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Slurry Stabilization and Reaction Chemistry Of Cement-Treated Soils

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CHAPTER 1

A REVIEW OF CEMENT SLURRY APPLICATION EQUIPMENT

SUMMARY

Although cement slurry application methods exist in many specification books, equipment in industry suited for application of cement by slurry is not widely available. TTI researchers surveyed what equipment in industry exists to deliver cement slurry and discovered a range of methods widely ranging in both cost and complexity. At the most basic level, concrete trucks can mix and even spread cement slurry, although uniform spreading with a concrete truck will be somewhat difficult. At the other extreme, microprocessor-controlled, on-site mixing plants exist that meter and deliver the cement slurry based on the desired application rate and the rate of travel of the stabilization train. This chapter summarizes known methods of applying cement by slurry. The methods presented below likely do not represent all techniques that exist; cement slurry application equipment in general seems to be regional and at times, even custom-built for specific project requirements.

TXI MOBILE BATCH PLANT

TXI recently completed work on its cement slurry product called Super Slurry. Approximately 60 percent cement solids by weight, admixtures including a suspension aid and a retardant reportedly allow this slurry to be transported, without continual mixing, for up to 8 hours. TXI recently designed and procured a new mobile production plant, shown in Figure 1.1, to produce the product. Load cells under the mixing tanks help ensure accurate proportioning of the slurry ingredients.



Figure 1.1. TXI's New Super Slurry Mobile Plant.

Conventional haul trucks as shown in Figure 1.2 transport and place the slurry. Standard field stabilizers incorporate the slurry into the pavement material. Figure 1.3 shows slurry placement and mixing taking place on a construction project.



Figure 1.2. Haul Trailer for Transporting and Spreading Super Slurry.



Figure 1.3. Placement and Mixing Super Slurry.
Note: photos courtesy of TXI.

Thus far the primary demand for Super Slurry has been with municipalities, county governments, and private entities needing environmentally friendly (dust-free) stabilization processes. However, this slurry product does conform to typical department of transportation (DOT) criteria. To date, no problems with equipment cleanout have been reported as the admixtures maintain slurry workability for extended lengths of time. A slate of projects currently awaits treatment with Super Slurry, and TXI plans to work with Tarrant County

and the City of Dallas to investigate field application methods utilizing the injection system on the field mixers.

OLMOS CONSTRUCTION RETROFITTED WATER TRUCK

Olmos Construction of San Antonio, Texas, also performs cement slurry work. Olmos uses a retrofitted water truck shown in Figure 1.4. The truck holds approximately seven tons of cement and is mixed on-site prior to spreading. A high-pressure butterfly valve system agitates the slurry, and Olmos reports the slurry is good for 30 minutes to 1 hour with the pumps operating. Figure 1.5 shows application of the slurry. A traditional field stabilizer blends the slurry with the pavement material.



Figure 1.4. Filling the Olmos Construction Slurry Truck with Dry Cement.
Note: photo courtesy of Olmos Construction.



Figure 1.5. Spreading Slurry with the Olmos Construction Slurry Truck.

Note: Photo courtesy of Olmos Construction.

ANGEL BROTHERS ENTERPRISES SPREADER BOX

Angel Brothers Enterprises of Baytown, Texas, recently performed cement stabilization on a full-depth-recycling (FDR) project requiring the use of cement slurry. To accomplish this task, arrangements were made to produce the cement slurry at a nearby concrete batch plant and then spread the slurry with a custom-made spreader box. A conventional reclaimer/stabilizer then mixed the slurry into the previously pulverized section. Figures 1.6 and 1.7 show the slurry loading and spreading operation employed by Angel Brothers. This method of slurry application was custom-created to meet this project's requirement for the application of cement by slurry.



Figure 1.6. Loading Slurry from Cement Truck into Spreader Box.



Figure 1.7. Spreading Slurry with the Angel Brothers Spreader Box.

WIRTGEN MOBILE SLURRY PLANT

Wirtgen manufactures perhaps the most advanced cement slurry application equipment. The WM 1000 suspension mixing plant holds 883 ft³ of cement and 2,906 gal of water, and produces slurry on-site while the stabilization train progresses. On-board controllers adjust the quantity of cement in accordance to the travel speed of the recycling train, and the cement slurry gets pumped and injected by a downstream stabilizer. Figure 1.8 shows this operation in progress. No units of this type are known to be in the U.S.; however, Cemex reportedly has units in operation in Mexico.



Figure 1.8. Wirtgen Mobile Slurry Mixing Plant.
Note: photo courtesy of the Wirtgen Group.

TEXAS SLURRY MOBILE BATCH PLANT

Texas Slurry, LLC, produces cement slurry on-site with a custom mobile batch plant. Figure 1.9 shows the plant, which injects the cement into the mixing chamber below the waterline after the chamber is filled with water. Load cells under the mixing chamber enable proportioning the water:slurry ratio, and Texas Slurry has experience with slurry percent solids ranging from 30 to 62 percent. Agitators maintain suspension of the cement until the slurry is pumped to water-trucks equipped with spreader bars for distributing the slurry. Figure 1.10 shows the slurry being pumped into a spreader truck, and Figure 1.11 shows the spreader bar arrangement on the spreader truck.



Figure 1.9. Texas Slurry Batch Plant.



Figure 1.10. Texas Slurry Batch Plant Offloading Slurry into Spreader Truck.



Figure 1.11. Texas Slurry Spreader Truck.

CHAPTER 2

TESTING PLAN TO EVALUATE CEMENT SLURRY APPLICATIONS FOR SOILS

SUMMARY

Clearly, methods exist to practically apply cement slurry during construction and reduce dusting. However, this project also investigated to determine if slurry applications produce the same stabilization result as a dry application. In particular, the following topics were posed:

- Does a slurry application produce the same strength result as a dry application?
- Does the percentage solids in the slurry impact the performance?
- How does the age of the slurry impact performance?
- How does the time delay between mixing the slurry into the soil and completion of compaction influence results?

TESTING PLAN

In order to evaluate these topics, the testing plan outlined in Table 2.1 was carried out on a low plasticity index (PI) soil ($PI = 14$) and a moderately plastic soil ($30 < PI < 35$). This factorial results in testing potential construction sequences with cement slurry that could range in duration from as little as 30 minutes to as long as 4 hours from the time of slurry mixing to final compaction. For all cases with a delay between slurry mixing and application of the soil, the slurry was agitated continuously during the delay time. For reference, researchers also performed the tests on specimens treated with dry cement powder with time delays of 30 minutes, 2 hours, 2.5 hours, and 4 hours between mixing the cement into the soil and soil compaction. Researchers performed each test in triplicate.

Table 2.1. Testing Factorial for Slurry Investigation.

Slurry Ratios	Slurry age from time of slurry mixing to incorporation of slurry with soil	Delay time from incorporation of slurry with soil to soil compaction	Test Performed
30% Solids	0 minutes	30 minutes	7-day Unconfined Compressive Strength (UCS)
50% Solids	2 hours	2 hours	7-day Seismic Modulus
70% Solids			

CHAPTER 3

PERFORMANCE INVESTIGATION OF CEMENT SLURRY APPLICATIONS ON A LOW-PI SOIL

SUMMARY

Using the test plan outlined in Chapter 2, TTI researchers first tested cement slurry application on an AASHTO A-6 soil with a PI of 14. The results support the following:

- Treatment of the soil by slurry produced results that at least met and often exceeded results obtained by dry powder application of cement. In general, treatment by slurry better stabilized the soil as evidenced by less sensitivity to delay time (the time between mixing the cement into the soil and compaction) and increased modulus values.
- Design tests should consider delay time, particularly if the field will employ a dry powder application of cement, to avoid under-design. A delay time of 2 hours should adequately safeguard the design process.
- Slurry age (the time between production of the slurry and its incorporation into the soil) did not impact mechanistic properties of the soil with slurry ages up to 2 hours.
- Slurry percentage solids can slightly impact results, with high percent solids (70 percent) producing slightly lower modulus values and in some cases slightly lower strengths.

BACKGROUND SOIL INFORMATION

TTI researchers first tested a low-PI material (liquid limit = 26, plastic limit = 12; plastic index = 14). This soil had 51 percent passing the #200 sieve, resulting in an AASHTO classification of A-6. Table 3.1 shows the particle size distribution.

Table 3.1. Particle Size Distribution for Low-PI Soil.

Sieve Size	Percent Passing
3/8	100.0
# 4	99.8
# 10	99.6
# 40	99.2
# 100	90.2
# 200	51.2

Using ASTM D 558, the researchers developed the moisture-density curve shown in Figure 3.1, indicating an optimum moisture content of 14.2 percent with a maximum dry density of 114.0 pounds per cubic foot (pcf). For developing the moisture-density curve cement was applied to the soil as a dry powder.

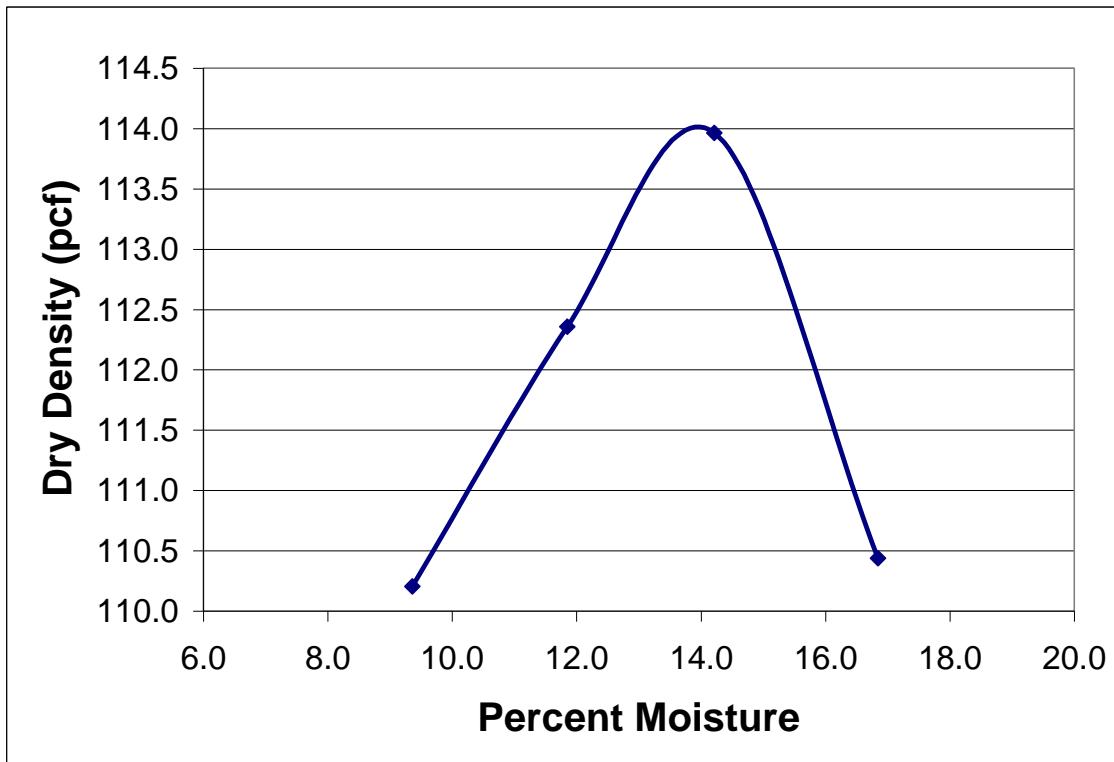


Figure 3.1. ASTM D 558 Result for Low-PI Soil with 6 Percent Cement.

SELECTION OF DESIGN CEMENT CONTENT

Following determination of the moisture-density curve, the 7-day unconfined compressive strengths of this soil were determined for cement treatment levels of 5, 6, 7, and 8 percent at a loading rate of 0.05 inches per minute. Cement was applied as a dry powder for these tests. The goal was to select a treatment level that would produce a 200 psi 7-day strength. Cylindrical, 4-inch diameter by 4.6-inch tall specimens were compacted then cured for 7 days. Immediately prior to testing, each specimen was soaked by complete submersion in water for 4 hours then capped. Each treatment level was tested in duplicate, and Figure 3.2 shows the average result for each level of treatment. The data show that, although 5 percent cement was sufficient to meet the target strength on average, that treatment level only slightly exceeded the 200 psi target. Therefore, given inherent variability in construction, researchers selected a design treatment level of 6 percent cement to better ensure the mixture consistently met the strength target.

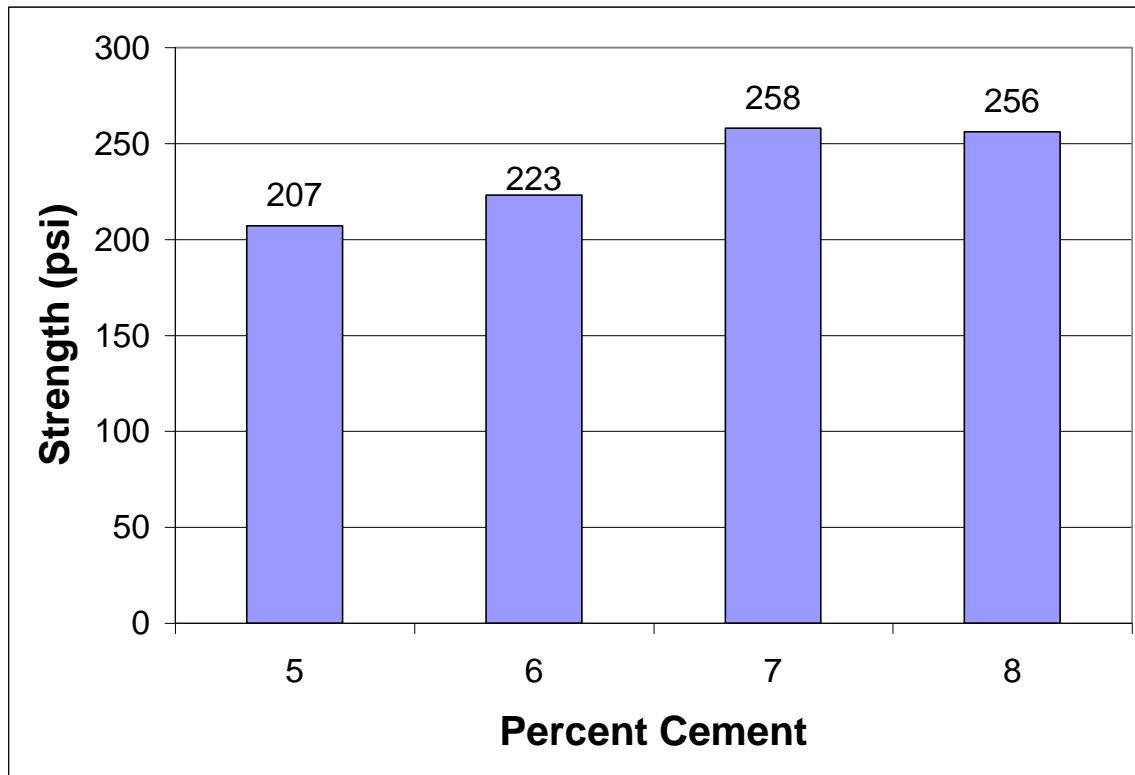


Figure 3.2. Strength Results from Low-PI Soil Cement Design.

STRENGTH TESTING RESULTS

Figure 3.3 presents the strength results from the reference samples treated with 6 percent cement applied as a dry powder. These results show a 34 percent decrease in strength between the delay times of 0.5 hours and 2 hours. From 2 to 4 hours delay time, the average strength did not change. Interestingly, with delay times of 2 or more hours between mixing the cement into the soil and compaction, on average the mixture did not meet the 200 psi strength target. These findings show the time anticipated in the field between mixing the cement into the soil and compaction should be considered when performing cement content design tests. Based upon these results, using a 2-hour delay time should adequately safeguard against under-design (since the average strength did not change from delay times of 2 to 4 hours).

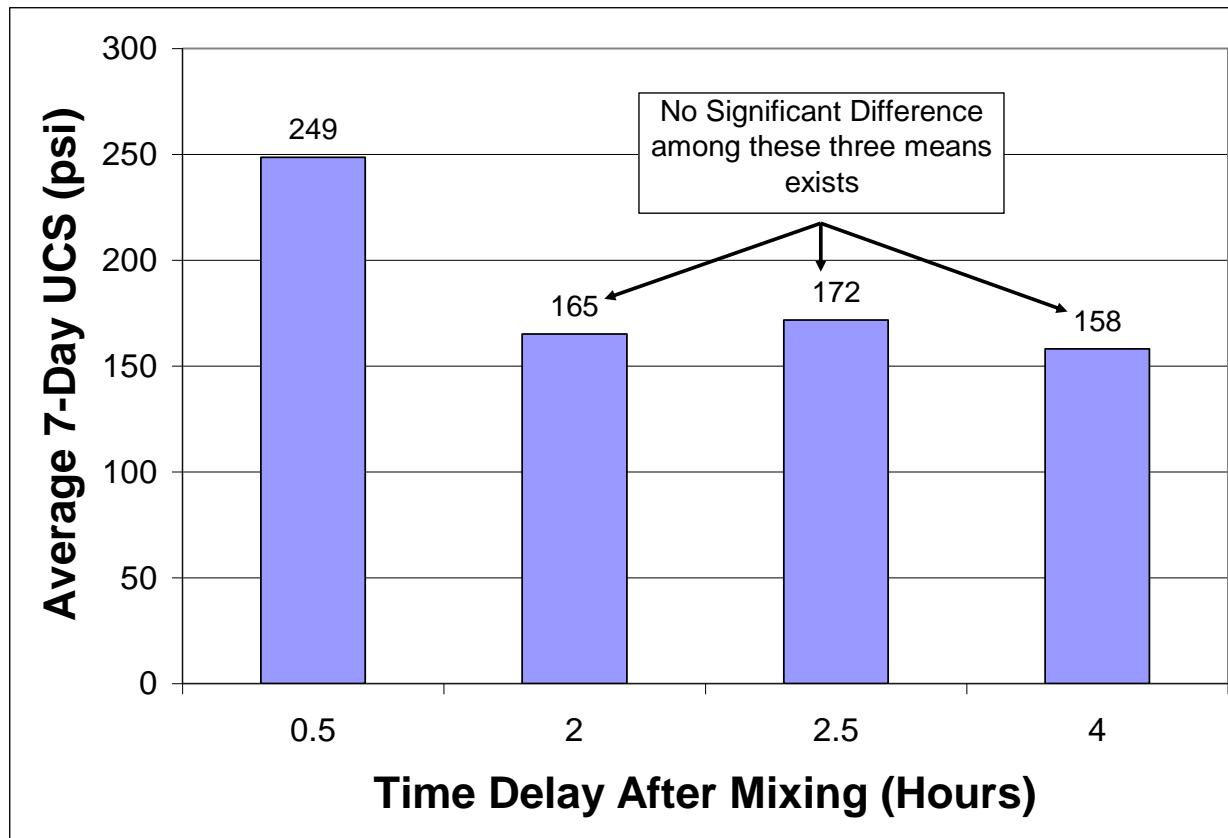


Figure 3.3. Low-PI Strengths with Cement Applied as a Dry Powder.

Table 3.2 presents the strength results from the low-PI soil treated with cement slurry, and Figure 3.4 summarizes these data from the slurry treatment. For reference, Figure 3.4 also notes the strengths obtained from the dry application with delay times of 0.5 and 2.0 hours. Visual inspection of the data reveals several noteworthy observations:

- For a given delay time between mixing the cement into the soil and compaction, application of cement by slurry resulted in strengths that met or exceeded the strengths obtained when cement was applied as a dry powder. This is evident in Figure 3.4 since slurry treatments A and C (both with a 0.5 hour delay time) always met or exceeded 249 psi (obtained with a dry application of cement and a 0.5 hour delay time), and slurry treatments B and D (both with a 2.0 hour delay time) always met or exceeded 165 psi (obtained with a dry application of cement and a 2.0 hour delay time).
- With one exception, all treatments with cement slurry met the 200 psi criteria on average.
- Increasing percent solids appears to result in decreased strength with the 0.5 hour delay time, as evidenced by the apparent negative slope in the plots of strengths from treatments A and C.
- Increasing percent solids does not appear to impact strength with the 2.0 hour delay time, as evidenced by the essentially horizontal plots of strength for treatments B and D.
- The longer delay time always produced a lower strength. This is evident in Figure 3.4 when comparing slurry treatment A to B (both with a slurry age of 0 hours but with delay

times of 0.5 and 2.0 hours, respectively), and when comparing treatment C to D (both with a slurry age of 2.0 hours but with delay times of 0.5 and 2.0 hours, respectively). The difference in strengths does appear to narrow as slurry percent solids increases.

- Slurry age did not appear to impact strength. For example, when comparing treatment A to C (both with a delay time of 0.5 hours but with slurry ages of 0 and 2 hours, respectively), the strengths produced from the two treatments are very near each other. Similarly, when comparing treatment B to D (both with delay time of 2.0 hours but with slurry ages of 0 and 2 hours, respectively), the average strengths produced again appear nearly identical.

Table 3.2. UCS (psi) Results for Low-PI Soil.

Treatment	Slurry Age (hr)	Delay Time (hr)	Slurry Percent Solids		
			30	50	70
A	0	0.5	378	367	316
			339	321	269
			315	343	257
B	0	2	219	187	234
			206	163	192
			211	163	232
C	2	0.5	371	183*	266
			322	316	265
			294	292	233
D	2	2	193	237	204
			210	219	209
			199	199	205

*data point omitted from further analysis due to suspect test result.

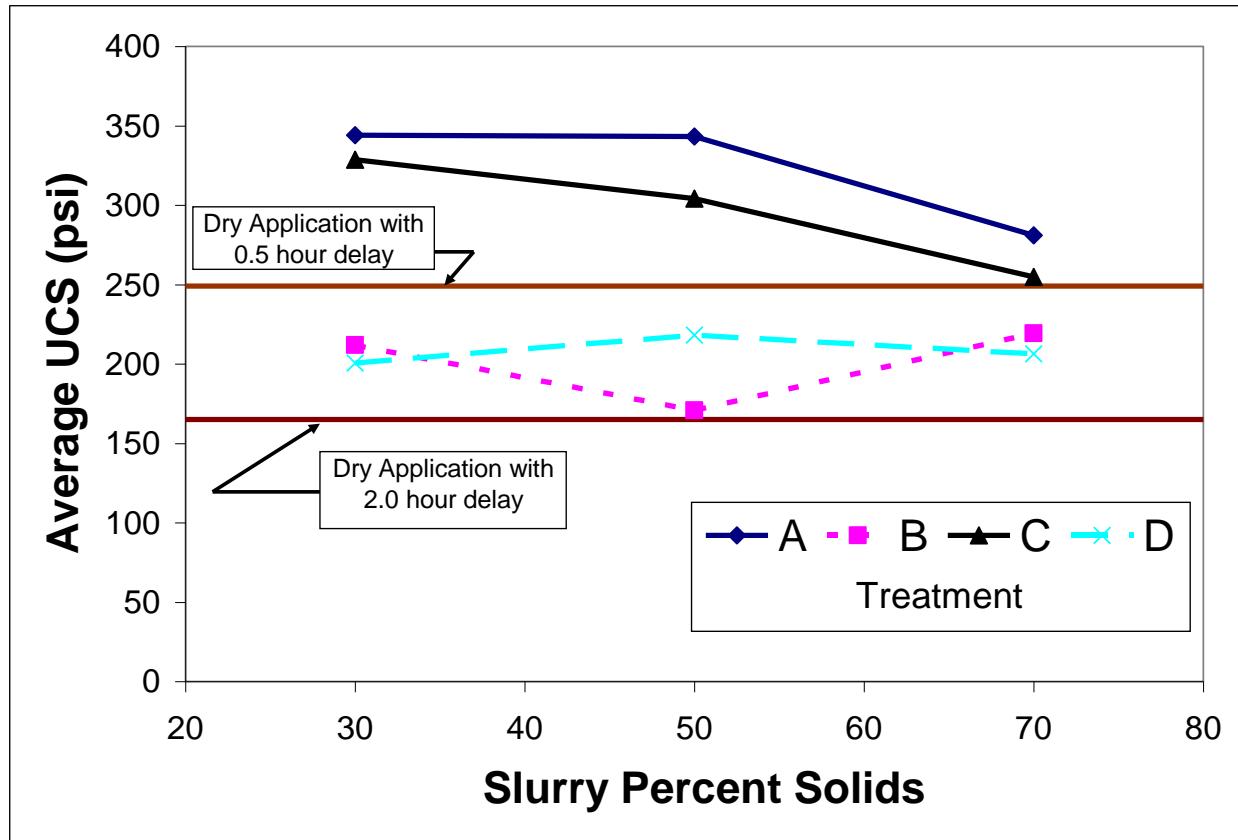


Figure 3.4. Summary of Strength Results for Low-PI Soil.

Although visual examination of the data shows several interesting points, these observations need to be supported by quantitative analysis. For a more thorough analysis, three-way analysis of variance (ANOVA) techniques provide a method to statistically and quantitatively investigate the factor effects of delay time, slurry percent solids, and slurry age. Table 3.3 presents the treatment average values $\bar{Y}_{ijk\bullet}$ where i , j , and k represent the treatment level for slurry age, percent solids, and delay time, respectively. Table 3.3 also presents the apparent main effects for each of the three factors. Some information gleaned from these data includes:

- The slurry age effect appears insignificant.
- Strength appears to decrease as slurry percent solids increases.
- Delay time appears to substantially impact the strength, with short delay times resulting in substantial increases in strength, and long delays resulting in substantial decreases in strength.

Table 3.3. 3-Factor Summary Results for Low-PI Soil Strengths.

Slurry Age (hours)	Slurry Percent Solids						AVG i... Main age effect	
	30		50		70			
	Delay Time (hr)	0.5	Delay Time (hr)	0.5	Delay Time (hr)	0.5		
0	344	212	343	171	281	219	262 5	
2	329	201	304	218	255	206	252 -5	
Overall AVG u....							257	
AVG .j..	271		259		240			
Main % Solids Effect	14		2		-17			
AVG ..k.	309	205						
Main Delay Time Effect	52	-52						

Although a glimpse of the data indicates slurry percent solids and delay time appear to impact strength, a full analysis is necessary to determine the true nature of the effects, particularly if interactions are present. For example Figure 3.4 showed how strength decreased with increasing percent solids with certain treatments, but not all treatments. Table 3.4 shows the 3-way ANOVA result. For each single test, the F-critical value was determined with a level of significance of 0.015 to yield a family level of significance to not exceed 0.10. The results show that, with a family confidence level of at least 90 percent, all of the following are true:

- no 3-way interactions exist,
- no two-factor interactions exist between slurry age and either percent solids or delay time,
- two-factor interactions do exist between slurry percent solids and delay time, and
- slurry age did not produce an effect on strength.

Table 3.4. 3-Way ANOVA Results for UCS with Low-PI Soil.

Source of Variation	SS	df	MS	F	P-value	F-Crit
Slurry Age	841	1	841	1.74281	0.199	6.86583
Slurry % Solids	5876	2	2938	6.08998	0.007	5.02837
Delay Time	98748	1	98748	204.7	0.000	6.86583
Age-Percent Solids Interaction	902	2	451	0.93521	0.406	5.02837
Age-Delay Time Interaction	2715	1	2715	5.62804	0.026	6.86583
Percent Solids-Delay Time Interaction	11114	2	5557	11.5193	0.000	5.02837
Age-Delay Time-Percent Solids Interact	3097	2	1549	3.21014	0.058	5.02837
Error	11578	24	482			
Total	134871	35				

Based upon the results from these tests, we may conclude that slurry age did not impact the UCS with slurry ages up to 2 hours. However, since interactions exist between slurry percent solids and delay time, the impact of these two factors cannot be estimated from the apparent

main effects shown in Table 3.3. Instead, the data must be analyzed by averaging the UCS over the slurry age for each level of percent solids and delay time, resulting in obtaining $\bar{Y}_{jk\bullet}$ values. This results in the dataset shown in Table 3.5 and graphically illustrated in Figures 3.5 and 3.6.

Table 3.5. Low PI UCS Results Averaged over Slurry Age.

	Slurry Percent Solids				
	30		50	70	
	Delay Time (hr)	0.5	2	0.5	2
AVG UCS (psi)	336	206	324	195	268
					213

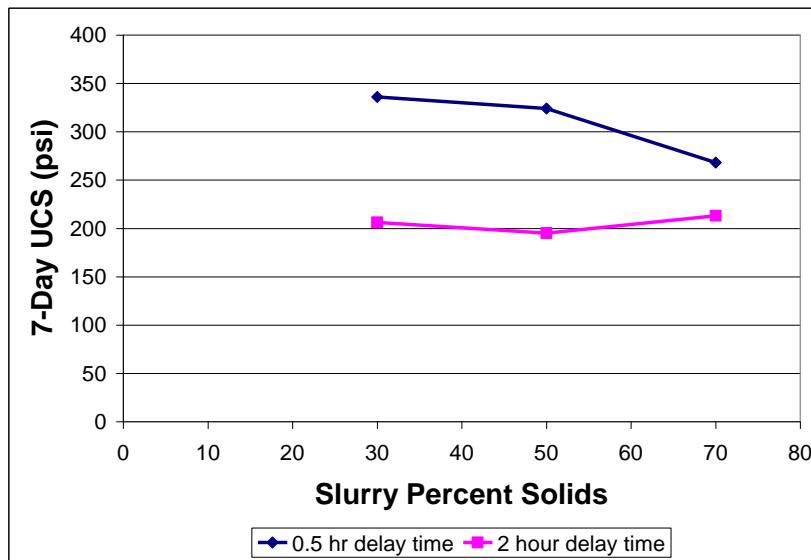


Figure 3.5. UCS with Varying Slurry Percent Solids for Low-PI Soil.

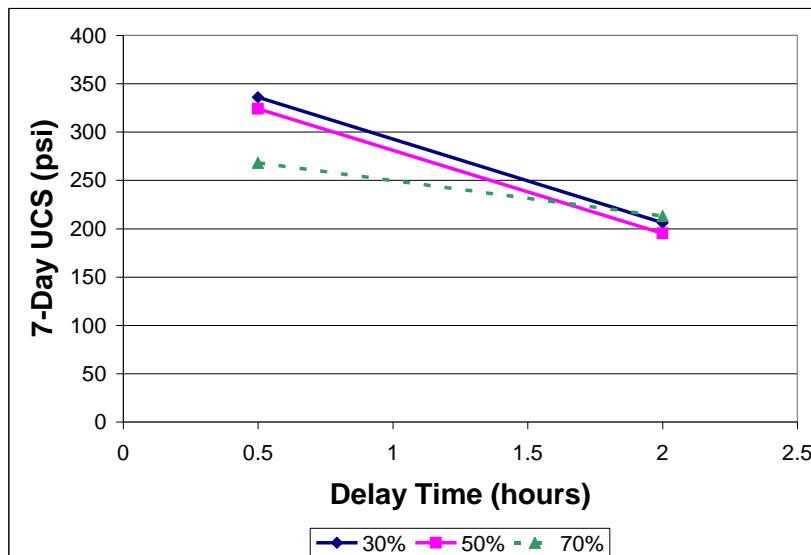


Figure 3.6. UCS with Varying Delay Time for Low-PI Soil.

Since slurry age has already been determined non-important to UCS (at least up to an age of 2.0 hours), and slurry percent solids and delay time interact, the pertinent questions become:

- For a given percent solids level, how was strength impacted by delay time?
- For a given delay time, how was strength impacted by percent solids?

The data shown in Figures 3.5 and 3.6 appear to illustrate sizeable decreases in strength when increasing the delay time from 0.5 to 2.0 hours for any given level of slurry percent solids. Additionally, with a delay time of 0.5 hours, the data indicate slurry with 70 percent solids produced a lower strength than the other two levels of percentage solids tested. No noticeable difference appears in strength among the varying levels of percent solids when the delay time was 2.0 hours.

To substantiate these observations, researchers used the Bonferroni procedure to evaluate contrasts of treatment means. Essentially, researchers differenced treatment means between delay times for a given level of percent solids, then treatment means were differenced between slurry percent solids for a given level of delay time, resulting in a family of nine tests. Researchers selected a test level of significance to produce a 90 percent family confidence coefficient. Table 3.6 presents the output from the analysis, including the observed differences and the lower and upper confidence limits. The data show, with a family confidence coefficient of 90 percent, that:

- With both 30 and 50 percent solids, increasing delay time from 0.5 to 2.0 hours reduced the UCS 90 to 169 psi, with an observed reduction of 130 psi.
- With 70 percent solids, increasing delay time from 0.5 to 2.0 hours reduced the UCS by 16 to 94 psi, with an observed reduction of 55 psi.
- With a delay time of 0.5 hours, strength was not impacted by changing the slurry percentage solids from 30 to 50 percent.
- With a delay time of 0.5 hours, treatment with slurry containing 70 percent solids produced reduced strength (by approximately 62 psi) as compared to treatment with slurries containing 30 or 50 percent solids.
- With delay times of 2.0 hours, varying slurry percentage solids did not impact strength.

Table 3.6. Output from Contrast of Treatment Means for Low-PI Soil UCS*.

		Observed Difference (psi)	Lower Conf Limit (psi)	Upper Conf Limit (psi)
Differences between delay times for given percent solids	L1 = y.11. - y.12.	130	91	169
	L2 = y.21. - y.22.	129	90	168
	L3 = y.31. - y.32.	55	16	94
Differences between percent solids for 0.5 hr delay time	L4 = y.11. - y.21.	12	-27	51
	L5 = y.11. - y.31.	68	29	107
	L6 = y.21. - y.31.	56	17	95
Differences between percent solids for 2.0 hour delay times	L7 = y.12. - y.22.	11	-28	50
	L8 = y.12. - y.32.	-7	-46	32
	L9 = y.22. - y.32.	-18	-57	21

*If the confidence interval contains zero, the observed difference is not significant.

SEISMIC MODULUS RESULTS

Analysis of the seismic modulus results follows the same process previously illustrated for investigating the impacts of slurry age, slurry percentage solids, and delay time factors on strength. Table 3.7 presents all the seismic modulus data, Table 3.8 presents the 3-factor summary results, and Table 3.9 presents the output of a 3-way ANOVA on the dataset.

Table 3.7. Seismic Modulus (ksi) Results for Low-PI Soil.

Treatment	Slurry Age (hr)	Delay Time (hr)	Slurry Percent Solids		
			30	50	70
A	0	0.5	960	866	741
			795	851	700
			715	767	793
B	0	2	529	646	579
			457	754	590
			595	553	560
C	2	0.5	910	769	721
			880	765	692
			754	814	703
D	2	2	572	620	510
			600	593	543
			706	631	540

Table 3.8. 3-Factor Summary Results for Low-PI Soil Modulus Values.

Slurry Age (hours)	Slurry Percent Solids						AVG i... Main age effect	
	30		50		70			
	Delay Time (hr)	0.5	Delay Time (hr)	0.5	Delay Time (hr)	0.5		
0	823	527	828	651	745	576	692 4	
2	848	626	783	615	705	531	685 -4	
Overall AVG u....						688		
AVG .j..	706		719		639			
Main % Solids Effect	18		31		-49			
AVG ..k.	789	588						
Main Delay Time Effect	101	-101						

Table 3.9. 3-Way ANOVA Results for Seismic Modulus of Low-PI Soil.

Source of Variation	SS	df	MS	F	P-value	F-Crit
Slurry Age	455	1	455	0.11087	0.742	6.86583
Slurry % Solids	43938	2	21969	5.35194	0.012	5.02837
Delay Time	363609	1	363609	88.5789	0.000	6.86583
Age-Percent Solids Interaction	21393	2	10697	2.60582	0.095	5.02837
Age-Delay Time Interaction	1495	1	1495	0.36422	0.552	6.86583
Percent Solids-Delay Time Interaction	15227	2	7614	1.85475	0.178	5.02837
Age-Delay Time-Percent Solids Interact	2737	2	1368	0.33335	0.720	5.02837
Error	98518	24	4105			
Total	547373	35				

The results show that, with a family confidence level of at least 90 percent:

- no interactions exist,
- slurry age had no impact on the seismic modulus,
- slurry percentage solids did impact the modulus, and
- delay time significantly impacted the modulus.

Since no interactions exist, the main factor effects of slurry percentage solids and delay time may be investigated on the basis of contrasts between the factor level means shown previously in Table 3.8. Figures 3.7 and 3.8 graphically present these data, and Table 3.10 presents the results of the Bonferroni procedure again used for evaluating contrasts of treatment means.

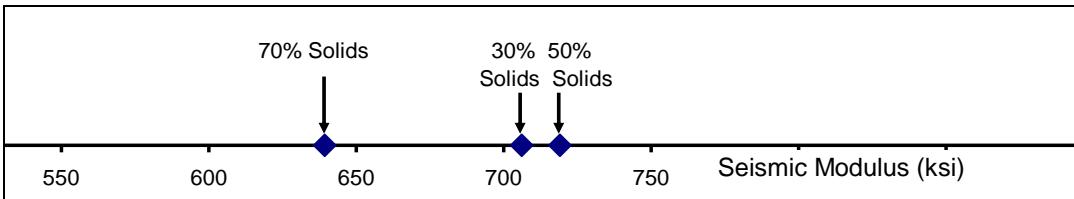


Figure 3.7. Impact of Percentage Solids on Seismic Modulus for Low-PI Soil.

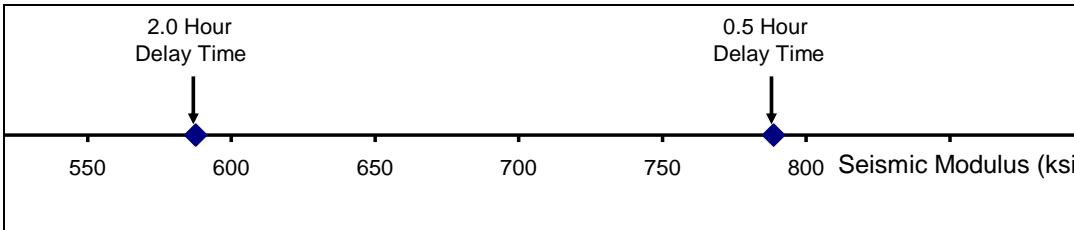


Figure 3.8. Impact of Delay Time on Seismic Modulus for Low-PI Soil.

Table 3.10. Contrast of Treatment Means for Low-PI Soil Seismic Modulus.

	Observed Difference (ksi)	Lower Conf Limit (ksi)	Upper Conf Limit (ksi)
Differences between varying percentage solids	L1 = y _{1..} - y _{2..}	-13	-77
	L2 = y _{1..} - y _{3..}	67	3
	L3 = y _{2..} - y _{3..}	80	16
Difference between delay times	L4 = y _{1..} - y _{2..}	201	149
			253

The Bonferroni method used was selected to provide a family confidence level of 90 percent. The results shown indicate:

- no difference in seismic modulus occurred by changing the slurry percent solids from 30 to 50 percent,
- using a slurry percentage solids of 70 percent reduced seismic modulus by between 3 and 144 ksi, with an observed reduction of approximately 73 ksi as compared to treatment using a slurry containing 30 or 50 percent solids, and
- increasing the delay time from 0.5 to 2.0 hours decreases the 7-day seismic modulus between 149 and 253 ksi, with the observed reduction being 201 ksi.

As with the strength results, the modulus results clearly indicate delay time most significantly contributes to reduced mechanistic properties of soil-cement mixtures. Of additional interest is whether slurry treatment produced a modulus comparable to that resultant from the application of cement by dry powder. Figure 3.9 shows such results obtained by dry application of cement to the low-PI soil out to a delay time of 4 hours. The results showed no significant difference in the seismic modulus after 7-days curing for delay times up to 4 hours, with an overall average value of 532 ksi. Comparison of this value with the results obtained by slurry application shown in Figure 3.8 indicate, and statistical tests confirm, that slurry treatment

resulted in a higher mean modulus value regardless of the delay time. With a 0.5 hour delay time, slurry produced an observed increase in modulus of 258 ksi, with the 95 percent confidence interval for this increase being 200 to 316 ksi. The increase in modulus by slurry treatment with a delay time of 2.0 hours was much more subtle, with an observed increase of 57 ksi and a 95 percent confidence interval of 4 to 110 ksi.

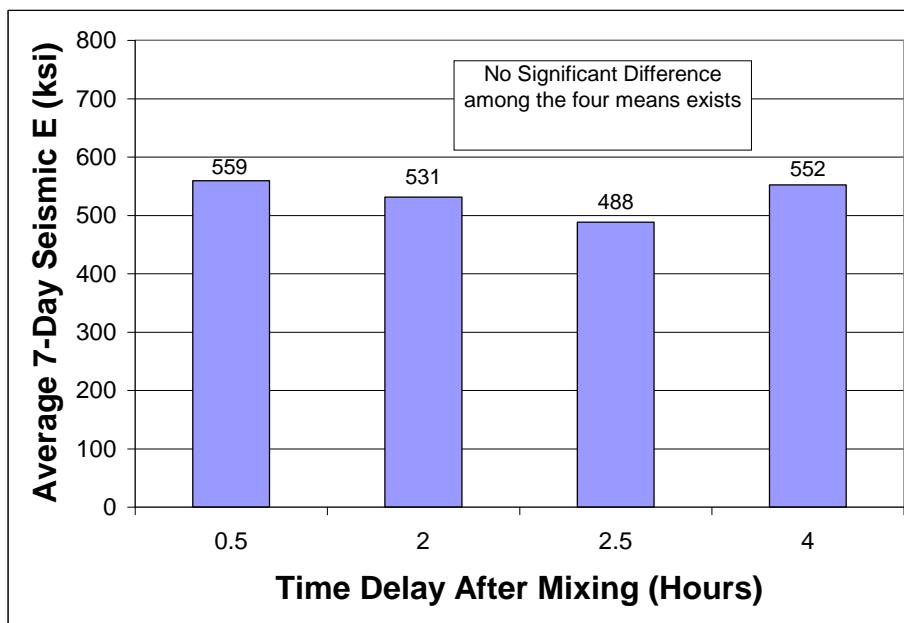


Figure 3.9. Seismic Modulus Results for Dry Application of Cement to Low-PI Soil.

CONCLUSIONS FROM LOW-PI SOIL TESTS

The strength and seismic modulus test results presented and analyzed from the low-PI soil indicate the following:

- Application of cement by slurry always resulted in strengths meeting or exceeding UCS values obtained from comparable dry cement powder applications.
- Slurry age (the time from slurry mixing to incorporation of the slurry into the soil) had no impact on soil strength or seismic modulus with slurry ages up to 2.0 hours.
- Increasing delay time (the time between mixing the cement into the soil and compaction) negatively impacted the UCS regardless of whether the cement was applied in powder or slurry form.
- With a 2-hour delay time, dry application failed to meet the design strength requirement. However, even with a 2-hour delay time, application of cement by slurry still resulted in strengths meeting the 200 psi criteria.
- With a short delay time (of 0.5 hours), treatment with slurry containing 70 percent solids resulted in a 62 psi reduction in strength as compared to treatment with cement slurries containing either 30 or 50 percent solids (which did not vary in strength).
- Varying slurry percentage solids did not impact UCS with delay times of 2.0 hours.

- Treatment of the soil with slurry containing 70 percent solids resulted in a reduced seismic modulus by approximately 73 ksi as compared to treatment with slurries containing 30 or 50 percent solids (which did not vary in modulus).

These findings suggest the following implications for industry:

- Design tests should consider delay time, particularly if the field application will employ dry powder cement. The data suggest a delay time of 2 hours will adequately safeguard against under-design.
- Cement treatment using slurry better stabilizes the soil as compared to a dry powder application, as evidenced by less susceptibility to delay time and increased modulus values.
- Slurry age had no impact on performance with slurry ages up to 2 hours.
- Slurry percentage solids can slightly impact results, with high percent solids (70 percent) producing slightly lower modulus values and in some cases slightly reduced strengths. However, from an engineering standpoint, these differences are quite subtle and selection of slurry percentage solids likely will be best determined according to equipment (workability), economic (haul costs), and project (in-situ soil moisture content) factors.

CHAPTER 4

PERFORMANCE INVESTIGATION OF CEMENT SLURRY APPLICATIONS ON A HIGH-PI SOIL

SUMMARY

To investigate the impact of slurry treatment on a high-PI soil ($30 < PI < 35$), the same testing factorial outlined in Chapter 2 was performed on a soil with a liquid limit of 57, a plastic limit of 22, and a PI of 35 (AASHTO A-7-6). As with the low-PI soil tests, the purpose of this factorial was to investigate the following:

- Does a slurry application produce the same strength result as a dry application?
- Does the percentage solids in the slurry impact the performance?
- How does the age of the slurry impact performance?
- How does the time delay between mixing the slurry into the soil and completion of compaction influence results?

Researchers employed the same methods for mixing and molding the soil that were used with the low-PI soil; i.e. for all cases with a delay between slurry mixing and application to the soil, the slurry was agitated continuously during the delay time. For reference, researchers also performed the tests on specimens treated with dry cement powder with time delays of 30 minutes, 2 hours, 2.5 hours, and 4 hours between mixing the cement into the soil and soil compaction. Researchers performed each test in triplicate. The results showed:

- Slurry age (the time from slurry mixing to incorporation of the slurry into the soil) had no impact on soil strength or seismic modulus with slurry ages up to 2.0 hours.
- Both strength and modulus peaked with slurry percentage solids of 50 percent.
- No differences in strength or modulus existed when comparing results from treatment with slurry containing 30 percent solids to 70 percent solids.
- Increasing delay time (the time between mixing the cement into the soil and compaction) negatively impacted the UCS regardless of whether the cement was applied in powder or slurry form.

BACKGROUND SOIL INFORMATION

The high-PI soil used had a PI of 35 and 99.4 percent passing the #200 sieve, resulting in an AASHTO classification of A-7-6. Using ASTM D 558, the researchers developed the moisture-density curve shown in Figure 4.1 with a treatment level of 5 percent cement, indicating an optimum moisture content of 25.2 percent with a maximum dry density of 93.8 pcf. Cement was applied to the soil as a dry powder when performing the moisture-density curve.

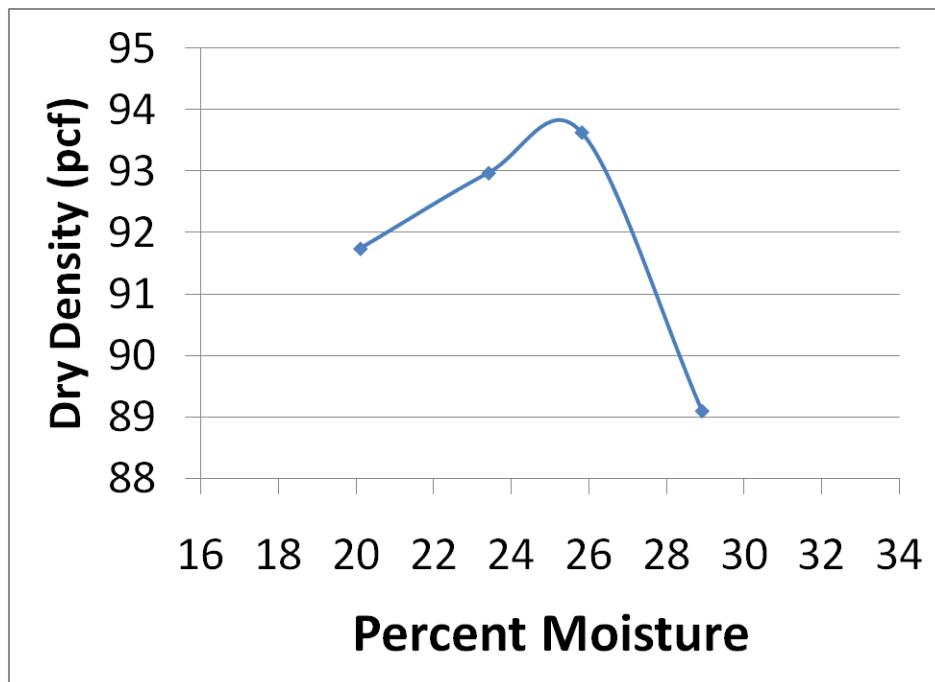


Figure 4.1. ASTM D 558 Result for High-PI Soil with 5 Percent Cement.

SELECTION OF DESIGN CEMENT CONTENT

Following determination of the moisture-density curve, the 7-day unconfined compressive strengths of this soil were determined for cement treatment levels of 8, 10, 12, and 15 percent at a loading rate of 0.05 inches per minute. Cement was applied as a dry powder for these tests. The goal was to select a treatment level that would produce a 200 psi 7-day strength. Cylindrical, 4-inch diameter by 4.6-inch tall specimens were compacted then moist cured for 7 days. Immediately prior to testing, each specimen was soaked by complete submersion in water for 4 hours then capped. Each treatment level was tested in duplicate, and Figure 4.2 shows the average result for each level of treatment. To reliably achieve the 200 psi target, 9 percent cement was selected as the design treatment level.

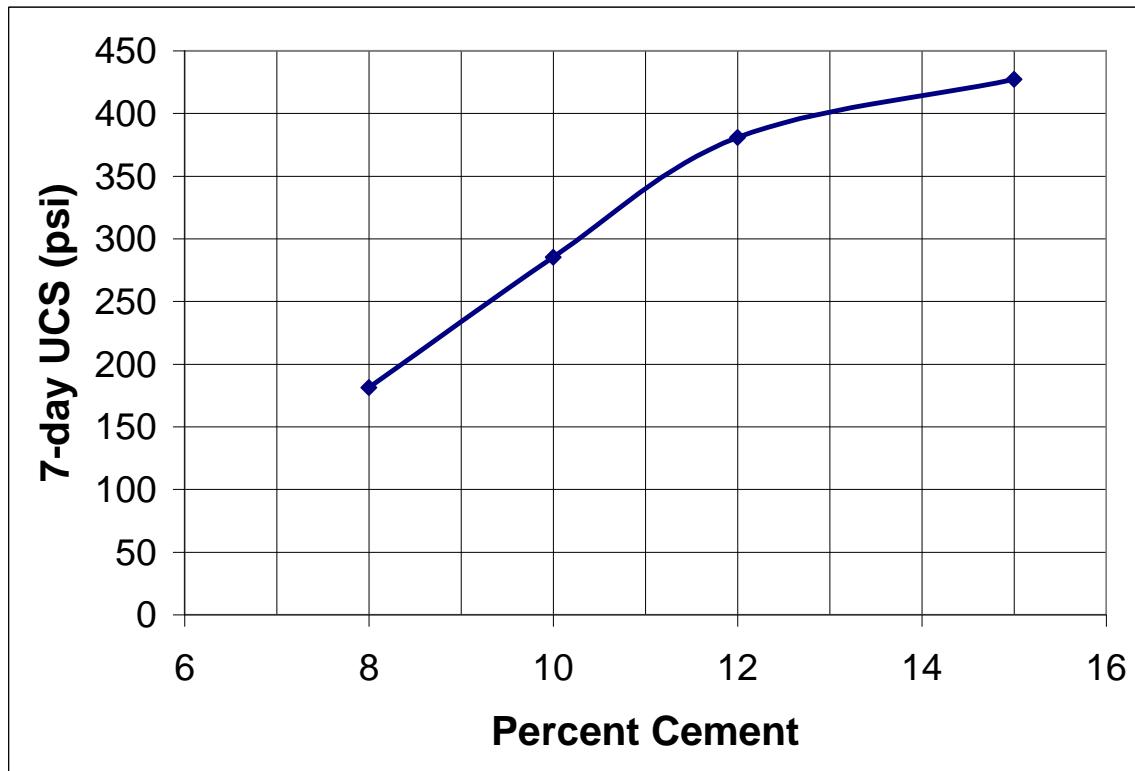


Figure 4.2. Strength Results from High-PI Soil Cement Design.

STRENGTH TESTING RESULTS

Figure 4.3 presents the strength results from the reference samples treated with 9 percent cement applied as a dry powder. These results show a 50 percent decrease in strength between the delay times of 0.5 hours and 2 hours. From 2 to 4 hours delay time, the average strength did not statistically change. With delay times of 2 or more hours between mixing the cement into the soil and compaction, on average the mixture did not meet the 200 psi strength target. These findings show the time anticipated in the field between mixing the cement into the soil and compaction should be considered when performing cement content design tests. Based upon these results, using a 2-hour delay time should adequately safeguard against under-design (since the average strength did not change from delay times of 2 to 4 hours).

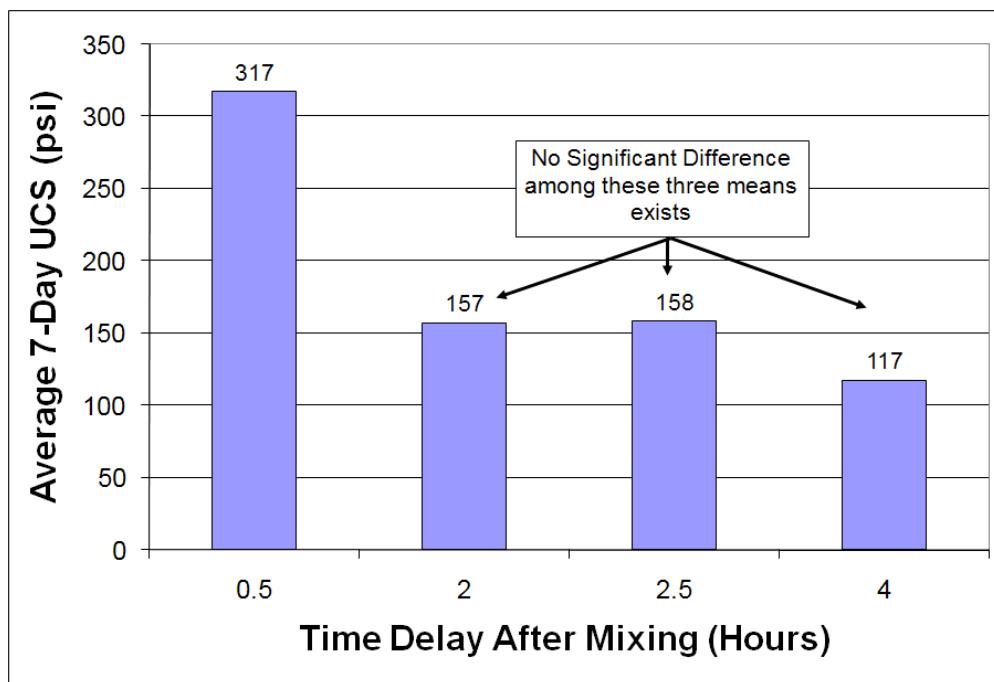


Figure 4.3. High-PI Strengths with Cement Applied as a Dry Powder.

Table 4.1 presents the strength results from the high-PI soil treated with cement slurry. As with the low-PI soil, researchers analyzed these results using three-way ANOVA to statistically and quantitatively investigate the factor effects of delay time, slurry percent solids, and slurry age. Table 4.2 presents the treatment average values \bar{Y}_{ijk} , where i , j , and k represent the treatment level for slurry age, percent solids, and delay time, respectively. Table 4.2 also presents the apparent main effects for each of the three factors.

Table 4.1. UCS (psi) Results for High-PI Soil.

Slurry Age (hours)	Slurry Percent Solids					
	30		50		70	
	Delay Time (hr)	0.5	Delay Time (hr)	0.5	Delay Time (hr)	0.5
Slurry Age (hours)	0.5	2	0.5	2	0.5	2
0	270	176	278	176	261	140
0	254	141	294	209	230	142
0	251	146	280	184	234	112
2	217	147	307	230	190	168
2	198	155	272	238	212	140
2	217	162	320	264	196	124

Table 4.2. 3-Factor Summary Results for High-PI Soil Strengths.

Slurry Age (hours)	Slurry Percent Solids						AVG i... 210 209	Main age effect 1 -1		
	30		50		70					
	Delay Time (hr)	0.5	Delay Time (hr)	0.5	Delay Time (hr)	2				
0	258	154	284	190	242	131				
2	211	155	300	244	199	144				
Overall AVG u....							209			
AVG .j..	195		254		179					
Main % Solids Effect	-15		45		-30					
AVG ..k.	249	170								
Main Delay Time Effect	40	-40								

Some information gleaned from these data in Tables 4.1 and 4.2 includes:

- The slurry age effect appears insignificant.
- Strength appears to peak at 50 percent solids.
- Delay time appears to impact the strength, with short delay times resulting in strength increases, and long delays resulting in strength decrease.

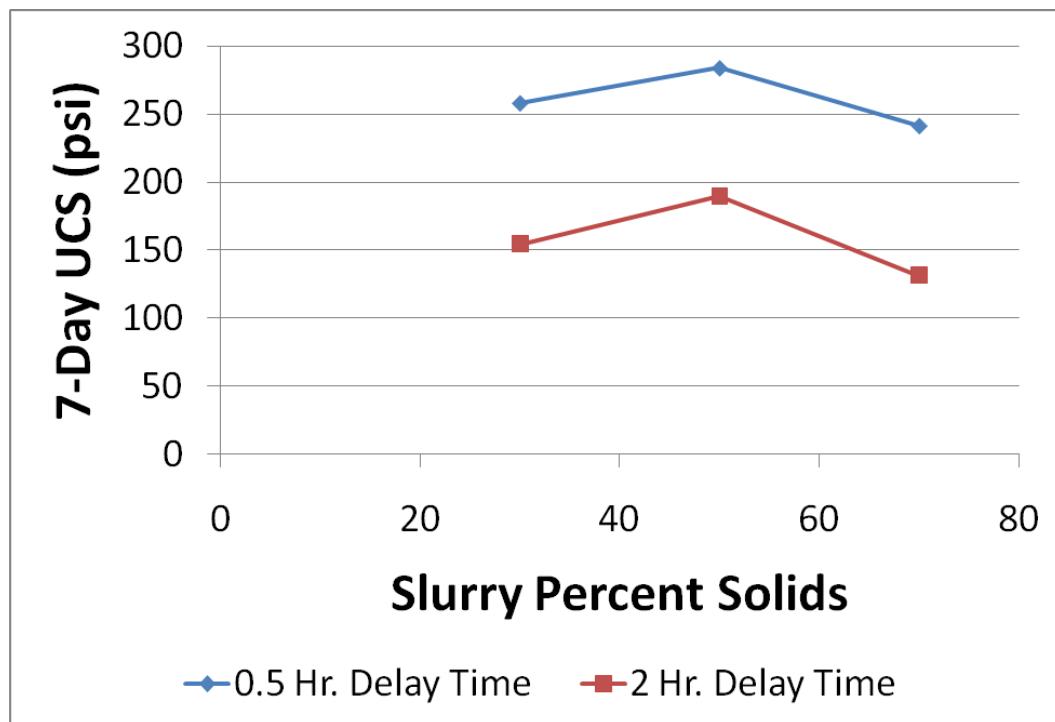
Although a glimpse of the data indicates slurry percent solids and delay time appear to impact strength, a full analysis is necessary to determine the true nature of the effects, particularly if interactions are present. Table 4.3 shows the 3-way ANOVA result. For each single test, the F-critical value was determined with a level of significance of 0.015 to yield a family level of significance to not exceed 0.10. The results show that, with a family confidence level of at least 90 percent, all of the following are true:

- slurry age did not produce a main effect,
- both slurry percent solids and delay time produced a main effect,
- slurry age and percent solids interacted,
- slurry age and delay time interacted,
- slurry percent solids and delay time did not interact, and
- no three-factor interactions exist.

Table 4.3. 3-Way ANOVA Results for UCS with High-PI Soil.

Source of Variation	SS	df	MS	F	P-value	F-Crit
Slurry Age	12	1	12	0.04689	0.830	6.86582
Slurry % Solids	37921	2	18961	72.57618	0.000	5.02839
Delay Time	56565	1	56565	216.5156	0.000	6.86582
Age-Percent Solids Interaction	6003	2	3002	11.48931	0.000	5.02839
Age-Delay Time Interaction	5017	1	5017	19.20521	0.000	6.86582
Percent Solids-Delay Time Interaction	94	2	47	0.180649	0.836	5.02839
Age-Delay Time-Percent Solids Interaction	101	2	50	0.19277	0.826	5.02839
Error	6270	24	261			
Total	111984	35				

Based upon the result from the ANOVA, we may conclude that slurry age did not impact the UCS with slurry ages up to 2 hours. However, since interactions exist between slurry age and slurry percent solids, and between slurry age and delay time, the impact of percent solids and delay time cannot be estimated from the apparent main effects shown in Table 4.2. Instead, the effect of slurry percent solids and delay time must be investigated individually for slurry ages of 0 and 2 hours. Figures 4.4 and 4.5 illustrate this evaluation for slurry ages of 0 and 2 hours, respectively.

**Figure 4.4. UCS with Varying Slurry Percent Solids and a Slurry Age of 0 Hours.**

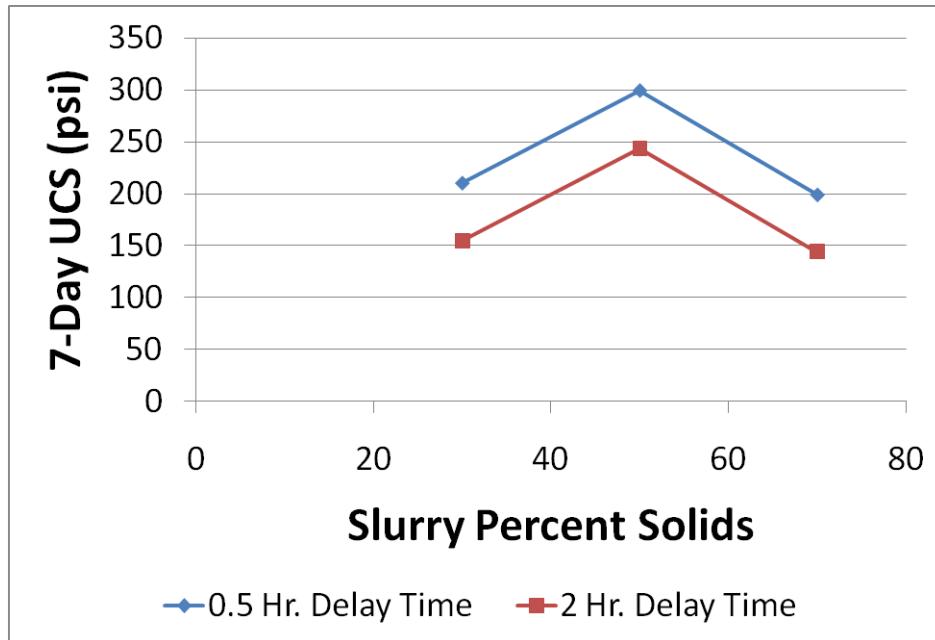


Figure 4.5. UCS with Varying Slurry Percent Solids and a Slurry Age of 2 Hours.

The data shown in Figures 4.4 and 4.5 illustrate sizeable decreases in strength when increasing the delay time from 0.5 to 2.0 hours for any given level of slurry percent solids. The data also indicate treatment with slurry containing 50 percent solids produced higher strengths than treatment with slurry containing 30 or 70 percent solids. To validate these observations, contrasts of treatment means as presented in Table 4.4 were conducted.

Table 4.4. Output from Contrast of Treatment Means for High-PI Soil UCS*.

	Observed Difference (psi)	Lower Conf Limit (psi)	Upper Conf Limit (psi)
Differences between delay times for 0 hr slurry age	L1 = y1.1. - y1.2. 103	89	117
Difference between delay times for 2.0 hour slurry age	L2 = y2.1. - y2.2. 56	41	70
Differences between percent solids for 0 hr slurry age	L3 = y11.. - y12.. -31 L4 = y11.. - y13.. 20 L5 = y12.. - y13.. 50	-53 -3 28	-8 42 73
Differences between percent solids for 2.0 hour slurry age	L6 = y21.. - y22.. -89 L7 = y21.. - y23.. 11 L8 = y22.. - y23.. 100	-112 -11 78	-67 33 123

*If the confidence interval contains zero, the observed difference is not significant.

The analysis presented in Table 4.4 supports the following conclusions:

- Delay time significantly impacted the UCS, where increasing the delay time from 0.5 to 2 hours resulted in strength reduction. With a slurry age of 0 hours, this strength decrease was 103 psi. With a slurry age of 2 hours, this strength decrease was 56 psi.
- With a slurry age of 0 hours, the strength produced with slurry containing 50 percent solids exceeded the strengths produced both by slurry containing 30 or 70 percent solids. The strength produced by treatment with slurry containing 50 percent solids exceeded the 30 and 70 percent solids strengths by 31 and 50 psi, respectively. No significant difference in strength existed when comparing treatment with slurry containing 30 percent solids to slurry containing 70 percent solids. Figure 4.6 further illustrates these findings.
- With a slurry age of 2 hours, the strength produced with slurry containing 50 percent solids exceeded the strengths produced both by slurry containing 30 or 70 percent solids. The strength produced by treatment with slurry containing 50 percent solids exceeded the 30 and 70 percent solids strengths by 89 and 100 psi, respectively. No significant difference in strength existed when comparing treatment with slurry containing 30 percent solids to slurry containing 70 percent solids. Figure 4.7 further illustrates these findings.

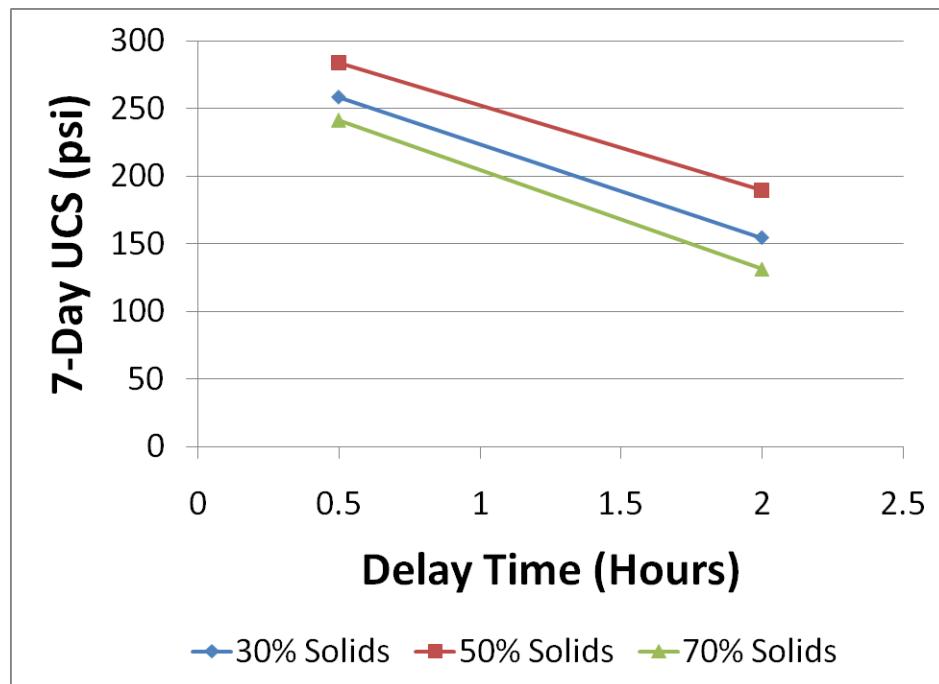


Figure 4.6. Impact of Slurry Percent Solids on High-PI Soil Strength with Slurry Age of 0 Hours.

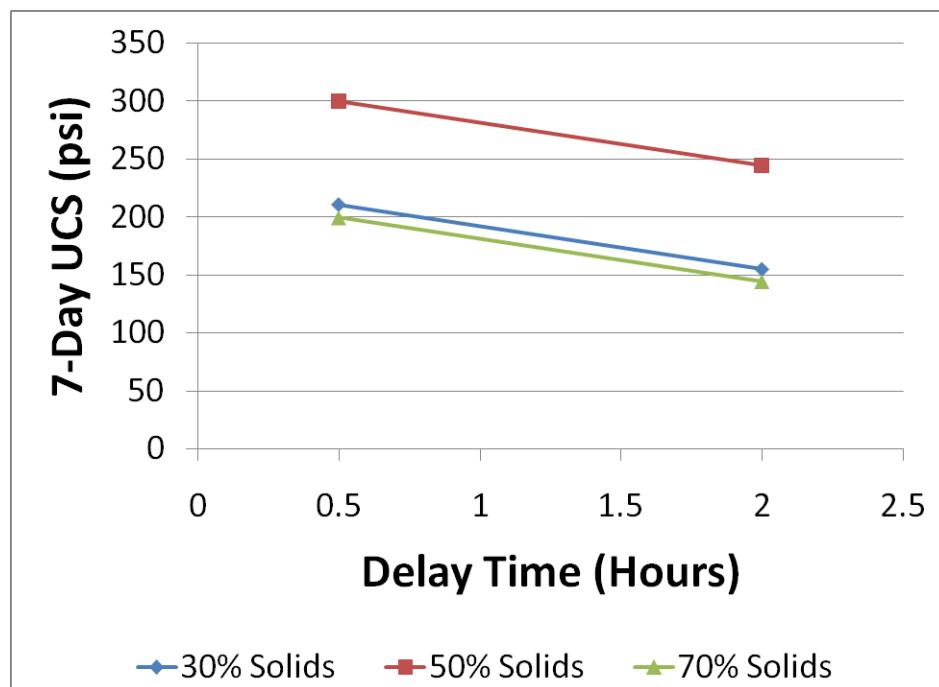


Figure 4.7. Impact of Slurry Percent Solids on High-PI Soil Strength with Slurry Age of 2 Hours.

SEISMIC MODULUS RESULTS

Analysis of the seismic modulus results follows the same process previously illustrated for investigating the impacts of slurry age, slurry percentage solids, and delay time factors on strength. Table 4.5 presents all the seismic modulus data, Table 4.6 presents the 3-factor summary results, and Table 4.7 presents the output of a 3-way ANOVA on the dataset.

Table 4.5. Seismic Modulus (ksi) Results for High-PI Soil.

Slurry Age (hours)	Slurry Percent Solids					
	30		50		70	
	Delay Time (hr)		Delay Time (hr)		Delay Time (hr)	
Slurry Age (hours)	0.5	2	0.5	2	0.5	2
0	420	351	447	428	426	349
0	408	287	451	436	406	272
0	405	310	491	349	378	249
2	388	300	561	419	456	312
2	378	368	496	435	362	320
2	399	312	583	482	363	376

Table 4.6. 3-Factor Summary Results for High-PI Soil Modulus Values.

Slurry Age (hours)	Slurry Percent Solids						AVG i... 381 406	Main age effect -12 12		
	30		50		70					
	Delay Time (hr)	0.5	Delay Time (hr)	0.5	Delay Time (hr)	0.5				
0	411	316	463	404	403	290				
2	388	327	547	445	394	336				
Overall AVG u....							394			
AVG .j..	361		465		356					
Main % Solids Effect	-33		71		-38					
AVG ..k. Main Delay Time Effect	434	353								
	41	-41								

Table 4.7. 3-Way ANOVA Results for Seismic Modulus of High-PI Soil.

Source of Variation	SS	df	MS	F	P-value	F-Crit
Slurry Age	5529	1	5529	4.148155	0.053	6.86582
Slurry % Solids	91193	2	45596	34.20633	0.000	5.02839
Delay Time	59473	1	59473	44.61625	0.000	6.86582
Age-Percent Solids Interaction	7230	2	3615	2.712011	0.087	5.02839
Age-Delay Time Interaction	546	1	546	0.409831	0.528	6.86582
Percent Solids-Delay Time Interaction	84	2	42	0.031413	0.969	5.02839
Age-Delay Time-Percent Solids Interaction	3997	2	1999	1.499353	0.243	5.02839
Error	31992	24	1333			
Total	200044	35				

The ANOVA results in Table 4.7 show that, with a family confidence level of at least 90 percent:

- no interactions exist,
- slurry age had no impact on the seismic modulus,
- slurry percentage solids did impact the modulus, and
- delay time significantly impacted the modulus.

Since no interactions exist, the main factor effects of slurry percentage solids and delay time may be investigated on the basis of contrasts between the factor level means shown previously in Table 4.6. Figures 4.8 and 4.9 graphically present these data, and Table 4.8 presents the results from evaluating contrasts of treatment means to validate the observed effects of varying slurry percent solids and delay times.

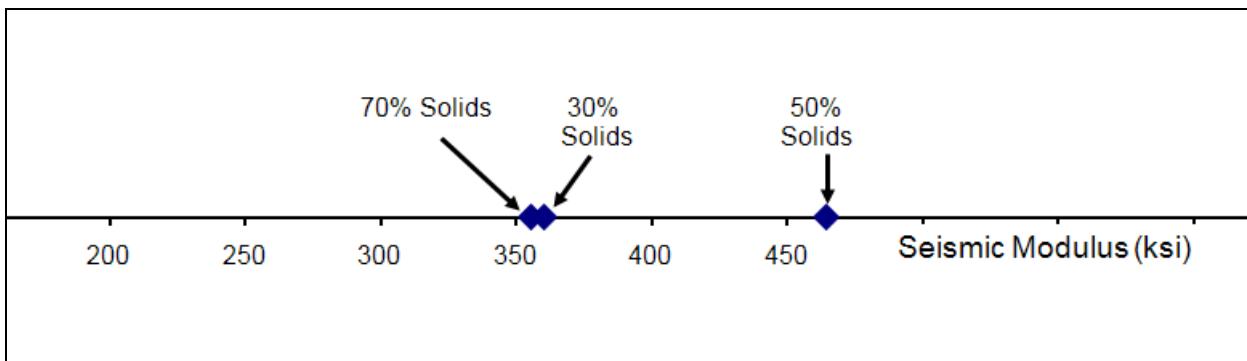


Figure 4.8. Impact of Percentage Solids on Seismic Modulus for High-PI Soil.

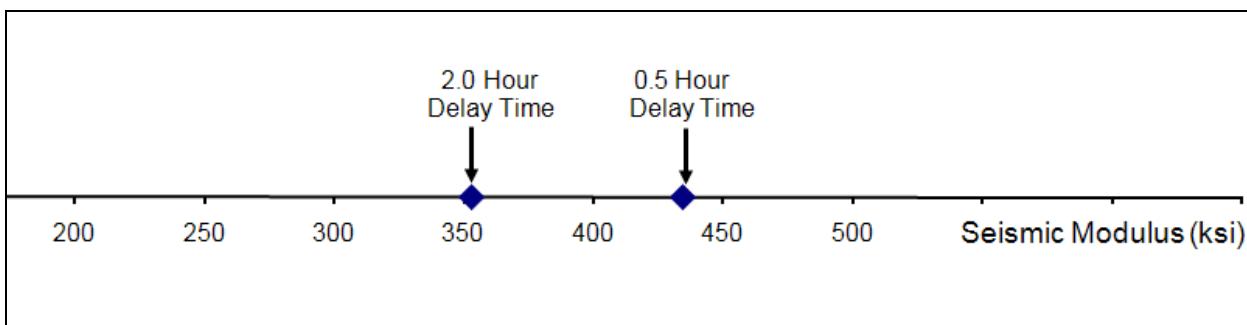


Figure 4.9. Impact of Delay Time on Seismic Modulus for High-PI Soil.

Table 4.8. Contrast of Treatment Means for High-PI Soil Seismic Modulus*.

	Observed Difference (ksi)	Lower Conf Limit (ksi)	Upper Conf Limit (ksi)
Differences between varying percentage solids	L1 = y _{1..} - y _{2..} -104	-128	-80
	L2 = y _{1..} - y _{3..} 5	-19	29
	L3 = y _{2..} - y _{3..} 109	85	133
Difference between delay times	L4 = y _{..1.} - y _{..2.} 81	60	102

*If the confidence interval contains zero, the observed difference is not significant.

The results in Figures 4.8 and 4.9, and Table 4.8 support the following conclusions:

- The seismic moduli produced by using slurry with 30 and 70 percent solids were statistically equivalent, averaging 358 ksi.
- Applying slurry with 50 percent solids produced a seismic modulus exceeding the values produced by slurry containing 30 and 70 percent solids by approximately 106 ksi.
- A 0.5 hour delay time produced a seismic modulus exceeding the value produced with a 2 hour delay time by 81 ksi.

As with the strength results, the modulus results showed peak mechanistic properties with slurry containing 50 percent solids and short delay times. Of additional interest is whether slurry treatment produced a modulus comparable to that resultant from the application of cement by dry powder. Figure 4.10 shows results obtained by dry application of cement to the high-PI soil out to a delay time of 4 hours. The results showed the seismic modulus with a 0.5 hour delay time exceeded the modulus observed with a 2.5 hour delay time. No other significant differences in mean seismic modulus existed.

Comparison of the dry-application seismic modulus values in Figure 4.10 with the values obtained by slurry treatment shown in Figure 4.9 indicate, and statistical tests confirm, that for an equal delay time no difference in seismic modulus existed between dry powder and slurry application methods.

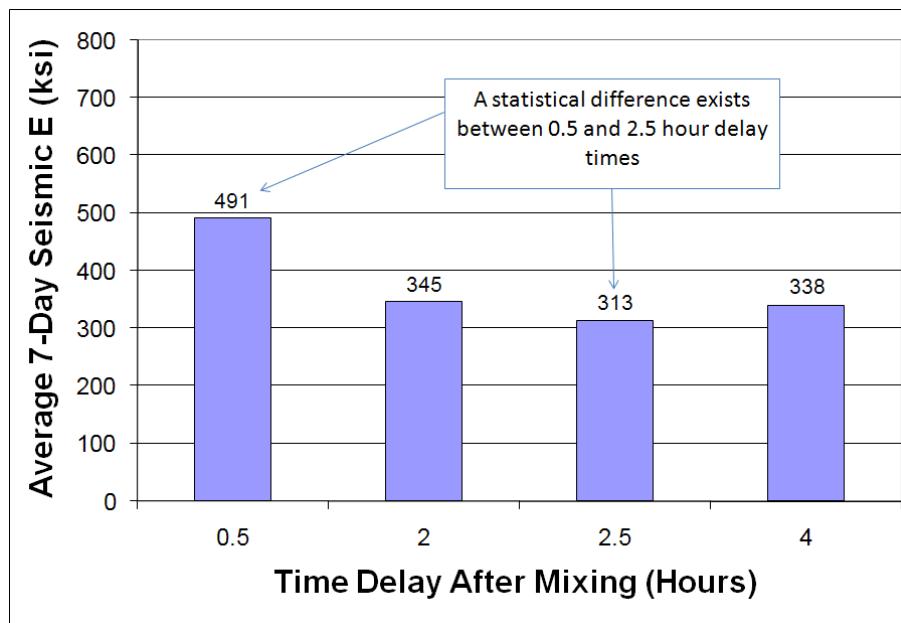


Figure 4.10. Seismic Modulus Results for Dry Application of Cement to High-PI Soil.

CONCLUSIONS FROM HIGH-PI SOIL TESTS

The strength and seismic modulus test results presented and analyzed from the high-PI soil indicate the following:

- Slurry age (the time from slurry mixing to incorporation of the slurry into the soil) had no impact on soil strength or seismic modulus with slurry ages up to 2.0 hours.
- Both strength and modulus peaked with slurry percentage solids of 50 percent.
- No differences in strength or modulus existed when comparing results from treatment with slurry containing 30 percent solids to 70 percent solids.
- Increasing delay time (the time between mixing the cement into the soil and compaction) negatively impacted the UCS regardless of whether the cement was applied in powder or slurry form.
- With a 2 hour delay time, dry application failed to meet the design strength requirement. With slurry application of cement to the high-PI soil, only treatment with slurry containing 50 percent solids met the 200 psi target.

These findings suggest the following implications for industry when treating plastic soils with cement:

- Design tests should consider delay time. The data suggest a delay time of 2 hours will adequately safeguard against under design. In the lab, substantial strength decrease occurred when increasing the delay time from 0.5 to 2 hours. Only treatment with slurry containing 50 percent solids still met the strength target with a 2 hour delay time.

- Cement treatment of the plastic soil using slurry provided performance on par with, but not necessarily exceeding, the performance obtained by cement treatment with dry powder.
- Slurry age had no impact on performance with slurry ages up to 2 hours.
- Slurry percentage solids can impact results, with slurry containing 50 percent solids providing the peak mechanical properties with the plastic soil tested. With the plastic soil tested, in some cases the percentage solids of the slurry made the difference in whether the mixture met the design strength. Therefore, when seeking to treat plastic clays with slurry, one should carefully consider the anticipated percentage solids of the slurry during design tests.

CHAPTER 5

EVALUATING SLURRY WORKABILITY THROUGH VISCOSITY MEASUREMENT

SUMMARY

The ability to pump or otherwise offload and spread cement slurry clearly must be considered as part of the solids content selection process. Industry personnel indicate approximately 65 percent solids represents the upper limit of slurry workability. Therefore, since laboratory performance investigations (previously detailed) included slurry ages of up to 2 hours, viscosity tests were conducted on slurries to determine if workability still existed after 2 hours. Tests revealed a small increase in viscosity, but it is suspected the observed increase is not sufficient to inhibit real-world workability.

TEST PLAN

TTI researchers used a Brookfield DV-III+ rheometer, a Model D helipath, and a model T-B t-bar spindle to measure slurry viscosity. With the spindle turning at 5 RPM, measurements were collected at 10-second intervals for one complete cycle of the helipath. This resulted in collecting 11 data points for each sample. TTI used the viscosity of slurry containing 70 percent solids at an age of approximately 5 minutes to represent slurry too viscous to work. Following this determination, researchers planned to use the identical equipment and test parameters to measure the viscosity of slurries containing 50 to 65 percent solids, in 5 percent intervals, at slurry ages between 5 minutes and 2 hours. For comparison purposes, Brookfield application engineers stressed the importance of using the same spindle and RPM for all the tests.

RESULTS

With the T-B spindle turning at 5 RPM, the Brookfield DV-III+ measurement range is from 8000 to 80000 centipoise (cP). With 70 percent solids, the measured slurry viscosity approached the upper measurement limit. With 65 percent solids, the slurry viscosity barely exceeded the lower measurement limit. Since changing spindles or RPM was not recommended for comparing results, TTI conducted only one additional test with slurries containing less than 65 percent solids. This test was with 50 percent solids aged for 2 hours, to evaluate if the viscosity increased enough to approach that of slurry containing 65 percent solids. The viscosity did not, and the measurements were well below the lower measurement limit of the test arrangement employed. Table 5.1 shows the results.

Table 5.1. Cement Slurry Viscosity Measurement Results.

Slurry Percent Solids	Slurry Age (minutes)	Viscosity (cP)		Average Percent Torque**
		Average	Standard Deviation	
70 – test 1	3	70760	5440	88.5
70 – test 2	7	70734	5309	88.4
65	5	8344	261	10.4
65	9	8704	379	10.9
65	38	8726	380	10.9
65	80	10154	841	12.7
65	80	10131	645	12.4
65	123	10816	448	13.5
65	127	11984	1642	15.0
50	120	*	*	*

*Viscosity below lowest measurement limit for test arrangement.

**Must be between 10 and 100 for valid results.

Figure 5.1 shows the average and 95 percent confidence interval of the slurry viscosity with time for slurry containing 65 percent solids. The results show the slurry does not change in viscosity for at least the first 40 minutes. At a slurry age of 80 minutes and 120 minutes, the slurry viscosity did increase. As compared to the initial viscosity, the viscosity increased approximately 25 percent after 2 hours. However, it is unknown if this 25 percent increase in viscosity after 2 hours is enough to make a difference in real-world workability, especially since the measurements were still only a fraction of the values measured with cement slurry containing 70 percent solids.

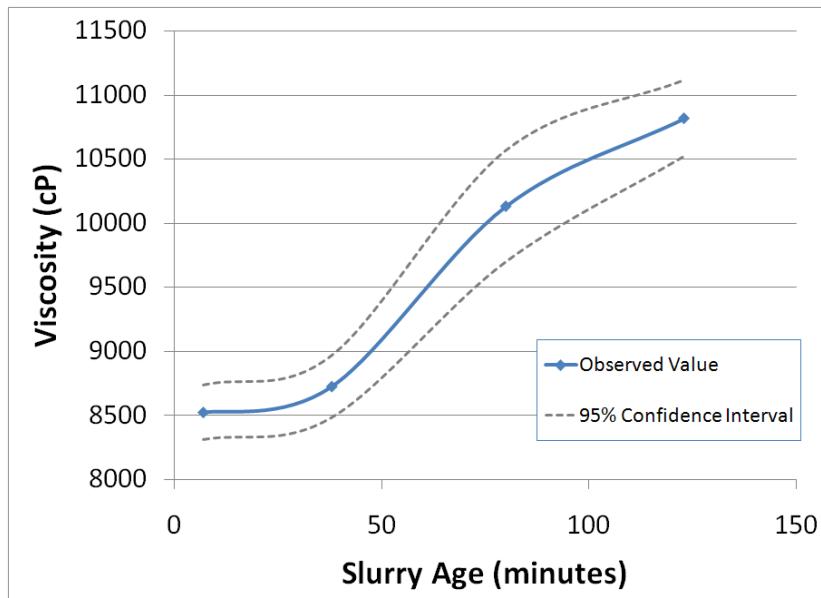


Figure 5.1. Slurry Viscosity with Time for Slurry Containing 65 Percent Solids.

CONCLUSIONS

With the test arrangement and slurry percentage solids used, slurry viscosity did not change up to an age of at least 40 minutes. The slurry viscosity did increase by 25 percent at an age of 2 hours; however, it is unknown if this increase would impact real-world workability. A less sensitive test such as a flow cone apparatus may better address whether the measured increase in viscosity has any practical significance.

CHAPTER 6

SUGGESTED CONSTRUCTION SPECIFICATION REVISIONS

SUMMARY

The Portland Cement Association (PCA) segments cement treatment into three categories: Cement-Modified Soil (CMS), Cement-Treated Base (CTB), and Full-Depth Recycling (FDR). The goal of CMS differs substantially from CTB or FDR; this project evaluated topics that most relate to CTB and FDR specifications. Based upon the results presented in Chapters 1-5, the following modifications specific to cement slurry operations are recommended for inclusion into the PCA's existing suggested construction specifications for CTB and FDR:

- Add a section on mixture design within the Materials section, recommending laboratory procedures incorporate a compaction delay time between preparing the soil-cement mixture and compaction.
- Allow the requirement for continuous agitation of slurry to be removed with approval of the engineer, if an approved suspension aid is used.
- Extend the allowable time from first contact of cement with water to application on the soil from 60 minutes to 90 minutes.

MIXTURE DESIGN

The following wording is suggested for a new Mixture Design section. This would be a new section 4.7 and 2.5 in the CTB and FDR specifications, respectively:

The engineer will determine the mixture design, including the target cement content and optimum moisture content, to produce a stabilized mixture meeting the strength requirements shown on the plans. Compaction density and optimum moisture content will be determined with ASTM D 558, and compressive strength will be determined with ASTM D 1633, except all soil-cement mixtures shall be covered and allowed to stand for not less than 55 minutes but not more than 65 minutes prior to forming a specimen by compacting the prepared soil-cement mixture in the mold.

If the proposed mix design is developed by the contractor or there is a suggested change to the mix design, it must be developed in accordance with ASTM D 558 and ASTM D 1633 as noted above and submitted to the engineer for approval at least two weeks prior to construction. This mix design shall include details on material gradation, cementitious materials, and required moisture and density to be achieved during compaction.

CEMENT PROPORTIONING

The proposed new wording for cement proportioning in section 5.3 of the CTB specification follows:

The cement meter for central-plant mixing and the cement spreader for in-place mixing shall be capable of uniformly distributing the cement at the specified rate. Cement may be added in a dry or slurry form. If applied in slurry form, the slurry mixer and truck shall be capable of completely dispersing the cement in the water to produce uniform slurry, and shall continuously agitate the slurry once mixed unless an approved suspension aid is used.

The proposed new wording for cement proportioning in section 3.3 of the FDR specification follows:

Cement may be added in a dry or slurry form. If applied in slurry form, the slurry mixer and truck shall be capable of completely dispersing the cement in the water to produce uniform slurry, and shall continuously agitate the slurry once mixed unless an approved suspension aid is used.

APPLICATION OF CEMENT

The proposed new wording for application of cement in section 6.3.3 of the CTB specification follows:

The specified quantity of cement shall be applied uniformly in a manner that minimizes dust and is satisfactory to the engineer. If cement is applied as slurry, unless an approved retarding admixture is used, the time from first contact of cement with water to application on the soil/aggregate shall not exceed 90 minutes. The time from cement placement on the soil/aggregate to start of mixing shall not exceed 30 minutes.

The proposed new wording for application of cement in section 4.2.3 of the FDR specification follows:

The specified quantity of cement shall be applied uniformly in a manner that minimizes dust and is satisfactory to the engineer. If cement is applied as slurry, unless an approved retarding admixture is used, the time from first contact of cement with water to application on the soil shall not exceed 90 minutes. The time from cement placement on the soil to start of mixing shall not exceed 30 minutes.

OTHER RECOMMENDATIONS

The first sentence of the second paragraph in Mixing and Placing (section 6.1.2 and 4.1.2 in the CTB and FDR specifications, respectively) should be reworded as follows to avoid conflict with the time requirement allowed for finishing:

The operation of cement application, mixing, spreading, and compacting shall be continuous and completed within 2 hours from the start of mixing.

In addition, the following guidelines on cement slurry proportioning and testing for the presence of sulfates in soils/aggregates are recommended for consideration in the guidelines for CMS, CTB, and FDR:

Slurry Proportioning

When performing cement treatment with slurry, the water-cement ratio should be considered from a performance, workability, and economic standpoint.

- If it is known in advance a project will use cement slurry, consider using slurry in the mix design phase with a percentage solids representative of the expected field product. In some cases, the slurry percentage solids can impact the performance of the stabilized material.
- In the field, select a percentage solids considering the following factors:
 - In-situ moisture content of soils/aggregates: the cement slurry must provide water to bring materials' water content up to, or just shy of, optimum.
 - Balancing workability and runoff: the cement slurry must be fluid enough to be workable with field application equipment yet if possible provide minimal risk of runoff and ponding.
 - Economics: it generally makes the most economic sense to haul slurry with as high a percentage solids as practical.

Sulfates

Sulfates present in soils and aggregates can interfere with successful stabilization by reacting with calcium (provided by the stabilizer) and forming expansive minerals, resulting in material swelling. If project soils/aggregates contain more than 3000 ppm sulfates, additional investigations should be performed to determine a course of action for stabilization.

CHAPTER 7

CHEMISTRY OF CEMENT-MODIFIED SOILS

SUMMARY

The researchers selected two soils to evaluate the chemical changes cement hydration has on the soil. The first soil was an artificial soil composed of 40 weight percent Gonzales Bentonite (smectite) and 60 weight percent quartz sand (Ottawa sand). The second soil was a moderate plasticity (PI= 35) soil from State Highway 6 in south Texas.

BACKGROUND SOIL INFORMATION

We measured physical and chemical properties of the natural soil to determine if there were any constituents that may affect chemical stabilization of the soil. We determined the plasticity, organic carbon content, sulfate content, and pH which are shown below (Table 7.1). There was no need to measure sulfates and organic carbon content for the artificial soil because we did not add these constituents to it. The sulfate concentration for SH 6 is an average of six samples; the measured sulfate concentrations are well within the limits of acceptable levels (up to 3000 ppm) for stabilization with calcium-based additives (Petry and Little, 1992; Harris et al., 2004). The percentage of organic carbon is below the one percent threshold cited in the literature as the level that is detrimental to stabilization with calcium-based additives (Little 1995).

Table 7.1. Physical and Chemical Properties of Soils.

Soil Name	Plastic Limit	Liquid Limit	Plasticity Index	Organic C %	Sulfate ppm	pH
Artificial	19	55	36	NA	NA	
SH 6	22	57	35	0.55	389	5.2

NA = Not Applicable

For the artificial soil, we determined the optimum moisture/density relationship using no stabilizer. The artificial soil maximum density was 103pcf at a moisture content of approximately 18 percent (Figure 7.1). We made moisture-density curves for the soil from SH 6 with no stabilizer and again with 5 percent cement (Figure 7.2). The addition of cement reduced the maximum dry density (by ~1.5 pcf) and increased the optimum moisture content (by ~4%) for the soil from SH 6.

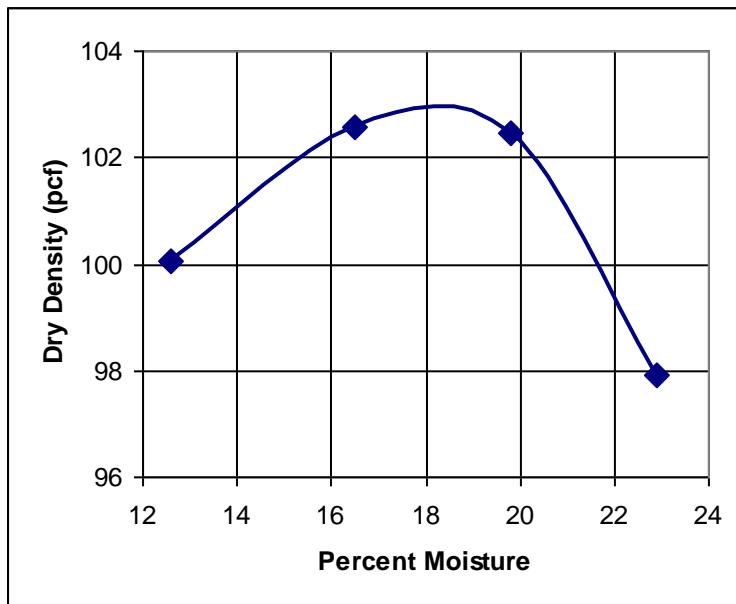


Figure 7.1. Moisture/Density Relationship for the Artificial Soil with No Stabilizer.

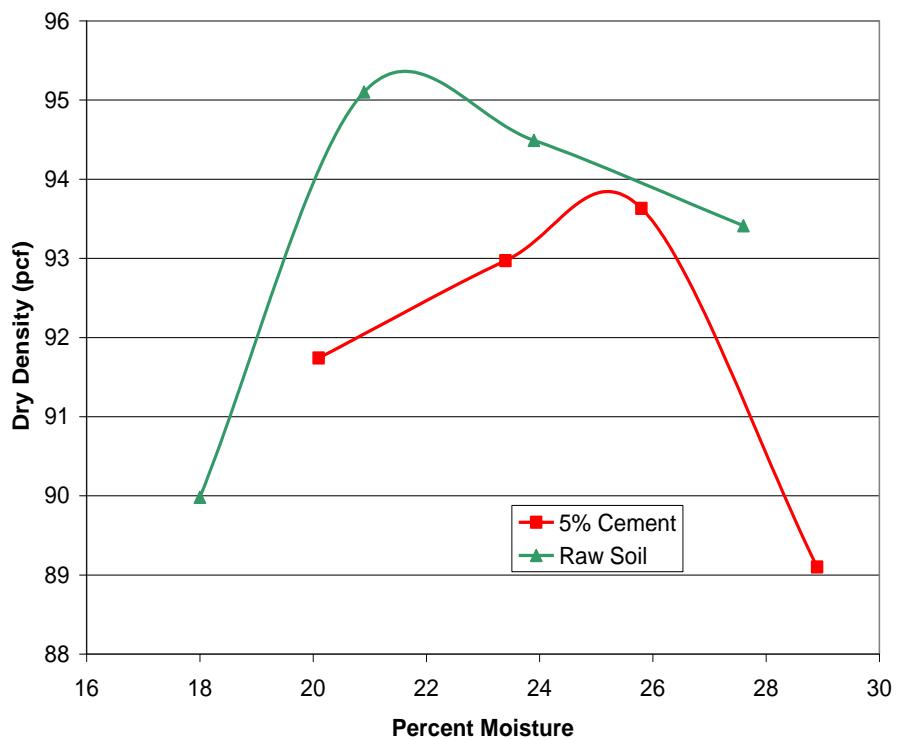


Figure 7.2. Moisture/Density Relationship for the SH 6 Soil.

To determine the optimum cement content we added different amounts of cement to each soil and compacted the soil in accordance with ASTM D 698. We then measured the strength gain following a 7-day moist cure plus a 10-day capillary rise for a total cure time of 17 days. The stabilized soil had to show a 50 psi strength increase over the unstabilized soil. All of the unstabilized samples failed during the capillary soak. The optimum cement contents were 5 percent for the SH 6 soil and 4 percent for the artificial soil. We used the Eades & Grim procedure to determine the optimum lime content for each soil as well. The optimum lime content for the SH 6 soil was 5 percent and 4 percent for the artificial soil.

TESTING PLAN

After we determined the optimum cement and lime contents, we added cement to each soil at 5 weight percent for the SH 6 soil and 4 weight percent for the artificial soil. To determine what chemical reactions were occurring during the first 24 hours, we mixed 5 weight percent cement into approximately 100 g of the air dry soil to ensure homogeneous mixing and then we added double-distilled water to bring the mix to the optimum moisture content. We immediately placed about 15 g into a 40 ml centrifuge tube and soaked it in liquid nitrogen for 5 minutes until the nitrogen stopped boiling. We then placed the centrifuge tube into a freeze dryer flask and freeze dried for 72 hours. This procedure should stop any chemical reaction at the time that the mixture is totally frozen. We froze the samples at times of approximately 10 minutes, 2 hours, 6 hours, and 24 hours. After freeze drying, we crushed the freeze-dried mixture in an agate mortar and pestle and passed the material through a # 325 sieve to run in the differential scanning calorimeter (DSC). We did not crush certain portions of the freeze-dried mixture so we could analyze them with a scanning electron microscope (SEM). The pieces saved for SEM analyses were placed in a dessicator until analyses were made.

We also measured pH on subsamples at the time samples were being frozen and placed on the freeze dryer (Table 7.2). Note how the pH rises from 10 minutes to 6 hours, but it drops at 24 hours with 4 percent cement, which is probably due to calcium hydroxide reacting with the clay. Note how the 8 percent cement retains a high pH because there was enough calcium hydroxide in the system to satisfy the exchangeable sites with the smectite with some left over to maintain the pH. The SH 6 soil has a similar pH response as the artificial soil indicating that the artificial soil is a good representation of a natural soil.

Table 7.2. pH of Cement-Treated Samples with Time.

Sample Name	Cement %	Time Cure	pH
Artificial Soil	4	10 min.	10.9
Artificial Soil	4	2 hrs.	11.3
Artificial Soil	4	6 hrs.	11.4
Artificial Soil	4	24 hrs.	10.7
Artificial Soil	8	10 min.	12
Artificial Soil	8	2 hrs.	12.3
Artificial Soil	8	6 hrs.	12.4
Artificial Soil	8	24 hrs.	12.3
SH 6 Soil	5	10 min.	11.7
SH 6 Soil	5	2 hrs.	12
SH 6 Soil	5	6 hrs.	12.1
SH 6 Soil	5	24 hrs.	11.8

SCANNING ELECTRON MICROSCOPE ANALYSES

We analyzed the cement reaction products with a JEOL 6400 scanning electron microscope at a 15 kV beam current and a working distance between 11 and 14 mm. A Princeton Gammatech energy dispersive spectrometer (EDS) is attached to the SEM for elemental analysis. We looked at samples cured for 10 minutes and 2, 6, and 24 hours to see if there were any visual differences in clay morphology, cement crystal morphology, or clay/cement interactions. After just 10 minutes curing, there were not any visual differences in clay morphology (Figure 7.3). You can see the nice crenulated texture of montmorillonite in Figure 7.3. The accompanying EDS pattern shows elevated calcium levels in the clay indicative of calcium uptake from the cement (Figure 7.4).

After 2 hours curing the cement had formed a coating on the clay not evident in the 10 minute cure samples (Figure 7.5). One can see hydration products after 2 hours curing. The elemental distribution (EDS pattern, Figure 7.6) for the paste coating the clay shown in Figure 7.5 is almost identical to the elemental distribution in the sample cured for 10 minutes (Figure 7.4).

Samples cured for 24 hours show more extensive development of coatings on the clay and larger crystalline hydration products (Figure 7.7). The arrow shows the location of the clay under the cement hydration product. After 24 hours curing the individual hydration products were large enough to resolve with the SEM; however they were still relatively anhedral, which is expected after such short curing times (Figures 7.9 and 7.10). Lea (1970) quotes depths of hydration for Portland cement particles at 7 μm for curing of 24 hours, so cement particles over 7 μm will still not be fully hydrated, which is roughly 75 percent of the cement clinker.

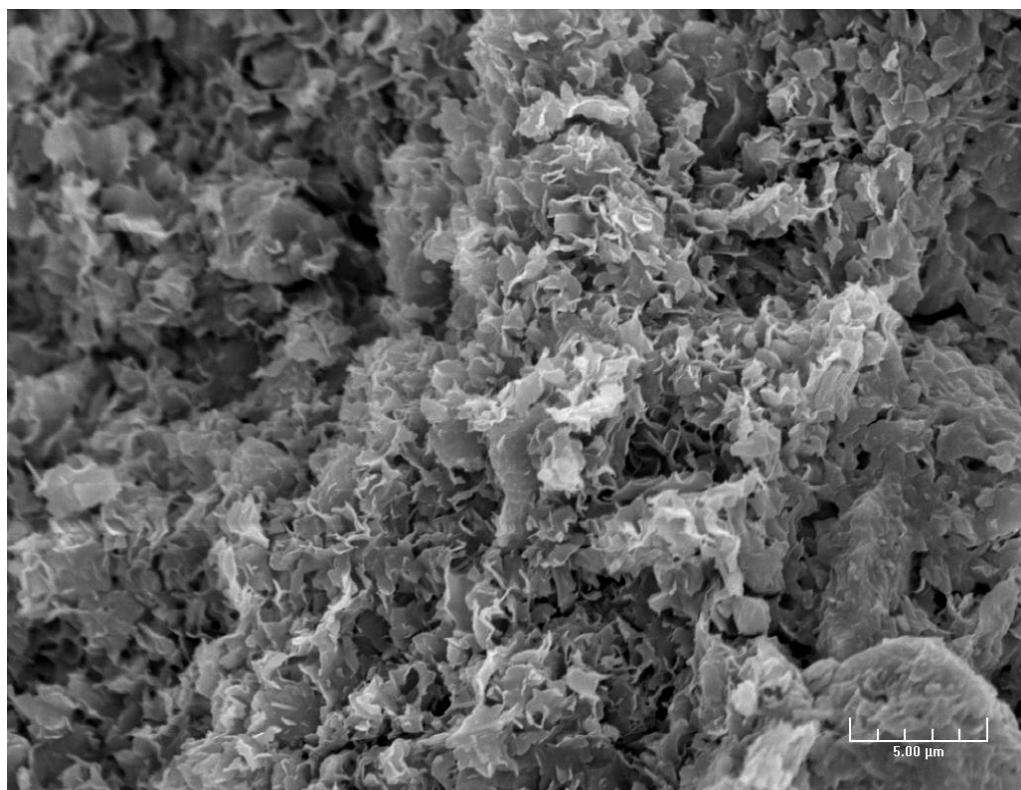


Figure 7.3. SEM Image of Bentonite at Cure Time of 10 Minutes.

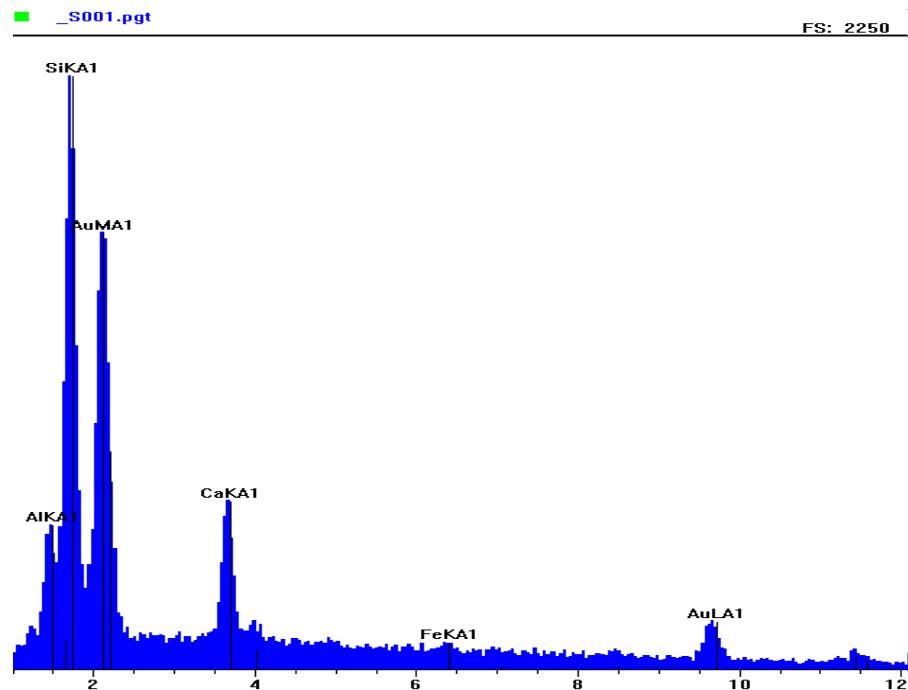


Figure 7.4. EDS Pattern of Clay Cured for 10 Minutes.

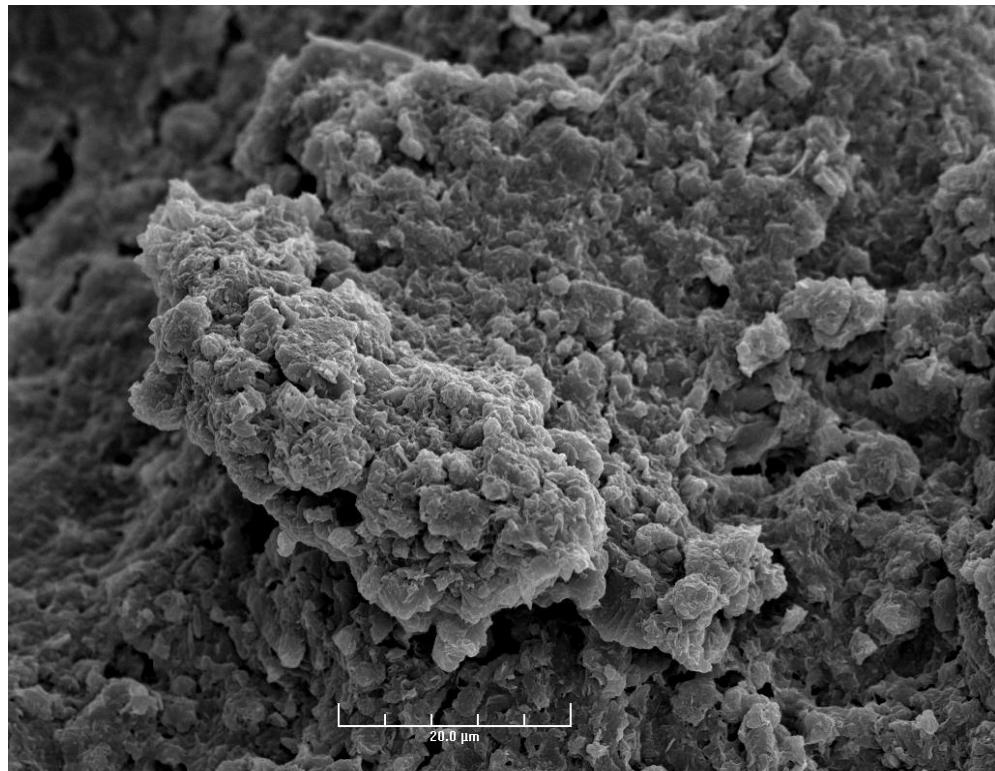


Figure 7.5. SEM Image of Paste Coating Clay at Cure Time of 2 Hours.

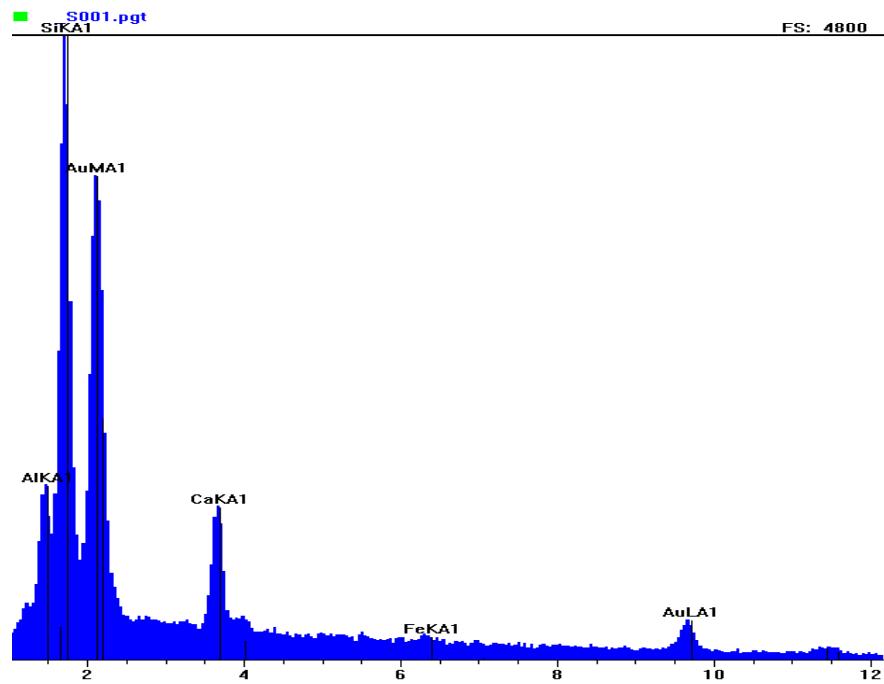


Figure 7.6. EDS Pattern of Paste Coating on Clay Cured for 2 Hours.

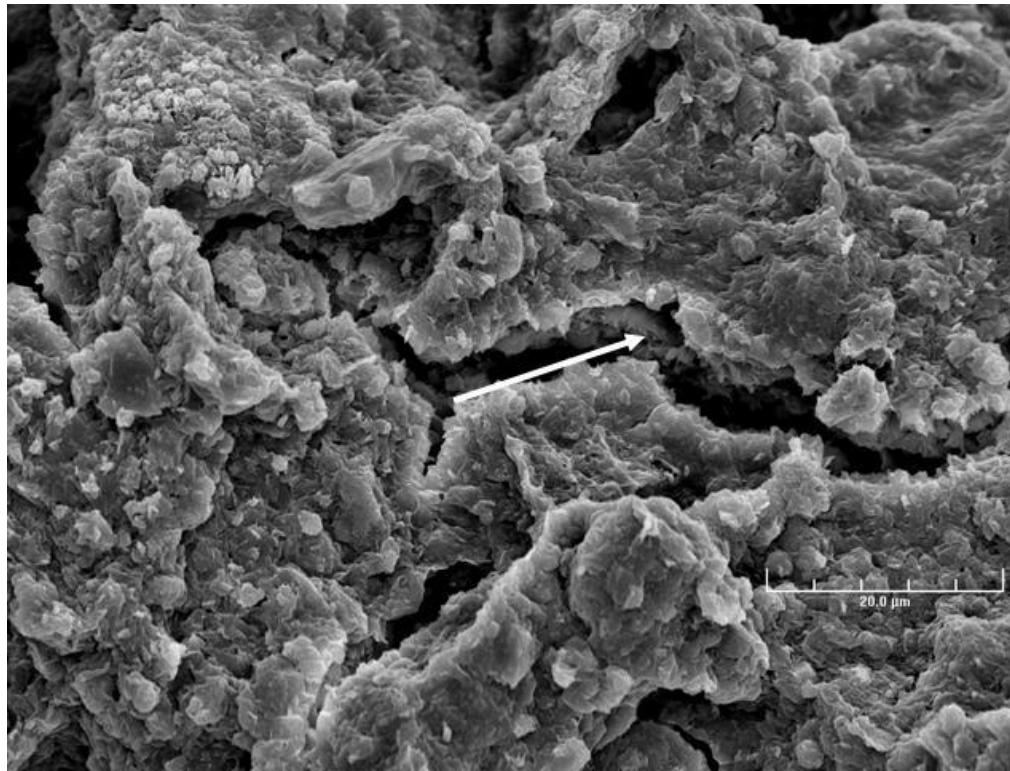


Figure 7.7. SEM Image of Paste Coating Clay at a Cure Time of 24 Hours.

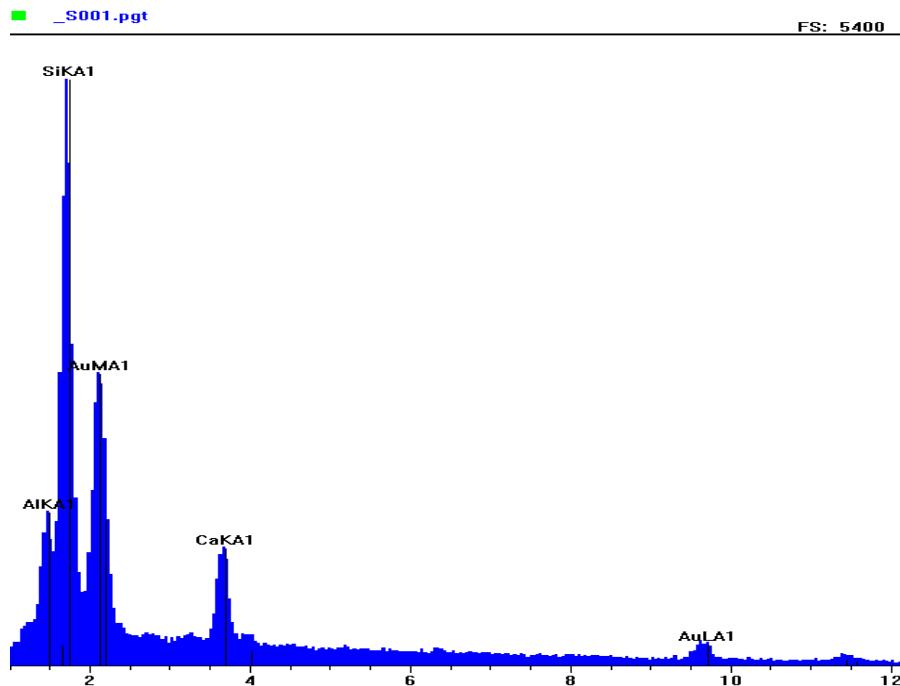


Figure 7.8. EDS Pattern of Paste Cured for 24 Hours.

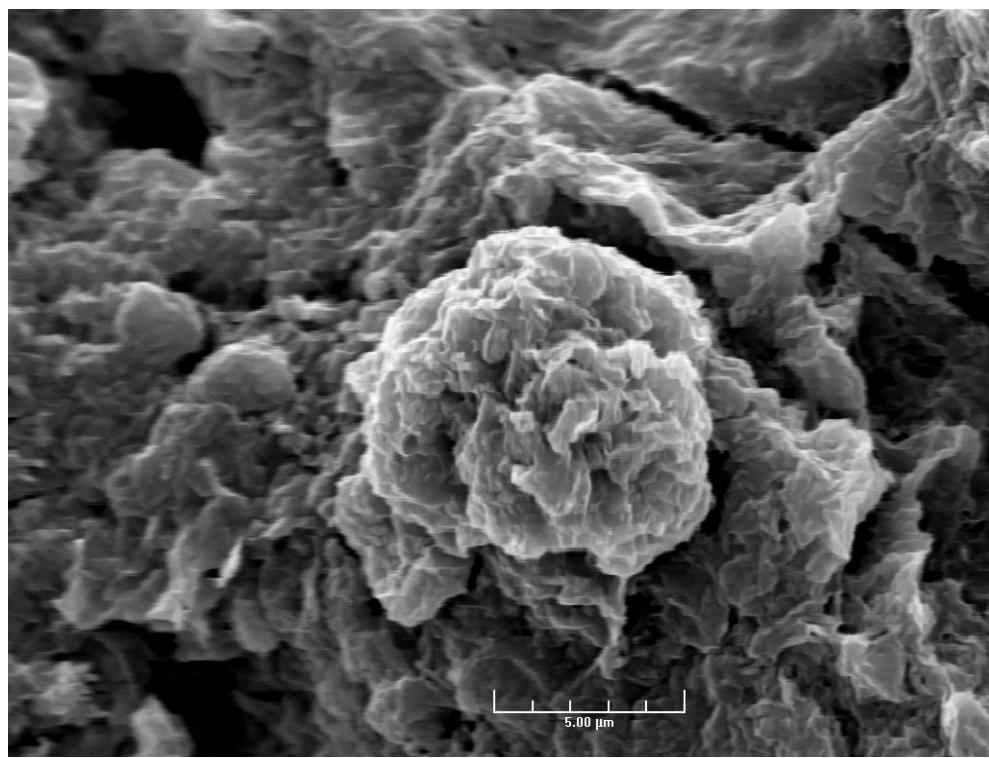


Figure 7.9. SEM Image of Cement Hydration Product after 24-Hour Cure.

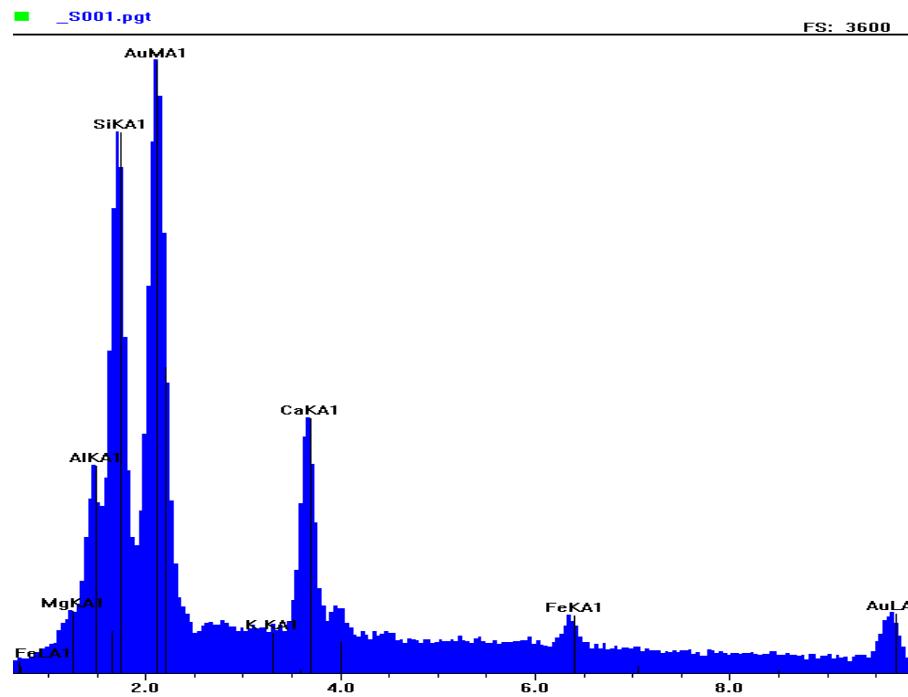


Figure 7.10. EDS Pattern of Cement Hydration Product in Figure 7.9.

DIFFERENTIAL SCANNING CALORIMETER RESULTS

TTI researchers used a SDT Q600 differential scanning calorimeter to measure heat flow (energy) as a function of temperature in samples cured for times ranging from 10 minutes to 24 hours to see what reaction products can be observed. This technique involves heating a sample at a constant rate to over 1000°C and measuring the heat transfer in the sample. This technique has two advantages over X-ray diffraction (XRD): (1) it works well in samples that are amorphous because XRD will not detect these phases, and (2) it can detect minute changes in a sample that are not always observed with XRD. If a phase is less than 5 percent in a sample it may not be detected by XRD.

We ran the freeze-dried samples at a rate of 10°C/minute up to a final temperature of 1030°C. We graphed the heat flow versus temperature and did not see any big changes, so we used software included with the instrument to calculate the second derivative of the data. The second derivative does two things to aid interpretation: (1) it normalizes the data for weight differences in samples so quantitative estimations regarding amounts of different materials can be made, and (2) it will show very small endothermic and exothermic reactions not observable in the original graph. The second derivative data show upward peaks as endothermic reactions and downward peaks as exothermic reactions. Generally phase transitions, dehydration, dehydroxylation, and some decomposition reactions produce endothermic peaks, while oxidation, recrystallization, and certain decomposition reactions generate exothermic peaks (Karathanasis, 2008).

We compared the second derivative curves of untreated bentonite with curves of bentonite treated with 4 weight percent Portland cement and 8 weight percent Portland cement. Cure times ranged from 10 minutes to 2, 6, and 24 hours.

Sha et al. (1999) identified three main reaction products (C-S-H, Ca(OH)₂, and CaCO₃) in ordinary Portland cement that they analyzed using DSC. We were able to identify these same reaction products in our samples.

We observed four trends regarding chemical reactions of the paste with the bentonite during the first 24 hours. First, we see a consistent decrease in the calcium silicate hydrate (C-S-H) dehydration peak (~130°C) as the paste cures for longer time periods. The C-S-H dehydration peak in Figure 7.11 is much larger than the same peak in Figure 7.12 indicating more C-S-H being present at early hydration ages. The second observation is an increase in calcium hydroxide (Ca(OH)₂) dehydroxylation (peak at ~430°C) as curing time increases which indicates more portlandite is produced as the cement cures up to 24 hours. Third is the decarbonation of calcium carbonate (CaCO₃) between 650° and 700°C. Karathanasis (2008) reports the decarbonation peak for calcite at 900°C but the calcium carbonate mineral we observe here is probably vaterite. Cole and Kroone (1959-60) reported that vaterite forms metastably in cement paste and later alters to aragonite and finally to calcite. Our carbonate endotherm is partially masked by a large endotherm from the degradation of the montmorillonite. The last trend we observed is the retardation of the recrystallization reaction of montmorillonite at approximately 1000°C. One can see in Figure 7.11 that there is not an obvious exotherm at 1000°C which is present in untreated montmorillonite. However, after 24 hours of curing the

recrystallization peak reappears but it is much smaller than for an unstabilized montmorillonite. Sha and Pereira (2001) observed a decrease in the size and temperature of the recrystallization peak of metakaolin as Portland cement was mixed with the metakaolin. They attributed this to the reaction of calcium hydroxide from the cement with the metakaolin which reduces the amount of amorphous metakaolin.

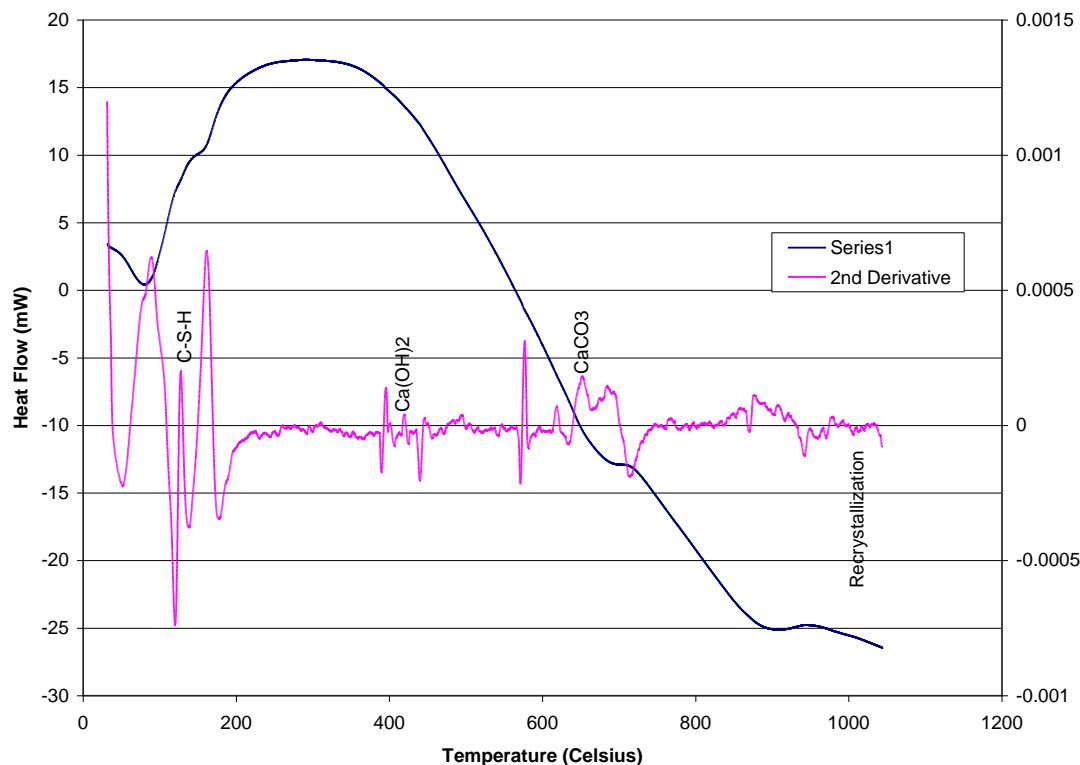


Figure 7.11. DSC Data for 8 Percent Cement Cured for 10 Minutes.

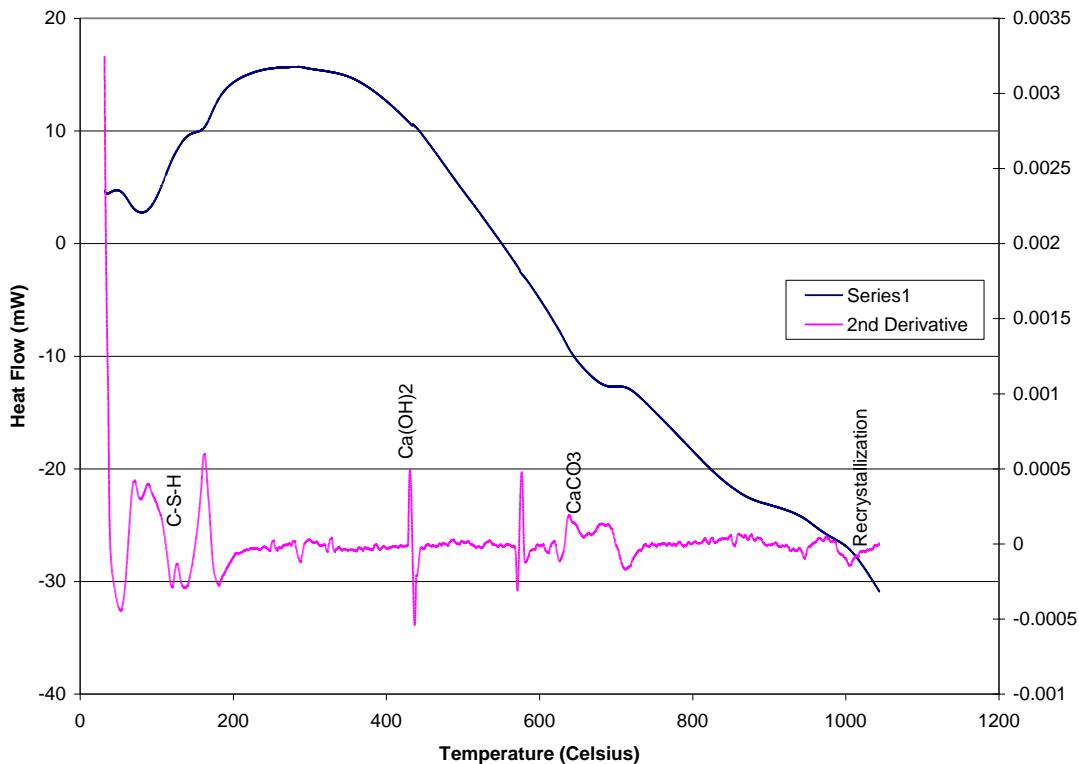


Figure 7.12. DSC Data for 8 Percent Cement Cured for 24 Hours.

X-RAY DIFFRACTION RESULTS

We analyzed some of the samples with XRD to try and confirm the reaction products that we were seeing in the DSC data. As stated earlier, XRD cannot see amorphous material. Much of the C-S-H and calcium hydroxide are amorphous so the X-ray pattern will not detect these phases (Ray, 2002). If a crystalline phase constitutes less than 5 percent of the sample, XRD may not detect it.

We are presenting two XRD patterns (Figures 7.13 and 7.14) that illustrate the two extremes as far as the crystalline phases are concerned. We present data from the 8 percent cement-treated bentonite cured for ~10 minutes and the 8 percent cement-treated bentonite cured for 24 hours. There are some notable differences in these two patterns. First, there is a high percentage of alite/belite (A/B) in the sample cured for 10 minutes. Alite and belite are dominant phases in Portland cement clinker (St John et al., 1998). The sample cured for 24 hours shows a drastic decrease in alite/belite due to these phases hydrating to form cementitious products (Figure 7.14). We detected gypsum (G) in the 10-minute cured material but it could not be definitely identified in the 24 hour cured material. We do not see any evidence of portlandite (P) in the 10-minute cured material but it is definitely present in the 24-hour cured material. Finally, we observe a shift in the montmorillonite (M) basal spacing

from 15.3\AA to 14.1\AA , which may be due to ion exchange reactions. Watson et al. (2009) report the conversion of Mg-montmorillonite to K-montmorillonite by reaction of cementitious water with bentonite. That could explain the decrease in the basal spacing of the montmorillonite in our samples. Quartz (Q), feldspar (F) and cristobalite (C) are all present in the bentonite and appear unchanged in the reacted bentonite/cement mixtures.

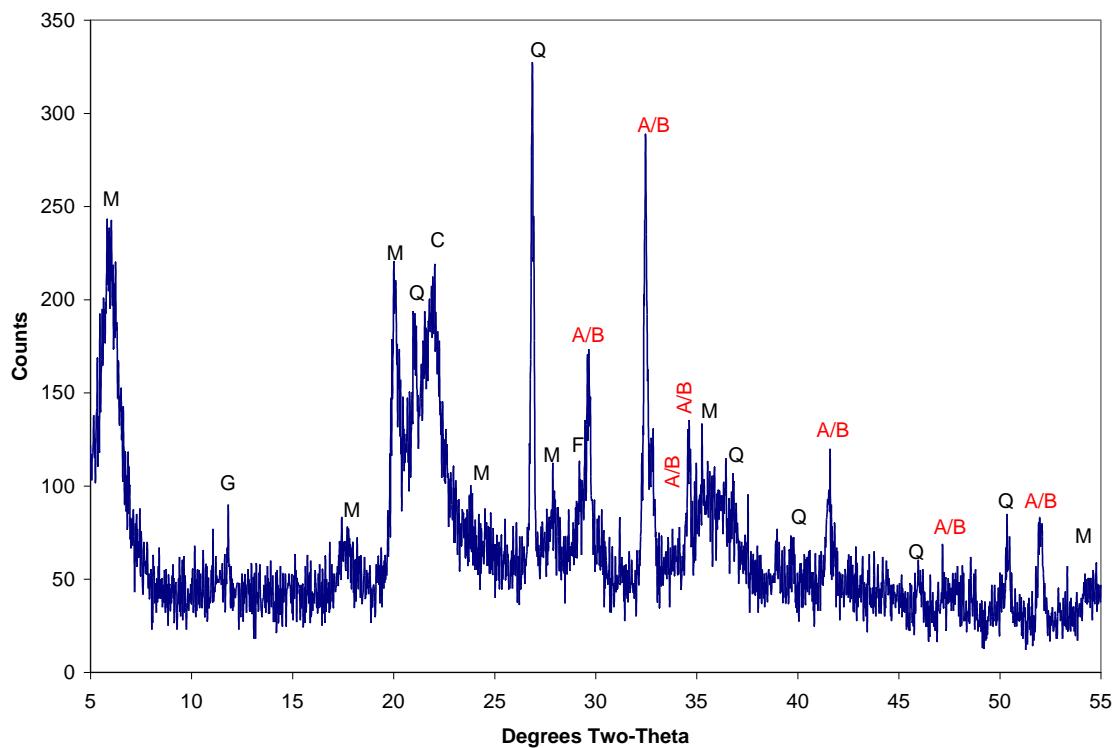


Figure 7.13. XRD Pattern of Bentonite with 8 Percent Cement Cured for 10 Minutes.

We did not see any evidence of C-S-H in the XRD patterns, even though the DSC data showed C-S-H. However C-S-H was not expected in the XRD patterns since C-S-H is generally amorphous until extended aging at which time the concentration may still be too low for the XRD to detect. We also did not see the calcium carbonate mineral vaterite, which was observed in the DSC data.

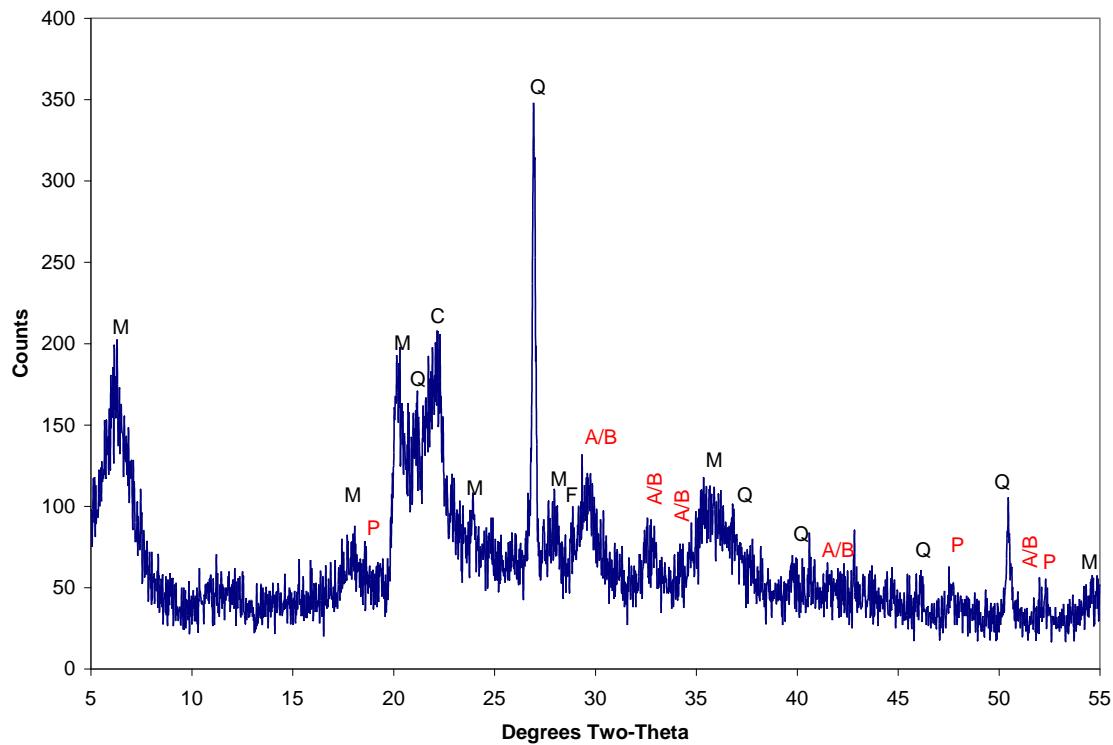


Figure 7.14. XRD Pattern of Bentonite with 8 Percent Cement Cured for 24 Hours.

CONCLUSIONS

The following observations were made for bentonite/cement mixtures cured up to 24 hours:

- The montmorillonite structure is apparently modified at an early age. Evidence includes high calcium observed in the SEM data for the 10-minute cure, DSC results show hindrance of the recrystallization reaction for montmorillonite at $\sim 1000^{\circ}\text{C}$, and XRD data show a shift in the basal spacing for the montmorillonite, suggesting a structural modification.
- We see less C-S-H as the bentonite/cement mixture cures. Evidence is the decreased peak intensity of the peak at $\sim 130^{\circ}\text{C}$ that represents the loss of water from C-S-H. SEM analyses show coatings forming on the montmorillonite after only 2 hours cure.
- We see more calcium hydroxide (portlandite) as the bentonite/cement mixture cures. DSC data show larger peaks at $\sim 430^{\circ}\text{C}$ (which is dehydroxylation of the portlandite) as the curing time is increased up to 24 hours. The XRD data show portlandite after a cure time of 24 hours.
- After 24 hours curing there is still a measurable amount of unreacted cement clinker. Evidence is the persistence of alite/belite in XRD patterns of the bentonite/cement mixture after a 24-hour cure.
- Calcium carbonate is an early reaction product in the bentonite/cement mixture. Decarbonation of calcium carbonate (vaterite?) was observed after a 10-minute cure in DSC data and appeared in all DSC data up to 24 hours.

CHAPTER 8 **CONCLUSIONS AND RECOMMENDATIONS**

CONCLUSIONS AND RECOMMENDATIONS FOR USING CEMENT SLURRY

Several industry companies can provide cement slurry stabilization. Application of cement by slurry can eliminate dusting and provides performance at least meeting, and in some cases exceeding, the performance provided by dry application of cement.

- Slurry age does not impact performance for slurry ages up to 2 hours.
- The slurry percentage solids can slightly impact performance in some cases.
- The compaction delay time between mixing cement into the soil and completion of compaction strongly impacts performance, with longer delay times resulting in reduced mechanical properties.

In light of these findings, the following recommendations as were detailed in Chapter 6 are proposed for revising the PCA's construction specifications for CTB and FDR:

- Add a section on mixture design within the Materials section, recommending laboratory procedures incorporate a compaction delay time between preparing the soil-cement mixture and compaction.
- Allow the requirement for continuous agitation of slurry to be removed with approval of the engineer, if an approved suspension aid is used.
- Extend the allowable time from first contact of cement with water to application on the soil from 60 minutes to 90 minutes.

CONCLUSIONS AND RECOMMENDATIONS FROM CHEMICAL INVESTIGATION OF CEMENT-MODIFIED SOIL

Analysis of the chemistry of cement-modified soils at curing ages up to 24 hours with DSC, SEM, and XRD revealed:

- Calcium carbonate is an early reaction product in the bentonite/cement mixture.
- The montmorillonite structure is apparently modified at an early age. XRD data show a shift in the basal spacing for the montmorillonite, suggesting a structural modification.
- As the bentonite/cement mixture cures, less C-S-H was observed and more calcium hydroxide (portlandite) was present.
- SEM analyses show coatings forming on the montmorillonite after only 2 hours cure.
- After 24 hours curing there is still a measurable amount of unreacted cement clinker.

These results show, in a controlled setting, chemical reactions taking place in cement-modified bentonite soil as early as 10 minutes after treatment. While the basic reactions taking place are assumed to also occur in field construction, other soil constituents and soil texture can affect reaction kinetics or even interfere with reaction products forming (such as organic matter).

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