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16. ABSTRACT

In December 1963, a project was initiated to develop modified mortar test procedures and/or volume change specifications for Portland cement intended for use in prestressed concrete, most of which is steam cured.

A series of tests were made to compare the volume change characteristics of Type II, low alkali cements and cements of the same type which had been modified by finer grinding. (The modified cements are used to produce a higher early strength product in casting prestressed steam cured concrete.)

Steam curing at 150*F was studied to determine its influence on the drying shrinkage of mortars and on the amount of gypsum required for minimum shrinkage of both types of cement.

On the basis of evidence obtained in these studies, volume change and SO3 (gypsum) content specification limits were established for the finer ground cements. These limits now appear as the 1964 California Standard Specifications for Type II, Prestress Cement. (1)

Subsequently, test results for routine cement samples were statistically analyzed to determine the degree of conformance with the new specifications.

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STATE OF CALIFORNIA
HIGHWAY TRANSPORTATION AGENCY
DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS

FINAL REPORT

VOLUME CHANGE OF MORTARS AS AFFECTED BY STEAM CURING

65-07

October, 1965

State of California
Transportation Agency
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

October, 1965
Laboratory Project
Work Order 451068

Mr. L. R. Gillis
Assistant State Highway Engineer
Division of Highways
Sacramento, California

Dear Mr. Gillis:

Submitted for your consideration is:

Final Report on

Volume Change of Mortars as Affected By

Steam Curing

Tests made by . . . . . Concrete Section
Under general direction of. . . . . D. L. Spellman
Work supervised by. . . . . R. W. Ford
Report prepared by. . . . . R. W. Ford

Very truly yours,

JOHN L. BEATON
Materials and Research Engineer

cc:ACEstep
CGBeer
JFJorgensen
PCSheridan
JEMcMahon
DLSpellman
Research Files

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VOLUME CHANGE OF MORTARS AS AFFECTED BY STEAM CURING

Synopsis

In December 1963, a project was initiated to develop modified mortar test procedures and/or volume change specifications for portland cement intended for use in prestressed concrete, most of which is steam cured.

A series of tests were made to compare the volume change characteristics of Type II, low alkali cements and cements of the same type which had been modified by finer grinding. (The modified cements are used to produce a higher early strength product in casting prestressed steam cured concrete.)

Steam curing at 150°F. was studied to determine its influence on the drying shrinkage of mortars and on the amount of gypsum required for minimum shrinkage of both types of cement.

On the basis of evidence obtained in these studies, volume change and SO_3 (gypsum) content specification limits were established for the finer ground cements. These limits now appear as the 1964 California Standard Specifications for Type II, Prestress Cement.[1]

Subsequently, test results for routine cement samples were statistically analyzed to determine the degree of conformance with the new specifications.

Introduction

During 1963, a number of cement samples failed to meet the specification requirement of not more than .048% contraction in air when tested in accordance with Test Method No. Calif. 527. Investigation of failing samples usually disclosed that they represented a kind of modified cement used at some prestressing plants in steam cured precast products.

These cements met all chemical requirements for Type II low alkali cement but had been modified during manufacture, most often by finer grinding, in order to increase early strength. The cement manufacturers stated that they could not consistently supply a product which would satisfy the prestressing plants' requirements for high early strength and still meet our volume change and chemical requirements for Type II low alkali cement. Since the use of the finer ground cements in precast prestressed products was apparently producing a satisfactory product, and the use of the modified cements made 24 hour casting schedules possible, which would in turn lower costs, it was believed that modifications to our specifications might be advisable.

Previous studies [2,3] indicated that steam curing reduced the drying shrinkage of the concrete. It was therefore assumed that the apparent higher shrinkage of the finer ground cements when measured after moist curing at 73±°F in the laboratory, might be tolerated in applications where steam curing is used. It was then necessary to consider modification of the specification limits and/or the test method for drying shrinkage applicable to the subject cements. The cements tested, hereinafter referred to as prestress cements, are those which are intended for use in steam cured products and represent typical products from five California cement companies.

Test data on Type II cements from 15 mills supplying cement to California highway contracts were used for the statistical analysis.

Findings

1. The drying shrinkage of mortar, as measured by Test Method No. Calif. 527, is less for steam cured mortars whether made from Type II Modified cement (Type II low alkali cement) or Type II Prestress cement (Type II low alkali cement which has been modified by finer grinding to obtain higher early strengths).

2. Type II Prestress cement has a higher optimum gypsum content than a comparable Type II Modified cement whether steam cured or moist cured.

3. The optimum amount of gypsum required for minimum volume change is higher for steam cured mortar than it is for mortar moist cured at 73±°F.

4. At the 95% confidence level, both Type II Modified and Type II Prestress cements meet present specifications (as modified by findings reported here) for drying shrinkage and SO_3 content.

Resulting Specification Changes

During the course of and as a result of this study, a recommendation was made and adopted which increased the Type II Prestress cement specification limit for contraction in air from 0.048% to 0.053%, and increased the maximum allowable SO_3 content from 2.5% to 2.8%. No further changes are recommended in the specification nor are any changes recommended in the Test Method No. Calif. 527.

Experimental Test Data

The chemical analyses and surface areas of the cements used in experiments for this project are given in Table 1, The "prestress" cements have essentially the same composition as the corresponding Type II cements, but they have surface areas generally from 700 to 2,000 square centimeters per gram higher than their counterparts.

Drying shrinkage of mortar after moist curing and after steam curing, was determined for samples representing five brands of Type II Modified and Type II Prestress portland cements. Drying shrinkage measurements were also performed on samples representing four brands of cement under the same conditions but with 0.5% and 1.0% SO_3 added. Three rounds of tests were made on each cement-gypsum-curing method combination in accordance with the procedure shown in Appendix 1.

The average shrinkage values for the three rounds of tests are shown in Table 2. The drying shrinkages of mortars containing Type II Modified cements and cured at 73±°F, generally were below the specification requirement of .048% maximum while mortars containing Type II Prestress cements exceeded that limit.

Steam curing of subsequent test specimens reduced drying shrinkages of mortars containing either type of cement. The drying shrinkage, after steam curing of mortar containing prestress cements, was well below the .048% limit. The addition of gypsum caused increases in the drying shrinkages of 73±°F moist cured mortar containing Type II Modified cements but produced decreases in 73±°F moist cured mortar containing prestress cements and in steam cured mortar, containing cements of either type. These results indicate, first, that the Type II Modified cements contained near-optimum gypsum and that the Type II Prestress cements contained somewhat less than optimum gypsum for a minimum drying shrinkage under the conditions specified in Test Method No. Calif. 527, and second, that steam curing increases the amount of gypsum required by both types of cement for minimum drying shrinkage.

Routine Test Data

During the course of this investigation, the specifications on portland cement were modified to permit up to .053% drying shrinkage of mortar containing Type II Prestress cement and to increase the allowable SO_3 content of Type II Prestress cement from 2.5% maximum to 2.8% maximum. Test data from routine samples were then analyzed statistically to determine the degree of conformance to these modifications.

The statistical analysis of routine test data summarized in Table 3, indicates that at the 95% confidence level, samples of both Type II Modified and Type II Prestress cement meet the corresponding current specification requirements for drying shrinkage (with an allowable tolerance of .005% for a single test or .003% for the average of two or more tests). As shown in Table 4, cements of both types have less than 2.50% SO_3 at the 95% confidence level, and with few exceptions, the Type II Prestress cements contain no more SO_3 than do the Type II Modified cements.

Bibliography

1. California Standard Specifications
July, 1964, Section 90-2.01

2. "A Preliminary Report on the Effect of Steam Curing on the Properties of Concrete,"
Materials and Research Department Laboratory Report
August 1, 1960

3. "Prestress Loss as Affected by Type of Curing,"
By J. A. Hannon, Journal of the Prestressed Concrete Institute, Vol. 9, No. 2, April, 1964

Appendix 1

Test Procedure

Three rounds of tests were performed at the rate of one round per week on each cement used according to the following procedure:

1. Mortars for moist-curing were fabricated and tested as directed in Test Method No. Calif. 527 using these mix designs:

Cement	750	742	734
Graded Ottawa Sand-grams	1500	1500	1500
Gypsum (48.9% SO_3) added-grams	0	8	16
SO_3 added-percentage	0	0.5	1.0
Water-Sufficient to produce a flow between 100 and 115			

2. Two days after fabrication of mortars for moist-curing mortars of the same mix designs were fabricated and tested on the following schedule:

   a. Immediately after molding, the specimens were stored in the moist-curing room for 5 hours. (A presteaming period of moist curing at average air temperature as necessary to obtain best results from steam curing.)

   b. The specimens, while still in the molds, were placed in a steam cabinet, pre-heated and maintained at 150°F±3°F, and steam cured for a period of 18 hours.

   c. Immediately following removal from the steam cabinet, the specimens were placed in water at 140°F and cooled in water to 73.4°±3°F within 30 minutes. They were maintained at 73.4±3°F for an additional period of 30 minutes.

   d. The specimens were then removed from the water, stripped from the molds, surface dried and measured on a comparator.

   e. The specimens were then stored in the 50% Relative Humidity drying room for a 96-hour period of drying before measuring, to determine drying shrinkage.

Table 1

Properties of Cements Used in Drying Shrinkage Experiment

Mill	4		6		11		12		13	
Type	II Mod.	II Prestress	II Mod.	II Prestress	II Mod.	II Prestress	II Mod.	II Prestress	II Mod.	II Prestress
Chemical Analysis Percent			(a)						(b)	(b)
SiO_2	22.4	22.7	23.7	21.7	22.4	22.0	24.3	24.2	21.8	21.5
Al_2O_3	4.6	4.9	4.7	5.0	4.1	4.1	3.4	3.4	4.5	4.5
Fe_2O_3	3.0	2.8	4.1	4.0	2.6	2.7	2.8	3.1	2.9	2.8
CaO	63.7	64.0	65.7	64.5	64.2	64.0	64.9	65.1	62.6	62.7
MgO	1.7	1.2	1.4	1.6	2.9	3.3	1.2	1.0	4.2	4.0
SO_3	2.30	2.40	2.05	1.93	1.96	2.18	1.81	1.85	2.08	2.31
Ignition Loss	1.4	1.2	1.0	1.1	1.2	1.0	1.1	1.0	1.5	1.5
Insoluble	.4	.6	.1	.1	.3	.2	.1	.1	.2	.1
Na_2O	.18	.24	.45	.58	.15	.15	.26	.28	.43	.37
K_2O	.46	.43	.01	.01	.40	.41	.17	.19	.26	.20
Eq. Na_2O	.48	.52	.46	.59	.42	.42	.37	.40	.60	.50
C4AF	9	9	12	12	8	8	9	9	9	9
C_3A	7	8	6	7	6	6	4	4	7	7
C_3S	48	45	----	53	55	55	47	49	49	51
C_2S	29	32	----	23	23	21	34	33	26	23
Surface Area, Blaine cm^2/gm	2943	4490	3519	5015	3339	5344	3438	4564	4394	5127

(a) Analyses for SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO and SO_3 were performed by x-ray emission spectroscopy instead of by the wet chemical method. Since SiO_2 and CaO appeared to be abnormal by this method, C_3S and C_2S were not calculated

(b) Analyses were not performed on samples used in this portion of the experiment. Analyses shown are representative of routine samples of the brand tested at the time the drying shrinkage experiment was performed.

Table 2

Experimental Shrinkage Data for Type II Modified and Type II Prestress Cements

Type II, Modified *Percent Contraction in Air, 4-days drying						
% SO_3 Added	Moist Cured at 73°F 0	0.5	1.0	Steam Cured at 150°F 0	0.5	1.0
Mill						
4	.0451	.0517	.0631	.0339	.0326	.0315
6	.0415	.0430	.0547	.0414	.0358	.0348
11	.0502	.0536	.0660	.0381	.0329	.0335
12	.0446	.0469	.0616	.0295	.0321	.0311
13	.0426	-----	-----	.0360	-----	-----
Avg. (all Mills Tested)	.0448	.0488	.0613	.0358	.0334	.0327

Type II, Prestress *Percent Contraction in Air, 4-days drying						
% SO_3 Added	Moist Cured at 73°F 0	0.5	1.0	Steam Cured at 150°F 0	0.5	1.0
Mill						
4	.0537	.0492	.0528	.0357	.0340	.0326
6	.0620	.0506	.0486	.0449	.0433	.0381
11	.0581	.0527	.0592	.0461	.0416	.0373
12	.0496	.0450	.0562	.0359	.0364	.0308
13	.0552	-----	-----	.0374	-----	-----
Avg. (all Mills Tested)	.0557	.0494	.0542	.0400	.0388	.0347

*See Test Method No. Calif. 527 and Appendix 1 for procedures used.

Table 3

Statistical Analysis of Drying Shrinkage Data

From Routine Cement Sample Tests

Percent Shrinkage by Test Method No. Calif. 527

Mill	No. Samples n	% Contraction Average $\bar{X}$	Standard Deviation σ	% Contraction at 95% Confidence Level $\bar{X}+1.645\ \sigma$
Type II, Modified				
1	49	.0409	.0037	.0470
2	81	.0389	.0047	.0466
3	48	.0480	.0034	.0536[a]
4	53	.0413	.0029	.0461
5	5[b]	.0360	-----	-----
6	31	.0401	.0033	.0455
7	23	.0408	.0041	.0475
8	46	.0390	.0037	.0451
9	1[b]	.0360	-----	-----
10	42	.0362	.0028	.0408
11	60	.0445	.0031	.0496
12	108	.0390	.0037	.0451
13	42	.0448	.0041	.0515
14	96	.0425	.0033	.0479
15	32	.0444	.0025	.0485
All 15 Mills	717	.0414	.0047	.0491
Mills 4[c] 6,11,12,13	294	.0415	.0042	.0484
Type II Prestress				
4	4[b]	.0510	-----	-----
6	2[b]	.0535	-----	-----
11	2[b]	.0565	-----	-----
12	25	.0462	.0032	.0515
13	8	.0420	.0042	.0480
Mills 4, 6,11,12,13	41	.0485	.0046	.0561

[a] The manufacturer has recently improved test facilities and appears to be controlling drying shrinkage at a more favorable level.

[b] Number of samples was too small for calculation of standard deviation

[c] Five brands of Type II, Modified corresponding to five brands of Type II, Prestress cement tested.

Table 4

SO_3 Content of Cements by Mill and Type

Mill	No. Samples n	Average[a] $\bar{X}$	Standard Deviation σ	SO_3 at 95% Confidence Level $\bar{X}+1.645\ \sigma$
Type II Modified Cements				
1	55	1.92	0.11	2.10
2	83	1.94	0.16	2.20
3	60	2.14	0.13	2.35
4	54	2.293	0.109	2.472
5	6	2.04	0.14	2.27
6	37	2.186	0.175	2.474
7	31	2.02	0.16	2.28
8	44	2.10	0.16	2.36
9	2[b]	2.05	----	----
10	52	2.07	0.15	2.32
11	75	1.974	0.123	2.176
12	110	1.811	0.145	2.050
13	32	2.159	0.183	2.460
14	92	1.99	0.14	2.22
15	29	2.02	0.17	2.30
All 15 Mills	762	2.047	0.181	2.345
Mills 4, 6, 11 12 & 13	308	2.026	0.218	2.385
Type II, Prestress Cements				
4	4[b]	2.32	----	----
6	2[b]	2.05	----	----
11	1[b]	2.93	----	----
12	25	1.75	0.18	2.05
13	11	2.18	0.14	2.41
Mills 4, 6, 12 and 13	42	1.925	0.253	2.341

[a]Averages and standard deviations were determined graphically for mills 1,2,3,5,7,8,9,10,14 and 15 and were calculated for mills 4,6,11,12,13 and for the accumulated totals of 5 and 15 mills.

[b]Number of samples was too small for calculation of standard deviation