

Update on Sulfate-Induced Heave in Treated Clays; Problematic Sulfate Levels

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A brief background on sulfate-induced heave in lime- and portland cement-treated clay soils and some examples of projects affected by this phenomenon are presented. The nature and details of the phenomenon are covered as known at this time. A type of swell test that may be used to indicate the nature of problems is introduced, as well as some results from known affected sulfate-bearing soils. The main thrust of discussion is the determination of soluble sulfates and how varied the results of such testing can be depending on the procedure used to extract sulfate from the soil. Recommended procedures for extraction and determination of sulfates are provided. Levels of sulfates that represent potential problems are discussed. The conclusions include the minerals that need to be controlled, recommended determination of sulfate levels, recommended testing for problematic behavior, recommended problematic sulfate levels, and recommended research to achieve solutions to the problem.

Since Mitchell brought attention to the phenomenon of sulfate-induced heave in treated clays in 1986 (1) and Hunter reported on sulfate-induced distress in Stewart Avenue in Las Vegas in 1987 (2), this phenomenon has been realistically considered the cause of distress in many transportation facilities. In the opinion of soil scientists, much of the contiguous United States west of the Mississippi River may have sufficient soluble sulfates close enough to the surface to facilitate sulfate-induced heave. Hunter and others have reported this phenomenon in highly active clay soils containing at least 10 percent clay (1–3) and where sufficient water is available to feed the minerals that expand. This scenario appears logical, especially since lime and portland cement are the stabilizers most often used in these materials. Others believe that clays with as much aluminum as silica (i.e., kaolins) are more likely to exhibit this problematic behavior. It is clear to all that the level of so-called soluble sulfates in the soil is the most important factor. In states like Texas, where most of the state has the potential for moderate to high levels of sulfates near the surface, the concern over this phenomenon has become extreme.

There have been cases of suspected sulfate-induced heave in Texas. The first was noted at U.S. Army Corps of Engineers–Texas State Parks project roads around Joe Pool Lake in Dallas County. The second was at a Laughlin Air Force Base auxiliary field near Del Rio. The third project was Texas FM 2499 near Flower Mound and Grapevine. The fourth was in southeast Arlington where distress was noted on a four-lane divided city loop street. There have been verbal reports

of many instances that are likely to be the result of sulfate-induced heave of lime-treated clay subgrades. As investigations of this phenomenon continue, the reports of it increase. This has led to an increasing need to find realistic procedures for the prediction of potential problems and development of methods to deal with the problems associated with stabilizing the soils involved.

Offered here is an update to the existing published information concerning identification of the potential sulfate-induced heave and approximation of the amount of heave to expect. Recommendations for testing and research into ways to prevent sulfate-induced heave will be explored as well.

BACKGROUND

Although there has been considerable study of the formation of ettringite in portland cement pastes (3–6), it was not recognized as a possible problem during lime or portland cement stabilization until Sherwood reported it in 1962 (6). The problem did not receive national recognition until 1986 (1). Hunter provided many details of the Las Vegas case and the overall sulfate heave phenomenon in his journal article of 1987 (2). Thomas et al. reported on pyrite-derived sulfate-induced heave in portland cement-stabilized minestone in 1989 (7).

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. However, the present working hypotheses are discussed in the following paragraphs.

The most often found expandable mineral resulting from these reactions is ettringite, $\text{Ca}_6\{\text{Al}(\text{OH})_6\}_2(\text{SO}_4)_3 \cdot 26\text{H}_2\text{O}$, since it is the first to form and can be transformed into thaumasite, $\text{Ca}_6\{\text{Si}(\text{OH})_6\}_2(\text{SO}_4)(\text{CO}_3)_2 \cdot 26\text{H}_2\text{O}$. This transformation happens when there is a sufficient source of carbonate and dissolved silica in the system and the temperature is between 59°F and 40°F (4). 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.

The stable forms of calcium-sulfoaluminate-hydrates in aqueous solutions are monosulfate hydrate and trisulfate hydrate (ettringite) (8). Consequently, these are the forms usually sought in the evaluation of sulfate reactions.

When lime is added in sufficient quantities to clay, the pH of the soil-lime slurry is raised to 12.45 (at 77°F). Once the pH exceeds 10.5, dissolution of the clay surfaces occurs, and siliceous and aluminous pozzolans are released. These poz-

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zolans may react with calcium ions from the lime to form the cementitious products of calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH). These reaction products bind the soil particles together and may result in high compressive strengths and soils of high stability. However, if sulfates, either in solid or groundwater form, are present in sufficient quantity, they may confound the reaction between the calcium and the pozzolans to form significant quantities of ettringite and, when conditions are right, thaumasite. Also, the high pH environment, which is optimal for the formation of CSH, drops substantially upon the formation of ettringite. A model is provided by Hunter (2) that describes the basic reaction kinetics.

Mehta and Klein (4) determined that the formation of monosulfate hydrates is favored in high alumina environments, but the formation of ettringite is favored in low alumina environments. This might indicate that monosulfate hydrates are favored in 1:1 type clay minerals like kaolinite, while the formation of trisulfate hydrates would be favored in 2:1 type clay minerals such as smectite. Or it may indicate that the form of calcium-sulfoaluminate-hydrate may be transient between the monosulfate form and the trisulfate form. This unstable nature is precarious as transformation from the monosulfate form to the trisulfate (ettringite) form entails a 241 percent expansion. Researchers further explain that monosulfate hydrate is only stable in moist conditions, whereas ettringite is stable in both wet and dry conditions. They also describe trisulfate hydrate as substantially expansive upon wetting, while monosulfate hydrate is not. They explain that during formation CSH crystals will grow to fill the void space only (which is also true of calcium hydroxide crystal), while ettringite crystals will continue to grow so long as the reaction materials are present. They found that, if restrained against growth, these crystals can expand with pressures up to 35,000 psi. Bogue et al. (9) found that once formed, ettringite expands to a volume equal to 227 percent of the total volume of reactant solids. In addition, Hanson and Offutt (3) determined that the ettringite formed is eight times the volume of calcium aluminate in portland cement.

In spite of its expansive behavior, ettringite is a necessary part of the process of hydration in portland cement. Schwiete and Niel (6) found that crystals of this mineral form after only 30 sec of hydration. Eventually, trisulfate hydrates become monosulfate hydrates as the cement hardens and sulfates are used up in the reaction process. It is the addition of sulfates from outside sources in an aqueous environment that causes the reformation of ettringite from monosulfoaluminate and triggers large volume increases and destroys concrete. The formation of trisulfate hydrates in cement-stabilized sulfate-bearing materials leads to the types of heaves reported by Thomas et al. (7).

The presence of ettringite in materials may be confirmed by one or more of three methods. These methods include X-ray diffraction (XRD), differential thermal analysis (DTA), and scanning electron microscopy (SEM) with or without dispersive X-ray (DXR) for determination of elements. Trisulfate XRD peaks were noted by Mehta and Klein (4) at 9.71, 5.61, and 3.88 Å. They found that there is a decrease in intensity on drying, although the locations of the peaks were unchanged. Another reason given for the lack of intensity by Schwiete and Niel (6) was the small size of the crystals. When

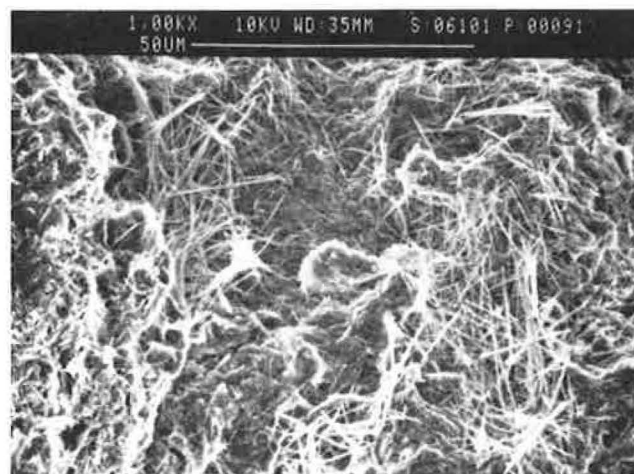


FIGURE 1 Typical SEM view of ettringite.

high percentages of ettringite were present, Mehta and Klein (4) found strong DTA endotherms at about 190°C (374°F) and weak endotherms at about 270°C (518°F), while when smaller percentages of ettringite were present these occurred at 150°C and 250°C, respectively. During SEM inspection of affected materials many have found that the crystals are well-grown, fibrous, or needlelike (4,6,7). Thomas et al. (7) identified needle-shaped crystals of ettringite by DXR during SEM, as has one of the authors. An example of a typical SEM view is shown in Figure 1. Mehta and Klein (4) noted that ettringite crystals formed by hydration of amorphous crystallites are more gel-like, while those formed by precipitates are more fibrous or needlelike. Schwiete and Niel (6) found that under prolonged irradiation, such as used in SEM, changes may occur, shifting the crystalline form to a more spherical nature. If this effect is combined with the fact that SEM environments are dry, it is easy to understand why a large variety of sizes and shapes of crystals may be seen. Of the identification modes discussed it is likely that XRD is the most reliable.

SWELL TESTING

Swell testing has been utilized by researchers to predict the nature of the volume increase caused by hydration of ettringite (10,11). This method of predicting behavior, if performed correctly, should reflect the physical and chemical influences of field conditions. The results will reflect actual behavior. In Las Vegas, Hunter (2) found heave where the levels of sulfates were as low as 700 ppm and no heave where the sulfate levels were as high as 20,500 ppm. The authors are acquainted with areas in Texas where high levels of sulfates occur in soils that have been very successfully stabilized with lime. It is, therefore, deemed necessary to perform swell behavioral tests as indicators of swell potential.

As part of an overall program to investigate the nature of the sulfate-induced failure at Lakeview Park beside Joe Pool Lake, which was funded by the U.S. Army Engineers Waterways Experiment Station, 138 specimens were taken for testing out of 56 borings at depths directly under the pavement to 3 ft below the pavement. Approximately one-half of the



FIGURE 2 Shear plane and overriding or subbase at Joe Pool Lake Park (courtesy of Corps of Engineers, Fort Worth).

specimens were taken from within the humps along the roadways. The others were from adjacent areas where no hump occurred. All specimens were nearly the same in water content and dry unit weight, with those in the humps slightly wetter because of more direct exposure. In addition, these materials exhibited lime-modified behavior and were fairly friable. Little evidence of pozzolan cementation was found. On the other hand, evidence of shear planes was found, and the material in the humps appeared to have been shoved one part over another, or buckled. Examples of these are shown in Figures 2 and 3. It is clearly evident, therefore, that the humps did not occur from concentrated vertical heave, but from horizontal heave, which caused layer overriding and buckling.

Because of the field behavior at the roads beside Joe Pool Lake and a need to simulate the heave noted there in a laboratory setting, it was decided to use a three-dimensional swell test. This test utilized a 6-in.-diameter, 4½-in.-high specimen consisting of soil at levels of pulverization simulating field conditions, treated with lime, and compacted using standard Proctor energy levels. The specimen was then placed over a porous stone and wrapped in a 4½-in.-high filter fabric. This assemblage was then placed inside a triaxial membrane and sealed, except for the porous stone and filter fabric, which



FIGURE 3 Buckling of subbase at Joe Pool Lake Park (courtesy of Corps of Engineers, Fort Worth).

were allowed to absorb water as the assemblage was placed into a bowl. Long-term swell tests were conducted as the height and circumference of the specimens were monitored daily. The swell tests were continued for periods exceeding 45 days. The results varied as the amounts of lime and test temperature conditions differed. However, simulations of actual field behavior were considered very successful. The minimum swell occurred in a specimen that had been treated with 6 percent hydrated lime, which exhibited 10.3 percent vertical rise and 6.3 percent horizontal diameter increase. The maximum swell was noted for a specimen stabilized with 9 percent lime, which exhibited 34.1 percent vertical rise and 12.5 percent horizontal heave. The averages for 30 specimens, all using soils with high levels of sulfates, were 19.6 percent vertical and 9.1 percent horizontal heave. When these specimens were inspected and sampled for testing, they appeared to be nearly identical to field samples of the stabilized layer. The success of this testing procedure has led the authors to continue its use on current projects where the phenomenon is expected or present.

SOLUBLE SULFATES

As stated by Ferris et al. (11), the sulfate content is the most important single property indicating the extent to which ettringite will form. The greater the soluble sulfates, the greater the potential for its growth. In addition, Mitchell and Dermatas (10) explain that it does not depend on the form of sulfate present. The sources can include oxidation of pyrite as described by Thomas et al. (7). It is interesting to note that significant pyrite levels were found in the soils adjacent to Joe Pool Lake. The levels of so-called soluble sulfates discussed as potentially problematic differ from one publication to another. Hunter (2) reported a level of 10 percent or 10,000 ppm as a cutoff, while Thomas et al. (7) found as little as 0.4 percent or 4,000 ppm to be a problem. Mitchell and Dermatas (10) determined ettringite formed in the presence of very small sulfate concentrations of 0.3 percent or 3,000 ppm. A recent British publication by Snedker and Temporal (12) concerning a highway distressed by this phenomenon describes sulfate levels as low as 0.37 percent or 3,700 ppm. The experience of the authors indicates that fairly low percents of sulfates can lead to problems.

If one studies the reports of so-called soluble sulfates, it becomes apparent that the various authors are not all describing the same type of sulfates or are using differing ways to extract sulfates from the soils they are testing. Hunter (2) reported that he used a 1 part soil to 50 parts water suspension to extract sulfates, while Mitchell and Dermatas (10) added the percent sulfates to soils without sulfates. The methods utilized by other authors to extract sulfates are unclear, since they are not mentioned in publications. As part of the project related to soils adjacent to Joe Pool Lake, the decision was made to use the standardized method to extract sulfates. However, no standardized method was found. In fact, several methods were found. These include utilizing hydrochloric acid at a pH of about 1.0 to digest sulfates, use of a buffered pH solution between 3 and 4 to extract sulfates, extraction of sulfates by centrifuging using Morgan's solution with a pH of 4.99, and extraction of sulfates using differing ratios of soil

TABLE 1 Soluble Sulfates by Differing Test Methods

Test Method	Mean Value (ppm)	Standard Deviation
pH=1.0	62,147	9,717
pH 3 to 4	26,207	1,515
pH=4.99	18,153	607
1:1 S/W	2,176	855
1:1 S/LW	640	235

to pure water. The ratios that were found in the literature were soil to water ratios: 1 to 1, 1 to 5, 1 to 10, and 1 to 50. It was decided to explore how the reported level of soluble sulfates could differ utilizing these varying methods, including extraction using one part soil to one part lime water. The results of six replications of testing on soils from two stations along the roads at Joe Pool Lake are presented in Table 1.

Subsequent testing to determine the effects of the soil:water ratio has been performed on soils from the project mentioned earlier in Arlington and soils sampled under the roads adjacent to Joe Pool Lake. These materials were sampled from the same geologic formation, the Eagle Ford, and come from locations within 3 mi of each other. Testing at soil/water ratios of 1:50 was not done, because it is believed that this represents an unrealistic situation compared to that of the field. The results are presented in Table 2, along with the number of samples tested.

Two facts are readily apparent from the information provided in Tables 1 and 2. First, it makes a great deal of difference how so-called soluble sulfates are extracted. Second, and equally important, the criterion for potentially problematic behavior, 10,000 ppm and 1 percent, must depend on the method of extraction. The same may be said for the criterion of 0.3 percent or 3,000 ppm. The value one must use is highly dependent on the method of extraction. This problem is further made difficult by the fact that many geotechnical engineering firms, which order sulfate testing from various testing companies, do not know how the sulfates are extracted, but use the previously published potentially problematic levels as standards. This part of the overall problem with sulfates in soils is receiving much attention by researchers in an effort to develop practical standards.

The authors are currently engaged in studies that include the use of soil/water extraction ratios of from 1:1 to 1:10. These ratios are believed to best represent the field situation yet provide adequate measures of sulfates. They also are believed to allow for sufficiently large soil sample sizes to accurately represent the soil. Because of the known variability of chemical properties of soils and the relative size of samples, even at these ratios, it is believed that as many samples as possible should be tested, and statistical analyses should be used. Potentially problematic behavior such as sulfate levels

of as little as 500 ppm (0.05 percent) for the 1:1 ratio to as little as 2,000 ppm (0.2 percent) for a 1:10 ratio are currently being investigated. It is evident that more data are required to provide assurance that these numbers are reliable.

SUMMARY

Heave induced by the presence of soluble sulfates in soils is caused by the interaction of these sulfates with lime-treated clays or portland cement-treated soils. The expandable minerals formed include ettringite and, when the conditions are correct, thaumasite. These minerals are known to expand significantly when they hydrate, and their expansion cannot practically be halted by the long-term live and dead loads associated with transportation facilities. The presence of water is, therefore, a necessary part of the phenomenon. It is not practical, in most cases, to stabilize the material with other, nonreactive chemical agents. Therefore, the phenomenon associated with these problematic minerals must be determined to the extent that it can be abated and suitable stabilization methodologies can be used.

A knowledge of the extent of soluble sulfates in subgrades is essential to ensure construction of a stable material. There are levels of sulfates that pose no potential problem, but the amount that may result in heave is generally less than previously reported and is highly dependent on the methodology used for extraction of sulfates from the soil. Significantly more testing is needed to provide an adequate data base upon which to set practical standards of potential problematic sulfate levels.

Swell testing is believed necessary to determine the extent to which the potential problems indicated by the levels of soluble sulfates actually represent field behavior. The test must allow for the measurement of horizontal as well as vertical volume increases, since the humping distress noted in field behavior is actually caused by horizontal swell. In addition, this test should utilize equipment normally available in testing laboratories and simple testing and measurement techniques. One such test is described in this report. The required length of this test is believed to be at least 30 days, with 45 days preferable. In the field, heave distress has oc-

TABLE 2 Soluble Sulfates by Differing Extraction Ratios

Test Method	Number	Mean Value (ppm)	Standard Deviation
pH=1 J.P.L.	56	41,481	14,835
pH=1 Arl.	12	34,594	9,215
1:1 S/W Arl.	12	1,011	35
1:5 S/W J.P.L.	56	6,340	1,367
1:10 S/W Arl.	12	13,632	375

curred in as short a time as 1 month but has happened much more slowly in most cases.

Several projects are under way to determine how these problematic soils can be stabilized with lime or portland cement and other additives. The greatest concentration is on the use of lime in highly active clays, since it is the major stabilizing agent used for these materials. Ideas currently being studied include the use of barium compounds to pretreat soils, first reported by Little and Deuel (13) and later used by Ferris et al. (11); double applications of lime, which have often been reported to the authors as being successful in overcoming the sulfate heave problem by personnel of the Texas Department of Highways and Public Transportation; the use of compounds to aid in sustaining pH's to promote pozzolans; and the addition of materials to promote pozzolans. Studies now in progress have resulted in some very promising possible remedies for sulfate-induced heave.

CONCLUSIONS

Based on the information provided in this report, past reports of many authors, and the current experience levels of the authors, the following conclusions are drawn:

1. The formation of ettringite is necessary for this phenomenon to take place, and control of this formation is a key to stabilization.
2. Heave caused by hydration and growth of ettringite cannot be controlled by loading but by the abatement of its formation.
3. Heave caused by this phenomenon cannot occur unless sufficient quantities of water are available.
4. The presence of soluble sulfates in sufficient quantities in either the soil or the water entering the soil is necessary for ettringite formation.
5. Soluble sulfates should be determined using extractions from mixtures of 1 part soil solids to 1 part pure water up to 10 parts pure water.
6. The levels of sulfates that represent potential problematic situations may be as low as 500 ppm for 1:1 S/W mixtures to 2,000 ppm for 1:10 S/W mixtures.
7. Three-dimensional swell tests must be performed using relatively large specimens, field gradations, and normal levels of compactive effort to enable prediction of field behavior.
8. Swell tests should be of extended length for as long as 30 days and possibly 45 days to accurately predict field behavior.
9. Stabilization of sulfate-bearing clay soils with lime is possible, yet more laboratory and field testing is required to be certain of the agents and methods needed.

10. Sulfate-bearing soils have been successfully lime stabilized in Texas for over 30 years. It is of paramount importance to identify construction and application techniques responsible for successful stabilization.

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REFERENCES

1. J. K. Mitchell. Practical Problems from Surprising Soil Behavior. *Journal of the Geotechnical Engineering Division, ASCE*, Vol. 112, No. 3, 1986, pp. 259-289.
2. D. Hunter. Lime-Induced Heave in Sulfate-Bearing Clay Soils. *Journal of the Geotechnical Engineering Division, ASCE*, Vol. 114, No. 2, 1988, pp. 150-167.
3. W. C. Hanson and J. S. Offutt. Gypsum Anhydrite in Portland Cement. *U.S. Gypsum*, 1962, p. 26.
4. P. K. Mehta and A. Klein. Investigations on the Hydration Products in the System $4\text{CaO} \cdot 3\text{Al}_2\text{O}_3 \cdot \text{SO}_3 - \text{CaSO}_4 - \text{CaO} - \text{H}_2\text{O}$. In *Special Report 90*, HRB, National Research Council, Washington, D.C., 1966, pp. 328-352.
5. P. K. Mehta and S. Wong. Expansion of Ettringite by Water Adsorption. *Cement and Concrete Research*, Vol. 12, 1982, pp. 121-122.
6. H. E. Schwiete and E. M. G. Niel. Formation of Ettringite Immediately After Gaging of Portland Cement. *Journal of the American Ceramic Society*, Vol. 48, No. 1, 1965, pp. 12-14.
7. M. D. A. Thomas, R. J. Kettle, and J. A. Morton. Expansion of Cement-Stabilized Minestone due to the Oxidation of Pyrite. In *Transportation Research Record 1219*, TRB, National Research Council, Washington, D.C., 1989, pp. 113-120.
8. W. Lerch, F. W. Ashton, and R. H. Bogue. Sulfoaluminates of Calcium. *Journal of the National Bureau of Standards*, Vol. 2, 1929, pp. 715-731.
9. R. H. Bogue, W. Lerch, and W. C. Taylor. *Industrial Engineering Chemistry*, Vol. 26, 1934, p. 1049.
10. J. K. Mitchell and D. Dermatas. Clay Soil Heave Caused by Lime-Sulfate Reactions. American Society of Testing Materials in Innovations and Users of Lime. San Francisco, Calif., June 1990.
11. G. A. Ferris, J. L. Eades, G. H. McClellan, and R. E. Graves. Improved Characteristics in Sulfate Soils Treated with Barium Compounds Before Lime Stabilization. In *Transportation Research Record 1295*, TRB, National Research Council, Washington, D.C., 1991.
12. E. A. Snedker and J. M. Temporal. 40 Motorway Banbury IV Contract-Lime Stabilization. *Highways and Transportation*, Dec. 1990, pp. 7-8.
13. D. N. Little and L. Deuel. *Evaluation of Sulfate-Induced Heave at Joe Pool Lake*. Chemical Lime Company, June 1989.

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