EVALUATION OF THE MINI SLUMP CONE TEST

Final Report for MLR-97-1

November 2000

Highway Division



Evaluation of the Mini Slump Cone Test

Final Report For MLR-97-1

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8. Abstract

Early stiffening of cement has been noted as contributing to workability problems with concrete placed in the field. Early stiffening, normally attributed to cements whose gypsum is reduced to hemi-hydrate or anhydrate because of high finish mill temperatures, is referred to as false setting. Stiffening attributed to uncontrolled reaction of C_3A is referred to as flash set. False setting may be overcame by extended mix period, while flash setting is usually more serious and workability is usually diminished with extended mixing. ASTM C 359 has been used to detect early stiffening with mixed results. The mini slump cone test was developed by Construction Technology Laboratories (CTL), Inc., as an alternative method of determining early stiffening.

This research examined the mini slump cone test procedure to determine the repeatability of the results obtained from two different testing procedures, effect of w/c ratio, lifting rate of the cone, and accuracy of the test using a standard sample.

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DISCLAIMER

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Introduction

Early stiffening of cement has been noted as contributing to workability problems with concrete placed in the field. Early stiffening, normally attributed to cements whose gypsum is reduced to hemi-hydrate or anhydrate because of high finish mill temperatures, is referred to as false setting. Stiffening attributed to uncontrolled reaction of C₃A is referred to as flash set. False setting may be overcame by extended mix period, while flash setting is usually more serious and workability is usually diminished with extended mixing. ASTM C 359 has been used to detect early stiffening with mixed results. The mini slump cone test was developed by Construction Technology Laboratories (CTL), Inc., as an alternative method of determining early stiffening.

This method uses high intensity mixing to simulate the high shear rate the paste is exposed to in a concrete mixer. The test is ran at a constant temperature of 73 °F with a recirculating bath attached to the mixing container. (Figure 1) The mini slump cone (Figure 2), having the same proportions as a concrete slump cone with dimensions of 19 mm (0.75 in.) top opening and a 38 mm (1.5 in.) bottom opening, is filled with paste and lifted at intervals of 2.5, 5, 15, 30, and 45 minutes. Information on flow is obtained from the area of the pat formed by the paste after the mini slump cone (Figure 3) has been lifted.

This research examined the mini slump cone test procedure to determine the repeatability of the results obtained from two different testing procedures, effect of w/c ratio, lifting rate of the cone, and accuracy of the test using a standard sample.

Objective

The objective of this research was to compare the effect on pat area of the modified mini slump cone test procedure with the original test procedure. Also, to determine, effect of lifting rate of the cone, effect of w/c ratio, and accuracy of the test using standard samples.

Materials

Cement from each of the following plants were used in this research.

Ash Grove I/II Louisville, NE

Holnam I Mason City, IA

Lafarge I/II Davenport, IA

Lehigh I Mason City, IA

The XRD plots of each source is included in Figure 4 of the Appendix.

Test Procedure

The original mini slump cone test procedure uses a 600 gram sample. After the material is introduced into the water, the mixing speed in increased from 1,200 rpm to 15,000 rpm for 1 minute. A sample is poured into a mini slump cone, rodded, struck off, and lifted at 2 minutes. The remaining paste is remixed at 15,000 rpm for 1½ minutes. Another sample is poured, rodded, struck off, and lifted at 5 minutes. The remaining paste is agitated at 1,200 rpm, 1 minute before each additional test at 15, 30, and 45 minutes. The area of each pat is determined and reported. The ratio of the 5 minute pat area to the 2 minute pat area, expressed as a

percentage, is calculated as the false setting index (FSI) and is an indication of the false setting tendency of a cement. The higher the false setting index the greater the false setting tendency of the cement.

The modified mini slump cone test procedure uses a 500 gram sample with a 13,000 rpm mixing speed and a 1,000 rpm agitation. CTL, Inc. had developed the original procedure and modified the procedure to more closely match the shear rate the cement particles experience in plant mixed concrete.

Early test results indicated a good correlation to false setting characteristics to the type of gypsum in the cement and false setting characteristics of the cement. (Figure 5) Lafarge and Lehigh show the highest degree of false setting characteristics while exhibiting the highest amounts of hemi·hydrate (bassanite) and anhydrate, as compared to Ash Grove and Holnam cements with mostly gypsum (Figure 4).

Scope

This research was basically divided into four different areas.

- 1) Compare the original mini slump test procedure to the modified mini slump test procedure and the effect on pat area and repeatability.
- 2) Determine the effect of lifting rate of the cone on pat area and repeatability.
- 3) Determine effect of w/c ratio on pat area.
- 4) Repeatability of same sample with single operator

Original vs. Modified Test Procedure

Each of the four cements were tested three times using the original test procedure and three times using the modified test procedure. Shear rate and sample size differed for each test procedure. The maximum, minimum, average, standard deviation, and range was tabulated for the pat areas at each time interval as well as the false setting index (Figure 6, 7).

With the exception of one cement, there was a lower standard deviation for the modified test method versus the original test method. The modified test procedure was used for the remainder of testing in this research.

Mini Slump Cone Lifting Rate

The lifting rate of the cone has been the subject of much controversy. The faster the lifting rate the larger the pat area. The Iowa DOT assumed that the cone should be lifted very carefully and the paste allowed to slump, similar to a concrete slump cone. Lifting the cone in this manner will be referred to as a "normal pull". Construction Technology Laboratories, Inc. refers to lifting "in a way that the gravitational force applied by the weight of the paste at the bottom of the cone is released instantaneously" or "vertically in a few tenths of a second, without jerking". Lifting the cone in this manner will be referred to as a "fast pull".

Two of the four cements were tested three times using the "normal pull" and three times using the "fast pull". The results were plotted for each of the two cements tested. (Figures 8, 9, 10, and 11) The false setting index was calculated and a standard deviation was determined.

For Holnam cement, the false setting index was comparable for both the normal and fast

pulls, ranging from 91 to 93 5 (Std. Deviation =2) for the normal pull and 90 to 99 % (Std. Deviation =9) for the fast pull. There was also a large increase in pat areas from the normal to the fast pull, ranging from 9.29 to 9.88 square inches for the normal pull and 13.06 to 14.19 square inches for the fast pull.

For Ash Grove cement, the false setting index was higher the normal pull verusu the fast pulls, ranging from 100 to 107 (Std. Deviation =7) for the normal pull and 76 to 93 % (Std. Deviation =11) for the fast pull. There was not as large of an increase in pat areas from the normal to the fast pull, ranging from 7.35 to 8.88 square inches for the normal pull and 8.97 to 9.62 square inches for the fast pull.

The normal pull was used for the remainder of the research since there was a lower standard deviation of the false setting index.

Effect of w/c ratio

Two of the four cements were tested ran at 0.37, 0.40, 0.43, 0.47, and 0.50 w/c ratios to investigate effect of w/c ratio on false setting characteristics. It was thought that the false setting index may vary with w/c ratio. The pat areas and false setting index was plotted for the w/c ratio at each time interval. (Figures 12 and 13)

As expected, the pat areas increased in size as the w/c ratio increased. The false setting index for Lehigh cement seemed to be comparable throughout the range of w/c ratios, varying from 77 to 88. For Ash Grove cement, the false setting index varied more widely as w/c ratio increased, varying from 86 to 107.

Accuracy of Test Method with Standard Sample

Two sets of seven test samples were obtained from CTL as part of a round robin study to determine interlaboratory evaluation of the test. These samples were tested by the same operator, using the modified test method and fast pull. Results from other laboratories in the round robin testing were never returned from CTL. The results are found in figures 14 and 15 of the appendix.

Results indicate a wider standard deviation in the average pat area for Set B versus Set C.

The average false setting index for Set B was 138 with a standard deviation of 12.8. The average false setting index for Set C was 65 with a standard deviation of 7.1.

As with the other testing in this research, the variability in test results seems to be dependant on the cement sample.

Conclusions

- 1) The modified test procedure produced less variability than the original test method.
- 2) Lifting rate of the cone produces variable results depending on cement sample.
- 3) The w/c ratio impact pat area and FSI depending on cement sample.
- 4) Using standard samples, the pat area and FSI was more variable depending on cement sample.

With somewhat variable results with some cements, it would be difficult to apply this test method as a specification requirement. It may be useful as a preliminary check to determine if

false setting may occur. CTL was working on some method to remove the variability of lifting rate of the cone, with no results as of this reseach.

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APPENDIX

- Figure 1 Mini slump cone test equipment
- Figure 2 Mini Slump Cone
- Figure 3 Paste pats and mini slump cones
- Figure 4 XRD plots of cements showing form of gypsum
- Figure 5 Mini slump cone test results of various cements
- Figure 6 Table of results High Shear Rate (Original Test Method)
- Figure 7 Low Shear Rate Table of Results (Modified Procedure)
- Figure 8 Holnam Cement Normal Pull
- Figure 9 Holnam Cement Fast Pull
- Figure 10 Ash Grove Cement Normal Pull
- Figure 11 Ash Grove Cement Fast Pull
- Figure 12 Lehigh Cement Various w/c Ratios
- Figure 13 Ash Grove Cement Various w/c Ratios
- Figure 14 Table of Results Standard Sample Set B
- Figure 15 Table of Results Standard Sample Set C



Figure 1 – Mini slump cone test equipment

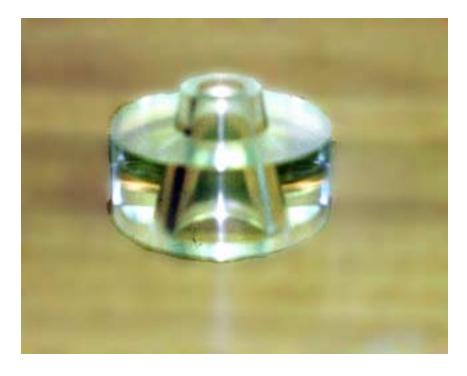


Figure 2 - Mini Slump Cone



Figure 3 – Paste pats and mini slump cones

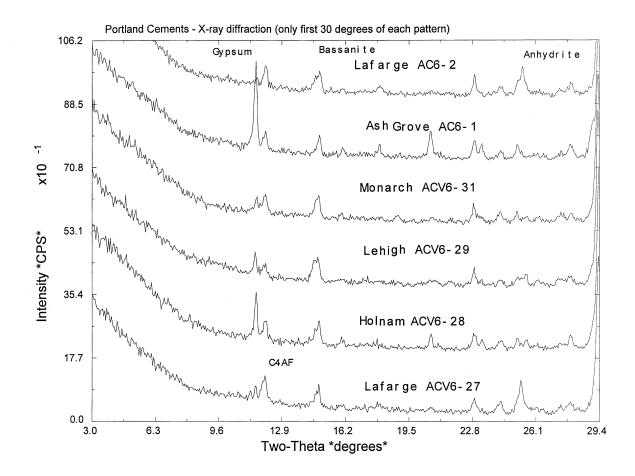


Figure 4 – XRD plots of cements showing form of gypsum

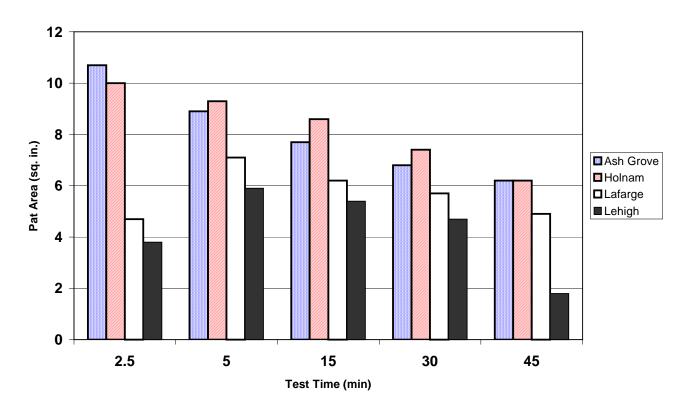


Figure 5 – Mini slump cone test results of various cements

Source	FSI (max.)	FSI (min.)	Std. Dev	2 min. Pat Area, sq. in.	2 min. Pat Area, sq. in.
				(max.)	(min.)
Ash Grove	118	108	5.08	7.99	7.69
Holnam	92	88	1.96	9.46	9.05
Lafarge	305	242	35.61	3.24	2.57
Lehigh	127	97	13.47	7.99	6.20

Figure 6 – Table of results High Shear Rate (Original Test Method)

Source	FSI (max.)	FSI (min.)	Std. Dev.	2 min. Pat Area, sq. in. (max.)	2 min. Pat Area, sq. in. (min.)
Ash Grove	107	100	3.28	8.88	7.35
Holnam	93	91	0.90	9.88	9.29
Lafarge	299	75	129.2	9.38	2.78
Lehigh	81	78	1.29	9.70	9.21

Figure 7 – Low Shear Rate Table of Results (Modified Procedure)

Holnam Cement - Normal Pull

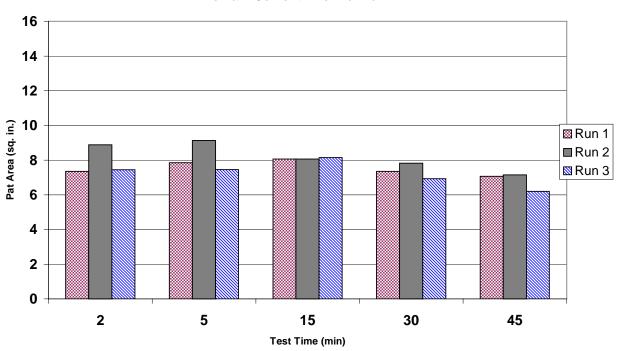


Figure 8 – Holnam Cement Normal Pull

Holnam Cement - Fast Pull

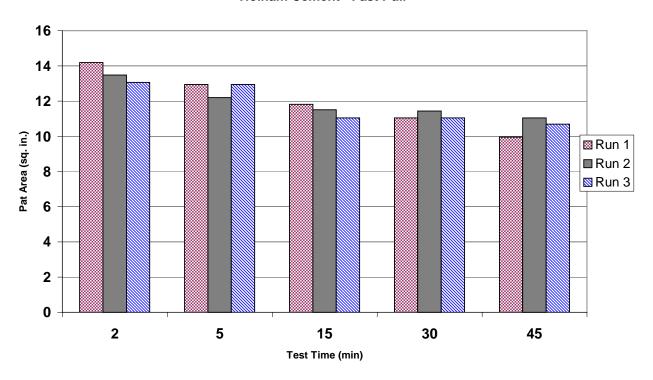


Figure 9 – Holnam Cement Fast Pull

Ash Grove Cement - Normal Pull

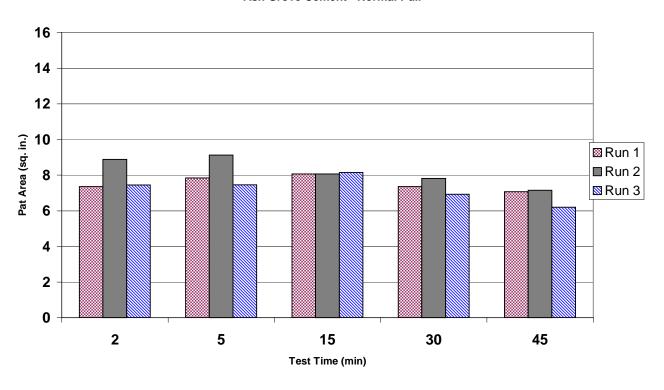


Figure 10 – Ash Grove Cement Normal Pull

Ash Grove Cement - Fast Pull

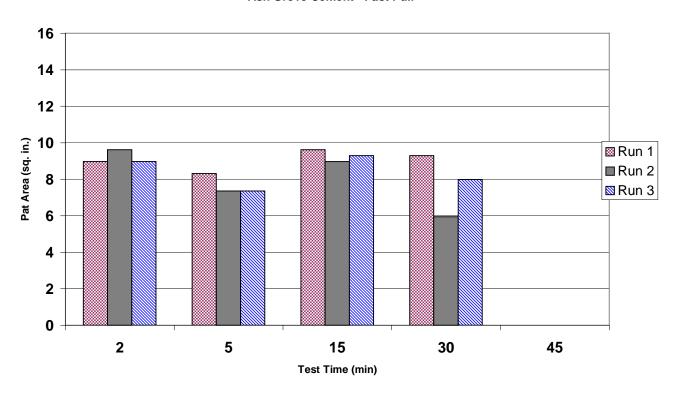


Figure 11 – Ash Grove Cement Fast Pull

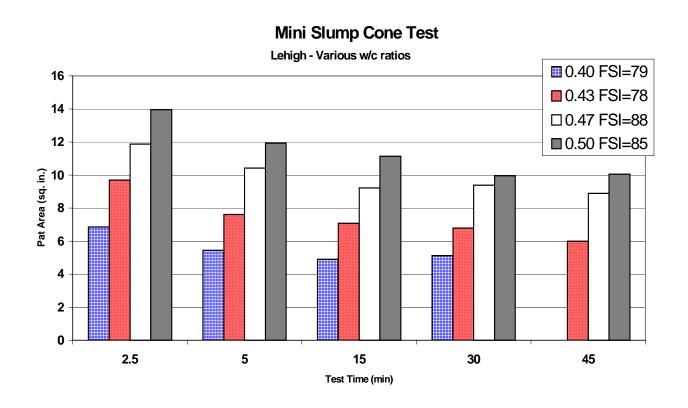


Figure 12 – Lehigh Cement Various w/c Ratios

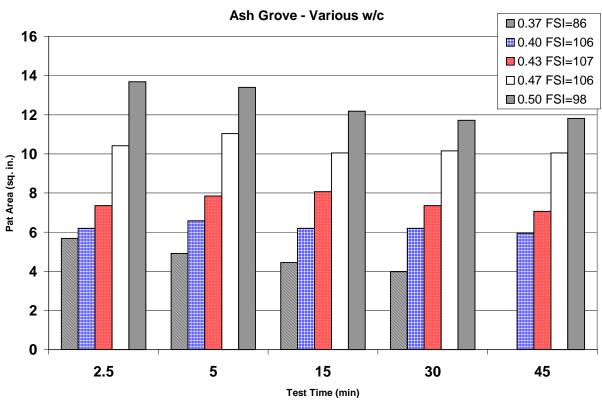


Figure 13 – Ash Grove Cement Various w/c Ratios

Run	2 min	5 min	15 min	30 min	45 min
1	15.90	21.40	18.70	17.70	17.30
2	16.30	23.63	19.87	15.59	17.72
3	12.10	19.28	17.06	17.17	13.59
4	13.59	18.70	18.17	10.78	12.38
5	14.83	17.72	14.19	12.57	13.89
6	13.79	17.50	15.90	13.79	14.39
7	12.60	17.61	16.12	12.66	10.35
Avg.	14.16	19.41	17.14	14.32	14.23
STD	1.59	2.31	1.93	2.58	2.60

Figure 14 – Table of Results Standard Sample Set B

Run	2 min	5 min	15 min	30 min	45 min
1	16.12	9.29	6.51	5.43	5.09
2	16.33	10.35	7.07	5.81	5.03
3	16.84	9.62	7.52	6.07	5.15
4	16.84	10.52	7.07	6.36	5.43
5	15.90	11.61	7.07	5.94	5.15
6	17.28	11.40	7.52	5.15	4.97
7	17.72	13.40	9.46	5.15	4.91
Avg.	16.72	10.88	7.46	5.70	5.10
STD	0.65	1.40	0.95	0.47	0.17

Figure 15 – Table of Results Standard Sample Set C