Accelerated Curing of Fly Ash–Lime Soil Mixtures

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The morphological, mineralogical, and stress-strain properties of compacted bentonite-fly ash-lime, bentonite-fly ash, and bentonite-lime cured at 23°C and 50°C were compared. The development of microstructure and cementitious crystals was observed by scanning electron microscopy, energy dispersive spectral analysis, and x-ray diffractometry. The elastic moduli and compressive strengths were obtained from unconsolidated, undrained triaxial and unconfined compression tests. Curing at 50°C increased the rate of strength development. Similar elastic moduli were obtained for specimens cured for one day at 50°C and 28 days at 23°C. However, strain at failure after one day of curing at 50°C was considerably higher than that obtained after 28 days of curing at 23°C. The samples cured at 50°C yielded higher compressive strengths relative to the samples cured at 23°C. Cementitious minerals formed at both curing temperatures were similar except that the cementitious minerals observed after 50°C curing had a higher degree of crystallinity. Short-term, high-temperature curing may be substituted for the prediction of long-term design parameters; however, the relationship between short-term, high-temperature curing versus long-term, low-temperature curing must be statistically established by further testing.

Laboratory tests for determining compressive strengths and elastic moduli use compacted and cured mixtures of soils and additives, such as fly ash or lime. Curing time, temperature, proportions of soil and additives, and compaction water content are important factors in this evaluation process. This study concentrates on the relationship between curing temperature and curing period. Higher curing temperatures generally increase the rate of strength development; thus elevated curing temperatures and shorter curing periods are sometimes used to predict engineering properties developed after longer curing periods at ambient temperatures.

The effects of elevated curing temperatures on the morphology, mineralogy, and stress-strain properties of test specimens must be studied to evaluate the viability of design parameters derived from tests performed on specimens cured at accelerated (one day at 50°C) temperatures. It is also essential to establish the relationship (or the similarity) of stress-strain properties, the mineralogy, and the morphology of mixtures that are subjected to accelerated curing before comparisons can be made of the properties of the two with any degree of confidence.

Thus the main objectives of this study were to compare the observed morphological, mineralogical, and stress-strain properties of fly ash-lime stabilized bentonite cured at 23°C and 50°C for 1, 28, 90, and 180 days and to use the results to evaluate the feasibility of an accelerated curing procedure.

A laboratory investigation was undertaken using bentonite-lime, bentonite-fly ash, and bentonite-fly ash-lime cured at 23°C and 50°C for periods of 1, 28, 90, and 180 days. As a follow-up study to provide results from even longer curing periods, bentonite-fly ash-lime mixtures were also cured for about 600 days. Unconsolidated, undrained triaxial tests were conducted to determine the stress-strain relationship. Mineralogical and morphological observations were accomplished by using x-ray diffractometry, scanning electron microscopy, and energy dispersive spectroscopy.

Bentonite, which is essentially a monomineralic montmorillonite, was chosen to eliminate variable mineralogical compositions associated with natural clays. For the accelerated curing test, 50°C for 24 hr was selected for its practicality. Other temperature and time combinations may produce similar results, but 50°C is probably the maximum temperature because other types of cementitious minerals may occur at higher temperatures.

The reactions between clay and lime cured at elevated temperatures have been studied by a number of researchers (*1–7*). The reaction products vary with the type of clay, reaction conditions (especially temperature), curing time, mixture proportions, and whether slurries, pastes, or compacted samples are used. Commonly the reaction products observed are composed of calcium silicate hydrates (C-S-H) and calcium aluminate hydrates. Poorly crystallized C-S-H (I) is the most frequently reported mineral (*1,2,8,7*). Tetracalcium aluminate thirteen hydrate is often observed at room temperature (*2,9,10*), whereas cubic tricalcium aluminate hexahydrate is more common in the mixtures that are cured at elevated temperatures (*3–5,11*).

The compressive strengths of clay-lime mixtures cured at elevated temperatures are higher than the compressive strengths of those cured at room temperatures for short and long time periods (*1,2*). In montmorillonite-lime mixture, the same cementitious minerals were observed at room and elevated temperatures. However, in the latter case, the compounds developed higher crystallinities and the mixtures had higher compressive strengths (*2*). Arabi and Wild (*1*) studied red marl–lime mixtures cured at temperatures up to 75°C. He reported higher unconfined compressive strengths for mixtures cured at elevated curing temperatures.

Anday (*12*) suggested that short-term curing at elevated temperatures simulated the long-term behavior of the lime-stabilized soils at normal temperatures. He recommended that two-day 50°C curing be used instead of long-term normal-temperature curing. However, no one has studied the response

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of combined fly ash-lime stabilization at the elevated temperatures, and because the reactions are more complex, previous results cannot be extrapolated for these mixtures.

PROCEDURE

Materials Used

The bentonite used for this study was obtained from NL Industries of Houston, Texas. Type C fly ash was obtained from the Cajun Electric Power Plant at New Roads, Louisiana. This fly ash contained a considerable amount of glassy spherical material and some free lime. The calcitic hydrated lime was obtained from the Dravo Lime Company of Baton Rouge, Louisiana.

Specimen Preparation, Curing, and Testing

Mixture proportions were determined by a variety of procedures (*13*). To determine the appropriate lime content for this study, plastic limit and pH values of different proportions of bentonite-lime mixtures were determined, and the percentage of lime that corresponded to the point at which no increase in pH or plastic limit occurred was used as the lime content for this study. Following determination of the lime content, 8 to 30 percent fly ash by dry weight was added to the bentonite-lime mixture, and compressive strengths were determined after 1 and 7 days of curing at 23°C. Increased fly ash percentages continued to produce increased compressive strengths within the range of the experiment; therefore, 20 wt% fly ash was arbitrarily selected as the highest practical amount to be included in a stabilization project. Twenty percent fly ash, 5 percent lime, and 75 percent bentonite (by dry weight) were selected as the mixture proportions. The effects of only lime or fly ash additives on microstructure development and strength were assessed by removing one additive at a time from this principal mixture, while maintaining the same dry weight ratios for the remaining materials. The mixture percentages, compaction water contents, and dry densities are presented in Table 1.

Cylindrical specimens having a diameter of 3.334 cm (1.313 in.) and a height of 7.153 cm (2.816 in.) were compacted in a Harvard miniature compaction device (using an 89 N-20 lb spring) (*14*). After compaction, all specimens were weighed and placed in practically airtight plastic bags and kept in an oven at 50°C or in a moisture-controlled room for ambient temperature curing (23°C, relative humidity greater than 95 percent) for up to 180 days. The moisture contents of cured specimens were determined before and after testing.

Compressive strength tests, with and without confining pressures, were conducted by using triaxial testing equipment at a strain rate of 0.25 mm/min (0.01 in./min). Six samples of the bentonite-fly ash-lime mixture and three samples of the other mixtures were tested for each curing temperature and time. The elastic moduli of the samples were calculated by using stress-strain values corresponding to 70 percent of the maximum deviator stress, which approximately represented the linear portion of the stress-strain curve (*15*). After the samples were fractured, representative subsamples were frozen and vacuum dried for mineralogical and textural analyses. Gold coating was used for scanning electron microscopy. X-ray diffraction (XRD) samples were prepared by a side-loading technique to obtain random orientation (*16*).

RESULTS

Stress-Strain Relationships

The relationships between unconfined compressive strength and curing time for the mixtures containing bentonite and additives are presented in Figure 1. In general, the compressive strengths for specimens cured at 50°C were higher than for those cured at 23°C. The compressive strength of the bentonite-fly ash-lime mixture reached 3947 kPa at 50°C curing after 624 days, which was 1.5 times the compressive strength of the mixture cured at 23°C for the same amount of time.

A similar response to elevated curing temperature was observed for the bentonite-fly ash mixture cured at 50°C. It developed a compressive strength of 1915 kPa after 180 days of curing, which is 1.4 times the compressive strength obtained at 23°C. The compressive strengths of the fly ash-bentonite mixtures after one day of curing at 50°C were higher than those cured at 23°C for 28 days. For samples cured at 23°C, the major portion of the compressive strength developed after the first 28 days of curing.

The strains at failure ranged from 0.92 percent to 1.65 percent for the one-day cured samples (Figure 2). After 28 days of curing, failure strains decreased to between 0.6 and 1.0 percent. No further decrease in strain was observed at normal and elevated curing temperatures. The failure strains of the samples cured at 50°C were consistently larger than those cured at 23°C. This difference, however, became negligible after 180 days of curing for most of the samples. The bentonite-fly ash-lime mixture cured at 50°C failed at an average strain of 0.98 percent, which is 1.3 times that of the 23°C cured sample after 624 days of curing. The elevated curing temperature generally produced higher failure strains for the samples containing fly ash after long curing periods, which in turn affected the elastic moduli.

TABLE 1 MIXTURE PROPORTIONS, COMPACTION WATER CONTENTS, AND DRY DENSITIES

Mixture	Proportion (%)				Dry Density (mg/m^3)
	Bentonite	Fly Ash	Lime	Water	
Bentonite + fly ash + lime	75	20	5	34	1.25
Bentonite + fly ash	79	21	0	24	1.40
Bentonite + lime	94	0	6	40	1.15
Fly ash + lime	0	80	20	20	1.65
Fly ash	0	100	0	11	1.90

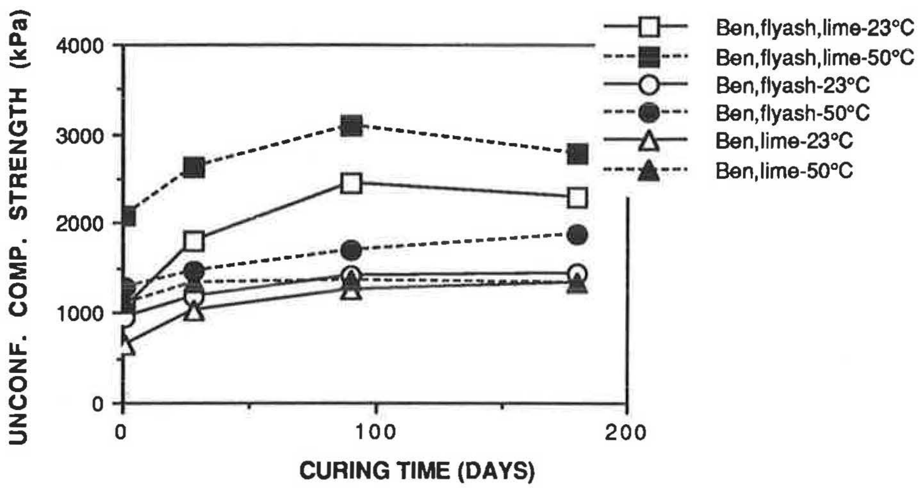

FIGURE 1 Average unconfined compressive strength (UCS) of bentonite-fly ash-lime, bentonite-fly ash, and bentonite-lime mixtures versus curing time. UCS for bentonite-fly ash-lime at 23°C and 50°C was 2600 and 3900 kPa, respectively.

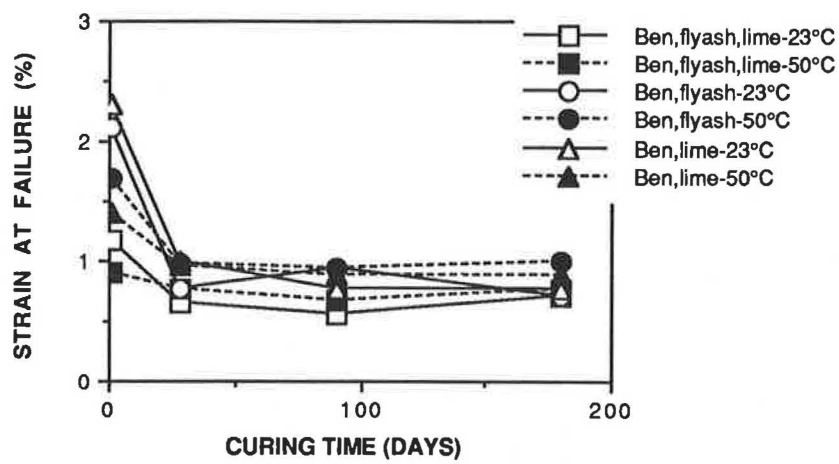

FIGURE 2 Average strain at failure for bentonite-fly ash-lime, bentonite-fly ash, and bentonite-lime mixtures versus curing time. Average strain for bentonite-fly ash-lime at 23°C and 50°C was 0.7 and 1.0 percent, respectively.

The changes in elastic moduli with time are presented in Figure 3. For the bentonite-fly ash-lime mixture, the average elastic modulus for the one-day curing period at 50°C was 349,250 kPa, which was 90 percent of the elastic modulus of the mixture cured for 28 days at 23°C; after 90 days, no significant difference was present. A sharp increase in elastic moduli was observed at 90 days of curing for both curing temperatures, which was then followed by a decrease. After 180 days of curing, the elastic moduli of the bentonite-fly ash-lime mixture were approximately twice that of bentonite-lime and bentonite-fly ash mixtures. After 90 days, the samples cured at 50°C produced comparable elastic moduli to the samples cured at 23°C.

Morphology of Cementitious Products

The morphological studies assisted in the interpretation of changes in the stress-strain relationships. In Figure 4, the fractured surface of the one-day cured (23°C) bentonite–fly ash–lime mixture contains mostly clay particles. The boundaries of the clay particles are not distinct—a feature typical of plastic materials such as montmorillonite-rich clays. After 28 days of curing at 23°C, a network of acicular crystals dominates the microstructure of the fracture surface as seen in Figure 5. These crystals are identified as C-S-H and ettringite.

The formation and the increase in the abundance of the acicular crystals are accompanied by a compressive strength increase from 1061 kPa to 1800 kPa and a decrease in strain at failure from 1.13 percent to 0.66 percent. The more rigid behavior is attributed to the formation of the three-dimensional network of rods and needles that supports the montmorillonite aggregates and the fly ash spheres, thus restricting the gliding of clay plates past one another and their relative displacement.

Figure 6 shows a representative fracture surface of the one-day cured bentonite-fly ash-lime mixture cured at 50°C. Well-developed acicular and lath-like crystals span the pores and

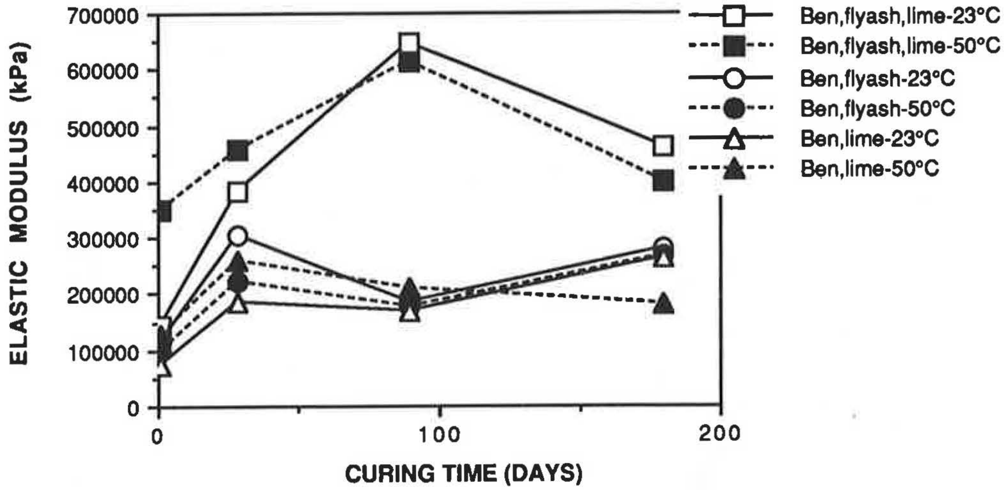

FIGURE 3 Average elastic modulus for bentonite-fly ash-lime, bentonite-fly ash, and bentonite-lime mixtures versus curing time. Elastic modulus for bentonite-fly ash-lime at 23°C and 50°C was 480 000 and 510 000 kPa, respectively.

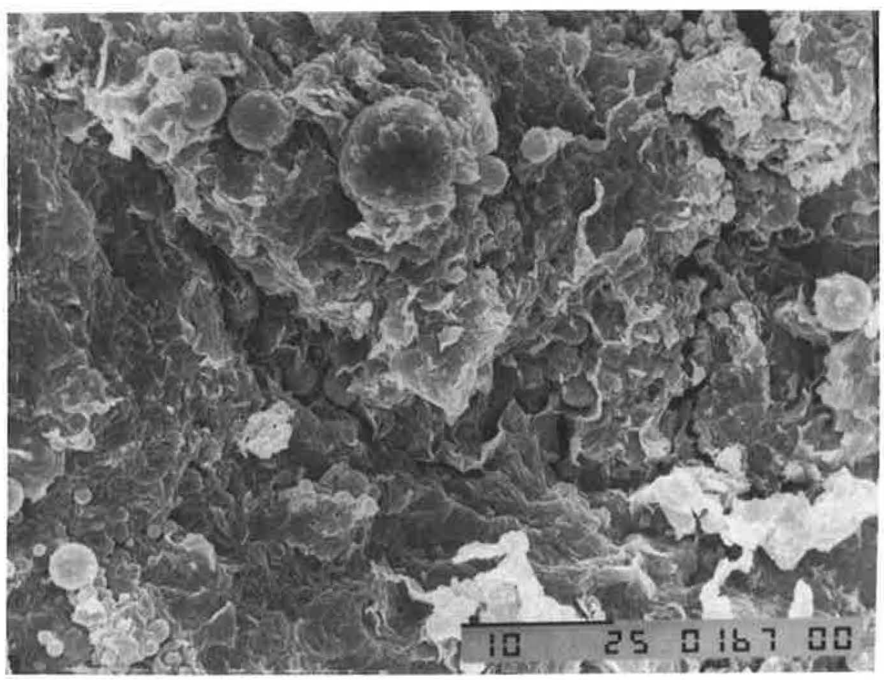

FIGURE 4 Representative fracture surface of the one-day 23°C cured bentonite-fly ash-lime sample. The fracture surface contains mostly clay particles.

support the fly ash spheres and the montmorillonite aggregates. As mentioned previously, the time of observation of the acicular crystals corresponded to a major increase in compressive strength and a decrease in failure strain. Although a quantitative measure was not possible, fewer acicular crystals were observed at 50°C curing, which may be the cause of the consistently larger failure strains at 50°C. A comparison of the morphologies of the 624-day cured samples at 23°C and 50°C supports this observation.

Figure 7 presents a representative fracture surface of the 624-day 23°C cured bentonite-fly ash-lime mixture. The fly ash spheres are embedded in a 50-μm-diameter montmorillonite aggregate and are interwoven with randomly oriented, tiny acicular crystals. At the center of the micrograph, a typical reacted shell of a fly ash grain can be seen. The mixture cured at 50°C had fewer acicular crystals; however, the degree of crystallinity of these acicular crystals was higher (Figure 8). The acicular crystals are thicker and longer and their outlines are sharper than those observed at 23°C.

The higher compressive strengths of the 50°C cured samples containing fly ash after long curing periods are attributed to the increased degree of reaction of the fly ash grains and glassy spheres found in fly ash, which provide calcium, silicon, and aluminum for the formation of more cementitious minerals.

The same morphological changes were also observed with the bentonite-fly ash mixture. In this mixture, the elevated

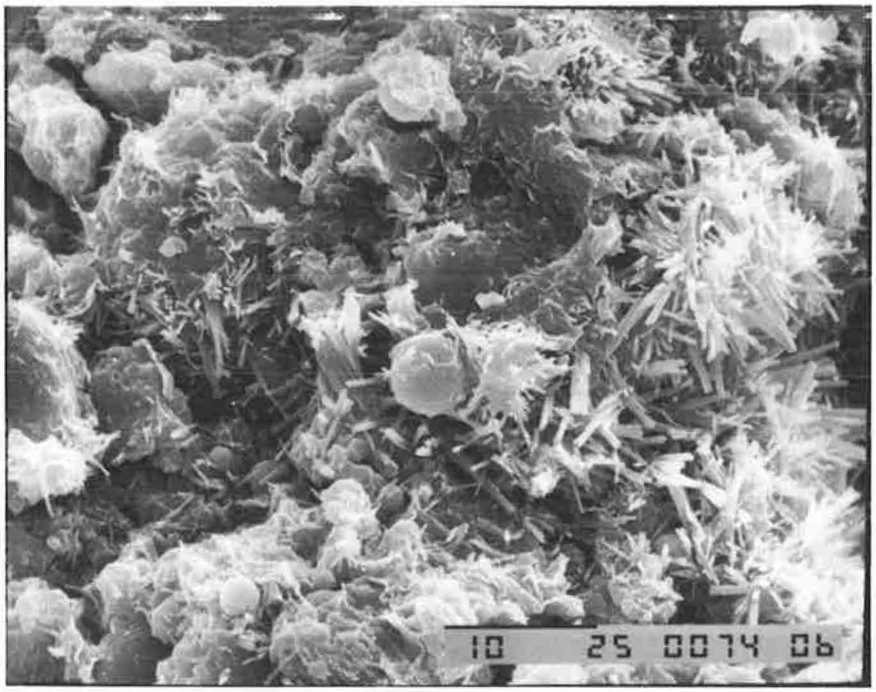

FIGURE 5 Representative fracture surface of the 28-day 23°C cured bentonite-fly ash-lime sample. Acicular crystals dominate the fracture surface.

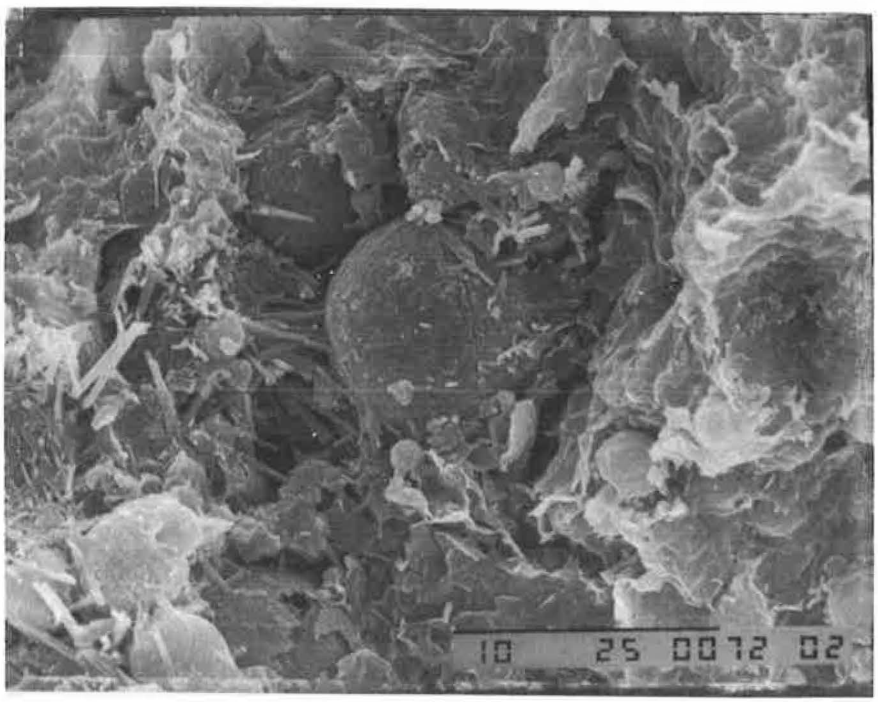

FIGURE 6 Representative fracture surface of the one-day 50°C cured bentonite-fly ash-lime sample. Acicular crystals span the pores and support the fly ash spheres and the montmorillonite aggregates.

temperature increased the rate of the reaction, and well-defined clay aggregate boundaries were observed after one day of curing at 50°C instead of 28 days of curing at 23°C. In bentonite–fly ash specimens, a larger number of reacted fly ash spheres was observed after curing at 50°C. A typical fracture surface of the bentonite-fly ash mixture cured for 180 days at 50°C is presented in Figure 9. The fly ash grain in the center has undergone more reaction relative to the fly ash grains observed at 23°C.

Mineralogy

The mineralogical observations support the observations made with the scanning electron microscope. The identifications of

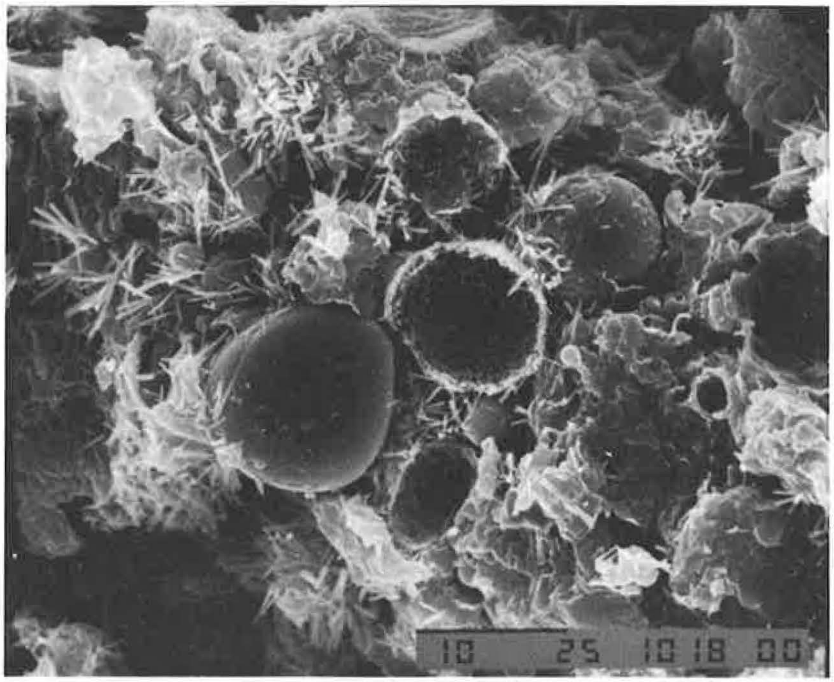

FIGURE 7 Representative fracture surface of bentonite-fly ash-lime cured for 624 days at 23°C.

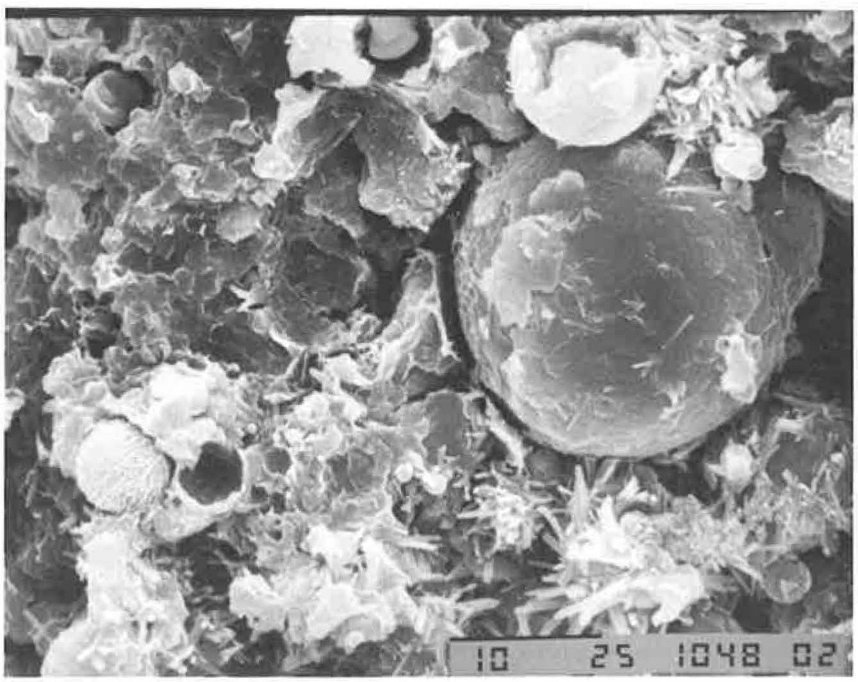

FIGURE 8 Representative fracture surface of bentonite-fly ash-lime cured for 624 days at 50°C.

the cementitious minerals are based on a limited number of d-spacings due to the overlapping of many peaks. Scanning electron microscope (SEM) observations and measured element ratios were used to aid in interpreting the x-ray diffraction patterns. The minerals recognized in the x-ray powder patterns are presented in Table 2. Ettringite is present after one day of curing in all mixtures. In the bentonite-fly ash-lime mixture, C-S-H and afwillite were observed after 28 days of curing at 23°C. In the bentonite-lime and bentonite-fly ash mixtures, afwillite was observed after one day of curing, while C-S-H was observed after 28 days in the bentonite-lime mixture and after 90 days in the bentonite-fly ash mixture at 23°C. At 50°C, the major cementitious minerals (afwillite, C-S-H, and ettringite) were observed after only one day of curing. In both cases, the formation of these minerals corresponded to major increases in strength as presented in Table 3. Addi-

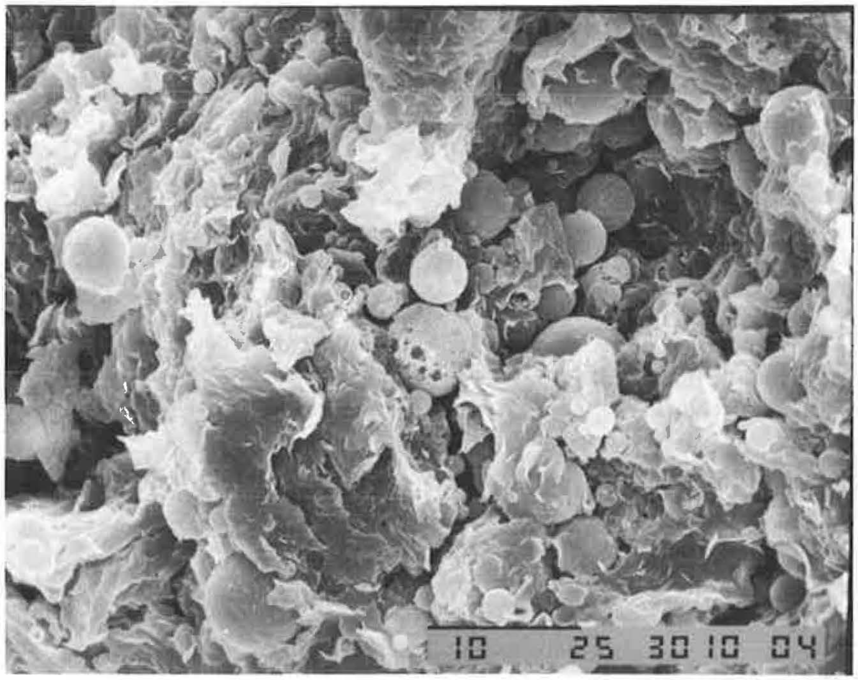

FIGURE 9 Representative fracture surface of bentonite-fly ash mixture cured for 180 days at 50°C.

tional similarities of the cured samples are revealed in the XRD patterns of Figure 10. The one-day 50°C sample and the 28-day 23°C sample are almost identical.

DISCUSSION OF RESULTS

In general, the specimens cured for one day at 50°C produced compressive strengths within ±15 percent of those cured for 28 days at 23°C (Table 3). The bentonite-fly ash-lime mixture had an unconfined compressive strength of 1800 kPa after 28 days of curing at 23°C. When the same mixture was cured for one day at 50°C, it developed a compressive strength that was 17 percent higher than the mixture cured at 23°C (2099 kPa). After 28 days of curing, the bentonite fly ash mixture failed at 1414 kPa, 15 percent higher than the compressive strength of the one-day 50°C cured sample. The bentonite-lime mixture yielded a compressive strength of 996 kPa after 28 days of curing at 23°C, which was 8 percent lower than that of the one day compressive strength of the same mixture at 50°C curing.

Although the compressive strengths were comparable for one-day 50°C and 28-day 23°C cured mixtures, the strains at failure were higher in the former case. The strain at failure after one day of curing at 50°C was 0.92 percent or 1.4 times the failure strain obtained at 23°C after 28 days. The same situation was also observed for bentonite-fly ash and bentonite-lime mixtures. The average strain at failure of the bentonite-fly ash samples cured for one day at 50°C was 1.36 percent, which was 1.9 times greater than the average failure strain of the sample cured for 28 days at 23°C (0.72 percent). The failure strain of the one-day 50°C cured bentonite-lime mixture was 1.49 percent, which was approximately two times the failure strain of the mixture cured for 28 days at 23°C (0.77 percent). In general, the calculated elastic moduli were lower after short-term high-temperature curing.

For most of the specimens, one-day 50°C curing produced compressive strengths similar to long-term compressive strengths of the mixtures cured at 23°C. One-day 50°C curing produced compressive strengths that were between 80 percent and 90 percent of the compressive strengths of bentonite-fly ash-lime, bentonite-fly ash, and bentonite-lime cured at 23°C for 28 days.

The morphological and mineralogical data support the interpretation that the physical behavior of the 23°C cured samples and the 50°C cured samples is roughly similar. The major cementitious minerals are the same in both groups of samples. The cementitious products form the same textural arrangements in the high- and low-temperature specimens. The cements form earlier and have better crystallinities at the higher temperature, suggesting that the rate of reaction is accelerated. Thus, it appears that laboratory curing of one day at 50°C could be substituted for 28-day tests at 23°C. More combined physical and mineralogical morphologies are required before these observations can be fully applied to similar mixtures.

CONCLUSIONS

1. The same cementitious minerals form at 23°C and 50°C curing temperatures, although their crystallinity may be of a higher degree at higher temperatures.
2. At 50°C curing, the rate of the strength-forming chemical reactions increased.
3. Curing at 50°C for one day developed elastic moduli comparable to those of samples cured for 28 days at 23°C.

TABLE 2 MINERALS IDENTIFIED BY X-RAY DIFFRACTION

	BENTONITE-FLY ASH	BENTONITE-LIME	BEN-LIME-FLY ASH
		(23°C) CURING TEMPERATURE	
1 DAY	Montmo.	Montmo.	Montmo.
	Quartz	Quartz	Quartz
	Calcite	Calcite	Calcite
	TCA	Hematite	TCA
	Periclase	Afwil.*	Periclase
	Hematite	Ettrin.*	Hematite
	Ettrin.*		Ettrin.*
	Afwil.*		
28 DAYS	Same	Same+	Same+
		CSH I*	CSH I*
			Afwil.*
90 DAYS	Same+	Same	Same
		CSH I*	
180 DAYS	Same	Same	Same
		50°C CURING TEMPERATURE	
1 DAY	Montmo.	Montmo.	Montmo.
	Quartz	Quartz	Quartz
	Calcite	Calcite	Calcite
	TCA	Hematite	Hematite
	Periclase	Afwil.*	CSH I*
	Hematite	CSH I*	Ettrin.*
	CHS I*	Ettrin.*	Afwil.*

NO CHANGE IN MINERALS WITH LONGER CURING TIMES

Note: (*) indicates the newly forming cementitious minerals. Montmo. = montmorillonite, Afwil. = afwillite, Ettrin. = ettringite, Portlan. = portlandite. FA = Fly ash, FL = Fly ash-lime, F = Bentonite-fly ash, L = Bentonite-lime, S = Bentonite-fly ash-lime, TCA = tricalcium aluminate.

TABLE 3 COMPARISON OF COMPRESSIVE STRENGTH TEST RESULTS OF SAMPLES CURED AT 23°C FOR 28 DAYS AND AT 50°C FOR ONE DAY

		CURED AT 23°C 28 DAYS	CURED AT 50°C 1 DAY
	Unconfined:		
BENTONITE	UCS[1] (kPa):	1800 (3.05)[2]	2099 (3.56)
FLY ASH	FAILURE STRAIN,% :	0.66	0.92
LIME	ELASTIC MODULUS (kPa):	386882	349250
	With 100 kPa Confinement:		
	CS[3] (kPa):	1957 (3.32)	2220 (3.76)
	FAILURE STRAIN,% :	0.89	1.31
	ELASTIC MODULUS (kPa):	402751	358066
	Unconfined:		
BENTONITE	UCS (kPa):	996 (1.69)	1084 (1.84)
LIME	FAILURE STRAIN,% :	0.77	1.49
	ELASTIC MODULUS (kPa):	217945	85861
	With 100 kPa Confinement:		
	CS (kPa):	1108 (1.88)	1144 (1.94)
	FAILURE STRAIN,% :	1.47	1.1
	ELASTIC MODULUS (kPa):	140063	207692
	Unconfined:		
BENTONITE	UCS (kPa):	1414 (2.40)	1234 (2.09)
FLY ASH	FAILURE STRAIN,% :	0.72	1.36
	ELASTIC MODULUS (kPa):	278289	140950
	With 100 kPa Confinement:		
	CS (kPa):	1277 (2.16)	1343 (2.28)
	FAILURE STRAIN,% :	1.11	2.03
	ELASTIC MODULUS (kPa):	237267	56697

[1]Unconfined Compressive Strength.

[2]Compressive Strength of Treated/Untreated Bentonite.

[3]Compressive Strength.

Samples cured at 50°C for one day failed at higher strains than those cured at 23°C for 28 days.

4. Major strength development and decrease in failure strain were observed after 28 days of curing at ambient temperatures. Comparable strength development was observed after only one day at 50°C.

5. Accelerated curing procedures may be used to determine the potential strength development of lime, fly ash, and montmorillonite mixtures. However, specimens cured for short terms at elevated temperatures yield higher failure strains relative to those cured at lower temperatures.

6. It is recommended that those involved in testing soil, lime, and fly ash or any combination perform comparative (curing time versus temperature) studies and develop statistically acceptable data. It may be possible to completely replace the 28-day, 23°C curing with the 24-hr, 50°C curing procedure.

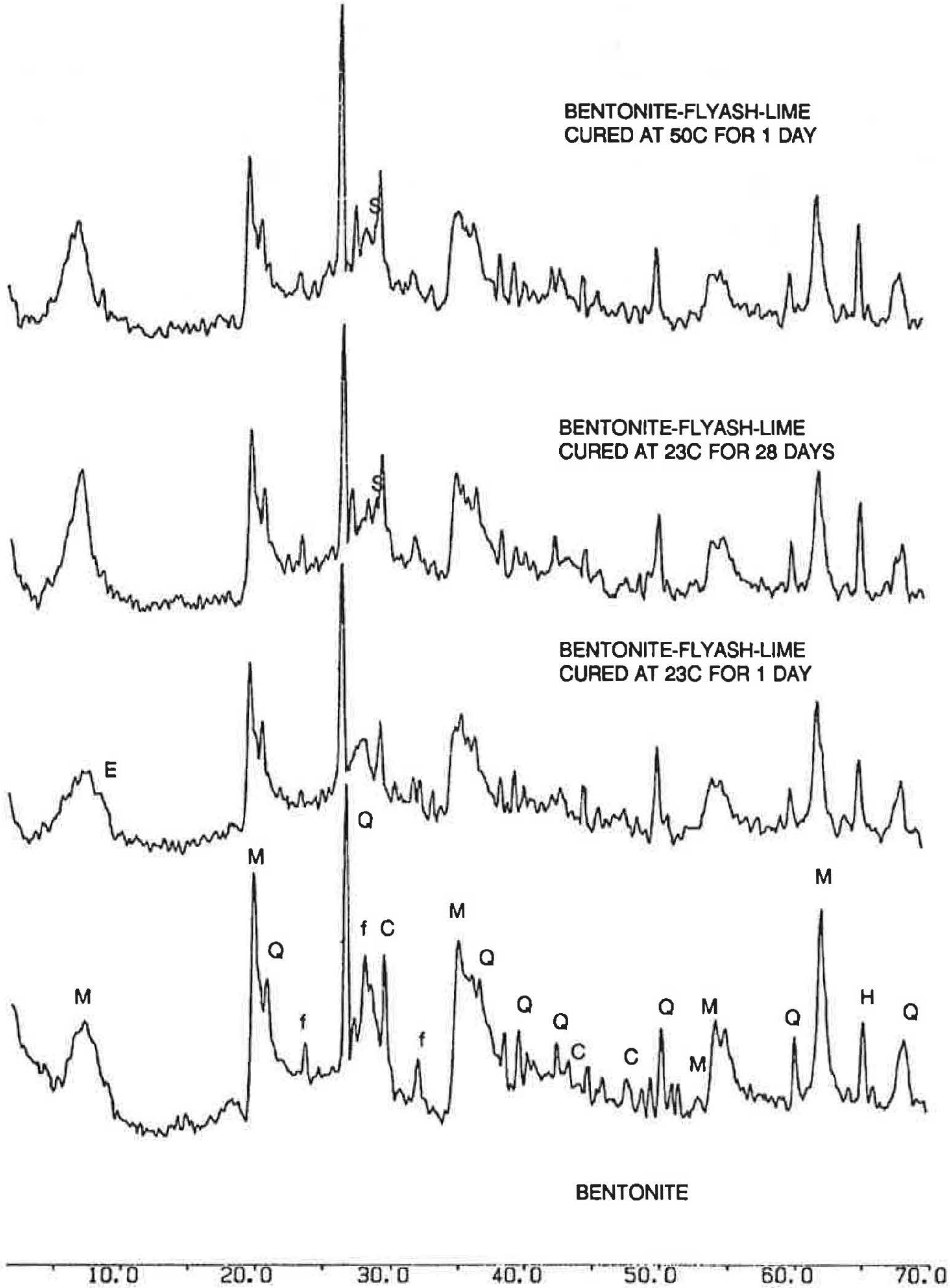

FIGURE 10 X-ray powder diffractograms of bentonite and bentonite-fly ash-lime cured for 1 and 28 days at 23°C and 1 day at 50°C, showing distinct differences from the starting bentonite and 1-day 23°C sample. (*M*, montmorillonite; *Q*, quartz; *f*, feldspars; *C*, calcite; *H*, hematite; *E*, ettringite; *S*, calcium-silicate-hydrate.)

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