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EXPANSION OF PORTLAND CEMENT MORTAR DUE TO INTERNAL SULFATE ATTACK

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ABSTRACT

An exploratory test program was designed to determine the expansive potential of portland cement mortar due to internal sulfate attack initially moist-cured at high temperature. Expansion of cement mortars made with six different portland cements was measured. The effect of different test parameters, such as, pre-storage time, curing temperature, type of sand, sand/cement ratio, specimen size and pre-treatment, on expansion behavior was determined. © *1997 Elsevier Science Ltd*

Introduction

Damage of concrete due to internal sulfate attack (often referred to as delayed ettringite formation (DEF)) has been reported primarily in concrete members subjected to high temperature curing followed by open-air weathering and hence drying/re-wetting cycles, e.g. precast front panels and railway sleepers (1). Well publicized cases have been observed in Europe and USA (1,2). Three critical conditions are reported to govern internal sulfate attack (3–5): (i) portland cement composition, i.e. high C_3A and SO_3 contents; (ii) high curing temperatures; (iii) exposure to a moist environment.

Standard mortar bars made with these cements and soaked in water for 14 days should have an expansion value less than 0.02% (ASTM C1038). The use of this standard test for the evaluation of internal sulfate attack has shortcomings. Lerch and Ford (7) have reported that some specimens exhibiting expansion less than 0.02% at 14 days have much higher expansion five years later. The objective of this study was to demonstrate the effects of different test parameters, such as, pre-storage time, curing temperature, type of sand, sand/cement ratio, specimen size and pre-treatment, on expansion behavior of mortar containing portland cements suspected of being potentially expansive due to internal sulfate attack.

EXPERIMENTAL

Materials

Portland Cements. Six commercially available, ordinary portland cements were investigated. Their oxide composition data is provided in Table 1. Their Blaine fineness values and Bogue compound compositions are given in Table 2.

TABLE 1
Oxide Composition of the Portland Cements Investigated

Cement	Oxide Composition (mass %)							
	SiO2	Al2O3	CaO	Fe2O3	MgO	Na2O + K2O	SO3	L.I.
C-1	21.42	4.10	62.43	2.76	3.73	.53	2.45	2.03
C-2	20.80	5.09	63.20	2.14	2.48	1.12	2.95	1.26
C-3	21.85	4.43	63.91	1.96	3.70	0.81	1.86	0.81
C-4	19.87	5.09	62.34	1.91	2.98	0.60	4.08	2.48
C-5	21.20	4.01	64.05	1.80	2.68	0.69	3.46	1.53
C-6	20.79	3.84	62.78	4.38	3.37	0.81	2.40	0.89

Natural River Sand (River). The grading curve of the natural river sand used in this study is shown in Table 3. The average composition (mass %) was: gneiss (13.5); feldspar (21.1); limestone (41.7); quartz (21.2); other (2.5).

Coarse River Sand (River-c). The coarse river sand used was separated from the natural river sand. The particle size range is, 0.85–3.33 mm. The composition (mass %) was: gneiss (19.0); feldspar (3.0); limestone (76.0); quartz (0.0); other (2.0).

Fine River Sand (River-f). The fine river sand used was separated from the natural river sand. It contains particles less than 0.85 mm. The composition (mass %) was: gneiss (14.0); feldspar (26.0); limestone (34.0); quartz (25.0); other (1.0).

Graded Standard Sand. This material conformed to the requirements for graded standard sand in ASTM Specification C 778.

20-30 Standard Sand. This material conformed to the requirements for graded standard sand in ASTM Specification C 778.

Mortar Mix Design. The mix proportions of the basic cement mortar mixes cured at 90°C for 12 h are given in Table 4. Mortars made with cement C-4 (highest sulfate content) were subjected to a variety of test conditions. The mix proportions are given in Table 5. The mix

TABLE 2
Cement Fineness and Bogue Compound Compositions

Cements	Blaine Fineness (cm^2/g)	Mineralogical Composition (%)			
		C2S	C3S	C3A	C4AF
C-1	3729	21.52	52.88	6.20	8.40
C-2	3611	19.25	53.54	9.87	6.51
C-3	3407	20.21	56.25	8.42	5.96
C-4	5682	16.07	54.21	10.26	5.81
C-5	5147	15.33	60.25	7.58	5.48
C-6	4198	15.35	58.67	2.77	13.33

TABLE 3
Particle Size Grading for Sands Investigated

	Percentage Passing				
Sieve	River	River-f	River-c	Graded	20-30
4.75-mm (No. 4)	100	-	-	-	-
3.33-mm (No. 6)	93	-	100	-	-
1.18-mm (No. 16)	81	-	62	100	100
850-μm (No. 20)	73	100	30	-	85–100
600-μm (No. 30)	60	45	-	98	0–5

proportions of cement C-1 mortars subjected to cycles of wetting and drying are given in Table 6.

Specimen Preparation. The mortars were mixed according to the procedure described in ASTM Method C 305. Two 1 × 1 × 6-in. (25 × 25 × 160-mm) test specimens for each cement type were prepared. The temperature of the preparation room, dry materials, and mixing water was maintained at 74.4°F (23°C). The relative humidity was about 50%.

The specimens were then placed in the moist curing room. All test specimens in the molds were kept in the moist room for at least 1 hour with upper surfaces exposed to the moist air but protected from dripping water. This pre-storage time was later varied from 1 to 5 h for cement C-4 mortars. The specimens were then placed in a plastic container after pre-storage. The container with the test specimens was tightly covered, sealed and placed in an oven. The temperature in the container was raised to the designated temperature (e.g., 176 or 194°F/80 or 90°C) in 60 min and maintained for 12 hours. The heating unit was turned off and the test specimens were cooled for 4 hours at ambient. The specimens were removed from the molds, properly identified, and placed in lime saturated water maintained at 73.4°F (23°C) for 6 hours prior to making the initial length measurement. This period also varied with pre-storage time for the cement C-4 mortars. Following this (after a total of 24 hours) the specimens were dried in an oven maintained at were then stored in saturated lime water at 73.4°F (23°C).

TABLE 4
Mix Proportions of the Cement Mortars Cured at 90°C for 12 Hours

	Mix Proportions (mass units)							
	Cement		* Sand			Curing Temp.	Drying	Results
Mortar	type	content	type	content	Water	(°C)	Cycles	Fig. No.
C-1	C-1	1	river[1]	2.75	0.48	90	1	1
C-2	C-2	1	river	2.75	0.48	90	1	1
C-3	C-3	1	river	2.75	0.48	90	1	1
C-4	C-4	1	river	2.75	0.48	90	1	1
C-5	C-5	1	river	2.75	0.48	90	1	1
C-6	C-6	1	river	2.75	0.48	90	1	1

TABLE 5
Mix Proportions of the Cement C-4 Mortars Subjected to Different Curing Conditions

Mortar	Mix Proportions (mass units)			Curing Temp. (°C)	Drying Cycles	Results Fig. No.	Remarks
	Sand						
	type	content	Water				
M-1	20-30[3]	2.75	0.48	80	-	2	1 h Pre-storage
M-2	20-30	2.75	0.48	80	-	2	2 h Pre-storage
M-3	20-30	2.75	0.48	80	-	2	3 h Pre-storage
M-4	20-30	2.75	0.48	80	-	2	4 h Pre-storage
M-5	20-30	2.75	0.48	80	-	2	5 h Pre-storage
M-6	river	2.75	0.48	90	1	3	90°C curing
M-7	river	2.75	0.48	80	1	3	80°C curing
M-8	graded	2.75	0.48	90	1	4	
M-9	20-30	2.75	0.48	90	1	4	
M-10	river-f	2.75	0.48	90	1	4	
M-11	river-c	2.75	0.48	90	1	4	
M-12	river	2.75	0.48	90	1	4	
M-13	river	1.00	0.48	90	1	5	s/c = 1.00
M-14	river	2.00	0.48	90	1	5	s/c = 2.00
M-15	river	2.75	0.48	90	1	5	s/c = 2.75
M-16	river	3.00	0.48	90	1	5	s/c = 3.00
M-17	river	2.75	0.48	80	1	6	D3 × 3 × 11 1/4
M-18	river	2.75	0.48	80	1	6	D1 × 1 × 6
M-19	river	2.75	0.48	80	0	7	
M-20	river	2.75	0.48	80	1	7	
M-21	river	2.75	0.48	80	3	7	
M-22	river	2.75	0.48	80	5	7	

Length Measurement. The specimens were removed from water storage, and wiped with a damp cloth. The length change of the specimens was measured using an electronic length comparator. The first reading was taken 24 h from the time the cement and water were mixed together. The subsequent measurements were carried out every 7 days. The average expansion value of two specimens was reported.

Results

Effect of Cement Composition. The development of expansion of the mortars made with different portland cements initially cured at 90°C is shown in Fig. 1. The data presented are for a period of 1 to 250 days. Mortars made with cement C-4 & 5 exhibited the most deleterious expansion. The mortar made with cement C-4 had the highest expansion, beginning after 14 days. Its 90-day expansion value was higher than 1.3%. The expansion of the cement C-5 mortar developed more slowly (after 21 days) than that of cement C-4 mortar. Its 250-day expansion value was still very high. The other cement mortars showed no significant expansion. Cements C-4 and C-5 had the highest sulfate contents at 4.08 and

TABLE 6
Mix Proportions of the Cement C-1 Mortars (Effect of Drying Cycles)

	Mix Proportions (mass units)					
	Sand					
Mortar	type	content	Water	Curing Temp. (°C)	Drying Cycles	Results Fig. No.
M-23	river	2.75	0.48	80	0	8
M-24	river	2.75	0.48	80	1	8
M-25	river	2.75	0.48	80	3	8
M-26	river	2.75	0.48	80	5	8

3.46% respectively. Cement C-3 with the lowest sulfate content had expansion of about 0.4%. Its MgO content was relatively high at 3.70%.

Effect of Pre-Storage Time. The effect of pre-storage time (before high temperature curing) on expansion of cement C-4 mortars is shown in Fig. 2. Cement C-4 was selected as it had the highest sulfate content. The expansion decreased significantly with the increase of pre-storage time. However the mortars pre-stored for 5 hours still exhibited deleterious expansion higher than 0.1% after 180 days. The mortar bars in this test were not pre-treated by thermal drying.

Effect of Curing Temperature. The effect of curing temperature on expansion of cement C-4 mortars is shown in Fig. 3. The expansion developed before 90 days for all specimens cured at 80 or 90°C. The increase of curing temperature resulted in a significantly increased expansion of the mortars.

Effect of Sand Type. The effect of different types of sand on the expansion of cement C-4 mortars initially cured at 90°C is shown in Fig. 4. The expansion of the mortar containing fine river sand showed the highest expansion. The overall composition of those two sands is

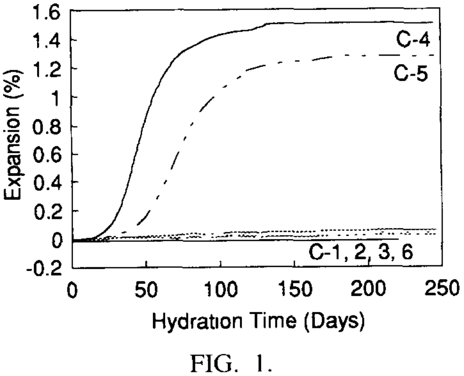

FIG. 1.
Delayed expansion of different cements.

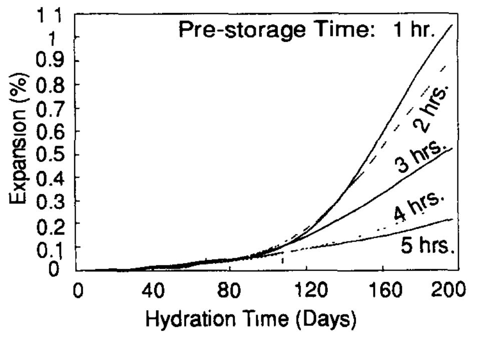

FIG. 2.
Effect of pre-storage time on the delayed expansion of C-4 cement mortars.

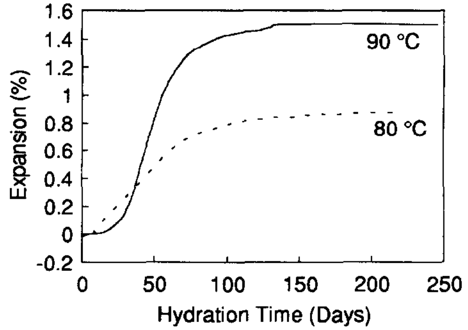

FIG. 3.
Effect of curing temperature on delayed expansion of C-4 cement mortars.

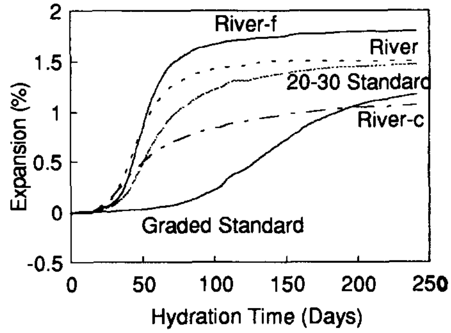

FIG. 4.
Effect of sand type on delayed expansion of C-4 cement mortars.

similar. The finer sand gives the highest expansion possibly due to a greater influence on microcracking during the pre-treatment stage. The coarse river sand contains little or no quartz and feldspar and results in a much lower expansion than the other two river sands. The expansion of the mortar made with 20-30 standard sand was less than those made with river sand and fine river sand but greater than that of coarse river sand. It contains primarily quartz and is finer than the coarse river sand but not the other sands. It reached a value of about 1.4% at 250 days. Expansion of the mortar made with graded standard sand developed much slower than the others.

Effect of the Sand/Cement Ratio. The effect of the sand/cement (s/c) ratio of the cement C-4 mortars initially cured at 90°C is shown in Fig. 5. Similar expansion was obtained in the mortars with s/c ratio of 2.00 and 2.75. A decrease of s/c ratio to 1.00 would reduce the expansion of the cement mortars before 100 days but increase the expansion at later times. The effect of s/c ratio on the expansion was relatively less significant than the effect of the other parameters.

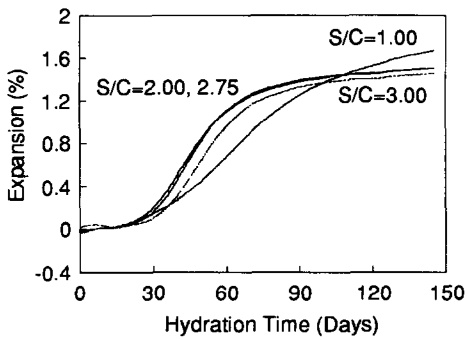

FIG. 5.
Effect of sand/cement ratio on delayed expansion of C-4 cement mortars.

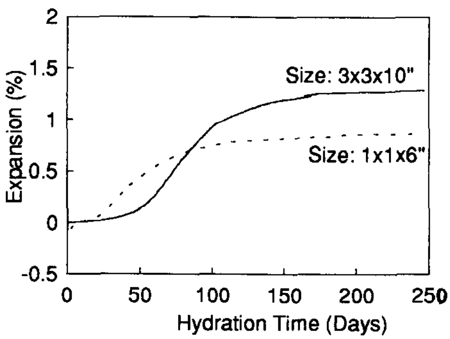

FIG. 6.
Effect of specimen size on delayed expansion of C-4 cement mortars.

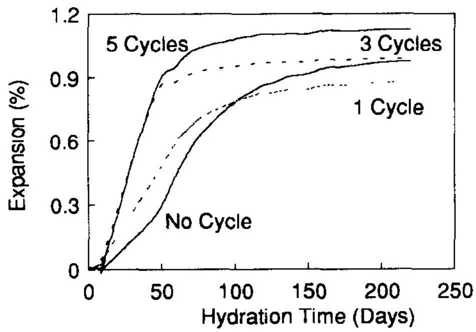

FIG. 7.
Effect of thermal drying on delayed expansion of C-4 cement mortars.

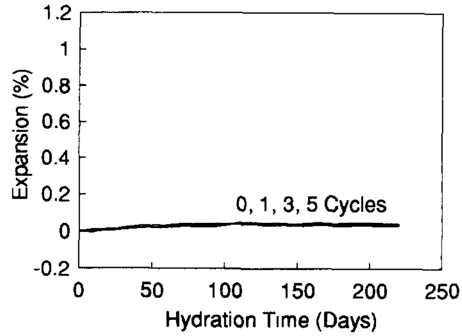

FIG. 8.
Effect of thermal drying on expansion of C-1 cement mortars.

Effect of Specimen Size. The effect of specimen size on the expansion of cement C-4 mortar initially cured at 80°C is shown in Fig. 6. The expansion of the large size (3 × 3 × 11-in.) specimen was less than that of the small size (1 × 1 × 6-in) specimen during the ages from 14 to 80 days. The expansion of the large size specimen exceeded that of the small size specimen after about 80 days.

Effect of Thermal Drying. The effect of thermal drying on expansion of mortars made with cements C-4 and C-1 initially cured at 80°C is shown in Figs. 7 and 8. A significant increase of expansion was found in the specimens containing cement C-4 subjected to 3 or 5 drying/re-wetting cycles. An increase of drying/re-wetting cycles accelerated the expansion of the mortars. The C-4 cement mortars without the thermal drying treatment also exhibited high expansion up to 0.9%. Little or no expansion was found in any specimens made with C-1 cement, Figure 8.

An appropriate test method may be useful in evaluating a cement for use in the precast concrete structures exposed to out-door weathering. The test parameters (related to the above critical conditions and procedures) evaluated in this study appear to be more severe than those that occur in some precast concrete production. It is noted that only some of the cements exhibited the potential for expansion due to internal sulfate attack. Selection of a durable cement to make sound concrete structures should be considered.

Conclusions

1. A significant delayed expansion of cement mortars, made with portland cement having a Blaine fineness greater than 5000 cm^2/g and SO_3 content greater than 3.5 mass % (e.g., cement C-4 and -5) and initially moist-cured at <85°C, occurs.
2. Delayed expansion of cement mortars made with portland cement that is suspect of being prone to internal sulfate attack is accelerated and enhanced by reducing the pre-storage time, increasing curing temperature and thermal drying.
3. The delayed expansion of cement mortars made with fine sand containing about 25% quartz and 34% limestone is significantly greater than sand containing predominantly limestone and little or no quartz.

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