Transportation Consortium of South-Central States

Tran-SET

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

Development of Environmentally-Friendly Stabilization Methods for Transportation Infrastructure Based on Geoploymers

Project No.17GTTAM02

Lead University: Texas A&M University

Collaborative Universities: University of Texas at Arlington

Preserving the Environment

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October 2018

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16. Abstract

Geopolymers (GPs) have received much attention as an eco-friendly and sustainable alternative to conventional chemical additives, since they can be processed at room temperatures from aqueous solutions by utilizing waste materials (e.g. fly ash) or abundant natural sources (e.g. clay). A collaborative research study is carried out by teams from Texas A&M University (TAMU) and University of Texas at Arlington (UTA) to investigate the effectiveness of GPs for stabilizing base and subgrade materials by considering the strength and shrink-swell characteristics. Two types of clay, obtained from the North Dallas area, were mixed with a potassium-based GP synthesized from metakaolin and silica fume, at a ratio of 8 wt% of dry geopolymer to dry soil, and cured for a period of 7 days. Moisture-density relationship tests conducted show that treated soils have a higher optimum moisture content and lower maximum dry density as compared to untreated soils. Treated soils exhibited significant reduction in shrinkage and swell properties as compared to untreated soils. Additionally, unconfined compression strength tests also show an increase in strength of treated soils.

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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)		
^oF	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	^oC
		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)		
^oC	Celsius	1.8C+32	Fahrenheit	^oF
		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

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ACRONYMS, ABBREVIATIONS, AND SYMBOLS

AASHTO	American Association of State Highway and Transportation Officials
ACerS	American Ceramics Society
ASTM	American Section of the International Association for Testing Materials
DSC	Differential Scanning Calorimetry
EDS	Energy Dispersive X-Ray Spectroscopy
GP	Geopolymer
LL	Liquid Limit
OMC	Optimal Moisture Content
OPC	Ordinary Portland Cement
PI	Plasticity Index
PL	Plasticity Limit
RH	Relative Humidity
RPM	Revolutions Per Minute
SEM	Scanning Electron Microscopy
TAMU	Texas A&M University
Tran-SET	Transportation Consortium of South Central States
TRB	Transportation Research Board
UCS	Unconfined Compressive Strength
USCS	Unified Soil Classification System
UTA	University of Texas at Arlington
XRD	X-Ray Diffraction

EXECUTIVE SUMMARY

Current soil stabilization methods are often limited by durability and leaching issues, and do not always offer sustainable treatments. This research explores the use of geopolymers to stabilize clays in the North Texas area. In recent years, the use of alumino-silicate polymers, commonly referred to as Geopolymers (GPs), has received much attention as an eco-friendly and sustainable alternative to conventional chemical additives, since they can be processed at room temperature from aqueous solutions by utilizing waste materials (e.g. fly ash) and/or abounded natural sources (e.g. clay).

Teams from Texas A&M University (TAMU) and University of Texas at Arlington (UTA) collaborated in this study to explore the concept of geopolymers, different methods to synthesize geopolymers, and to effectively synthesize a geopolymer composition to stabilize clayey soils. Material characterization of geopolymers as well as treated and untreated soils were conducted using x-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. Additionally, engineering characterization tests were conducted to investigate the effectiveness of GPs for stabilizing base and subgrade materials. Effects of GP composition, dosage rates, and curing time and temperature on properties of GP stabilized base and subgrade materials were studied to optimize the use of GPs derived from local waste and natural materials. Two subgrade soils from North Texas were treated with GP mix at a ratio of 8 wt% dry GP to dry soil. GP is shown to reduce swelling and shrinkage potential of soil considerably while an increase in unconfined compressive strength is observed as well. Therefore, further studies are recommended to understand the mechanism of GP and soil bonding resulting in said changes.

IMPLEMENTATION STATEMENT

The analyses and results of this research are planned to be disseminated to various agencies and industry research partners. The progress of this research has already been presented at the 2018 Transportation Research Board (TRB) and 2018 Tran-SET conferences. Results were also presented to attendees of the Innovation Day 2018 outreach at UTA, which garnered much interest. Further findings from the research are also planned to be presented at the 2019 GeoCongress, 2019 TRB, and 2019 American Ceramics Society (ACerS) conferences, in the form of papers and oral presentations. GPs are to be introduced as a soil stabilizing material in the ‘CE334: Soil Mechanics’ and ‘CE5374: Ground Improvement’ courses at UTA, and in the ‘MSEN625: Mechanical Behavior of Materials’ and ‘MSEN410: Materials Processing’ courses at TAMU.

1. INTRODUCTION

Transportation infrastructure in Texas and its neighboring states has been frequently built on highly compressible soils, which lack the strength to support structures during their construction or service life. Conventionally, chemical stabilization techniques using cementitious materials and polymers have been used to increase strength and stiffness properties of these soils. Although these chemical stabilization techniques are widely used, they are limited by durability and leaching issues, resulting in infrastructure failure. In addition, currently used soil stabilizers do not offer sustainable and eco-friendly treatments. As such, there is a need for new and improved ground improvement solutions that are sustainable, durable, and enhances the engineering properties of soils for transportation infrastructure. This research explores the use of geopolymers as a soil stabilizer for subgrade soils commonly found in North Texas.

Alumino-silicate binders, known as Geopolymers (GPs), are proposed to be a sustainable and eco-friendly alternative to conventional chemical stabilization techniques *(1–3)*. GPs harden at ambient temperatures in a relatively short amount of time *(4)* and can be synthesized by curing activated solutions of various alumino-silicate sources including natural minerals (e.g. clay), their products (e.g. metakaolin), and waste materials (e.g. fly ash, furnace slag, etc.). GPs are known for their high compressive strength and low shrinkage properties, and have been used in recent years as a sustainable alternative to ordinary Portland cement (OPC) in concrete structures, including pavements, bridges, etc. GPs have a much lower carbon footprint than lime and OPC *(5)*, and is therefore more environmentally friendly than other conventional additives used for soil stabilization. This eco-friendly nature of GPs over conventional chemical stabilizers prompted the present research team to investigate the feasibility of GP for effective stabilization of pavement bases and subgrades.

2. OBJECTIVES

The overall objective of this study is to develop an innovative, sustainable, and eco-friendly solution to provide resilient stabilized base and subgrade foundation support for pavements in Region 6, using natural and waste materials that abound in the region.

More specific objectives of the research are to:

- Select composition of Geopolymers with optimum workability and properties for soil stabilization;
- Select appropriate base and subgrades in Region 6 suitable for Geopolymer treatment;
- Validate the selection through comprehensive characterization of Geopolymers treated base and subgrades; and
- Optimize composition of Geopolymers and solid stabilization parameters.

3. SCOPE

The scope of this research includes the treatment of native soils obtained from Region 6 with environmentally-friendly, sustainable, and inexpensive Geopolymer-based methodology for soil-stabilization. A comprehensive literature review was conducted in this study to understand the nature and background of geopolymer materials, their synthesis and use in soil stabilization. Geopolymers are a relatively new material that are used to stabilize soils, as such, extensive literature is not available in this regard. Standard tests used for evaluating the effectiveness of chemicals for soil stabilizations based on ASTM D4609-94, such as unconfined compressive strength, shrinkage limit and 1-D swell tests were conducted to evaluate the efficiency of geopolymers as a soil stabilizer. Individual limitations of the tests are discussed in the following sections of this report. Geopolymers used in this research were synthesized using a specific proportion of ingredients prepared by the research team based on the literature review, and may vary in strength and durability properties if not replicated exactly. Soils used for testing geopolymer treatment were obtained from the North Texas area, therefore analyses and results from this research may not be applicable for soils from different regions or geopolymers with varying compositions than which is described in this report.

4. METHODOLOGY

The primary focus of this research is to evaluate the effectiveness of GP-based soil stabilization for local soils. To this end, the research was categorized into six tasks, which for the most part, are also in their chronological order of execution: (1) literature review, (2) synthesis and characterization of GP, (3) acquisition of local soils, (4) testing program for characterization of soils, (5) GP treatment of soils, and (6) evaluation of sustainable benefits and life cycle assessments. While most of the literature review was conducted initially and was beneficial in developing an effective research strategy, it was an on-going process that consistently molded our approach and understanding of GPs based on the expectations and outcomes of each task. Once the local soils were obtained and the ratios for GP synthesis was finalized, the soils were subject to testing programs for their characterization. The testing program is divided into three categories: basic, engineering characterization, and material characterization tests. XRD and SEM tests were also conducted as part of the material characterization tests. The following sections detail the procedures and outcomes of the tasks conducted to evaluate the effectiveness of GPs as a soil stabilizer.

4.1. Literature Review

The term 'Geopolymers' was coined by Joseph Davidovits in the 1970s for alumino-silicate polymers synthesized from rock-forming minerals *(1)*, which were used as fire-resistant coating materials. This initial work was then expanded and is now present in various applications such as: fire protection for cruise ship *(6)*, resin of high-temperature carbon-fiber composite *(7)*, thermal protection for wooden structure *(8)*, and more *(9)*. Moreover, geopolymers have been further developed and now find a wide range of applications such as: construction material alternative to OPC *(9)*, nuclear waste immobilization *(10)*, water purification (heavy metal immobilization) *(10)*, and low-energy processing route to ultra-refractory ceramics powder (such as SiC and Si_3N_4) *(11)*.

During the geopolymerization process, reactive alumino-silicate minerals dissolve in highly alkaline solutions in the presence of an alkali hydroxide and silicate solution to form common Si and Al species, which in turn form chains of Al-O-Si and Si-O-Si bonds during a polycondensation process during which water is expelled *(1)*. The polycondensation process continues to develop 3-D net like features, and ultimately into an amorphous rigid gel known as geopolymer *(3)*. Previous research shows that GP-stabilized soil has unconfined compression strength (UCS) values ranging from 2-7 MPa after 7 days of curing *(2,12)*. However, it is difficult to reproduce these results due to the differences in both soil and precursor (e.g. fly ash) composition. Additionally, most methodologies from existing literature are heavily application-driven and does not thoroughly explore the fundamental science behind the results. It has proved challenging to create an experimental procedure that would allow a systematic study to thoroughly understand how the different parameters affect GP's effectiveness as a soil stabilizer efficiently. In both papers by Zhang et al. *(3,12)* the group conducted well-organized studies on GP's effectiveness as a soil stabilizer, with the first paper finding an effective amount by focusing on mechanical properties, and the second paper further investigating other engineering soil properties at and around the effective amount. It is evident from previous literature that GP works as soil stabilizer and is superior to OPC and lime in terms of durability, but still lacks the attraction due to coming up short in terms of mechanical properties. Based on the review of literature, it is suggested that the usage of GP as soil stabilizer would be more attractive if the same mechanical properties can be achieved while utilizing equal or lesser amount of GP to that of OPC and lime. We have followed Zhang et al.'s *(3,12)* work by starting out with metakaolin since their work contains the most description in methodology, and it is the better choice to gain fundamental understandings on how GP work as soil stabilizers.

4.2. Geopolymer Synthesis and Characterization

The GPs used in this research were synthesized by researchers using potassium hydroxide (Mallinckrodt Chemicals, NJ), 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 alumino-silicate 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 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 Si/Al 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 3 minutes at 400 revolutions per minute (RPM) to create a homogenized mixture, known as GP. Various small samples of GP were also synthesized to observe the viscosity and curing time at different compositions.

Since GP has multiple parameters (i.e. chemical composition, curing time and temperature) that depend on each other, the preliminary study primarily determined the relationship between mixability and water ratio for both sodium and potassium-based GP. Table 1 shows the different compositions of geopolymers synthesized to determine optimum GP composition, wherein it is evident that ambient curing time is dependent on the various parameters (cation, water content, and silica content). It is worth noting that increasing water and silica content decreases viscosity and increases curing time, and that sodium-based GP are more viscous than potassium-based GP at the same water content. Taking into account all these different parameters as well as the mechanical properties from Lizcano *et al. (4)*, it was decided that GP with molar ratio of Si/Al = 2 and Al/K = 1 (GP-ID: K421, K431, K441) would produce the best candidates to maximize both strength and mixability/workability for soil stabilization.

Table 1. Summary of different geopolymer compositions synthesized.

GP- ID	Cation	Si:Al	H_2O:Solid	Al:Cation	Curing time (days)
Na241	Na	1	4	1	1
Na251	Na	1	5	1	1
Na261	Na	1	6	1	1
Na271	Na	1	7	1	6
Na281	Na	1	8	1	6
Na291	Na	1	9	1	24
Na2(10)1	Na	1	10	1	Did not cure
Na341	Na	1.5	4	1	5
Na441	Na	2	4	1	7
K241	K	1	4	1	2
K251	K	1	5	1	4
K261	K	1	6	1	18
K271	K	1	7	1	21
K341	K	1.5	4	1	7
K421	K	2	2	1	7
K431	K	2	3	1	10
K441	K	2	4	1	14

All the GPs used for soil treatment in this research have molar ratios of Si:Al = 2, Water:Solids = 3, and Al:K = 1. Additionally, pure geopolymers were synthesized and characterized by SEM and XRD to validate that the methodology produces geopolymers.

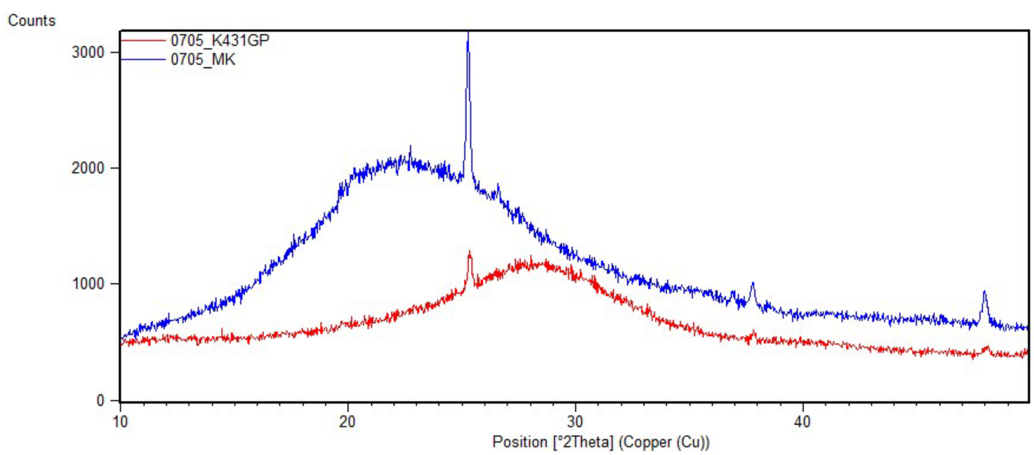


Figure 1. XRD of metakaolin (blue, top) and K431 geopolymer (red, bottom).

The XRD results in Figure 1 demonstrate that selected methodology of synthesizing pure GP indeed does produce GP. As can be seen, the characteristic amorphous hump from $2\theta_{max} \approx 22^o$ for the metakaolin to the $2\theta_{max} \approx 27\text{-}30^o$ for the cured geopolymer *(13)*. While the sharp peaks at $2\theta \approx 27.5^o$, $2\theta = 38^o$, and $2\theta = 48^o$ correspond to the crystalline non-reactive TiO_2 in anatase phase (PDF number = 00-034-0180) which is a known impurity that was already in the metakaolin *(4)*. The SEM results of pure metakaolin and K431 GP are presented later for morphology comparison with untreated and treated soils.

4.3. Soils

Two clay subgrade soils commonly found in North Texas were obtained from Lewisville, TX and Alvarado, TX (Figure 2) to be stabilized with GP. The main criterion for the selection of subgrade soils was based on their Plasticity Index (PI), which ranges between 10 to 60%. Additionally, masonry sand was obtained from a materials supplier in the Dallas-Fort-Worth area as well. All subgrade soil testing was conducted on oven-dried, crushed and pulverized soil, and is explained in detail in the following sections.

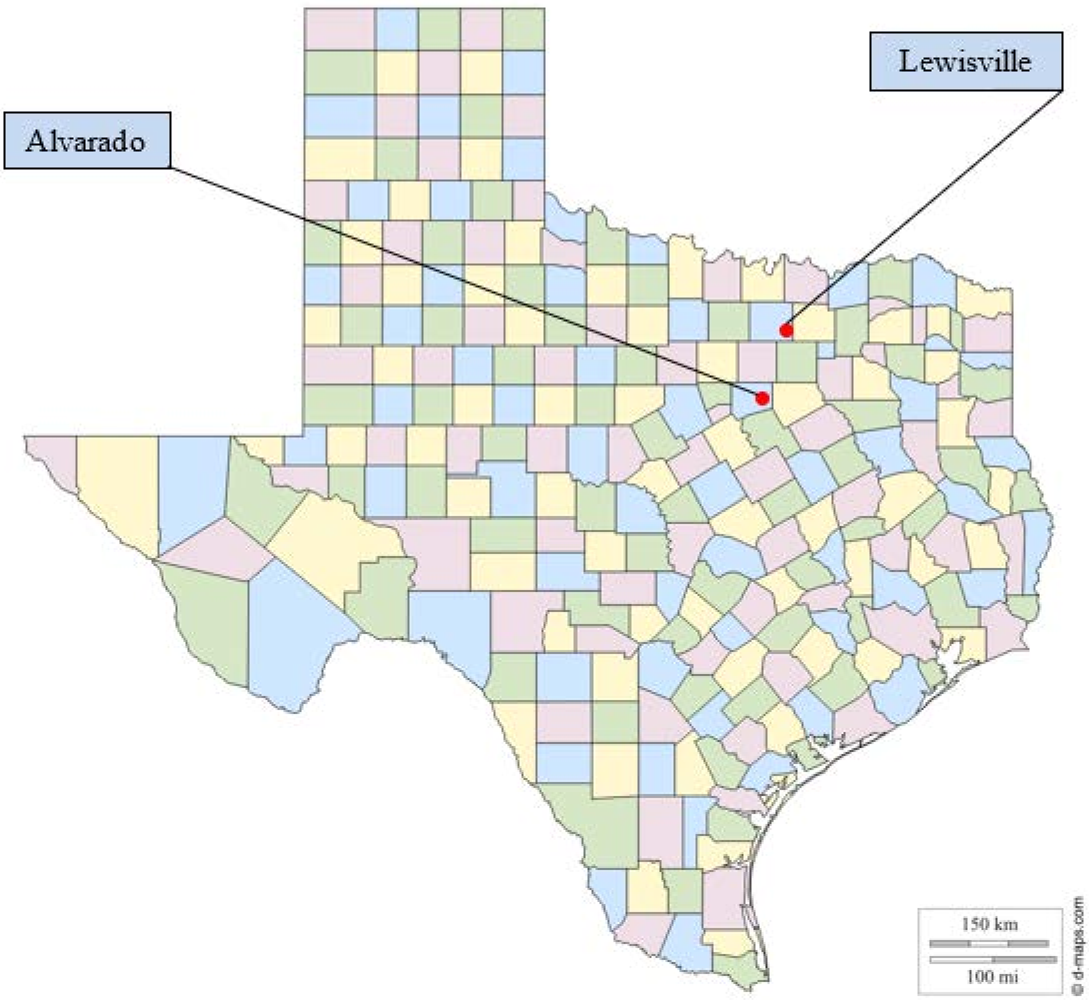


Figure 2. Map showing location of subgrade soils.

4.4. Testing Program

A testing program which includes basic soil index tests and engineering characterization were conducted based on approved testing standards. Basic index tests conducted include particle size distribution tests (sieve analysis and hydrometer), Atterberg limits, water content and specific gravity, while engineering characterization tests conducted include compaction tests, UCS, free vertical (1-D) swell, and linear shrinkage bar tests. In addition to these tests, material characterization tests such as SEM, and XRD were conducted for untreated and treated soils, and pure GP.

4.4.1. Particle Size Distribution

The particle size distribution test provides a distribution of grain sizes within a soil mass and is used to classify soils for engineering purposes according to the Unified Soil Classification System (USCS). The sieve analysis and hydrometer tests on untreated soils were performed as per ASTM D6913/D6913M-17 *(14)* and D7928-17 *(15)*, respectively. Based on the particle size distribution tests, the clay from Lewisville, TX of the Eagle Ford geological formation was classified as a high-plasticity clay (CH), while the clay obtained from Alvarado, TX was classified as a low-plasticity clay (CL), and the masonry sand obtained from the DFW Metroplex was classified as a poorly-graded sand (SP), as shown in Table 2. Further tests of Atterberg limits confirmed these results. Henceforth, the three types of soils will be addressed by their USCS classification.

Table 2. Summary of gradation tests: Sieve analysis and hydrometer tests.

Soil Location	Gravel	Sand	Silt	Clay	USCS Classification
Lewisville	0.0%	9.8%	34.3%	56.0%	CH
Alvarado	0.0%	33.6%	33.9%	32.5%	CL
DFW Metroplex	0.0%	99.2%	0.7%	0.1%	SP

4.4.2. Atterberg Limits

The Atterberg limits provide an insight into the plasticity and therefore the shrink/swell potential of cohesive soils. The moisture content at which the cohesive soil passes from a liquid state to a plastic state is known as the liquid limit of the soil. Similarly, the moisture content at which the soil changes from a plastic to a semisolid state and from a semisolid state to a solid state are referred to as the plastic limit and the shrinkage limit, respectively. The Atterberg limits, which include the liquid limit (LL), plastic limit (PL), and PI of the cohesive subgrade soils, were determined on the soil fraction that passes the 425-µm (No. 40) sieve (see Table 2)., as per ASTM D4318-17 *(16)*.

4.4.3. Specific Gravity

Specific gravity (G_s) is the ratio of the weight of a given volume of a material to the weight of the same volume of distilled water, and is important to determine weight-volume relationships of soils. The specific gravity tests of the three untreated soils of this study were conducted as per ASTM D854-14 *(17)*, and determined using a water pycnometer of soils passing the 4.75 mm (No. 4) sieve. The specific gravity values are shown in Table 3 and are found to be consistent with the expected values.

Table 3. Summary of Atterberg limits and specific gravity tests.

Soil Type	LL (%)	PL (%)	PI (%)	G_S
CH	80	27	53	2.78
CL	42	25	17	2.69
SP	n/a	n/a	n/a	2.65

4.4.4. Moisture-Density Relationships

Moisture-density relationships of untreated soils were determined using standard proctor and Harvard Miniature compaction tests, as per ASTM D698-12 *(18)* and GR-84-14 *(19)* respectively. The compaction tests were conducted to determine the optimum moisture content (OMC) at which the soils are compacted to its maximum dry density (MDD). The compaction test results of the three untreated soils are given in Table 4.

Table 4. Summary of moisture-density relationship tests of untreated soils.

Soil Type	MDD (g/cm^3)	OMC (%)
CH	1.57	24.2
CL	1.72	19.9
SP	1.60	8.8

4.4.5. Unconfined Compressive Strength

The unconfined compressive strength (UCS) of a cohesive soil provides a quick estimate of the undrained shear strength of the soil. The relationship between the UCS (q_u) and the undrained shear strength (c_u) of a soil is famously given by the relationship by Das *(20)*:

$$q_u = \frac{c_u}{2} \quad [1]$$

UCS tests were conducted in accordance with ASTM D2166/D2166M-16 *(21)* and ASTM STP479-EB (22) by performing a strain-controlled test where a constant strain rate was selected, and load was applied to a cylindrical soil specimen without any confining pressure (Figure 3). The strains corresponding to the different stresses were recorded. The stress at which the specimen fails is known as the UCS of the soil, which is also the maximum value of the stress-strain curve obtained from the UCS test. Samples were compacted to two types of dimensions, larger samples with approximate diameter and height of 2.8-in. and 5.8-in. respectively, and smaller samples of approximate diameter and height of 1.3-in. and 2.8-in. respectively. All samples have a diameter to height ratio of at least 2 to reduce the end effects of the sample. Results reported here are from smaller samples, as they utilize much less soil than required to mold larger samples and yield representative results as well.

The subgrade soils were mixed with water, so they reach their OMC values and set in a moisture room to be equilibrated for a minimum of 24 hours. The larger samples were molded using static compaction in three equivalent layers at 95% of its MDD value. The smaller samples were molded using the Harvard Miniature apparatus in three equivalent layers using 25 tamps per layer. The molded samples were extruded and set to equilibrate in a moisture room with 100% relative humidity for a period of 24 hours. The specimens were then tested in a UCS testing machine and values were recorded.

(a) (b)

Figure 3. UCS test of CL: (a) before failure and (b) after failure.

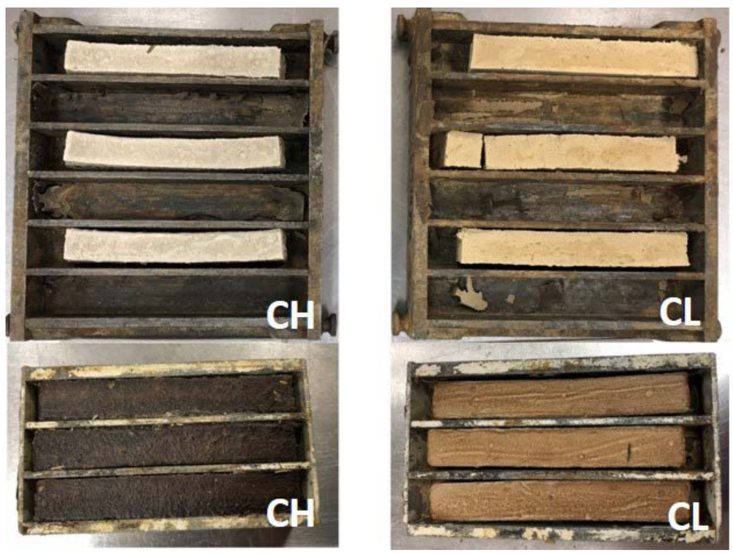
4.4.6. One-Dimensional Swell Pressure

The one-dimensional (1-D) swell test (ASTM D4546-14 *(23)*) provides an estimate of the swell capacity of cohesive soils. A load of 7 kPa (1 psi) was applied, instead of the recommended 1 kPa (0.145 psi) load, as the 1 kPa load would result in very high swell for the heavily expansive soils in Texas. Samples of dimensions 2.5-in. diameter and 1-in. height were placed in oedometers (Figure 4), and the 7 kPa load was applied. Once the load was applied, the swell indicator dials were adjusted to zero, and the sample was inundated with water. Swell dial readings were recorded for a period of 24 hours or until there was no significant increase in swell. The recorded vertical strain was then plotted with respect to time on a semi-logarithmic scale to obtain the swell curve.

Figure 4. The 1-D swell test being conducted in consolidometer.

4.4.7. Linear Shrinkage Bar Tests

This test was conducted in accordance with TEX-107-E *(24)* and is used to determine the bar linear shrinkage of soils (Figure 5). The soil fraction passing the 425-µm (No. 40) sieve was mixed thoroughly with water until it reached the required consistency. The proper molding consistency of the soil required for this test was tested by shaping the soil sample into a smooth layer about 0.5-in. thick and making a groove with the grooving tool. Once the material flowed on its own accord and just closed the groove, it was ready for the test. The insides of the bar mold were greased with petroleum jelly to make sure the soil did not adhere to the mold walls. Then soil was poured in, gently jarred to cause trapped air bubbles to escape, and smoothed with a straightedge. The mold with the soil was then air dried for a few hours and then oven-dried till there was no change in mass. Once cooled, the length of the bars was measured to determine the percentage of linear shrinkage.



(a) **(b)**

Figure 5. Shrinkage of untreated (top) and treated (bottom) samples: (a) CH and (b) CL.

4.4.8. Materials Characterization

CL was characterized using x-ray diffraction (XRD) to obtain an understanding of its mineralogical composition, while all samples (i.e. GP treated/untreated CL, metakaolin, and pure GP) were characterized with SEM to better understand the microstructure. XRD analysis was carried out using a Bruker D8 Advance (Bruker AXS Inc, WI) diffractometer with Cu-Kα radiation source generated at 40 mA and 25 kV, in the 2θ range of 10-75° with 2θ step of 0.02° and at the rate of 0.4 seconds per step. The results of XRD were analyzed with the X'Pert HighScore Plus version 2.2.2 software. SEM analyses of all samples were carried out with the JEOL JSM-7500F (JEOL USA Inc, MA) FE-SEM to study the microstructure of the samples. Samples were obtained from the center of the compacted samples, crushed into fine powder, and then mounted onto aluminum stubs with double-sided carbon tape. They were then sputter coated with 5 nm of platinum-palladium alloy to avoid charging and to enhance the quality of the analysis.

The XRD results in Figure 6 shows that the main crystalline minerals present in CL are low quartz and calcite. From the software's Rietveld analysis, CL is calculated to contain 77% of low quartz and 23% of calcite. The analysis could identify the major phases within CL, but was having trouble identifying the minor phases, that most likely composes less than 5% of CL (i.e. the 3 small unidentified peaks).

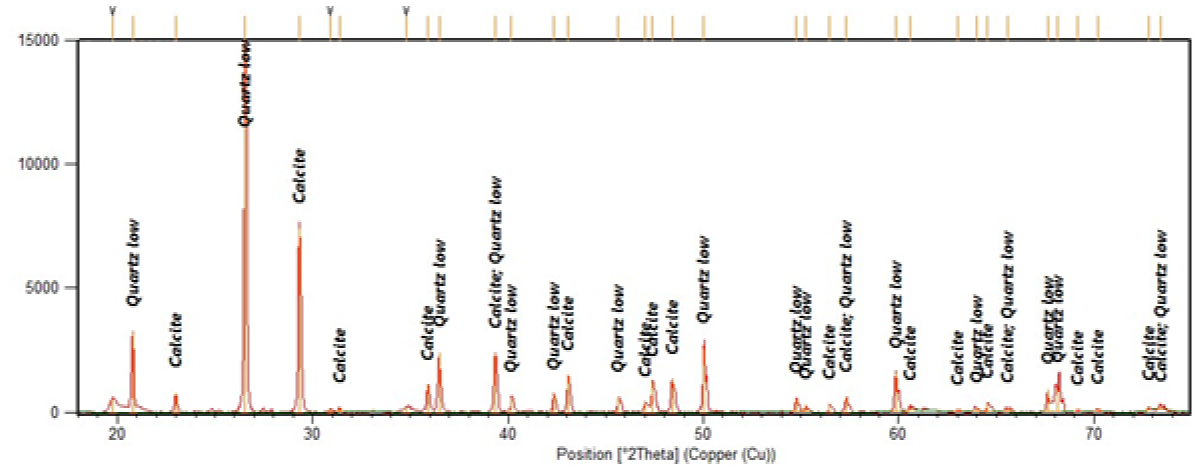


Figure 6. XRD analysis of untreated CL soil.

4.5. Geopolymer Treatment of Soils

The CL subgrade soil was chosen to assess the effects of GP on it, primarily due to the availability of a larger batch of CL to work with. A preliminary study was done to assess the mixability of GP and CL. Subsequently, the three different GP compositions (GP-ID: K421, K431, K441) were mixed with CL and compacted by hand into plastic molds with a diameter and height of 1.25 inches and 0.75 inches, respectively. It was observed that both K431 and K441 mixed well with CL, but K431 was chosen for the first set of testing to maximize strength.

Two methods of mixing were investigated to see how well soil and GP can be combined. In the first method, water was initially added to the dry soil to bring it to its OMC, then covered and placed in a moisture room overnight so the moisture equilibrates throughout the soil. Once the soil was removed from the moisture room, the GP mixture was finally added to the soil which was at its OMC, and mixed thoroughly for about 5 minutes at a ratio of 8 wt% of dry geopolymer to dry soil. In the second method, the water required to be added to a mass of dry soil to bring it up to its OMC, was added to the GP mix instead of the dry soil. The diluted GP mixture was then added to the dry soil and mixed thoroughly for around 5 minutes. The mixture was then statically compacted in 3 layers to make cylindrical samples for the UCS test. Note that the second method of mixing was only investigated and experimented recently, and therefore most of the data in this report are from treated samples mixed using the first mixing method.

The K431 GP mix was used to stabilize CL at an OMC of 20%, at a concentration of 6.6% by weight of the soil at OMC. Henceforth, the term 'treated samples' indicates samples treated with the K431 GP mix at the said concentration. Treated samples were cured and tested for

UCS, shrinkage, swell and Atterberg limits. For UCS testing, treated CL samples were cured for a period of 7 and 28 days, under two different curing methods. One set of treated CL samples were cured for 7 days in a moisture room by misting at 100% relative humidity (RH), while the second set of treated samples were cured in 100% RH for 3 days and then air-dried at about 21°C (70°F) for the remainder of the 4 days. The same was done for samples cured for 28 days as well, where some samples were cured for 28 days in 100% relative humidity (RH), while others were cured in 100% RH for 14 days and then air-dried at about 21°C (70°F) for the remainder of the 14 days. Untreated CL samples were also cured in 100% RH for 7 days, after which both treated and untreated samples were subject to UCS testing.

4.6. Sustainability Benefits and Life Cycle Assessment

Sustainability benefits of a structure are governed by its environmental and socio-economic impacts related to the construction, operation and maintenance of the system. Life cycle assessments of infrastructure systems can quantify these benefits. According to The AASHTO Guide for Design of Pavement Structures *(25)*, life cycle costs can be roughly divided into agency costs and user costs. Agency costs include: initial construction costs, future construction or rehabilitation cost, maintenance costs recurring throughout the design period, salvage or residual value at the end of the design period (a negative cost), engineering and administrative costs, and traffic control costs. User costs include: travel time, vehicle operation, accidents, discomfort, and time delay and extra vehicle operating costs during resurfacing or major maintenance. Socio-economic factors include cost-benefit analysis, local policy constraints, noise and vibrations from construction machinery. Environmental factors include acidification potential, human toxicity potential, eutrophication potential and carbon emissions released as part of construction operations.

Geopolymers are known to have a much lower carbon footprint *(9)*, since it uses by-products of the industry for raw materials (metakaolin, fly ash, etc.), unlike lime. GPs are expected to have a higher socio-economic impact compared to lime, as some of the ingredients needed for the synthesis of GP (silica fume, KOH) have high market values currently. Regarding this, it is important to consider that a few decades ago, lime and cement production were also considered extremely expensive, but once a standard of production was established, the production costs were lowered. Note that construction costs vary depending on labor and material costs, contractor fee, equipment availability and capabilities, project size, availability of work, potential weather delays and many other factors. Likewise, general economic inflation and recession pressures also affect costs from year to year

Consider a hypothetical pavement section on a low-volume road built on treated subgrade. Note that low-volume roads in North Texas are built by constructing a concrete road over treated subgrade *(26)*. This hypothetical road has an average dimension of each field section of 10 ft × 10 ft × 0.75 ft. One of the field sections is treated with 8% GP, while the other is treated with 8% lime. Each stabilizer was added at a rate of 5 lbs per square feet, to a depth of 9 inches (0.75 ft). The soil mixture should be compacted with sheep foot rollers to a density equivalent to 95% of the maximum dry density (MDD) of the composition. It is calculated that about 500 lbs each of the GP and lime are required for each of the field sections.

Based on this information, a sustainability benefits analysis is being evaluated using a multi-criteria assessment of factors such as resource consumption, socio-economic impact, as well as environmental repercussions of the stabilization construction operations *(27)*. The analysis works toward evaluating the sustainability index (I_{SUS}) of a treatment method, which is determined by assigning weights to different impact categories based on their relevance to the treatment method in question *(28)*. The treatment method with the lowest value of I_{SUS} is assigned to be the most sustainable stabilization method. Currently, the assessment of sustainability benefits and life-cycle costs of GP stabilization is ongoing, as such conclusive results cannot be presented in this report.

5. FINDINGS

The basic index and engineering characterization tests conducted on procured subgrade soils revealed the need for stabilization to enhance the strength of these soils. Moisture-density relationship curves were established for both treated and untreated subgrade soils. It is observed that treated soils have a higher OMC and lower MDD values as expected, and is shown in Figure 7.

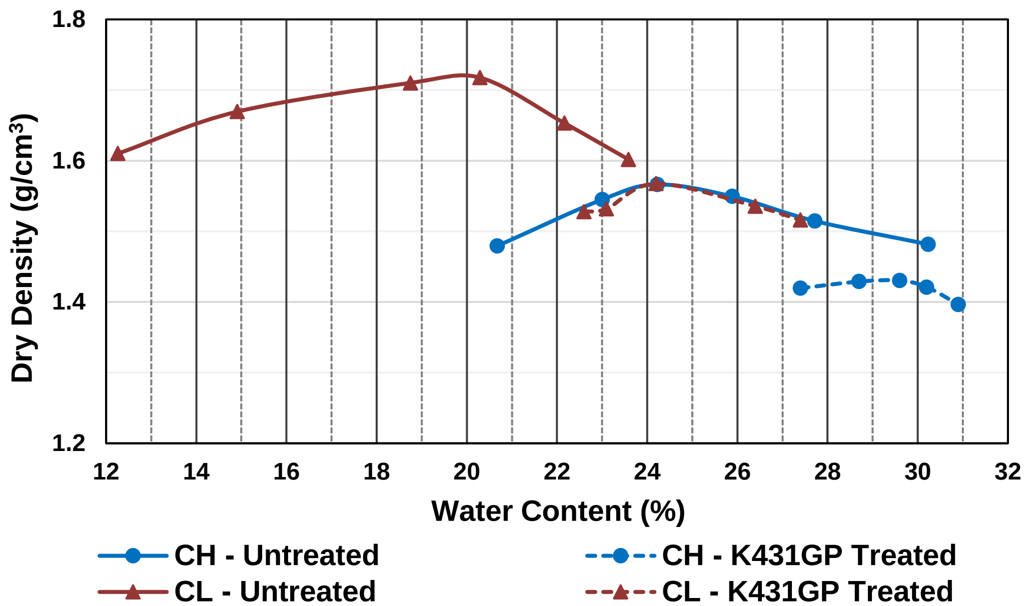


Figure 7. Moisture-density curves for treated and untreated subgrade soils.

Linear shrinkage tests were conducted on treated CL as well as CH samples, both of which indicated that GP treatment resulted in significant reduction of shrinkage compared to untreated samples, as is visually observed in Figure 5, shown earlier. Treated CH and CL samples showed a decrease in shrinkage from 21.6% to 7.3% and from 16.7% to 13.1%, respectively, as shown in Figure 8.

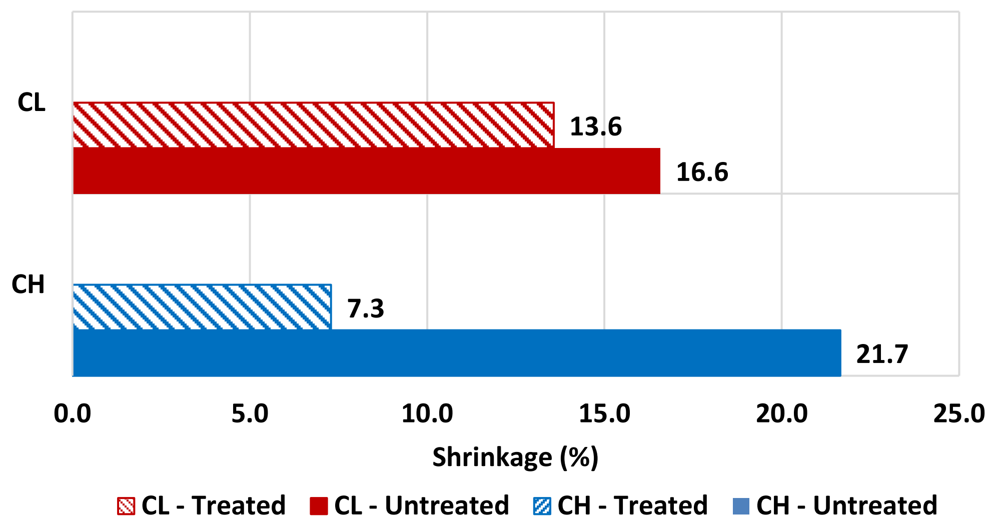


Figure 8. Shrinkage plot of untreated and GP treated subgrade soils.

Atterberg limit tests conducted on treated CL samples showed that GP treatment of CL resulted in a 72% reduction in its PI, from 25 to 7. In addition, Swell tests on treated CL and CH samples indicated a significant reduction in vertical swell strain from 0.9% and 8.8%, respectively to 0.04%.

The UCS of treated CL samples cured in 100% RH for a period of 7 and 28 days showed some strength increase (in the range of 50-75%) compared to the UCS of untreated samples, while the second set of samples that were air-dried for part of the curing period showed significant increase in UCS (in the range of 400-700%) compared to untreated samples (Figure 9). Samples cured for 28 days show that the UCS plateaus after 7 days as there is only about an 18% increase from the strength of 7-day cured samples. Elastic modulus was calculated from the linear portion of the UCS stress-strain curves and was found to be significantly higher (in the range of 100-300%) for GP treated samples as compared to untreated samples at OMC. The increase in UCS strength of air-dried specimens ranges from 75 psi to 100 psi, which is higher than the minimum requirement for the chemical treatment to be considered effective (50 psi as per ASTM D4609-94). Note that, the treated and cured specimen were observed to disintegrate and slake when soaked in water.

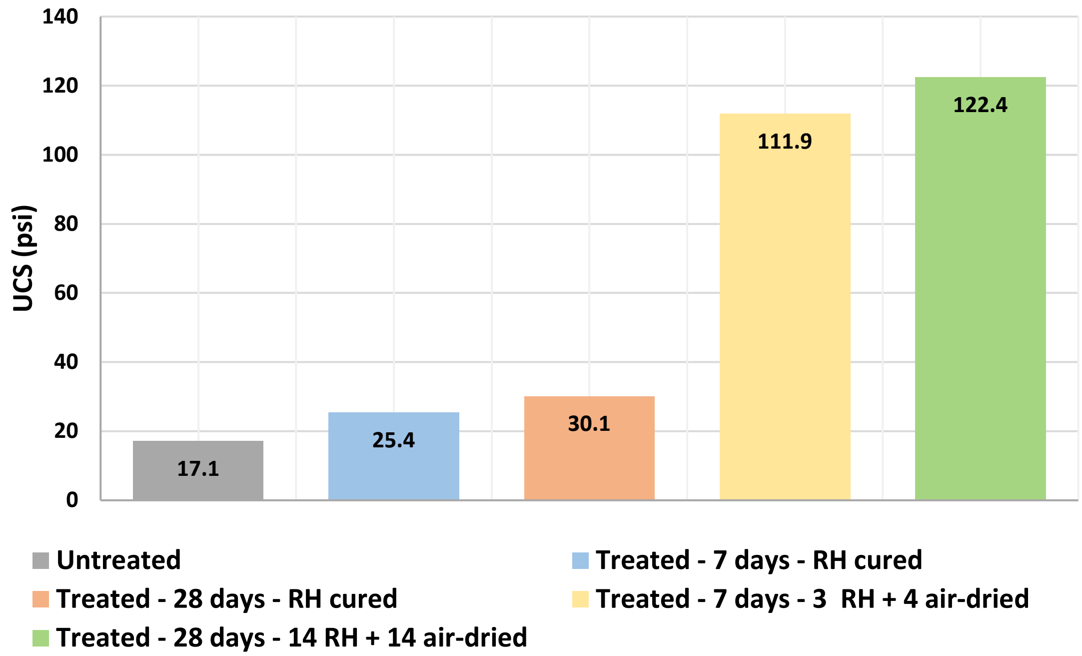


Figure 9. UCS test results of untreated and treated samples cured for 7 days using 2 curing methods.

On comparing the two mixing methods used for treating CL soil with GP, unexpected differences were observed in both physical appearance and strength. Specimen made using mixing method 1, where GP is added to soil at OMC, are observed to have more soil clumps and roughness (see Figure 10-a, c), while specimen made using method 2, where GP and water are added to dry soil, have a more uniform surface with no apparent cracks (see Figure 10-b, d). Although, the cross-section of the specimen made using method 2, shows that GP was not well-mixed into the soil and have instead formed spots of GP. As expected the non-homogeneous distribution of GP resulted in considerably lower strength in specimen made using method 2, as seen in Figure 11.

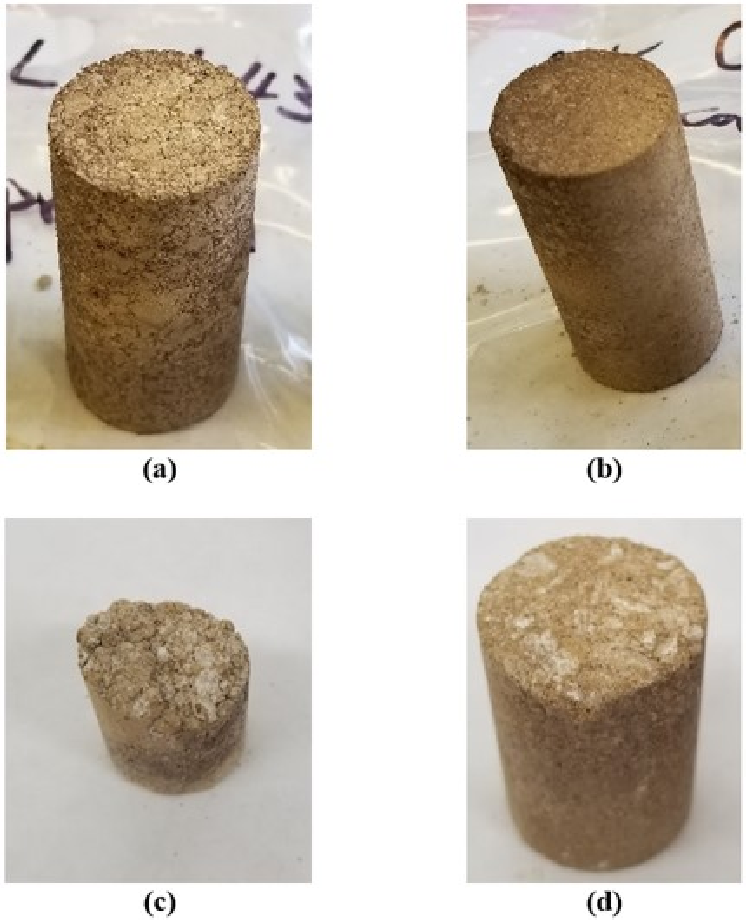


Figure 10. CL specimen mixed with 8wt% K431 GP using: (a) Method 1, (b) Method 2, (c) Method 1 - cross-section, and (d) Method 2 - cross section.

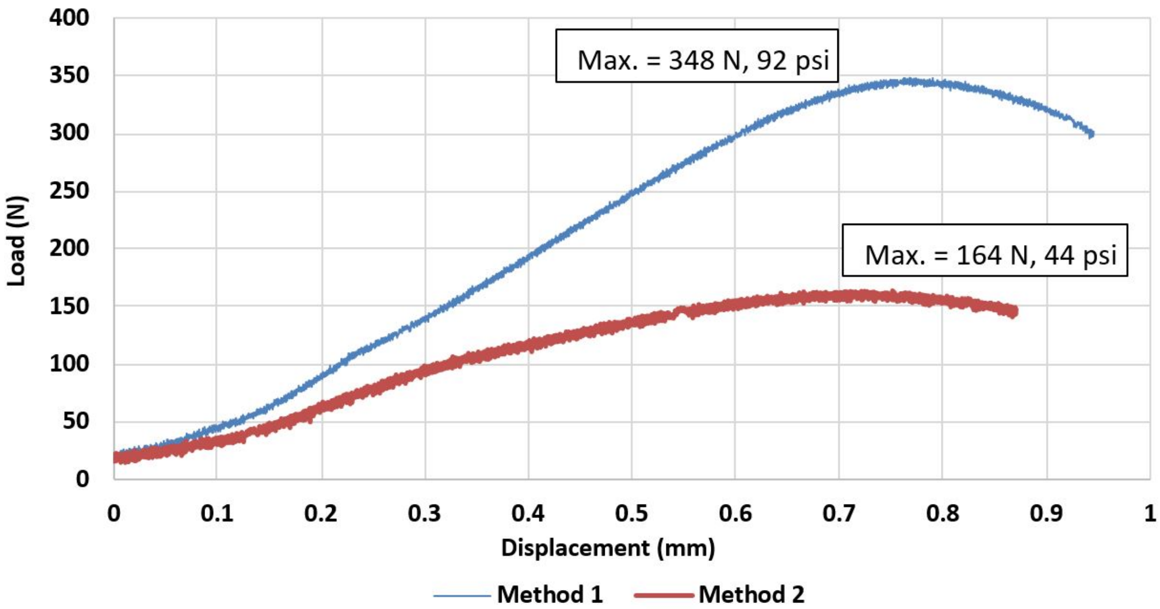
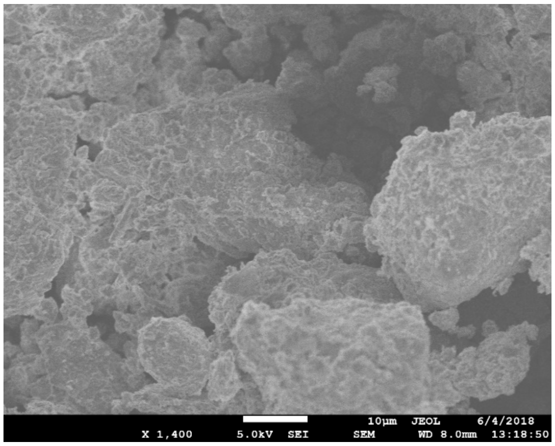
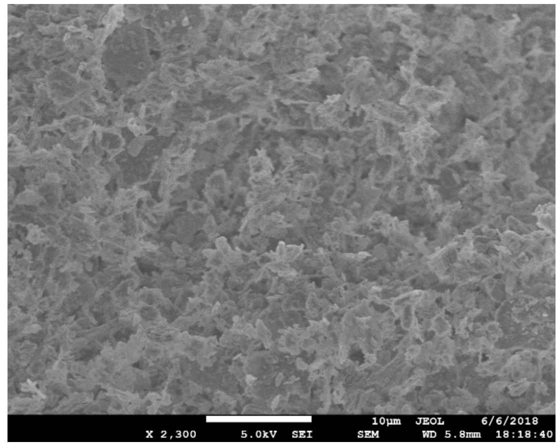


Figure 11. UCS results of two mixing methods for 8 wt% K431 GP treated CL, cured in RH for 3 days and air dried for 4 days.

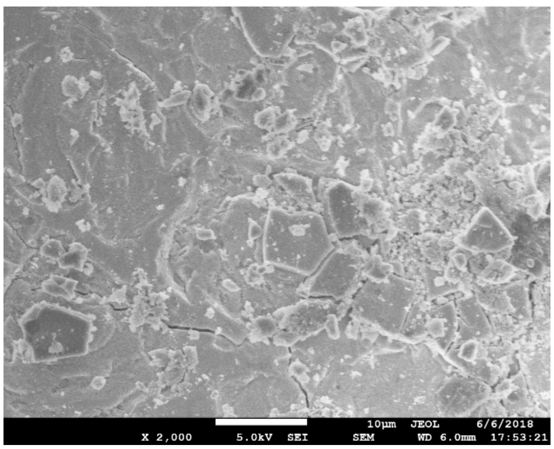
The findings in Figure 10 can be further reinforced by looking at the SEM images in Figure 12. It can be observed that both CL and metakaolin (unreacted GP) have unbounded and porous morphology, while GP shows the continuous and gel-like morphology. By comparing the two mixing methods, it can be clearly seen that method 1 shows a more gel-like morphology, which can then be assumed that GP has been formed and distributed well within the structure. On the other hand, method 2 exhibits a somewhat intermediate morphology once again showing that the GP was not well distributed throughout the structure, which contributes to the decrease in strength.



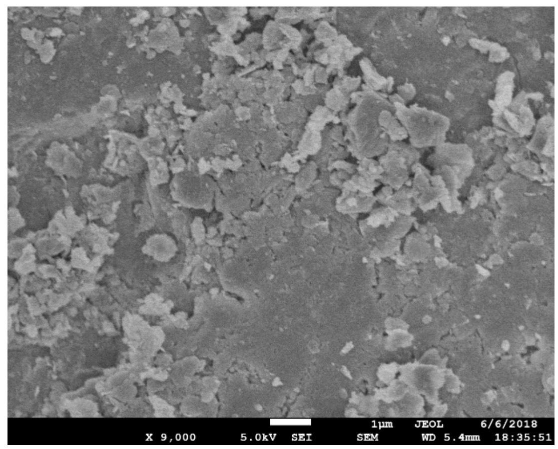
(a)



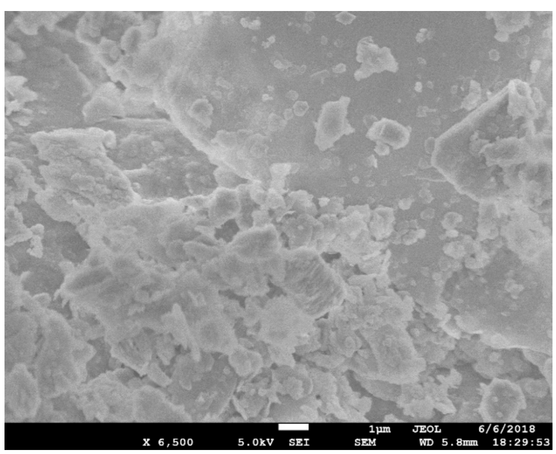
(b)



(c)



(d)



(e)

Figure 12. SEM images of: (a) CL, (b) Metakaolin, (c) K431 GP, (d) K431 GP + CL - Method 1, and (e) K431 GP + CL - Method 2.

6. CONCLUSIONS

This pilot study explores the use of metakaolin-based geopolymer for the stabilization of native North Texas subgrade soils. Two subgrade soils from North Texas were acquired for this study, a high-plasticity clay and a low-plasticity clay. The low-plasticity clay was chosen to study the preliminary effects of geopolymers, based on unconfined compression strength (UCS), swell, and shrinkage tests. Soils were mixed with the K431 GP mix at a ratio of 8 wt% dry GP to dry soil. The following conclusions can be drawn from the study results:

- Shrinkage tests show that geopolymer treated soils are efficient in reducing shrinkage, without developing cracks.
- Swell tests show that the swell potential of the soil is mitigated within acceptable limits, on treating with GP.
- GP treatment of soils is shown to have an increase in the UCS of subgrade soils.
- GP treatment of soils was found to reduce the shrink-swell potential of soils significantly, which is a major concern for high PI soils.

Further studies are recommended to validate the wide-scale application of GP as a sustainable soil stabilizer for high PI soils.

7. RECOMMENDATIONS

Based on this study, it is recommended that parameters affecting GP strength, such as dry GP to dry soil ratio, GP composition, processing methods, and alkali-activator, be varied, to observe its influence on the engineering characteristics of GP treated subgrade soils. Micro-characterization tests like Differential Scanning Calorimetry (DSC) would enable in providing a more conclusive direction into the behavior of the material particles. Furthermore, durability studies as well as sustainability metrics and life-cycle cost analysis studies would be useful in practical implementation of this soil stabilization method.

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