| Si K | 11.82 | 10.94 |
| K K | 2.71 | 2.63 |
| Ca K | 0.51 | - |
| Ti K | 0.15 | - |

**Figure 12. SEM-EDS result on area with GP.**

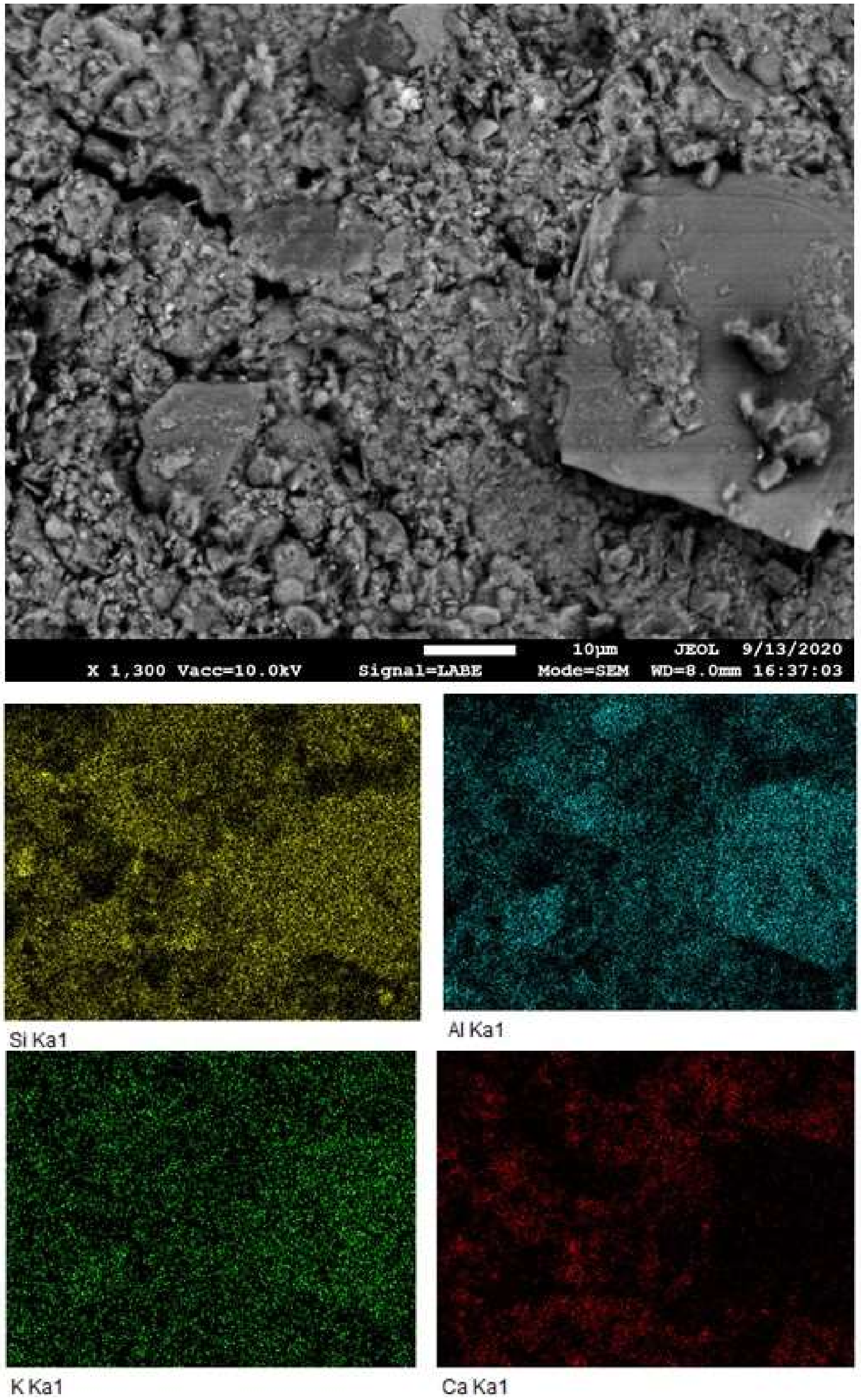


**Figure** 13. **SEM-EDS Elemental Mapping of GP treated Soil.**

XRD analysis is conducted on untreated and GP-treated soil to observe any potential phase changes within the soil after the treatment. Below in Figure 14 shows the XRD spectra of both untreated and 30% GP-treated soil, and when comparing the peaks, the 2 spectra show no difference, meaning there are no formation of new phases in the soil after the GP treatment. The only observable difference is that the overall intensity in the GP-treated soil is lower when compare to the untreated soil. This is an expected change since GP is known to be XRD amorphous.

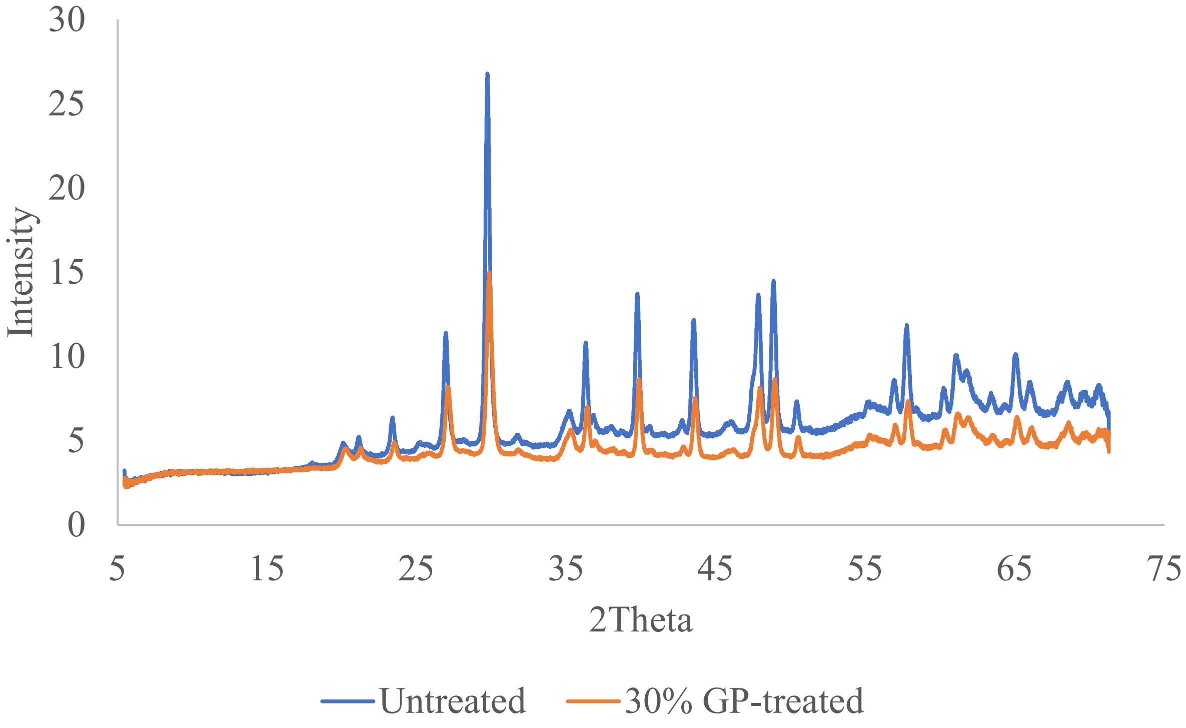


**Figure 14. XRD Spectra of Untreated and GP-treated Soil.**

# 6. SUMMARY AND CONCLUSIONS

This research study aimed to evaluate the suitability of utilizing metakaolin-based geopolymer to stabilize expansive soils with low to high soluble sulfate levels. Extensive characterization tests, engineering tests, mineralogical and microstructural tests were performed to study the behavior of sulfate soils treated with lime and geopolymer. The following conclusions can be drawn based on the findings of the research study:

1) Lime treatment was ineffective for stabilizing sulfate-rich soils. Although lime treatment suppressed the clay mineral swelling, the presence of sulfates resulted in the formation of ettringite and associated heaving. The swell test results affirm that geopolymer treatment is more effective in stabilizing expansive soils with low to high sulfate contents compared to traditional calcium-based stabilizers.
2) The shrinkage test results indicated that both lime and geopolymer treatment are effective in reducing the shrinkage potential of the soil. The shrinkage strain of the soil was reduced with an increase in geopolymer dosage and curing period. Therefore, geopolymer treatment is effective in reducing swell-shrink characteristics of expansive soils.
3) The strength properties of lime and geopolymer treated soil were similar for low geopolymer dosage. With an increase in geopolymer dosage and curing period, the strength properties increased appreciably compared to lime treated material. Hence, geopolymer treatment can improve the strength properties of the treated material.
4) The improvement in engineering properties of the geopolymer treated soils suggests that geopolymer has a strong potential for utilization as a sustainable and eco-friendly soil stabilizer.

As a part of the implementation phase (Phase II), mineralogical and microstructural studies are in progress to study the changes in the behavior of untreated and chemically treated soil specimens. Moreover, life cycle assessment studies will be conducted by identifying and quantifying all the socio-economic factors, environmental factors, and life cycle inventory factors for constructing a hypothetical field section.

# 7. RECOMMENDATIONS

Geopolymer-treated soils with low to high sulfate contents exhibited appreciable improvement in engineering and mechanical properties in controlled laboratory environment. Further field implementation studies are needed to assess the performance of the treated material exposed to seasonal fluctuations. The suitability of utilizing different aluminosilicate-rich waste materials for geopolymer synthesis and soil treatment needs to be explored.

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