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16. Abstract

Anhydrite ($CaSO_4$) beds occur as a cap rock on a salt dome in Winn Parish in north Louisiana. Locally known as Winn Rock, it has been quarried for gravel for road building. It has been used as a surface course for local parish and logging roads. Stabilization of these roads with Type I portland cement followed by an overlay of asphaltic concrete sometimes resulted in heaving. The causes of heaving and possible solutions were investigated.

In the laboratory 2" x 4" molds of Winn Rock containing soil were stabilized with various cementitious mixes and cured in a 40°C water bath, in a 100 percent-relative-humidity room, a sealed plastic bag at room temperature, and air. The mixes contained 5 percent to 20 percent cementitious material. The cementitious materials were Type I portland cement, lime, and supplementary cementitious materials (SCM) such as granulated blastfurnace slag (BFS), Class C fly ash, silica fume and amorphous silica. The expansion of the specimens over time was monitored. Mineralogical and micro-structural analysis of the specimens was also performed over time and correlated to expansion.

The Winn Rock gravel partially weathered in the soil to gypsum, which was detected in all size fractions. The highest amount of expansion occurred in Winn Rock soil stabilized by lime cured at 40ºC in a water bath. The magnitude of the expansion is directly proportional to the amount of Type I portland cement, the amount of available moisture, and the curing temperature. Replacement of a part of the portland cement by BFS reduced the expansion by almost an order of magnitude even at the highest moisture content. No expansion was detected when a mixture of Class C fly ash and amorphous silica was used as a partial replacement.

The costs of portland cement and blast furnace slag mixtures are less than that of portland cement only. The addition of amorphous silica or silica fume to the cementitious mix will increase the cost significantly.

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Stabilization Techniques for Reactive Aggregate in Soil-Cement Base Course

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ABSTRACT

Anhydrite ($CaSO_4$) beds occur as a cap rock on a salt dome in Winn Parish in north Louisiana. Locally known as Winn Rock, it has been quarried for gravel for road building. It has been used as a surface course for local parish and logging roads. Stabilization of these roads with Type I portland cement followed by an overlay of asphaltic concrete sometimes resulted in heaving. The causes of heaving and possible solutions were investigated.

Representative soil from the affected area was obtained. In the laboratory 2" x 4" molds of Winn Rock containing soil were stabilized with various cementitious mixes and cured in a 40°C water bath, in a 100 percent-relative-humidity room, a sealed plastic bag at room temperature, and air. The mixes contained 5 percent to 20 percent cementitious material. The cementitious materials were Type I portland cement, lime, and supplementary cementitious materials (SCM) such as granulated blastfurnace slag (BFS), Class C fly ash, silica fume and amorphous silica. The expansion of the specimens over time was monitored. Mineralogical and micro-structural analysis of the specimens was also performed over time and correlated to expansion. The characterization methods included X-ray diffractometry, thermal analysis, and scanning electron microscopy.

The Winn Rock gravel partially weathered in the soil to gypsum, which was detected in all size fractions. Anhydrite was also detected in the finer size fractions but in a lesser amount. In one soil, the sulfate in the -#270 (50 μm) size fraction was more than 90 percent gypsum. Apart from anhydrite and gypsum, ettringite derived from the cement from the previous attempt at stabilization was also present.

The highest amount of expansion occurred in Winn Rock soil stabilized by lime cured at 40ºC in a water bath. The magnitude of the expansion is directly proportional to the amount of Type I portland cement, the amount of available moisture, and the curing temperature. Replacement of a part of the portland cement by BFS reduced the expansion by almost an order of magnitude even at the highest moisture content. No expansion was detected when a mixture of Class C fly ash and amorphous silica was used as a partial replacement. Mixtures of portland cement, blast furnace slag, and silica fume also eliminated any expansion.

The sulfate necessary for the expansive reactions in the Winn Rock containing soil was mainly provided by gypsum.   Abundant free calcium hydroxide is necessary for the expansive reaction.   The addition of a SCM to the cementitious mix reduces available calcium hydroxide for the reaction and thus the amount of expansion.   The ettringite crystal size distribution varies widely in the stabilized soil. Expansion occurs due to the formation of small ettringite crystals (gel theory). These evolve in size over the first few days. When a portland cement component is present in the stabilization mix,   ettringite crystals can be detected within an hour of mixing by X-ray diffractometry.

ACKNOWLEDGMENTS

IMPLEMENTATION

Soils containing sulfate minerals can be stabilized successfully with portland cement mixed with supplementary cementititious materials. The key to a successful stabilization effort is in the reduction of free calcium hydroxide in the hydrated mixture. This can be effected by the addition of pozzolans such as granulated blast furnace slag (BFS), amorphous silica, or silica fume.

Blended cements containing a 1:1 to 1:3 cement:BFS ratio can reduce the expansion of soils with sulfate minerals significantly but not completely. The best result is obtained with mixtures containing cement, Class C fly ash, and amorphous silica, or with 1:3 cement:BFS and silica fume. No expansion is detected with these mixtures, and the strength requirement for pavement base is also easily met.

Cement:BFS mixtures cost less than portland cement alone (less than $70/ton). These mixtures can be used for any type of soil, with or without sulfate minerals. The addition of amorphous silica to the mixture will almost double the cost. A mixture of cement, BFS, and 5 percent silica fume, which is expected to work very well, will cost about $100/ton. The latter cost, though expensive, has to be weighed against any future rehabilitation cost, if necessary.

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INTRODUCTION

Winn Rock, quarried in Winn Parish, Louisiana, has been used quite extensively in road base stabilization locally and in near-by parishes as a surface course. The Louisiana Department of Transportation and Development (LADOTD) used it for shoulder surface course in some rural highways. Some of these parish roads were later taken into the LADOTD owned and maintained system. Many of these roads were overlaid with asphaltic concrete or received asphalt surface treatment as a means of improvement. These roads have reached a point where rehabilitation is required.

The rehabilitation process entails stabilizing the existing base course with portland cement and constructing an asphaltic concrete overlay. Some bases that contain Winn Rock experienced heave after the portland cement stabilization process within a day in some cases. In one instance, a stretch of a road several miles long had to be abandoned (unpublished data, R. Mays, LADOTD District 08 Engineer). Figure 1 shows heave in a newly-surfaced road and shoulder in north Louisiana. The ball-point pen in the center provides the scale.

La 882 near Ebarb

US 71 near Campti
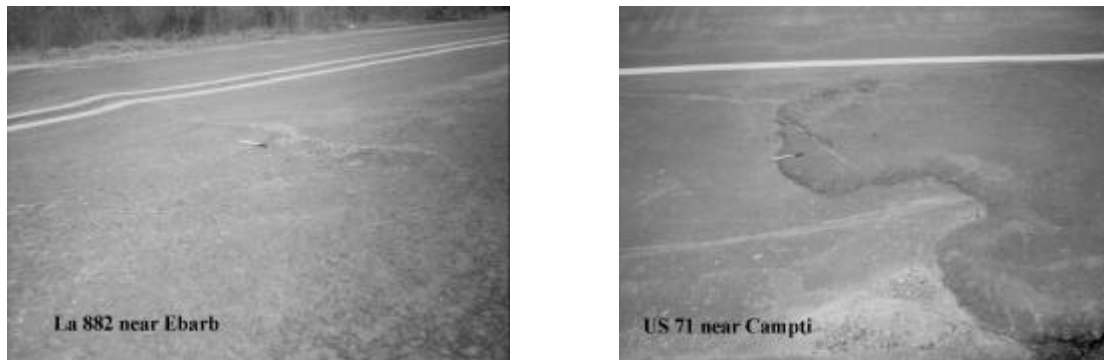
Figure 1

Heaving of freshly surfaced road and shoulder in north Louisiana

Winn Rock is essentially composed of anhydrite (Table 1). The heave reported for the Winn Rock containing base is most likely caused by the crystallization of an expansive mineral phase called ettringite (Table 2). Sulfate minerals in soils are known to cause expansion when stabilized with lime or cementitious materials *[1]*. The usual sulfate mineral is gypsum (Table 1), but anhydrite may also be common. Gypsum may be an original component of the

soil, which is common in many parts of the world and the US, or it could be a weathering product, as is often the case in Texas [2]. However, even if the reaction mechanisms are similar, in all the reported instances none had a large internal source of calcium sulfate. There is also the possibility of anhydrite expanding by acquiring water of crystallization to form gypsum. The experiments conducted in the laboratory by LTRC with crushed Winn Rock gravel stabilized by Type I portland cement (ca. 4 percent C_3A), however, did not show any expansion (unpublished data, K. Gaspard).

Table 1
Important calcium sulfate phases and their solubility [3]

Mineral Name	Formula	Solubility (grams/liter)	Cement Chemist's Notation
Gypsum	$CaSO_4.2H_2O$	2.4	$C\check{S}H_2$
Bassanite (hemihydrate)	$CaSO_4.\frac{1}{2}H_2O$	~6	$C\check{S}.\frac{1}{2}H$
Soluble anhydrite	$CaSO_4$	~6	$C\check{S}$
Insoluble (natural) anhydrite	$CaSO_4$	2.1	$C\check{S}$

Cement Chemist's Notation $CaO = C$; $SiO_2 = S$; $Al_2O_3 = A$; $H_2O = H$; SO_3, $S = \check{S}$.

Table 2
Common mineral phases in portland cement before and after hydration

Name	Chemical Formula	Cement Chemist's Notation
	Cement Phases	
Tricalcium silicate	$CaO.3\ SiO_2$	C_3S
Dicalcium silicate	$CaO.2SiO_2$	C_2S
Tricalcium aluminate	$CaO.3Al_2O_3$	C_3A
Tetracalcium alumino-ferrite	$2CaO.0.5Al_2O_3.0.5Fe2O3$	C_4AF
Gypsum	$CaSO_4.2H_2O$	$C\check{S}.2H$
HYDRATED PHASES		
Calcium Silicate Hydrate	$CaO\text{-}SiO_2\text{-}H_2O$	$C\text{-}S\text{-}H$
Calcium Hydroxide	$CaO.H_2O\ (Ca(OH)_2)$	CH
Ettringite	$CaO.Al_2O_3\ .3\ CaSO4.\ 32H2O$	$C_3A.3\ C\check{S}\ .H_{32}\ (AFt)$
Monosulfate	$CaO.Al_2O_3.CaSO_4.1\ 2H_2O$	$C_3A.C\check{S}.H_{12}\ (AFm)$

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OBJECTIVE

The objectives of this research are

- to characterize fresh, un-weathered Winn Rock,
- to characterize Winn Rock containing soil,
- to identify the mineralogical properties of soil-cement bases which have heaved or can potentially heave,
- to simulate expansion of cement-stabilized soil in the laboratory,
- to identify the concentration of Winn Rock that will cause heave when stabilized by cementitious agents,
- to correlate expansion with the microstructural and mineralogical properties of Winn Rock containing soil after cementitious stabilization, and
- to identify cost effective stabilization agents for soils which contain Winn Rock.

SCOPE

Winn Rock has been used widely for road construction in north Louisiana. All soils containing Winn Rock cannot be studied since a detailed study of a particular soil requires a considerable amount of time and effort. A few representative soils containing Winn Rock were obtained with the help of LTRC and LADOTD personnel.

In the field, expansion of sulfate-containing soil is controlled by many, often transient, variables. For example, the moisture content in a soil is likely to vary over time and can exhibit cyclic behavior. Simulation of such behavior in the laboratory is difficult. Thus only the most important variables can be studied.

The number of cementitious mixes that can potentially reduce expansion is large. If this number is considered along with the environmental variables, the total number of specimens to be molded can get extraordinarily large. The study was thus limited to the simulation of the maximum and the least possible expansion. The maximum expansion simulation would allow understanding the process of expansion, and the least expansion simulation would allow identification of cementitious mixes that can perform well in the field.

METHODOLOGY
Soil Cement

The addition of lime or portland cement to a soil usually improves its engineering properties such as strength and durability *[4]*. For most applications, Type I portland cement is used. Cement requirements vary depending on desired properties and type of soil. Generally, as the clayey portion of the soil increases, the quantity of cement required also increases.

Many supplementary cementititious materials (SCM) used in concrete can potentially be used in soil stabilization. Fly ash is a widely-used SCM in concrete. Both Class C fly ash alone and Class F fly ash mixed with lime have been used in soil stabilization *[5],[6]*. Granulated blast furnace slag is a by-product of the iron and steel industry. It can be ground separately or with portland cement clinker in various proportions. For typical applications, it provides enhanced durability, including high resistance to sulfate attack in concrete. Ground granulated blast furnace slag can also be incorporated in lime-stabilized soil *[7],[8]*. The studies by Wild and others have shown that the addition of BFS to lime during the stabilization of sulfate-bearing soils substantially reduce the damaging swelling and has no significant deleterious effect to strength development *[7],[8],[9],[10]*. Silica fume is a by-product of silicon alloy manufacturing. The average particle size of silica fume is a few hundred nanometers and the surface area ranges from 15,000 to 30,000 m^2/kg. Its use as a SCM in concrete is now firmly established as it confers many beneficial properties to the concrete *[11]*. Amorphous silica is a by-product of amorphous silica production *[12],[13]*. The average grain size of this material is only a few tens of nanometers (compared to a few hundred for silica fume) and the surface area is in high tens of thousands of m^2/kg. For example, an amorphous silica used as a SCM in concrete at Louisiana State University in Baton Rouge had an average grain size of 30 nanometers and a surface area of 95,000 m^2/kg. Silica fume and amorphous silica, because of their recent availability and high relative cost, have not been used in soil stabilization.

Sulfate Attack in Portland Cement

Sulfate attack in concrete has been studied in great detail. For example, Miller et al. found 402 references on sulfate attack in concrete for their 1952 review (in Heller, *[14]*). Sulfate attack of conventional portland cement is a widely recognized phenomenon, and appropriate

methods of protection against sulfate attack have been established as a function of the sulfate exposure level, but the actual chemical reactions and products involved have not been completely understood.

In the literature, researchers have postulated at least four types of sulfate attack depending on the type and concentration of sulfate in concrete *[15],[16],[17]*. The different types can work alone or overlap.

1.   The most common type of sulfate attack that is discussed in the literature is one which is accompanied by the formation of the expansive mineral ettringite. Due to the influx of sulfate ions any unreacted tricalcium aluminate in the concrete or monosulfate that formed from earlier ettringite can form new ettringite. However, no clear correlation has been shown between the amount of ettringite and the amount of expansion *[14]*.

$$3CaO.Al_2O_3.CaSO4.12H_2O + CaSO_4 + H2O = 3CaO.Al_2O_3.3CaSO_4.32H_2O \qquad (1)$$
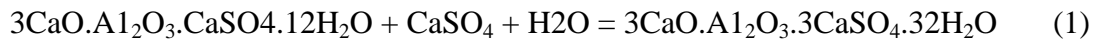
2.   In another type of sulfate attack, a sulfate solution (e.g., sodium sulfate, which has high solubility in water) reacts with the calcium hydroxide in the concrete to form gypsum, which can lead to expansion.

$$2Na^+ + SO_4^{2-} + Ca(OH)_2 + 2H_2O = 2Na^+ + 2OH^{-1} + CaSO_4.2H_2O \qquad (2)$$
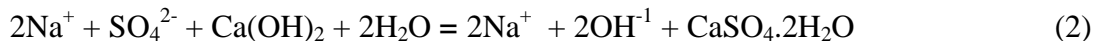
3.   The calcium silicate hydrate phase, the main hydration product of portland cement, can also react with sulfate ions to produce gypsum. The resultant gypsum can then form ettringite or itself cause expansion.

4.   The formation of thaumasite, which has the same crystal structure as ettringite but has Si instead of Al in the six-fold position, has sometimes been implicated in expansion of concrete, particularly at very low, near-freezing temperatures. The reaction of thaumasite formation is the same as in equation 1.

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Expansion Mechanism Due to Sulfate Attack in Concrete

The mechanisms of expansion due to sulfate attack have been investigated by a number of investigators (for example, Mehta *[18]*). Numerous theories and models aimed at explaining the mechanism of formation of ettringite and expansion due to ettringite have been suggested and can be divided into two major schools of thought: the crystal growth theory and the gel (swelling) theory *[19]*.

According to the crystal growth theory school, expansion begins when reaction zones of ettringite formation contact others, continue growing, and mutually exert pressure. According to the gel theory school, expansion is caused by the swelling of the ettringite particles which are of colloidal or gel size. The swelling occurs in the presence of free calcium hydroxide.

Sulfate Attack on Soil Cement

Sherwood was the one of the early investigators who noticed the problem concerning sulfate attack on soil cement *[1]*. He conducted an experiment to determine the effect of the presence of sulfate ions in soils on the durability of cement- and lime-stabilized soils *[20]*. The method of investigation consisted of observing the behavior of specimens of stabilized soil when totally immersed in water. When tested in this way, cement- or lime-stabilized clay mixtures containing calcium, magnesium, or sodium sulfates disintegrated within a few days of being immersed, whereas cement-stabilized sand mixtures containing the same proportions of sulfates were unaffected even after being immersed for one year. This suggested that the effect was related to the clay content of the soil. It was proposed that sulfate attack on cement-stabilized soils is due principally to the reaction involving clay minerals and that the relatively slow combination of the sulfates with the cement is of secondary importance. Cordon conducted a similar laboratory test concerning sulfate attack resistance of soil cement *[21]*. Type I, Type II and Type V portland cements and a coarse-grained soil and a fine-grained soil were used for specimen fabrication, which would be immersed in sulfate solution. A photographic record, taken at different time intervals, was used as a method of demonstrating progress of the sulfate attack. Major conclusions included (1) that soil cement is subject to sulfate attack much in the same manner as cement concrete, but deterioration in soil-cement is more rapid than in cement concrete; (2) soil cement specimens fabricated with Type V and Type II cements were more resistant to the sulfate

attack than soil cement specimens fabricated with Type I cement; and (3) soil cement specimens made with fine-grained soils deteriorated more rapidly.

Mitchell brought attention to the engineering phenomenon of sulfate-induced heave in treated soils *[22]*. An investigation was carried out concerning a section of a major arterial street with lime treated expansive soil base in Las Vegas, Nevada. The completed construction appeared to be of good quality and the initial performance was excellent. However, two years after construction, signs of distress began to appear in the form of surface heaving and cracking. The investigation of the failed pavement indicated that the   structural design of the pavement section was adequate and the failure was not traffic-induced. However, in the failed zones the soil had a much lower density and higher water content than the intact treated soil. Further tests provided the following information: the soil contained significant amounts of soluble sodium sulfate (up to 1.5 percent by weight); samples taken from intact and un-failed zones swelled if exposed to water; clay minerals were present in the soil; a significant amount of ettringite was indicated by X-ray diffraction in both the failed and un-failed zones.

Petry and Little reviewed the background on sulfate-induced heave in lime- and cement-treated clay soils and some examples of projects affected by this phenomenon *[23]*. According to the literature, even though the total reaction and favorable environment for formation of expansive minerals resulting from the interaction of lime and sulfate-bearing clay soils or portland cement and sulfate-bearing soils are not completely understood, the most often found expandable mineral resulting from these reactions is ettringite. Therefore, the formation of ettringite is necessary for the sulfate-heave phenomenon to occur, and curtailment or elimination of its formation would dramatically reduce the volume increases noted. When lime or cement is added in sufficient quantities to clay, the pH is raised. Once the pH exceeds 10.5, dissolution of the clay surface occurs, and Si and Al ions   are released. If sulfates, either in solid or groundwater form, are present in sufficient quantity, they may initiate a reaction between the calcium and the silica and alumina released from clay minerals to form significant quantities of ettringite or monosulfate hydrates. Mehta and Klein determined that the formation of monosulfate hydrates is favored in high alumina environments, but the formation of ettringite is favored in low alumina environments *[24]*. This might indicate that monosulfate hydrates are favored in 1:1 type clay minerals like kaolinite, while the formation of ettringite would be favored in 2:1 type clay minerals such as

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smectite. They also describe ettringite as substantially expansive upon wetting, while monosulfate hydrate is not.

As pointed out by Ksaibati and Huntington, there are some significant differences between sulfate attack in concrete and soil *[25]*. Heave experienced in cementitious stabilization of Winn Rock containing soil is similar to the problem of sulfate attack in portland cement concrete *[17],[19],[26]*.   In concrete, the excess sulfate can be supplied by the groundwater (at concentrations as low as 1000 ppm of SO_4^{-2}) or some mineral phase in the concrete itself. Some authors have thus defined the problem in terms of internal and external sulfate *[27]*. For Winn Rock containing soil, the source of sulfate will be the anhydrite. Unlike concrete, however, the ratio of sulfate to cement, where typically 10 percent by weight of cement is used for stabilization, will be quite high.

The thermal and chemical stability of ettringite, under varying conditions of pH value, has been evaluated by a number of investigators, for example Gabrisova et al.   *[28]*. In a calcium alumino-sulfate system, ettringite is the stable phase at room temperature with a pH value larger than 10.7. It is stable at sulfate concentrations above 3×10^{-6} moles per liter.

Factors Affecting Sulfate Attack

Similar to cement concrete, the pH, moisture availability, temperature, sulfate levels, and clay mineralogy may all affect sulfate attack of cement-stabilized soils. These factors should therefore be determined when stabilized soils are susceptible to sulfate attack.

Mitigation of Sulfate Attack

The problem of stabilizing sulfate bearing soils as well as plausible stabilization schemes were recently reviewed by Kota et al. *[2]*. Among the stabilization schemes identified were double treatment with lime, low-calcium stabilizers, non-calcium-based stabilizers, treatment by barium chloride, and others. However, these suggested solutions were somewhat speculative. Many of the methods have no prior history and are not necessarily economical.

The gel theory of Mehta provides a testable hypothesis for reducing or eliminating expansion in stabilization of sulfate-containing soil *[29]*. It is well known that the addition of a pozzolan or a SCM to portland cement reduces the calcium hydroxide content of the system

as it is consumed in the pozzolanic reactions. Thus the addition of a SCM to a stabilizing mixture can reduce the amount of free calcium hydroxide and in the process mitigate sulfate attack.

The pozzolanic reactivity generally depends on the grain size and the surface area of a pozzolan. The surface areas of Class C fly ash, BFS, silica fume and amorphous silica are 350, 400, 20,000, and 95,000 m^2/kg, respectively. An increased amount of a SCM, such as BFS, in the stabilizing mixture will reduce the amount of free calcium hydroxide available. Similarly the addition of a small amount of highly reactive silica fume or amorphous silica can remove the free calcium hydroxide very quickly *[12]*. Thus these SCMs thus can be used to reduce or stop expansion.

Experimental Methods

Materials

　　Lime. Calcium oxide (CaO) reagent, synthesized by Mallinckrodt Chemical, Inc., Kentucky, was used for lime stabilization of the Winn Rock containing soil.

　　Cement. Type I low alkali portland cement manufactured by Lone Star Industries, Inc., at its plant in Cape Girardeau, Missouri, was used to prepared stabilized samples. This cement contains 51.4 percent tricalcium silicate (C_3S) and 5.7 percent tricalcium aluminate (C_3A).

　　Supplementary Cementitious Materials. Several supplementary cementitious materials including BFS, Class C fly ash, silica fume, and an amorphous silica were used in this study.   Their relevant chemical and physical properties are given in Table 3. The BFS was supplied by Lone Star Industries, Inc.; the Class C fly ash, meeting ASTM C-618, was obtained from Bayou Ash Co., Erwinville, Louisiana; and the amorphous silica was supplied by the Silica Products Division of PPG Industries, Lake Charles, Louisiana, respectively**.**

Table 3

Properties of the supplementary cementitious materials

Composition (percent)	BFS	Class C fly ash	amorphous silica
SiO_2	34.5	47.5	95.0 – 98.0
Al_2O_3	9.5	20.6	0.12
Fe_2O_3	1.3	5.2	0.06 – 0.21
CaO	39.6	16.2	0.06 – 0.30
MgO	10.9	2.5	0.02 – 0.04
K_2O	1.26	0.7	0.01 – 0.06
Na_2O	0.48	0.3	0.65 – 3.29
Special Surface Area (m^2/Kg)	380	350	95000

Experimental Conditions

Curing Temperature. Two curing temperatures, 40ºC and 23ºC (room temperature), were used. The higher temperature was used to accelerate ettringite formation. This temperature is not unrealistic, and can be reached during cement hydration under an asphaltic cover. This temperature is well within the stability range of ettringite, since the high temperature stability limit of ettringite is 70º to 90ºC in concrete *[17]*. The 40ºC temperature was attained in a water bath. The specimens were sealed in plastic bags and stored in the water bath.

Moisture Content. The specimens were cured at three moisture levels. These moisture levels represent the range that can be observed in the field. The highest moisture level, which was attained in a 100 percent relative-humidity room at room temperature, can occur when the water table is high or after a heavy rainfall. This level can also occur after an asphaltic overlay, which can trap moisture. An intermediate moisture level is the moisture available in a sealed plastic bag. This level is the most likely scenario in the field. A small pool of water was observed in these bags after several weeks. The lowest moisture level was attained during air curing. Such an environment can be expected in well aerated sections of a road base.

Drying of Soil. The traditional method employed in geotechnical engineering for moisture determination is oven drying. It is suitable and economical provided minerals with low dehydration temperature are absent. However, the presence of gypsum in the Winn Rock containing soil is well known. Taking into account the author's previous experience with phosphogypsum, air drying was determined to be the best method of drying the soil to avoid its dehydration.

For small sample masses many special procedures have already been developed by researchers in soil sciences just for this problem and they can be easily adopted by the geotechnical personnel *[30]*. Nelson has described many different methods of quantifying gypsum and carbonate in soils *[31]*. Drying over P_2O_5 appears to leave the water of crystallization of gypsum undisturbed but moisture from clay minerals is removed. Unless otherwise stated, the small samples in this study were dried over P_2O_5 or air dried.

Mold Preparation. Since 2" x 4" molds were used for stabilization, the maximum size of the coarsest fraction was limited to –4 sieve size (suggested by Kenneth Johnston, LTRC). The soil was crushed and mixed thoroughly at LTRC. Two sets of specimen cylinders were prepared: one for compaction and unconfined compressive strength tests, and the other for linear expansion measurements.

The details of sample preparation are provided in Table 4. The sample abbreviations listed in the first column are used throughout the rest of the text. For the compaction and unconfined compressive strength tests, the Winn Rock containing soil was first thoroughly mixed with the required amount of cementitious material (5 to 9 percent). The calculated amount of water, obtained from the compaction test, was then added and the material was mixed again to achieve a homogeneous mass. The compaction test was conducted with the standard Proctor method. For the unconfined compressive strength test, specimen cylinders with maximum dry density and optimum moisture content were kept in the wet room with 100 percent relative humidity at 20°C and submerged in water for four hours before testing. For each test, three specimen cylinders were fabricated. The unconfined compressive strength test was conducted according to ASTM D 2166-91 with a United Compression Model SFM-30E load frame.

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For the linear expansion test, the calculated amounts of soil and 5 or 9 percent cementitious materials and water were first mixed. The mixture was poured in the 2" x 4" ($\phi 50 \times 100$ mm) mold and compressed to reach an exact volume with 95 percent of maximum dry density. The specimens fabricated for linear expansion measurements were subjected to different curing conditions. Their linear dimensions were measured and recorded at regular intervals.

Table 4

Specimen preparation matrix

	Composition (weight percent)							
Sample Name	Winn Rock Soil	ALF Soil	Portland Cement	Blast Furnace Slag	Class C Fly Ash	Amorphous Silica	Silica Fume	Water
WRS	100		-	-	-			11.4
C5	95		5	-	-			12.4
C9	91		9	-	-			13.2
C:BFS 1:1	91		4.5	4.5	-			12.3
C:BFS 1:3	91		2.25	6.75	-			11.6
C:CFA 1:1	91		4.5	-	4.5	-		11.5
C:CFA:AS 1:0.75:0.25	91		4.5	-	3.375	1.125		14.5
WRS:ALF 1:3	22.75	68.25	9					14.5
C:BFS:SF 4% SF	91		2.88	5.76			0.36	14.5
C:BFS:SF 8% SF	91t		2.76	5.52			0.72	14.5
C:BFS:SF 12% SF	91		2.64	5.28			1.08	14.5

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Table 5
Curing environments

Polythene bag for 1 day,	Polythene bag for 1 day,	Polythene bag for 1 day,	Polythene bag for 1 day,
followed by curing in a water bath with 100 percent relative humidity at 40°C	followed by curing in a wet room with 100 percent relative humidity at 20°C	followed by curing in a ziplock bag at 20°C	followed by exposure to air at about 20°C

At present, there is no standard method for linear expansion measurement of soil cement. However, standard ASTM tests for linear expansion measurements are available from other cement-related fields. For example, ASTM C 157 has been developed for monitoring the length change of hardened hydraulic cement mortar and concrete. Similarly, ASTM C227 measures length change due to potential alkali reactivity of cement aggregate. These tests can be used as references.

Following fabrication, the specimens were demolded immediately and stored in sealed plastic bags. After 24 hours, the first value of cylinder length, L_o, was measured. The specimens were then stored in different curing environments and their lengths L_i were measured at intervals. The percent ratio of increased length ($L_i - L_0$) to the original length L_o is defined as linear expansion. The length was measured with a vernier micrometer and the average value measured from three specimen cylinders was used as the linear expansion magnitude.

Analytical Methods

Several analytical methods were used to characterize the Winn Rock containing soil, before and after cementitious stabilization. These included X-ray diffractometry, thermal analysis (differential scanning calorimetry, thermogravimetry, and derivative thermogravimetry), scanning electron microscopy, energy dispersive X-ray spectrometry, and X-ray absorption near edge structure. A brief description of the type of information obtained from each technique is provided in Table 6.

Table 6

Information obtained by each analytical technique

Technique	Information obtained
Scanning Electron Microscopy (SEM)	The microstructure of the Winn Rock containing soil, before and after stabilization, was studied. Properties, such as the morphology of the individual minerals, the spatial relationship between them, etc., were studied. The specimens were prepared by freeze-drying so that the alteration of the microstructure, at high vacuum during gold coating or observation, was minimal. The morphology and distribution of the ettringite crystals were indicative of the type of sulfate attack.
Energy Dispersive X-ray Spectrometry (EDS)	Micro-analysis in the SEM provided chemical composition from a micrometer-diameter volume. Thus a chemical composition can be associated with a particular volume and different mineral phases having the same morphology can be distinguished. For example, calcium hydroxide and calcium aluminate hydrate, both have hexagonal shape. Though their morphology is identical, their chemical composition can be used to distinguish between them.
X-ray Diffractometry (XRD) Quantitative X-ray Diffractometry (QXRD)	X-ray diffractometry helped in the identification of the crystalline phases in the soil and the stabilized soil. This technique was also used in the quantification of the minerals in the Winn Rock containing soil.
Thermal Analysis - Thermogravimetry (TG) Derivative Thermogravimetry (DTG)	Thermal analysis is particularly suitable for materials that undergo dehydration, de-carbonation, or phase change during heating. This technique was used in the identification and quantification of the clay minerals, gypsum, ettringite, calcium hydroxide, and calcium carbonate in the Winn Rock containing soil, before and after stabilization.
Fourier Transform Infrared Spectroscopy (FTIR)	Along with XRD and thermal analysis, this technique was used for the identification of phases. This technique allows easy differentiation between gypsum ($CaSO_4.2H_2O$) and hemi-hydrate ($CaSO_4.1/2H_2O$).
X-Ray Absorption Near Edge Spectroscopy (XANES)	Distribution of sulfate among different phases (e.g., anhydrite, gypsum, and ettringite) was studied by XANES. The spectrum for sulfate was slightly different in each mineral species and curve-fitting was used in quantification of the different sulfate-containing minerals. This technique also showed that all the sulfate in the soil was present in the sulfate form.

X-Ray Diffractometry. A Siemens D 5000 diffractometer was used for X-ray diffractometry runs. Cu ka radiation was used. The runs were usually from 3º to 70º 2θ,

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with 0.02º step size, and 2 second counting at each step. A sample spinner was used so that the effective grain diameter could be lowered *[32]*.   The entrance slit width was kept constant. For quantitative X-ray diffractometry, a known amount of Silicon powder was added to the crushed soil as an internal standard. The powder was thoroughly mixed with the soil in an agate mortar and pestle. No micronizing mill was used for crushing the soil to a finer grain size because of the possibility of gypsum dehydration.   The run conditions were very similar as above except that the counting time was increased to four seconds at each step, and data were collected up to 80º 2θ.

In order to detect ettringite in specimens of low ages, scans were conducted over the range 7 to 11º 2θ with 0.02º step size and one minute of counting time at each step. With a typical count of 1400/sec at the ettringite peak, the statistical error counts is 0.3%, compared to 2.67% with 1 second counting time.

Thermal Analysis. For thermal analysis (TG) the samples were run up to 1000ºC with a TA Instruments 2950 High Resolution instrument. The temperature program was equilibrate at 40ºC for 5 minutes, and then raised up to 1000ºC with a resolution of six at 50ºC/min. The program allows a fast run rate if no peaks are present but a lower run rate results when the breakdown of a phase, particularly overlapping ones, occurs. The higher the resolution number, the better the separation is between two overlapping reactions. This program allows a run to be completed in less than two hours, whereas runs at a constant rate (resolution zero) would take at least three hours without any loss of information. The sample amount ranged from 15 to 40 mg.

The DSC runs were conducted from 40º to 275ºC at 3ºC/min. Tens of mg of samples were put in aluminum pans and then sealed with aluminum lids.

Fourier Transform Infrared Spectroscopy. Fourier-transform infrared (FTIR) spectroscopy was performed with a Mattson Gold FTIR spectrometer equipped with a triglycine sulfate (TGS) detector. Optically transparent discs were prepared in the mini-press from powdered samples mixed with potassium bromide (KBr). The samples were dried over P_2O_5 before KBr discs were made.   The scanning range varied from 4000 to 400 cm^{-1} and the number of scans averaged were at the minimum five at the scan speed of 0.4 cm/sec and resolution of 4 cm^{-1}.

Scanning Electron Microscopy. Scanning electron microscopy was performed with a JEOL 840 microscope. The operating voltage was usually 20 keV. The JEOL 840 was fitted with a Tracor Northern energy-dispersive X-ray spectrometer for X-ray micro-analysis. The samples were freeze-dried so that the effect of the high vacuum on the microstructure in the sample preparation and observation chambers was minimized.

Crystal size analysis from the scanning electron microscope images was assisted by *ImageTool,* which was developed at the University of Texas Health Science Center in San Antonio, Texas, and is available from the Internet by anonymous FTP from maxrad6.uthscsa.edu.

X-Ray Absorption Near Edge Structure. The X-ray absorption near edge structure spectra were obtained at the Double Crystal Monochromator beamline of the J. Bennett Johnston Sr. Center for Advanced Microstructures and Devices (CAMD), Louisiana State University, Baton Rouge. Finely powdered samples were uniformly smeared on Kapton tapes and the S K edge spectra were measured in transmission. Si 111 crystals were used in the monochromator. A step size of 0.6 eV was used for scans from 50 eV below the edge to 200 eV above the edge. The normalization was performed by drawing a line through this higher energy range. Helium was used as the ionization gas in the chambers.

DISCUSSION OF RESULTS

Winn Rock

Winn Rock Quarry

Winn Rock comes from the cap rock of a salt dome. A salt dome is formed when the lower-density salt beds move up from great depths (several miles) through higher density rocks due to density instability. The rocks that are dragged up from depths by the upward-moving salt beds are known as cap rocks. The Winn Rock quarry has supplied gypsum to cement manufacturers in the past. A variable proportion (20 percent to 85 percent) of the anhydrite tailings at the quarry has been converted to gypsum by the percolating rain water over the past several decades.

Winn Rock is a dense dark gray rock.   However, the exposed surface of Winn Rock was often covered with a white crust (efflorescence). The floor of the Winn Rock quarry near Winnfield was also covered with a milky liquid, particularly after a rain. Winn Rock aggregate kept in a moist condition for several months in the laboratory also develops this crust. X-ray diffractometry showed that fresh Winn Rock was composed of only anhydrite. The white crust (efflorescence) observed on Winn Rock was gypsum.

Scanning Electron Microscopy

In the scanning electron microscope, calcium sulfate phases can be identified by their morphology (shape).   Table 1 shows the crystal system in which different calcium sulfates crystallize. Figure 2 shows the morphologies of anhydrite (left) and gypsum (right).   The anhydrite crystals are almost cubic, with all their dimensions being somewhat similar, whereas the gypsum crystals are more plate like, with two of their dimensions being much larger that the third. Energy dispersive X-ray spectrometry in the electron microscope will show identical chemical analysis for these minerals as the water cannot be detected, but the morphology can be used to identify the minerals.
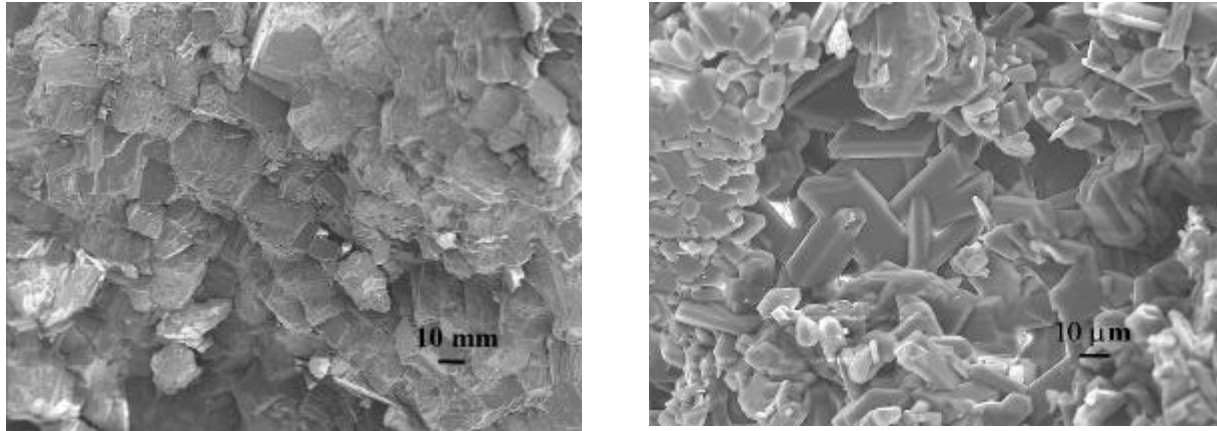
Figure 2

SEM photomicrograph of Winn Rock and its weathering product

Winn Rock Containing Soil

For the Winn Rock-containing soil, the possibility exists of anhydrite converting to gypsum. Hydration of anhydrite to gypsum may yield molar volume increases up to 62.6 percent and dehydration of gypsum to anhydrite may yield molar volume decreases up to 38.5 percent *[33]*. The conversion of anhydrite to gypsum is a slow process, that may take years. The information brochure from the Winn Rock quarry reports variable amounts of conversion of anhydrite tailings to gypsum (20 percent to 85 percent) over the last thirty years. Many anhydrite mineral specimens in museums have altered to gypsum at the surface if the humidity is high. The alteration usually occurs on the surface and in the cracks, whereas in the interior, the anhydrite stays unaltered. A survey of the available literature suggests that this conversion is a slow process (kinetics), even though it is thermodynamically feasible.

The conversion of anhydrite to gypsum, though possible, is unlikely to have occurred in the Winn Rock containing soil for the following reasons:

- only water is necessary for this conversion and it has been available in the soil in abundance, but no expansion occurred prior to cementitious stabilization;
- the expansion should have occurred without any temporal relationship to cement stabilization.
- the high pH (produced by cement) is not necessary for this transformation to occur; and

- the expansion need not be associated with ettringite crystallization (which also requires high pH).

The fact that the heave occurred only after cementitious stabilization of the soil followed by asphaltic concrete overlay suggests that anhydrite to gypsum transformation was not responsible for the heave noticed in the soil.

Mechanical Properties

The specific gravity of the Winn Rock containing soil is 2.64. The particle size distribution of Winn Rock containing soil, based on sieve analysis, is shown in Figure 3. The particle size distribution curve indicates a well-graded distribution. Percentages passing No. 10 sieve (2.00mm), No. 40 sieve (0.425mm) and No.200 sieve (0.075mm) were about 80 percent, 40 percent and 15 percent, respectively. Hydrometer analysis indicated that approximately 2 percent of the soil mixture is less than 0.001 mm. As expected for this silty soil, consistency testing showed it has no plasticity (NP). According to the American Association of State Highway and Transportation Officials (AASHTO M 145) classification system, the soil is classified as A-1-b group.

Table 7
Index properties of Winn Rock containing soil

Property	Winn Rock containing soil
Passing sieve No. 4 (percent)	100
Passing sieve No. 10 (percent)	80
Passing sieve No. 40 (percent)	40
Passing sieve No. 200 (percent)	15
Clay content (percent)	2
Liquid limit (LL) (percent)	NP
Plastic limit (PL) (percent)	NP
Plastic index (PI)	NP
Optimum water content (percent)	14.2
Maximum dry density (lb/ft^3)	117.13
Classification (AASHTO)	A-1-b
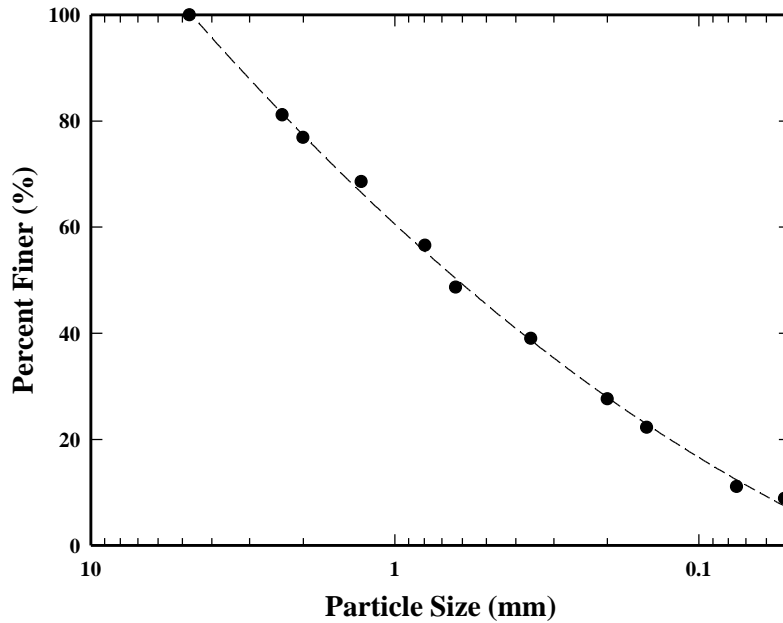
Figure 3

Particle size distribution of Winn Rock containing soil

The values of dry density (γ_d) determined from a series of compaction tests is plotted against the corresponding moisture contents (Figure 4) to obtain the maximum dry density and the optimum moisture content for the specimen, which are 117.90 lb/ft^3 and 14.2 percent, respectively. The unconfined compressive strength (UCS) of the Winn Rock containing soil (untreated) was determined after   seven days and twenty-eight days of curing.   The strength of the soil increased from 18 psi at seven days to 78 psi at twenty-eight days. The strength increase was likely due to the hydration of anhydrite to form interlocking crystals of gypsum.

From a mechanical point of view, the Winn Rock containing soil, which is classified as A-1-b, is suitable as a pavement base material. It has a certain amount of compressive strength and is easily mixed with cement to produce a soil cement to provide capacity. The well-graded distribution curve of the mixture means that it has a dense soil structure, which is defined as the geometric arrangement of soil particles with respect to one another. This behavior of dense structure may lead to a high dry density and stability, as aggregates that contain some fines fill all the voids between the aggregate grains resulting in a higher strength and shear resistance from grain contact. However, the highly dense structure may also aggravate sulfate expansion, as less

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pore space provides less room for the growth of expansive minerals and may result in more overall expansion pressure *[2]*.
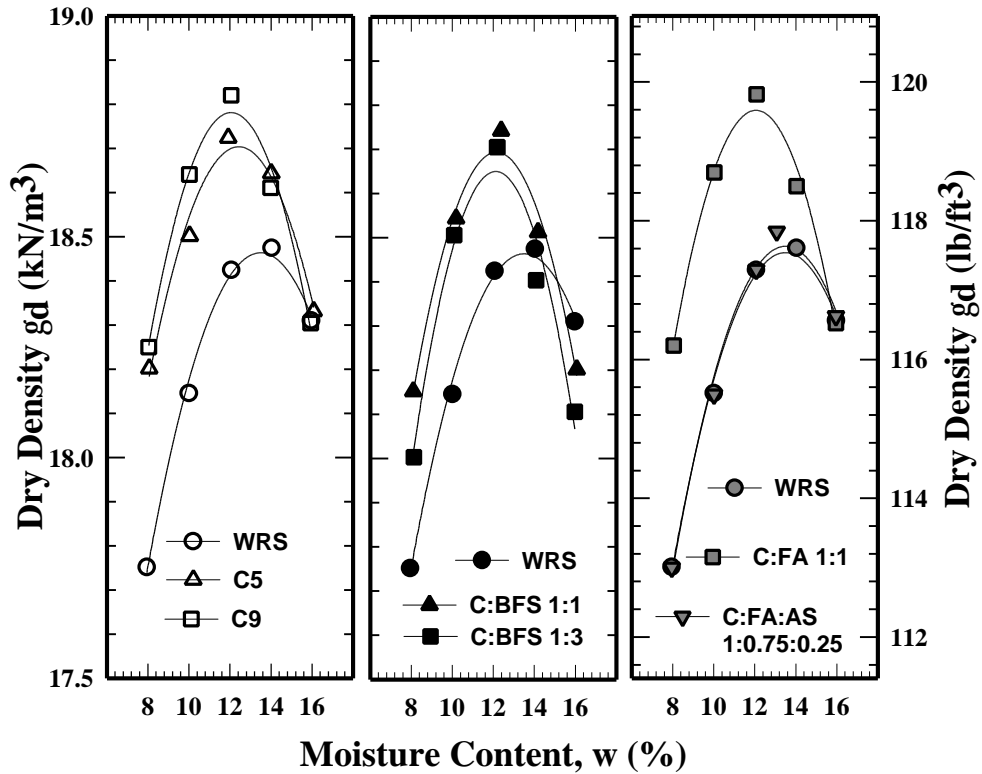
Figure 4

Standard Proctor test results for Winn Rock containing soil

Mineralogical Properties

X-ray Diffractometry. The X-ray diffraction pattern of the Winn Rock containing soil is shown in Figure 5.   Quartz (SiO_2) is the most dominant mineral in the soil (PDF# 33, 1161 (Powder Diffraction File)).   The anhydrite 100 percent peak (*200 reflection plane*) is the second strongest peak but other peaks of anhydrite are not that strong, suggesting preferred orientation of the anhydrite crystals.   Gypsum ($CaSO_4.2H_2O$) (PDF # 33,311; # 6, 46) and anhydrite ($CaSO_4$) (PDF# 6,226) are both present in the soil. A minor amount of ettringite ($3CaO.Al2O_3.3CaSO_4.26H_2O$)   (PDF # 31, 251) is present. Some amount of   calcium carbonate was also detected in the soil. No bassanite was detected in the soil.   The 00l (low 2θ) peak of any of the important clay mineral phases could not be identified with certainty in the XRD pattern and thus they are present in low amounts.   Several varieties of feldspar (albite 0.319 nm

and microcline (0.324 nm) were identified *[34]*.
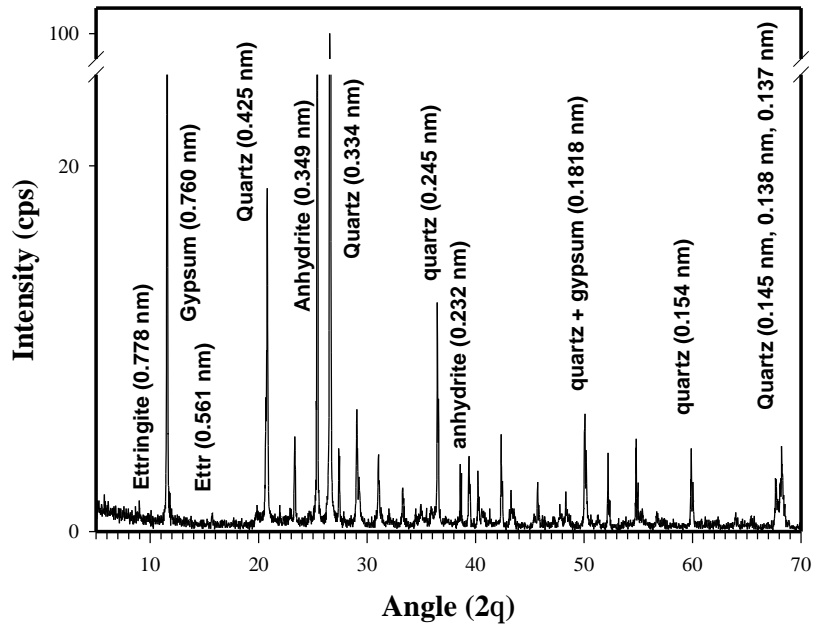
Figure 5

X-ray diffraction pattern of Winn Rock Soil
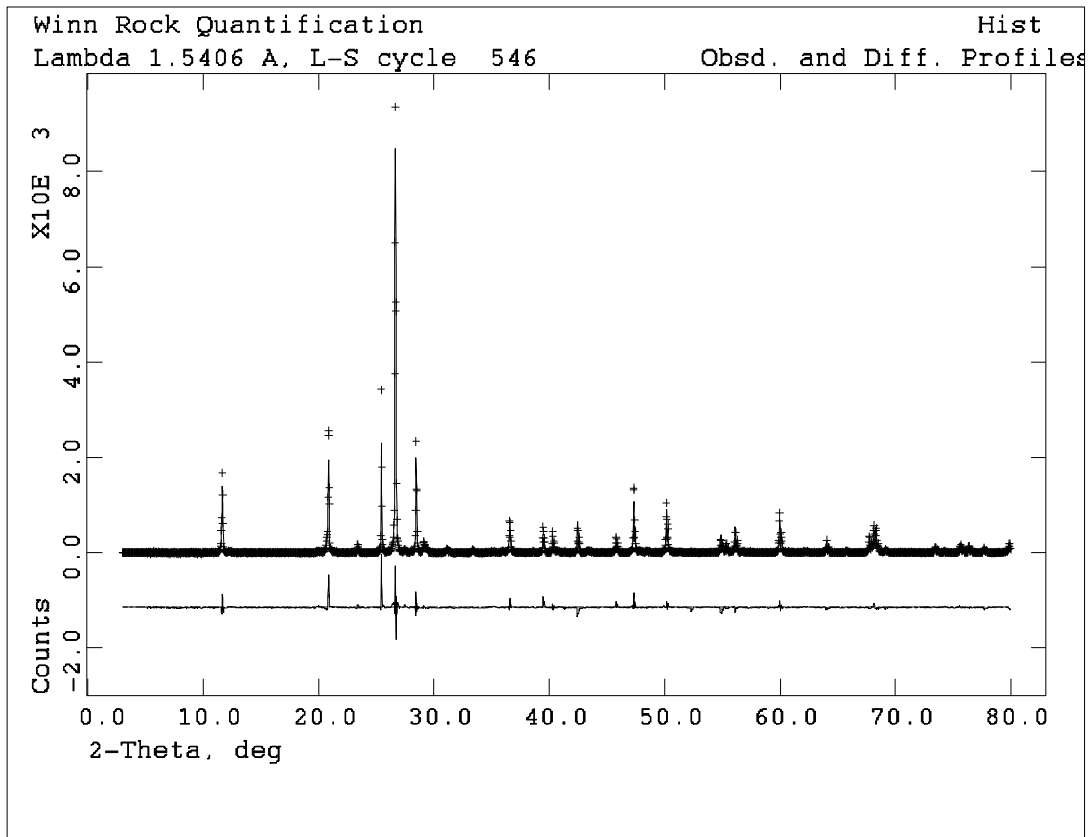
Figure 6

Quantitative Rietveld analysis of Winn Rock containing soil

X-ray diffractometry is a powerful method for identification and quantification of sulfate phases. *[35]*. Quantification of different phases in the Winn Rock containing soil was attempted by the Rietveld method *[32]*. There are several pieces of public domain software for this purpose. Quantitative Rietveld analysis of the Winn Rock Soil was performed by GSAS *[36]*. The experimental pattern (top) and the difference between the experimental pattern and the simulation are shown (bottom) in Figure 6. Since all the minerals were not considered (particularly the clay minerals), i.e., the total amount of the minerals does not add up to 100 percent, a silicon internal standard was added. The residual R_p (an indicator of the goodness of fit) for the fitting was 0.1516, which is partly due to the fact that not all phases were considered in the fit. Quantitative analysis of the soil yielded the following proportions (in percent) for the relevant minerals:

Quartz	Gypsum	Anhydrite	Calcite
54.9 ±0.2	12.92±1.1	0.9±0.2	1.8±0.7

The amount of gypsum in the soil obtained by quantitative XRD is 50 percent higher than that   obtained by derivative thermogravimetry. However, it appears reasonable and could be due to sample variability.   The weight fractions of quartz and anhydrite also appear acceptable. Anhydrite can be identified and quantified easily only by X-ray diffractometry.

Clay Minerals in Winn Rock Containing Soil. Our understanding of sulfate attack on soils suggests that clay minerals can be important sources of Al ions necessary for ettringite formation. The clay minerals in the Winn Rock containing soil should be thus identified in greater detail. EDTA (ethylenediaminetetraacetic acid) treatment was first used to remove the sulfate fraction from the soil *[34]*. It was not successful in the removal of the sulfate. Hydrochloric acid was then used to dissolve the sulfate phases.   The clay fraction was separated by centrifugation.
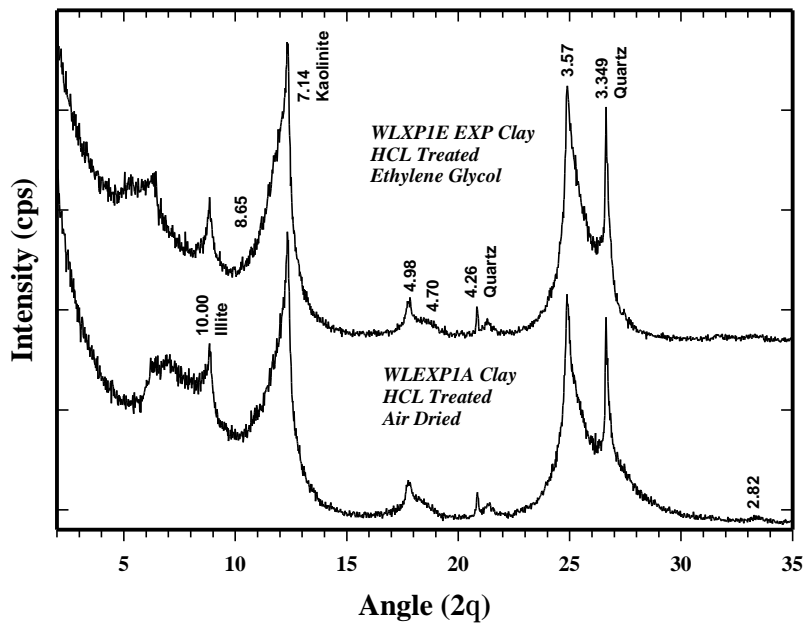
Figure 7

XRD patterns of clay fraction of Winn Rock containing soil

Figure 7 shows the XRD pattern of the clay fraction of the Winn Rock containing soil. At the bottom, the XRD pattern after HCl treatment is shown.   The clay peaks are much more pronounced here compared to the bulk pattern (Figure 5). The broad peak in the range 6º to 8º 2θ is due to smectite. The amount of smectite is very low. There is a shift in the peak position

from 7º 2θ to about 6º 2θ after ethylene glycol treatment (which enters the interlayer of smectite and expands its d spacing by pushing apart these alumino-silicate sheets), but the peak height is very low. The peak corresponding to 1 nm (10 Å) d-spacing is due to illite. Kaolinite and some amount of quartz can also be identified.

Fourier Transform Infrared Spectroscopy. FTIR is a rapid method for identification of calcium sulfate phases, particularly those with water molecules *[37]* . For example, the presence of any basssanite or hemihydrate (same as Plaster of Paris) in a soil can be easily checked by this method.   Figure 8 shows the FTIR patterns of reagent grade gypsum, ground Winn Rock dried for two hours at 45ºC, and two Winn Rock containing soils sampled at two different times.   The OH (hydroxyl) peaks in the range 3650 cm^{-1} to 3300 cm^{-1} have their intensities reversed for bassanite and gypsum.   Bassanite has stronger 3609 cm^{-1} peak and less intense 3555 cm^{-1} peak. Gypsum has a less intense 3540 peak and more intense 3400 cm^{-1} peak. There is also a weak 3483 peak for gypsum. Similarly for the molecular water peak around 1600 cm^{-1}, bassanite has a single peak at 1600 cm^{-1} whereas gypsum has a peak at 1620 cm^{-1} and a weaker shoulder at 1686 cm^{-1}.   The peaks in the range 700 to 600 cm^{-1} are also different for these two phases. Whereas bassanite has peaks at 662 cm^{-1} and 606 cm^{-1}, gypsum has peaks at 671 cm^{-1} and 608 cm^{-1}.

FTIR shows that the drying of   weathered Winn Rock in an oven for two hours even at a relatively low temperature of 45ºC makes gypsum lose some of its molecular water, resulting in the formation of bassanite. The nature of the soil is thus altered, in the process changing its water requirement. Extreme care is thus necessary for drying Winn Rock containing soil, air drying being a better method than any type of heating.
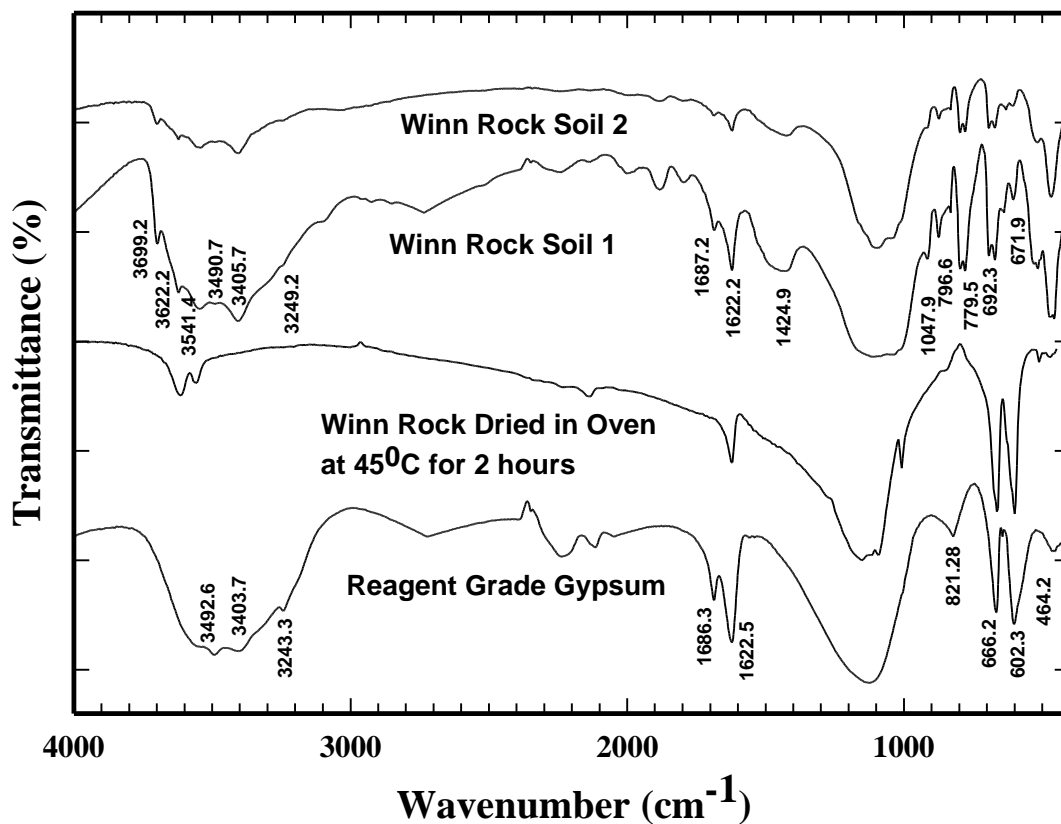
Figure 8

FTIR patterns of gypsum and Winn Rock containing soil

Differential Scanning Calorimetry. Differential scanning calorimetry can easily distinguish between gypsum and bassanite.   Gypsum ($CaSO_4.2H_2O$) loses one and one-half molecules of water around 140°C to form bassanite and the remaining half molecule is lost around 190°C (Figure 9). The ratio of the areas of these two peaks is 3:1. If this ratio is lower, the presence of bassanite ($CaSO_4.1/2H_2O$) is indicated.   Since anhydrite has no molecular water, it cannot be detected by this method.
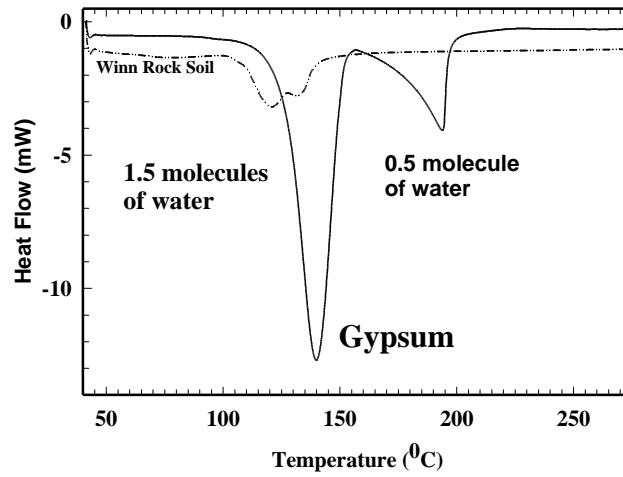
Figure 9

DSC of Winn Rock containing soil
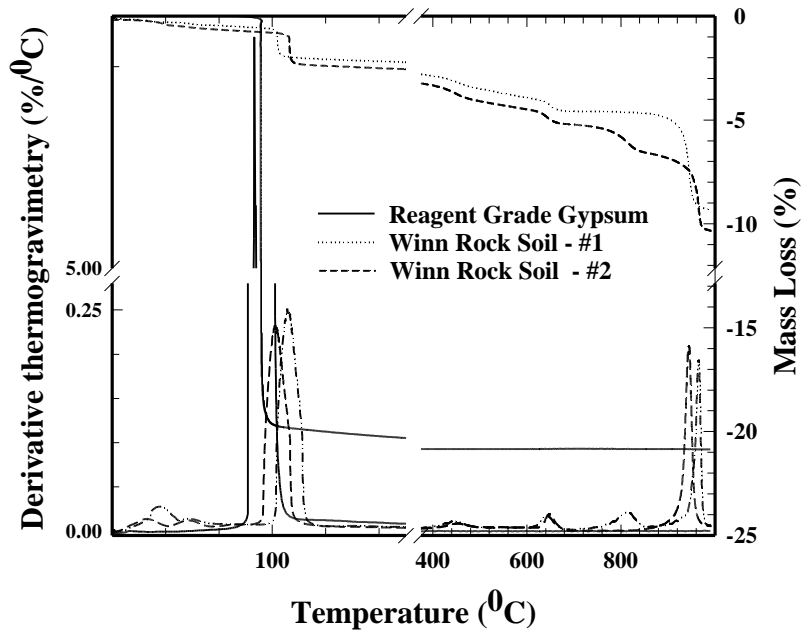
Figure 10

DTG of Winn Rock containing soil

Thermogravimetry. Thermogravimetry is a convenient technique for quantification of hydrated and carbonated phases *[38]*. The mass loss due to dehydration, for example from gypsum, can be accurately measured and related to the amount of that phase present. However, gypsum and bassanite cannot be separated by this method because they dehydrate over the same temperature range and anhydrite cannot be identified at all because it does not contain any water or carbon dioxide. The mineral causing the expansion, ettringite ($3CaO.Al2O_3.3CaSO_4.26H_2O$), can also be detected and quantified by this technique. However, the water loss steps of ettringite are very complex and highly overlap with the moisture loss from the sample itself. Thus it is difficult to quantify ettringite by thermogravimetry. Ideally, the loss of two molecules of water from $CaSO_4.2H_2O$ leads to 20.88 percent loss in mass.

Figure 10 shows the TG and DTG curves of two different samples of the Winn Rock containing soil. Gypsum dehydration spans the temperature range 98ºC to 116ºC, with the maximum dehydration occurring at 105ºC. The average amount of gypsum in the soil from analyses at four different times is 7.85 ± 0.39 percent (8.2, 8.0, 7.9, 7.3 percent). The peak at around 400ºC best corresponds to kaolinite. The peak at 800ºC could not be identified with certainty.
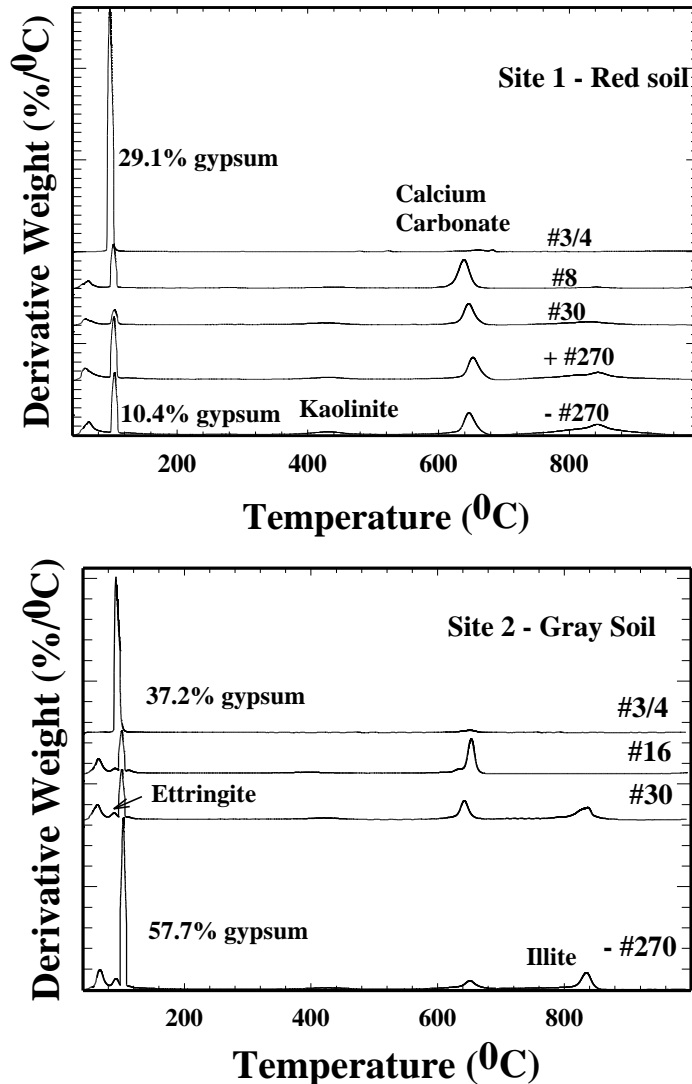
Figure 11

DTG curves of Winn Rock soil fractions

The DTG curves of two samples of the Winn Rock soil from two sites are shown in Figure 11. The different size fractions, obtained by sieving, were individually analyzed. First of all, all size fractions have gypsum. Since there is no naturally-occurring gypsum in the soil in this region, the initial anhydrite from the Winn Rock present in the soil must have hydrated to form the gypsum. The amount of gypsum in different size fractions has been quantified. The reddish soil shows a large amount of gypsum in the coarse size fraction and about one-third in the #-270 size fraction. The grayish soil, in contrast, has much more gypsum in the finer size (#-270) fraction than in the coarsest fraction. The finest size fraction in the gray

soil also has a distinct but small ettringite peak just before the gypsum peak. Ettringite cannot be detected in the reddish soil. A variable amount of calcium carbonate is also present in all but the coarsest size fraction of the soil samples.
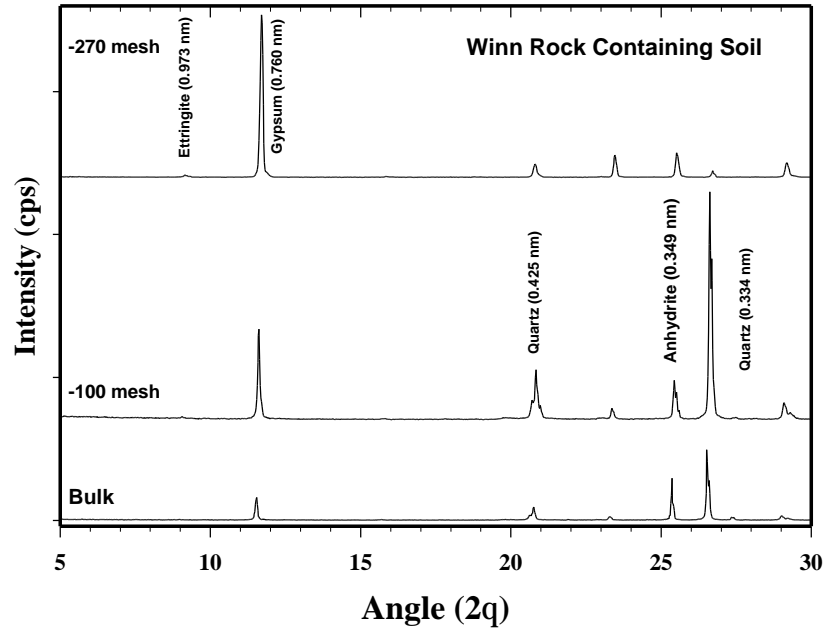
Figure 12

XRD patterns of Winn Rock containing soil

Figure 12 shows the XRD patterns of different size fractions of the Winn Rock containing soil. The ratio of the peak heights of gypsum to anhydrite gradually increases from the bulk to the finer size fractions. The amount of gypsum thus increases in the finer size fraction. However, anhydrite is still present in the finest size fraction. Ettringite is undetectable in the bulk but can be clearly detected in the finest size fraction.

Scanning Electron Microscopy. A scanning electron photomicrograph of a Winn Rock containing soil particle is shown in Figure 13. Ettringite crystals are abundant in the particle. Crystallization of ettringite requires pH at least above 10.7 *[28]*. Such pH normally does not occur in soil. However, some pre-existing portland cement, from the previous attempt to stabilize the soil, can provide the high pH necessary for ettringite's crystallization.

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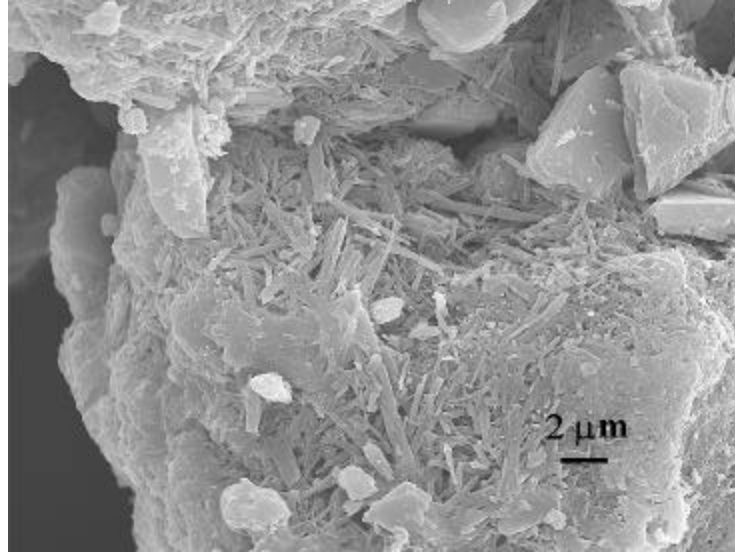
Figure 13

SEM photomicrograph of Winn Rock containing soil

X-Ray Absorption near Edge Structure. X-ray absorption near edge structure (XANES) is a spectroscopic technique which provides coordination and oxidation state information around an atom *[39]*. Subtle variation in the crystal structure of the sulfate phases can be used in their identification. The XANES spectra of a sample can be fitted to quantitatively obtain the different sulfate phases.
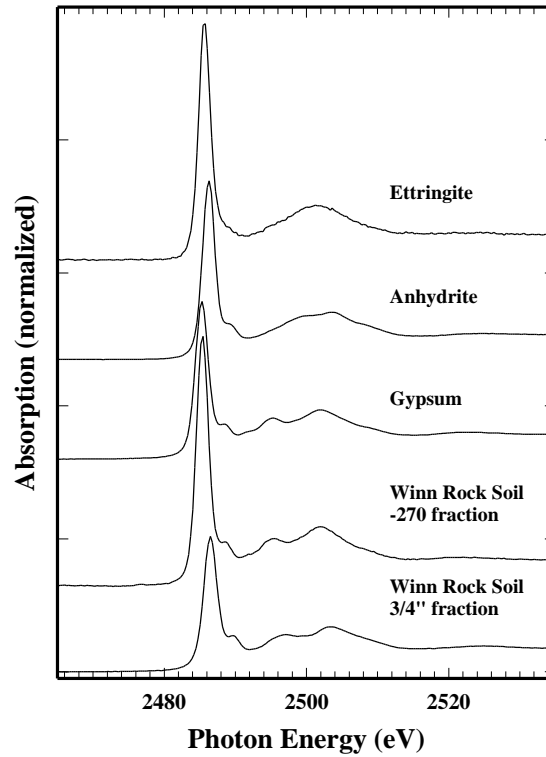
Figure 14

XANES spectra of some sulfate minerals and Winn Rock containing soil

XANES spectra of some relevant sulfate minerals and two size fractions of Winn Rock containing soil are shown in Figure 14. The S atom has slightly different environment (and crystal system) in each of the phases, and thus slightly different XANES spectra. The S K edge XANES spectra of the soils are sums of the spectra of the different sulfate species. Curve-fitting shows that 90 percent of the sulfate in the Winn Rock Soil finer than the # -270 sieve is gypsum.

Effect of Kinetics

Kinetics of Sulfate Dissolution. James and Lupton developed a method for dissolution rate analysis of sulfates and determined the following values *[40]*:

	Gypsum	Anhydrite
Rate Equation	$dM/dt = KA(c_s-c)$	$dM/dt = KA(c_s-c)^2$
K	2.9×10^{-6} m/sec	5.4×10^{-8} m/sec

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Symbol	Explanation
M	mass of calcium sulfate dissolved at time t
c_s	concentration of substance in saturated solution
c	concentration of substance in solution at time t
A	area of mineral surface exposed to aqueous solution
K	rate constant

It follows from these equations that for the same surface area in water containing no sulfate (c=0, for example, rapidly flowing water), the dissolution rate of gypsum is 3,000 times more than that of anhydrite. The dissolution rate being directly proportional to the surface area, it also follows from the above equations that for a given mass or volume, the finer grains with higher surface area per unit mass will dissolve faster than the coarser grains. Thus gypsum, which was often found in higher concentrations in the finer size fractions, would more readily dissolve compared to anhydrite, which was more often concentrated in the coarser size fractions *[31]*.

Stabilized Soil

Mechanical Properties

Moisture Content and Dry Density. The dry density (γ_d) of specimens C5 (see Table 4 for sample descriptions) and C9, determined from compaction tests, are plotted against the corresponding moisture contents in Figure 4. The dry density of Winn Rock containing soil is also shown for comparison. These plots show that the maximum dry densities for Winn Rock containing soil stabilized at different cement contents, C5 and C9 are 117.13 lb/ft^3, 119.04 lb/ft^{3}and 119.68 lb/ft^3, respectively.

The addition of cementitious materials to a soil changes both its optimum moisture content and the maximum dry density for a given compaction effort. The flocculating action of the cementitious materials tends to produce an increase in optimum moisture content and decrease in maximum density *[41]*. However, as indicated in Figure 4, the addition of cement increases the maximum dry density of the specimens C5 and C9. This may be attributed to the high specific gravity of cement relative to the soil mixture, and its small size particles easily filled in the voids of the soil mixture.

Compaction tests revealed the relationship of dry density and moisture content of each mixture of the Winn Rock soil stabilized with C:BFS (Figure 4). The values of maximum dry density of the specimens C:BFS 1:1 and C:BFS 1:3 are 119.04 and 119.04 lb/ft^3, respectively. There are no basic differences in the compaction behavior between the soil stabilized by portland cement and those stabilized by C:BFS.

BFS is a SCM which can be activated not only by calcium hydroxide but also by other materials such as sodium silicate and gypsum *[42]*. Activators can be both alkaline and slightly acidic. They will lead to the formation of C-S-H gel and ettringite. Both cement and gypsum presented in Winn Rock containing soil can function as "activators" for the hydration of BFS. Generally speaking, the strength of C:BFS is lower than that of portland cement, especially in the early stage of hydration. On the other hand, BFS is acknowledged to have a higher resistance to sulfate attack. Considering both the factors, the high strength value of specimens C:BFS 1:1 and C:BFS 1:3, compared to specimens C5 and C9, must partially be contributed to this sulfate-attack-resistant property. It is worthwhile to indicate that specimen C:BFS 1:3 with less portland cement content reached 450 psi at 7 days, suggesting that BFS has a profound effect on the strength development of sulfate-bearing soil cement matrix. In other words, the presence of a significant amount of gypsum present in the Winn Rock containing soil, plus calcium hydroxide released by cement, activated the latent hydraulic activity of BFS and led to a high strength development.

Winn Rock Soil Stabilized by Portland Cement with Class C fly ash and amorphous silica. Compaction test results also revealed the relationship of dry density and moisture content of each mixture of the Winn Rock containing soil stabilized by portland cement plus Class C fly ash and amorphous silica (Figure 4). The values of maximum dry density of specimens C:CFA 1:1 and C:CFA:AS 1:0.75:0.25 are 119.04 and 117.77 lb/ft^3, respectively.

It is apparent that the addition of Class C fly ash or Class C fly ash and amorphous silica to soil cement changes its compaction characteristics to some extent. However, the general compaction behavior is essentially the same as that of soil cement mixture C5. Class C fly ash acquired high maximum dry density at low optimum moisture content, and this may be attributed to its dispersion effect. In contrast, with the limited amount of amorphous silica added, the mixture obtained a low maximum dry density. This may be due to the high pozzolanic activity of amorphous silica that leads to a strong flocculating action.

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Linear Expansion. No standards are available to indicate an acceptable limit value and severity of sulfate expansion for soil cement. For cement concrete, Stark visually evaluated the performance of test specimens by using a numerical rating system with a range from 1.0 to 6.0 (with 1.0 indicating no evidence of deterioration and 6.0 indicating failure) *[43]*. Samarai recommended 0.1 percent expansion as a safe margin for determining the maximum sulfate expansion percentage that can be introduced into mixtures without causing any deterioration *[44]*. Crammond used 0.1 percent expansion after six months as the limit above which expansion becomes significantly deleterious *[45]*. The Federal Highway Administration (FHWA) accepts up to 0.1 percent expansion in concrete which suffers from the alkali silica reaction. Based on these suggestions, Wang developed a scale of severity of sulfate expansion (Table 5) *[46]*. Expansion up to 0.1 percent (negligible, Table 5) will thus be considered acceptable for this study.

Table 8
Severity of sulfate attack on soil cement specimens (after *[46]*)

Linear expansion (%)	Severity
0	zero expansion
0 to 0.1	negligible
0.1 to 0.5	light
0.5 to1.0	medium
1.0 to 2.0	strong
Larger than 2.0	very strong
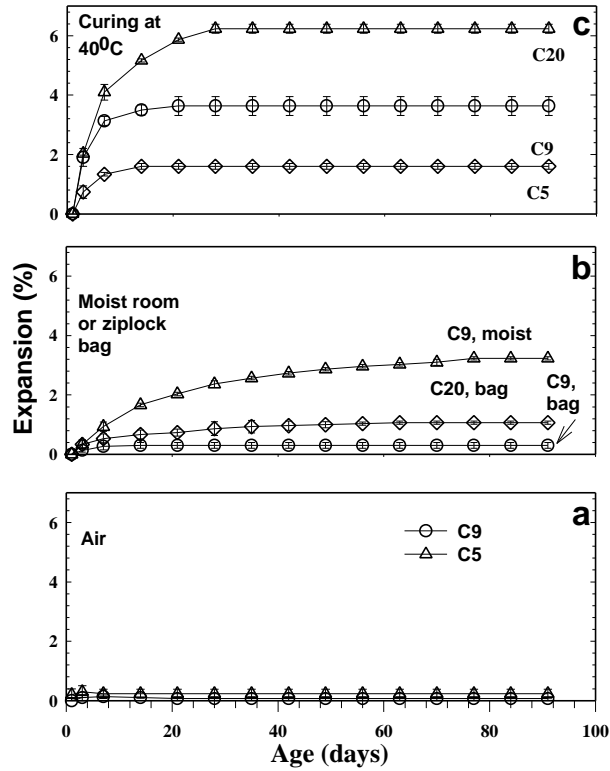
Figure 15

Expansion in portland cement-stabilized Winn Rock containing soil

       Winn Rock soil containing soil stabilized by Type I portland cement. Figure 15 shows the expansion in Winn Rock containing soil stabilized with Type I portland cement and cured under various conditions. A comparison of the curing environments shows that the expansion is highest in a moist environment at higher temperature, and the least when air cured at room temperature.   Figure 15c shows the effect of cement content. The amount of expansion is directly proportional to the amount of cement. The magnitude of the expansion is as high as 6 percent.   The expansion is initiated immediately at the beginning of the curing period and continues for a length of time in direct proportion to the amount of cement. Curing in a moist room at 20ºC and in a sealed ziplock bag also produces significantly different expansion, indicating the importance of continued availability of moisture. For the same 9 percent cement content, the amount of expansion is an order of magnitude less for the ziplock bag where only a fixed amount of water is sealed into the bag at the beginning of the curing period. Air curing produces a negligible amount of expansion. The expansion period can also be discussed in terms of a rate.   The rate of expansion is high at the highest cement

concentration, higher temperature, and highest moisture availability. The gradient becomes less steep with lower cement content, less moisture and lower temperature. A plateau in expansion is reached usually within a month. For 9 percent cement stabilized soil, cured in a moist room, the expansion appeared to continue for a longer time but does reach a plateau. The expansive reactions probably stop when the reactants are completely consumed.

Winn Rock Containing Soil Stabilized by Cement and SCMs. The effect of adding a SCM to portland cement for stabilization of Winn Rock containing soil can be seen in Figure 16. Compared to portland cement alone, the magnitude of expansion is almost an order less. The maximum amount of expansion (0.5 percent) is observed when a mixture of portland cement and fly ash is used. The curing environment does not have a major influence on expansion, with almost identical results in the water bath at 40ºC and in a sealed ziplock bag at 20ºC. The rate of expansion, however, is different. Most of the expansion occurs early in a high moisture environment, whereas the same magnitude is reached slowly in a limited moisture environment. For C:BFS mixtures, increasing the amount of BFS reduces the amount of expansion, again being almost similar in two very dissimilar environments. The rate of expansion also shows a pattern similar to the cement and Class C fly ash mixtures. The addition of amorphous silica or silica fume to the stabilization mixture reduces expansion to below detection limit.
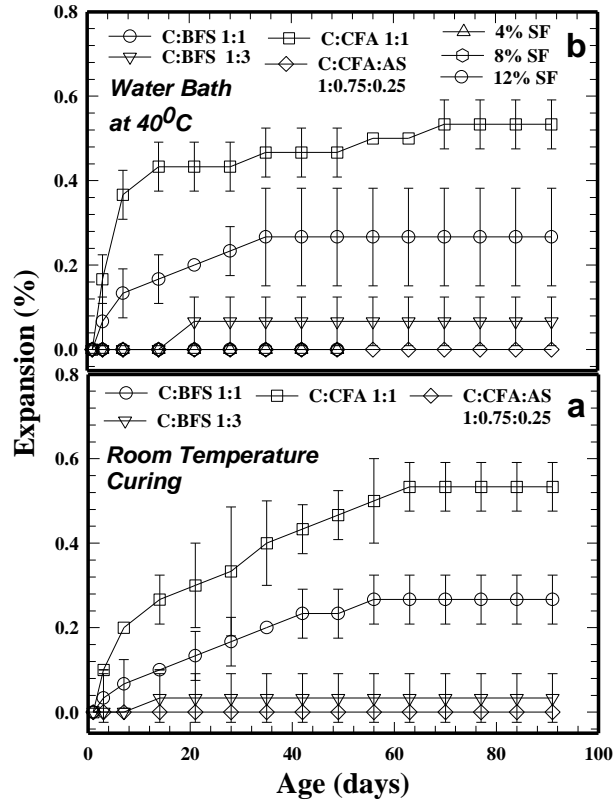
Figure 16

Expansion in Winn Rock Soil stabilized by SCMs

Figure 17 compares expansion of Winn Rock containing soil at the same 9 percent cementitious material level, under different curing conditions. The effect of temperature on expansion can be seen from the portland cement data. The general pattern of expansion, for all stabilization mixtures, is that there is an early period of expansion, followed by no expansion, at least over the six month period that they were monitored.
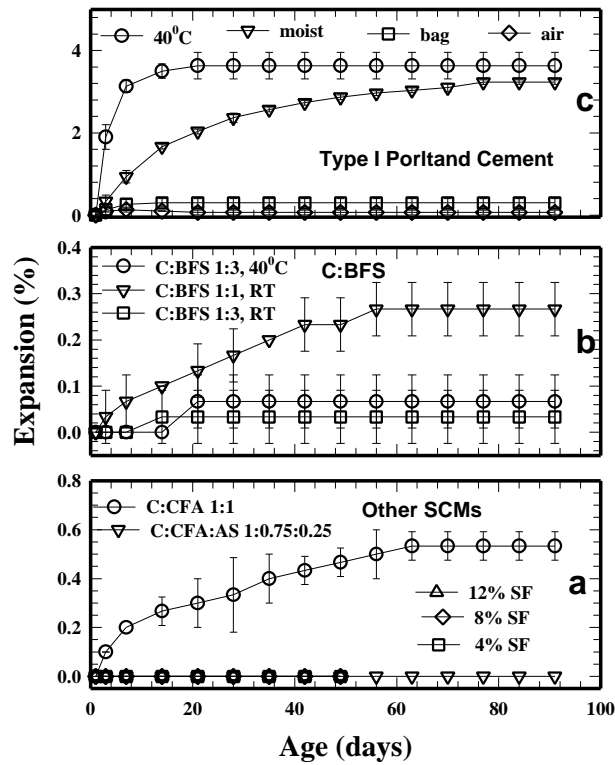
Figure 17

Expansion in cementitious material stabilized Winn Rock containing soil

Correlation between Expansion and Sulfate Content of Soil

Figure 18   shows the effect of Winn Rock concentration on expansion when stabilized by Type I portland cement. After fabrication, the specimens were cured in a 100-percent-relative- humidity room. The amount of Winn Rock or Winn Rock-derived products (e.g., gypsum) was diluted by mixing the soil with that from the ALF site. The mixing of these two soils does not significantly alter the grain size distribution as both soils have very similar properties. Mineralogically they are also similar except for the presence of Winn Rock and its weathering products, as they are both produced by the similar Mississippi River alluvial processes.
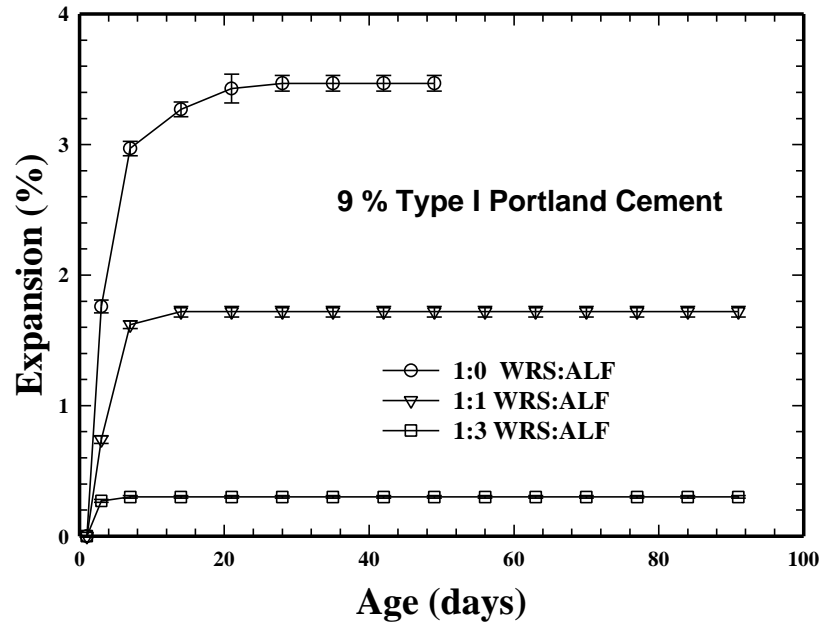
Figure 18

Effect of Winn Rock concentration on expansion

The specimens were prepared with 9 percent by weight of Type I portland cement. They were cured at 40°C in a water bath to simulate the high moisture availability condition. Figure 18 shows that diluting the Winn Rock containing soil with the ALF soil in 1:1 and 1:3 ratios reduces expansion by half and an order of magnitude, respectively. All soils exhibit early expansion, followed by little or no expansion at later age. In fact, the early expansion period is directly proportional to the amount of Winn Rock containing soil in the mixture.

Susceptibility of a Soil to Sulfate Attack

Some tests are available for testing the susceptibility of a soil to sulfate attack. These tests are usually based on the solubility of the sulfate component in a certain volume of water (see *[2]*). However, the solubility of calcium sulfates in water (Table 2) is low; therefore only a certain amount of calcium sulfate can be detected in a soil, if present. For example, the solubility of gypsum is 2 gm per liter. Thus if more than 2 gm of gypsum is present in a soil, the excess amount will not be detected. In such situations mineralogical testing by X-ray diffractometry and thermal analysis can be performed. It may also be necessary to quantify the sulfates in different size fractions since this study showed that the finer size fractions are more reactive than the

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coarser ones. Detection and quantification can be performed by the procedures outlined in the Analytical Methods section. X-ray diffraction pattern of the soil needs to be obtained to identify the minerals present in the soil. Thermogravimetry will be easier to perform than quantitative X-ray diffractometry, though the latter method can provide quantitative data for all types of sulfates.

Unconfined Compressive Strength

Unconfined compressive strength (UCS) was determined for specimen cylinders of at seven days and twenty-eight days. The unconfined compressive strength of specimens increased with increasing cement content and curing time (Figure 19). The Winn Rock containing soil itself shows several fold strength increase after mixing with water. This is presumably due to hydration of anhydrite to gypsum, which forms interlocking crystals in the process.
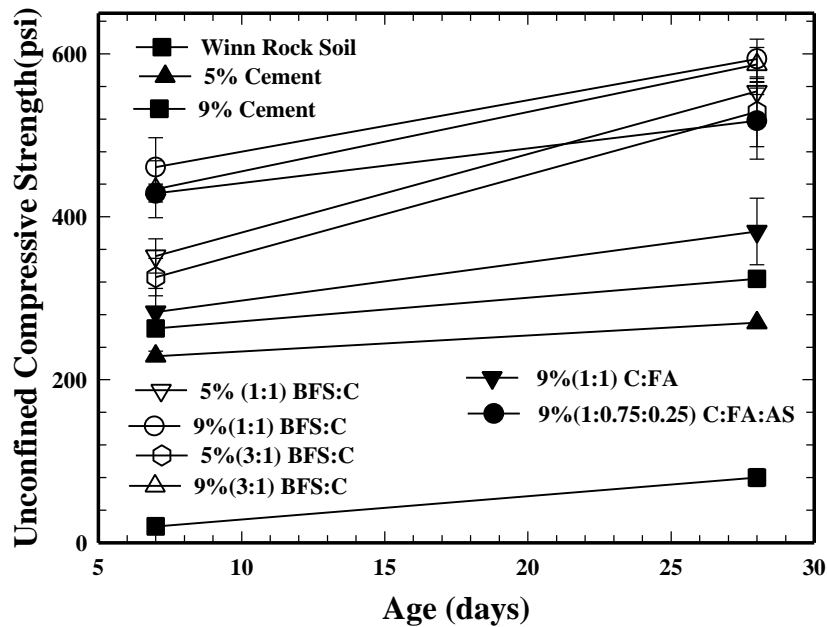
Figure 19

UCSs of Winn Rock soil cement mixture cylinders

Stabilization of Winn Rock containing soil with 5 to 9 percent Type I cement yields only about 250 psi strength at seven days. There is barely any strength gain of the stabilized soil at these cement concentration levels at twenty-eight days. The likely reason for this low strength is the development of abundant cracks in the stabilized soil due to sulfate attack as seen in the photograph of the molds (Figure 20). The typical unconfined compressive

strength values of sandy and gravelly soils stabilized with 5 to 9 percent cement may be up to 300-600 psi for seven-day curing and 400-1000 psi for twenty-eight-day curing *[4]*. The relationship between sulfate attack and strength loss is poorly known. Nevertheless, Sherwood found that sulfate contents as low as 0.2 percent resulted in losses of strength of more than 50 percent in clay stabilized with 10 percent cement *[20]*.

Stabilization by 5 percent 1:1 BFS:C mixture increases the strength slightly compared to Type I portland cement alone at seven days. However, stabilization by 9 percent of the same mixture increases the strength to above 400 psi at seven days. For the 3:1 BFS:C mixture, both 5 and 9 percent cement yielded more than 400 psi strength. The rate of strength gain up to twenty-eight days in the presence of BFS is higher than that of portland cement only. In concrete, C:BFS shows similar strength gain behavior. No cracks were observed in the molds stabilized by C:BFS mixtures.

Stabilization of the Winn Rock containing soil by Class C fly ash (specimen C:CFA 1:1) or Class C fly ash and amorphous silica (specimen CFA:AS   1:0.75:0.25) also produced an increase in strength. However, a noteworthy difference between them is that the strength increased about 30 percent with incorporation of 1.125 percent amorphous silica instead of Class C fly ash, as indicated in Figure 19. This strength was achieved by seven days and changed little thereafter. This dramatic improvement must be attributed to the high pozzolanic properties of amorphous silica *[12],[13]*. Since it has a high pozzolanic activity, amorphous silica will react with calcium hydroxide, released by the hydration of cement, to form C-S-H gel and lead to an increase in strength. At the same time, as calcium hydroxide is converted to the C-S-H gel, the risk of sulfate attack is reduced, for little calcium hydroxide is available for the formation of gel-like ettringite.
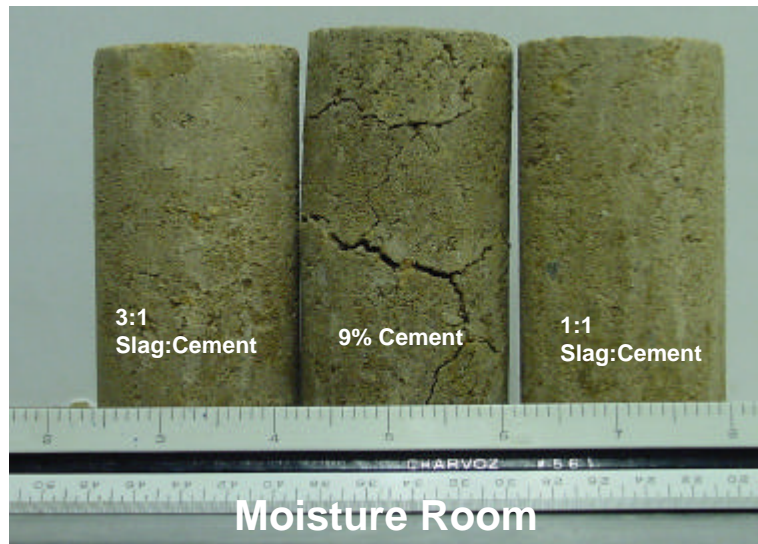
Figure 20

Integrity of cementitious material stabilized Winn Rock containing soil

Mineralogy and Microstructure
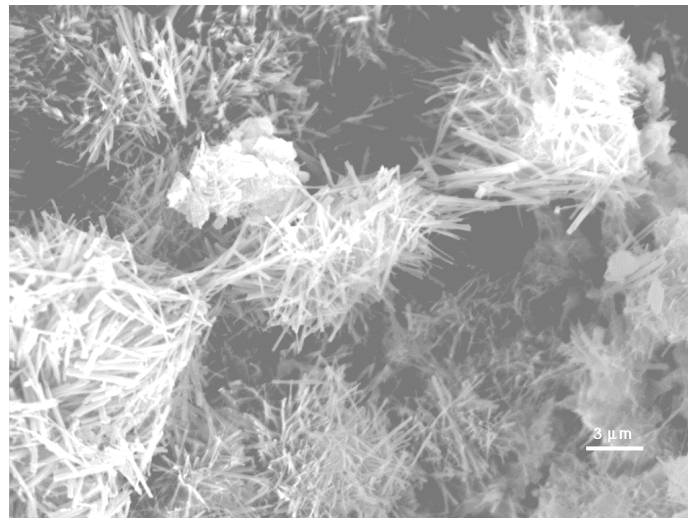
Figure 21

SEM photomicrograph of lime-stabilized soil

Winn Rock Soil Stabilized by Lime. An SEM photomicrograph of the Winn Rock soil stabilized by 9 percent lime and cured at 40°C in a water bath for three months is shown Figure 21. There are abundant ettringite crystals. The crystals show a wide size distribution.
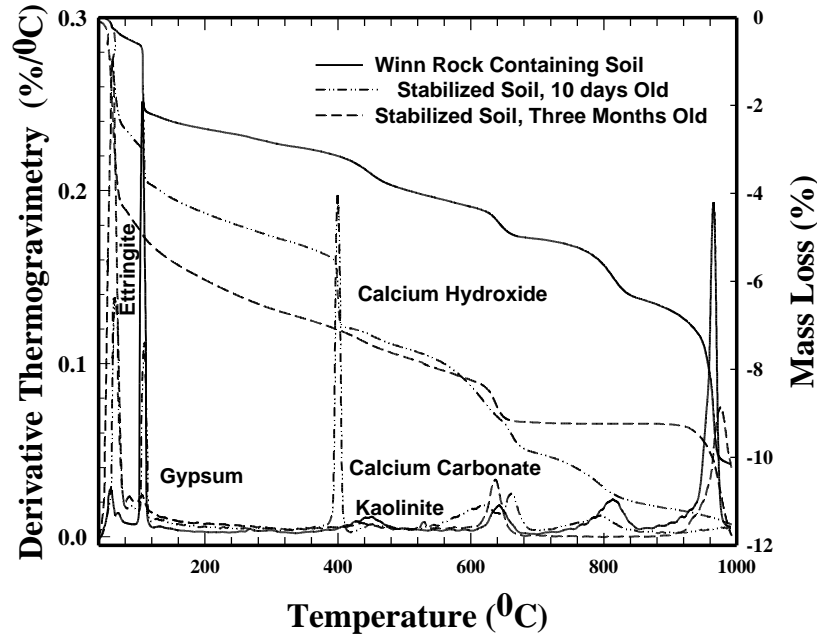
Figure 22

DTG curves of Winn Rock containing soil stabilized by lime

The thermal analysis patterns of the lime-stabilized, Winn Rock containing soil shows how gypsum reacts with lime. In the ten-day-old specimen, considerable amounts of lime and gypsum remain. Even in this early period, a large amount of ettringite has formed, and a significant amount of the gypsum has reacted. The broad peak below the calcium carbonate peak is due to smectite. This is absent in the three-month-old stabilized soil. By the end of three months, the lime peak cannot be detected and the gypsum peak is barely detectable. The peak at 800°C could be due to illite. This peak shows a gradual reduction from ten days to three months.

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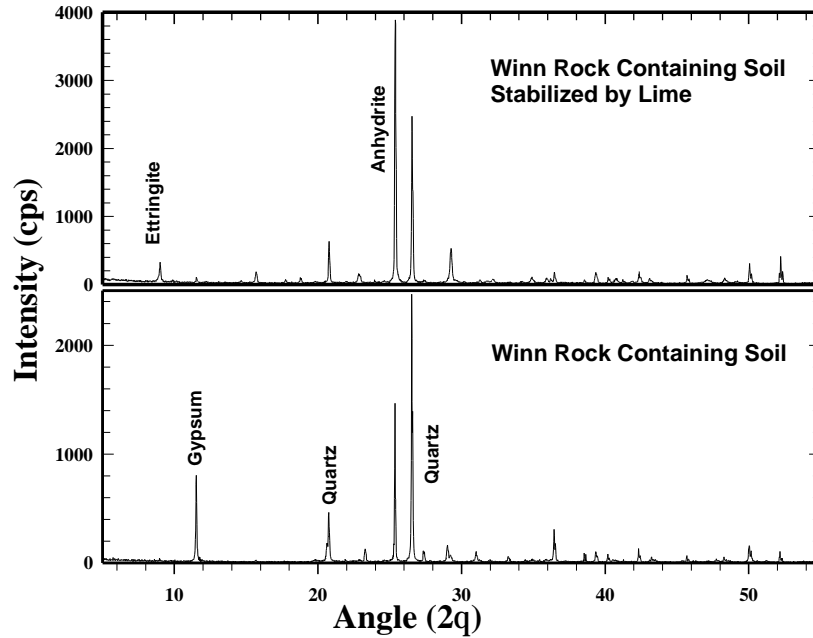
Figure 23

XRD pattern of Winn Rock containing soil compared

A comparison of the XRD patterns of the Winn Rock containing soil in Figure 23 before and after lime stabilization shows similar evidence as thermal analysis. However, anhydrite cannot be detected by thermal analysis and thus its role cannot be identified. Gypsum is barely detectable in the three-month-old stabilized soil whereas a large peak for ettringite can be seen. The two patterns are not strictly comparable as 9 percent by weight of lime has been added to the soil, along with some amount of water. However, the absolute amount of quartz should be the same in both specimens. Relative to quartz, the amount of ettringite has definitely increased in the stabilized soil. This may have happened as one crystalline component, gypsum, was nearly completely consumed in the pozzolanic reaction and thus the total amount of crystalline components has been decreased.

Stabilization of Winn Rock containing soil by lime and curing at high temperature in a high moisture environment are expected to simulate the worst possible scenario. At the same time, Winn Rock containing soil and lime provide a simpler system for investigation as more complicated hydration reactions of cement are absent. The rapid rate of expansion seen from the length measurements of the specimens can be clearly correlated with the mineralogical changes. The early expansion that is observed correlates very well with rapid consumption of gypsum and a large increase in ettringite crystallization.

Winn Rock Soil Stabilized by Portland Cement
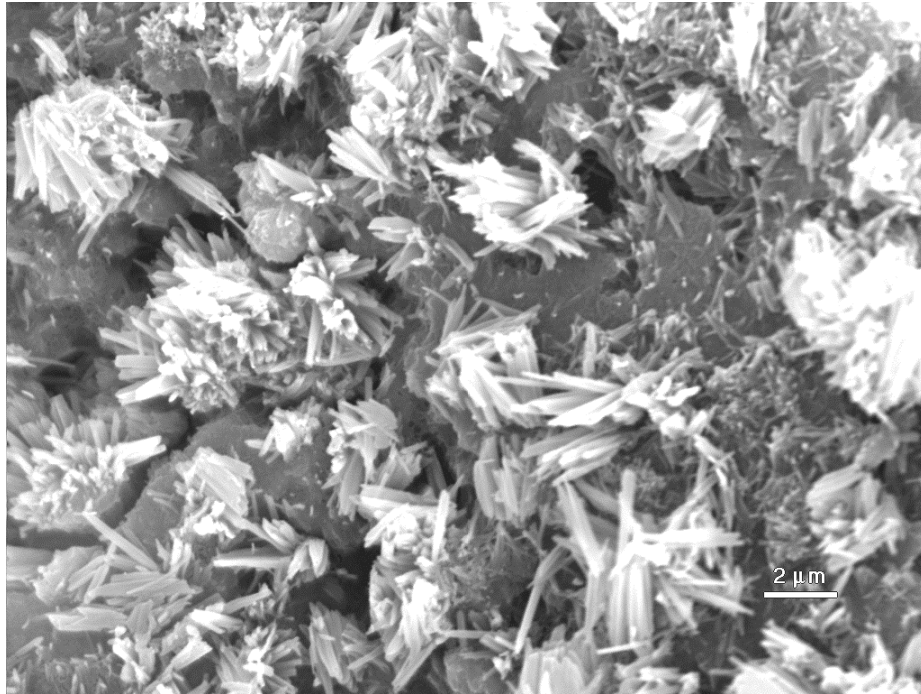
Figure 24

SEM photomicrograph of portland cement-stabilized soil

A representative SEM photomicrograph of the Winn Rock containing soil stabilized by 9 percent Type I portland cement and cured at 40ºC in a water bath is shown in Figure 28. Abundant ettringite crystals are present. They are also very homogeneously distributed. The length of the ettringite crystals shows a wide variation. Broadly, two morphologies are observed. First, there are the longer, wider diameter crystals, approximately 5 μm long, which occur in bundles. Second, there are the smaller, individual crystals embedded in other phases, about a μm in length.
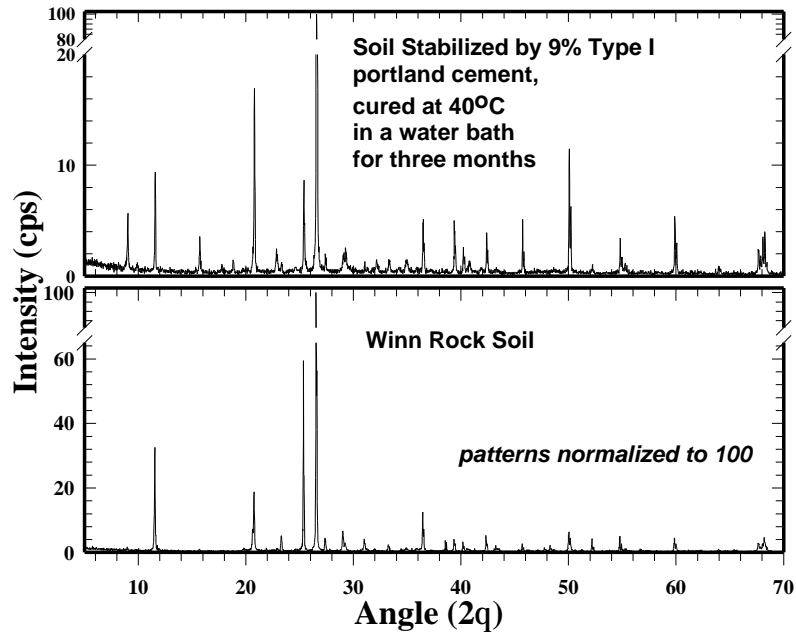
Figure 25

XRD patterns of stabilized soil

The XRD patterns in Figure 25 show the mineralogical changes after stabilization by Type I portland cement at high temperature and high humidity. Compared to the soil, a large amount of ettringite is present in the stabilized soil. The amount of gypsum has been reduced by approximately half in the stabilized soil. The amount of anhydrite shows a dramatic reduction in the stabilized soil. The intensity of the anhydrite peak has been reduced from close to 60 percent in the Winn Rock containing soil to less than 10 percent in stabilized soil. A part of this reduction is due to the addition of 9 percent cement by weight but that alone cannot account for this amount of reduction. The XRD patterns suggest that along with gypsum, anhydrite is also participating in the expansive reactions.

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Winn Rock Soil Incorporated with Portland Cement plus Class C fly ash as well as Amorphous Silica
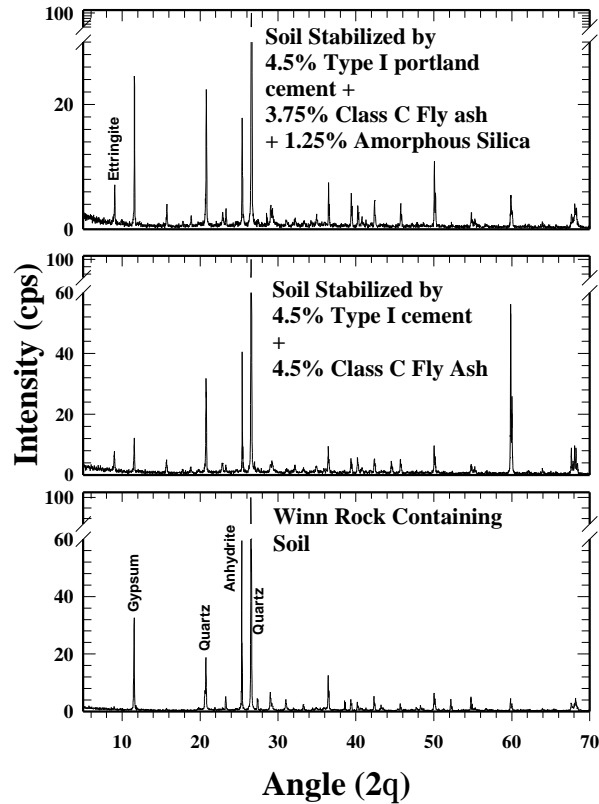
Figure 26

XRD patterns of Winn Rock containing soil compared with stabilized ones

A comparison of the XRD patterns in Figure 26 above shows that amount of gypsum is similar in the soil and after stabilization by the addition of cement, fly ash, and amorphous silica. The anhydrite peak intensity, in contrast, is reduced by about half when all these SCMs were added. In the presence of cement and Class C fly ash, the reduction in gypsum peak is very significant, whereas the anhydrite peak intensity has not been reduced much. The ettringite peak intensities are very similar in the stabilized soil, even though the magnitude of expansion is several times less with the presence of AS.

Relationship between Ettringite Crystals and Expansion

The relationship between expansion in concrete and the total amount of ettringite present has been a vexing issue. Very often there is no correlation between the two variables. In fact,

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Famy and Taylor recently proposed that the presence of large amounts of ettringite in failed concrete is more an effect than a cause *[47]*. The authors' present work shows that abundant ettringite crystals are present in most samples without any correlation to the magnitude of expansion. The left photomicrograph in Figure 27 shows the typical ettringite morphologies in lime-stabilized Winn Rock containing soil. The right photomicrograph shows the same feature when the stabilization mixture contained Type I portland cement, fly ash, and amorphous silica. Qualitatively, the ettringite crystals in the lime-stabilized soil are thinner in diameter whereas they are thicker in the latter. Similar ettringite morphologies were also observed by Tasong et al. *[10]*.
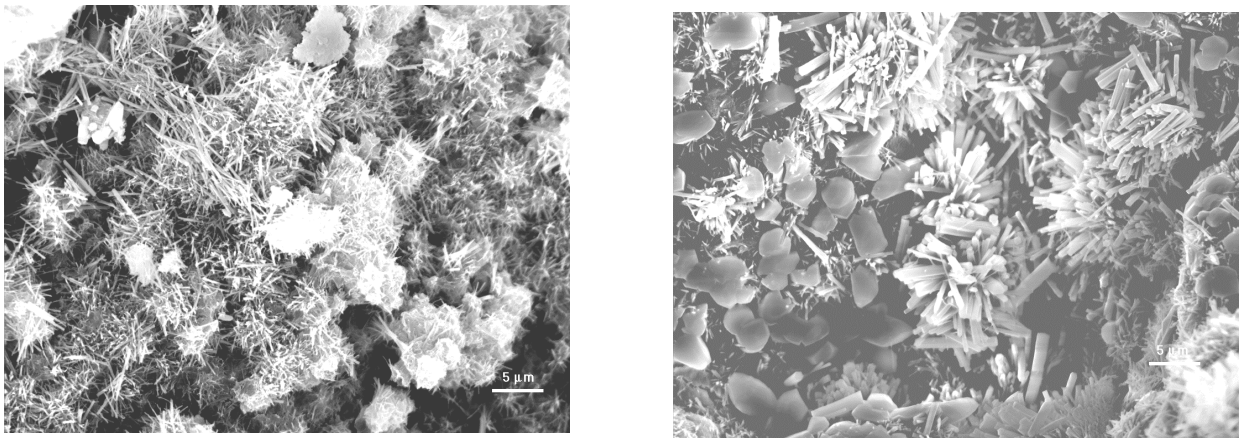
Figure 27

Scanning electron photomicrographs of ettringite crystals

Figure 28 shows the distribution of the length of the ettringite crystals in these stabilized soils of one hour to three months of age. The length of the ettringite crystals was measured with the image analysis software UTHSCA ImageTool. The assumption is that the lengths measured are the full lengths. Since a part of ettringite needles is often embedded within other phases, this assumption is not fully valid. The length measured is probably the minimum length.

Many differences can be noticed in the grain size distribution of these two stabilized soils

(Figure 28). When stabilized by lime, the average grain size is less than a µm at one hour of age. The average grain size increased up to seven days of age but stayed unimodal. At three months of age there is a wide variation in grain size, without any dominant mode. The number of crystals observed was also much higher compared to the cementitious mixture group. The other group, stabilized by portland cement, Class C fly ash, and amorphous silica, at one hour had an average ettringite grain size slightly coarser than that of the lime stabilized soil. The size variation was larger even at one day and is distinctly poylmodal by seven days. Fewer crystals were observed in these specimens compared to the lime-stabilized one.
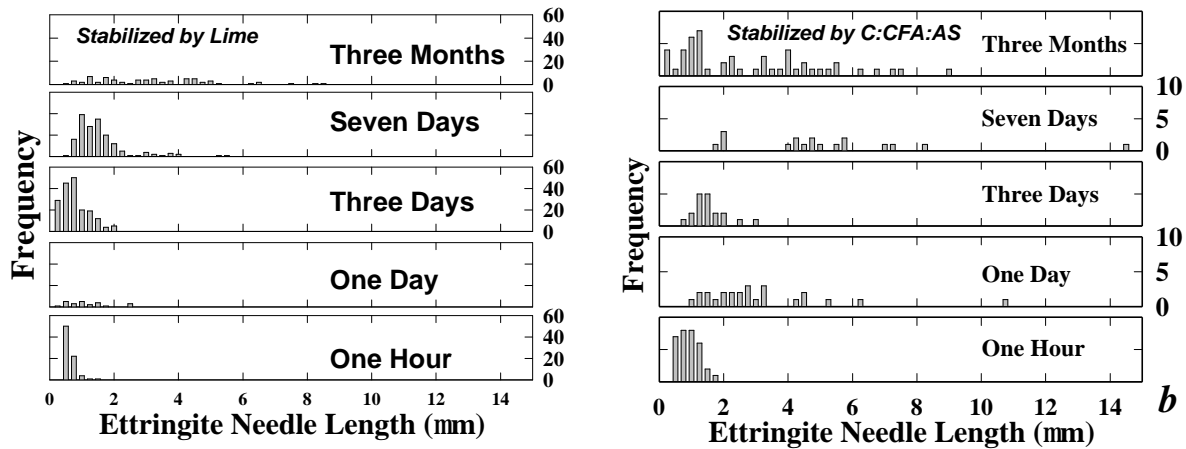
Figure 28

Distribution of ettringite crystal lengths in stabilized soils

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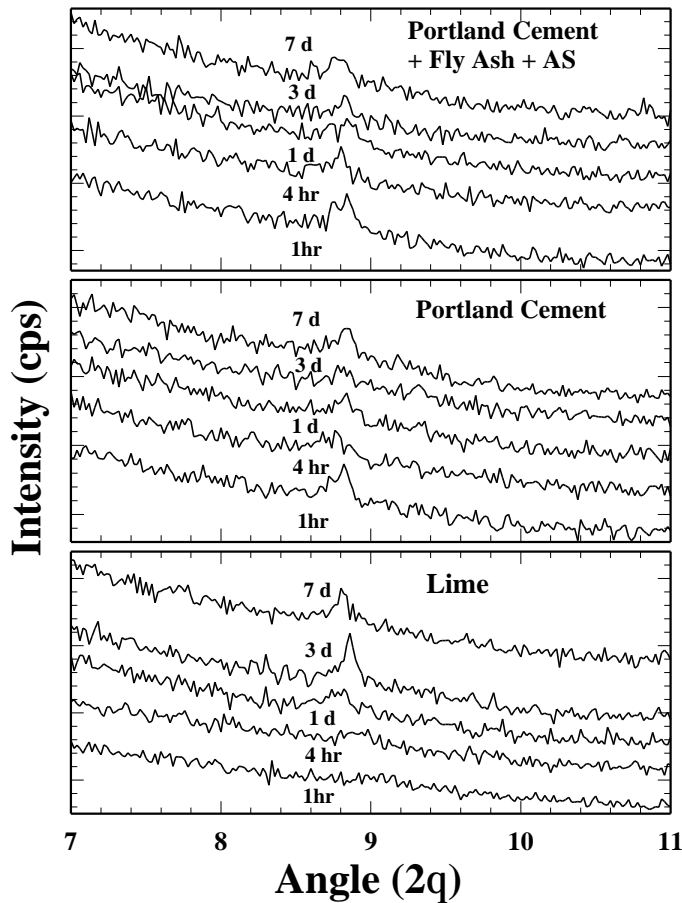
Figure 29

Early-age XRD patterns of soil stabilized by different SCMs

Figure 29 shows the early age XRD patterns of the Winn Rock containing soil stabilized by different binders. The XRD pattern in this range for a typical run is usually extremely noisy. The standard deviation for discrete counts is given by the formula $\sqrt{N}/N$, where N is the total number of counts. An increased number of counts will thus reduce the noise. The counting time was increased to one minute at each step instead of two seconds for a typical run (see Analytical Methods Section).

The lime-stabilized soil has a very broad ettringite peak at one hour age, which shifts to lower 2θ with age. The peak becomes sharper and more well-defined when three days old. The portland cement stabilized soil has a well-defined ettringite peak even at one hour. The peak becomes wider but stays discernible through seven days of age. The soil stabilized by the

cementitious mixture containing amorphous silica has X-ray diffractometry patterns over time very similar to those of portland cement only. The X-ray diffraction peak width of a phase is related to its average grain size by the Scherer equation *[32]*. The higher the width, the smaller is the grain size. For the lime stabilized soil the ettringite crystals up to one day are extremely small and become larger as time progresses. In contrast, the ettringite crystallite size does not vary much in portland cement and cement + Class C fly ash + amorphous silica stabilized soils.

Qualitatively, the expansion behavior of the lime-stabilized Winn Rock containing soil was very different from that of the cement stabilized or cement + Class C fly ash + amorphous silica stabilized soils. The lime stabilized soil showed too much expansion and could not be stabilized at all. The cement stabilized soil had high expansion whereas the soil stabilized by the cementitious mix containing amorphous silica had practically no expansion.   The amount of free lime gradually decreased from the stabilizing mix: lime > 20% Type I portland cement > 9% Type I portland cement > 1:1 C:BFS > 1:3 C:BFS > 4.5 percent C: 3.375 percent Class C fly ash: 1.125 % amorphous silica. The amount of expansion observed in the Winn Rock containing soil stabilized by these mixes also decreased in the same order. The X-ray diffraction patterns of the lime-stabilized soil is in agreement with the ettringite gel formation theory of expansion, where most of the expansion is effected by very small crystallites. These are not detected in a typical fast X-ray diffraction run because of the noise in the pattern. Crystallization of ettringite of relatively large grain size as seen in mixes containing portland cement   produces a lot less expansion compared to the mix stabilized by lime.

Effect of Supplementary Cementitious Material Addition

The substitution of the portland cement in the stabilizing mix by BFS, Class C fly ash, amorphous silica, and silica fume generally reduced the expansion of the stabilized soil by as much as an order of magnitude compared to portland cement alone (Figures 15 to 17). For C:BFS mixtures, the magnitude of the reduction is in direct proportion to the amount of BFS present. Silica fume and amorphous silica effected even higher reduction in expansion (Figure 17). In comparison, when only lime was used in stabilization, the amount of expansion was the maximum.

This line of evidence suggests that there is direct correlation between the free calcium hydroxide available in the stabilized soil and the amount of expansion. A SCM reduces the available lime in the stabilized soil by reacting with it, in the process producing calcium silicate hydrate. The reactivity of amorphous silica is very high. For example, Anderson et al. showed that amorphous silica starts reacting with the calcium hydroxide released by cement hydration reaction within hours [12]. In a 90:10 portland cement:amorphous silica paste, more than 50 percent of the calcium hydroxide produced by cement hydration was consumed within hours. Silica fume, because of its smaller surface area and larger grain size relative to amorphous silica, will have a slightly slower reaction rate. The reaction rate of BFS or Class C fly ash, because of their larger surface areas, would be significantly slower. Thus all these SCMs would react to consume lime but in the following sequence: amorphous silica > silica fume>>BFS>Class C fly ash. The reduction in expansion effected by the presence of a SCM follows the same sequence.

In cementitious systems, the pH of the system is buffered by calcium hydroxide at around 12.5. If all the calcium hydroxide is consumed, and the buffering capacity destroyed, the pH will be lowered below this value. The lower stability limit of ettringite in terms of pH is 10.7 [28]. In the model system, there is a relationship between the pH and the morphology of ettringite crystals [18]. If the pH remains high, the ettringite crystals are usually shorter and of small diameter. If the pH is reduced by consumption of the calcium hydroxide, the ettringite crystals becomes longer and wider in diameter. The latter also leads to less expansion.

Economic Analysis

The use of blended cement, i.e., mixtures of Type I portland cement and BFS, is not only helpful in mitigating sulfate attack, but it is also economical. Table 7 provides the prices of different mixtures as provided by Darrell Elliot of Lone Star Industries. The blends containing silica fume are not commercially available but can be prepared according to specifications in a pneumatic mill at a cement plant. The 1:3 mixture of C:BFS proved better than 1:1 blended cement in reducing expansion of the Winn Rock containing soil. It is also the cheapest. However, expansion was not completely eliminated by this mixture. The addition of silica fume to the 1:3 C:BFS mixture eliminated expansion completely. The addition of silica fume to the 1:3 C:BFS blend increases the price per ton by 50 percent.

However, a silica fume-containing mixture may still be cheaper than   removal of the sulfate-containing top soil and its replacement with a non-reactive soil.

Table 9

Price per ton of cement mixtures

Cement (percent)	BFS (percent)	silica fume (percent)	Amorphous Silica (percent)	$/ton
50	50			70
25	75			65
50	45	5		100
25	70	5		95
25	72	3		83
25	73	2		76
25	74	1		70
50	37.5		12.5	140

CONCLUSIONS

Winn Rock Containing Soil

Winn Rock was used as gravel to improve the strength of the roads around Winn Parish, Louisiana. Over time, both mechanical and chemical weathering of Winn Rock occurred. The mechanical weathering resulted in the breakdown into smaller anhydrite grains in the finer size fractions of the soil. The chemical weathering resulted in the formation of gypsum, which was found in all size fractions of the soil in significant amounts. In some cases, its amount increased in the finer size fraction. In one soil, more than 90 percent of the sulfates in the -#270 size fraction (50 μm) is gypsum. Apart from anhydrite and gypsum, the other sulfate phase in the soil is ettringite which was derived from the cement from the previous attempt at stabilization.   The clay minerals present in the soil are illite, kaolinite, and a small amount of smectite.

A routine sieve analysis of a suspected sulfate-containing soil is inadequate for the detection of the problem. If the sulfate minerals are present in significant amounts (higher than the solubility limits of the sulfates), X-ray diffractometry and thermal analysis will be necessary to detect and quantify these sulfates. Dissolution of the sulfate minerals in a certain volume of water in a fixed soil to water ratio is also inadequate for this purpose.

Winn Rock Containing Soil Stabilized by Cementitious Materials

A review of literature of sulfate-related problems in soil and concrete suggests that up to 0.1 percent expansion in soil can be tolerated.

In the laboratory, expansion of Winn Rock containing soil observed in the field can be simulated with stabilization by making cementitious mixes have high cement content and curing in a   high moisture and high temperature environment.   Cement:BFS mixtures, in 1:1 to 1:3 ratios, can reduce the expansion significantly but some expansion still does occur. The reduction in expansion is in direct proportion to the amount of BFS. When silica fume or amorphous silica is added to the stabilization mix, no expansion is observed even when cured in a high humidity and high temperature environment.

The most cost-effective mixture is 1:3 C:BFS mixture. Even at 40ºC curing in a water bath , it produced less than 0.1 percent expansion, which is well within the acceptable limit. The addition of silica fume to this mixture will reduce expansion to below detection limit, but will increase the cost of stabilization by 50 percent compared to the blended cement mixtures.

The sulfate necessary for the expansive reactions in the Winn Rock containing soil is mainly provided by gypsum because of its higher solubility and much higher dissolution rate than anhydrite.

Abundant free calcium hydroxide is necessary for the expansive reaction.   The addition of a SCM to the cementitious mix reduces the available calcium hydroxide for the reaction and thus reduces the amount of expansion.

The ettringite crystal size distribution varies widely in the stabilized soil. At early ages, in the presence of lime, a unimodal distribution of relatively smaller size crystals is seen. In the presence of amorphous silica in the stabilization mix, the crystal size is initially smaller, coarser and unimodal, but over time becomes at least bimodal. The number of crystals is also larger when stabilized by lime.

Expansion occurs due to the formation of very small ettringite crystals (gel theory). These evolve in size over the first few days. When a cement component is present in the stabilization mix, the ettringite crystals appear within an hour of mixing and do not vary much in size.

RECOMMENDATIONS

A soil should be analyzed for sulfate minerals if the presence of sulfates is suspected. The amount of sulfate minerals in the soil can be quantified by thermal analysis and quantitative X-ray diffractometry. Thermal analysis will be easier to perform, but the data will be limited only to gypsum. The procedures are outlined in the Analytical Methods section.

Instead of portland cement, blended cement (portland cement and granulated blastfurnace slag) should be used for stabilization of Winn Rock-containing soil.

Laboratory tests showed that cementitious mixes with SCMs are effective in reducing expansion of sulfate containing soils. A test section should be built to test this hypothesis in the field.

The cheaper price of C:BFS blended cement may make its wider use possible without any reference to sulfate attack. Just like portland cement concrete, the use of blended cement can confer improved properties to the stabilized soil.

The effect of variable moisture content on expansion should be studied.   Specimens should be initially stored in a low humidity environment, followed by a high humidity environment.

ACRONYMS, ABBREVIATIONS, & SYMBOLS

ASTM	American Society for Testing Materials
BFS	Granulated Blast Furnace Slag
C-S-H	Calcium Silicate Hydrate. The dashes in between means that there is no definite stoichiometric relationship between the three constituents.
DSC	Differential Scanning Calorimetry
DTG	Derivative Thermogravimetry
EDS	Energy Dispersive X-Ray Spectrometry
TG	Thermogravimetry
FTIR	Fourier Transform Infrared Spectroscopy
LADOTD	Louisiana Department of Transportation and Development
LTRC	Louisiana Transportation Research Center
PDF	International Center for Crystal Diffraction , Powder Diffraction File
QXRD	Quantitative X-Ray Diffractometry
SCM	Supplementary Cementitious Material
SEM	Scanning electron microscopy
UCS	Unconfined Compressive Strength
XANES	X-Ray Absorption Near Edge Structure
XRD	X-ray Diffractometry

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