Quality Control Tools to Identify Source Variability of Class C Fly Ash and Its Impact on Freshly Mixed Cement-Fly Ash Paste

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ABSTRACT

The objective of this research was to identify tools available for quality control (QC) of as delivered class C fly ash. Tools investigated included penetration type devices and test procedures including the Iowa Set Time Test, Gillmore Needle, and Vicat Needle. Class C fly ash samples were obtained from 11 sources available to Louisiana Department of Transportation and Development (LADOTD). Samples were obtained twice a week for ten weeks for a total of about 20 samples per source. Statistical modeling was used to determine if a relationship existed between the various initial and final set times and the maximum temperature of the fly ash paste and the fly ash chemistry and fineness. Set time devices yielded similar results for both initial and final set. The test methods pointed out significant differences in set times between buckets within a source, but further testing when incorporating portland cement showed these differences to be negligible in effect. A suitable correlation was found to exist between the calcium oxide and sulfur trioxide content and the maximum temperature of the fly ash temperature results.

INTRODUCTION

Many entities currently use fly ash in PCC pavements and structures. Although the body of knowledge is great concerning the use of fly ash, several projects per year are subject to poor performance where fly ash is named as the culprit. Generally the "bad" projects arise due to:

1. Poor understanding of how fly ash affects concrete pavement construction and performance or
2. A switch of fly ash sources midstream during the construction project.

Although there may be several "bad" projects per year, there is general agreement that the use of class C fly ash has the following effects on concrete:

- Improved workability and finishability.
- Increased time of setting and has caused unpredictable change in time between initial and final set. (This is of particular concern for saw cutting operations.) The use of class C fly ash has also shown false or flash set tendencies in field construction operations.
- Despite early strength reduction, beyond 7 days concrete incorporating fly ash tend to show increased overall strengths over Portland cement concrete.
- The use of fly ash has been shown to reduce early rate of heat generation.
- Permeability is reduced in mature concrete and resistance to sulfate and chloride attack is improved.
- Freeze thaw resistance, modulus of elasticity and resistance to de-icing salts are all about the same as in ordinary Portland cement concrete.
- The use of fly ash and other supplementary cementitious materials (SCMs) in concrete helps reduce permeability and thus reduces chloride penetration leading to reduced corrosion of reinforcing steel.

The majority of electricity produced in the United States is produced from the combustion of coal at coal-fired utilities. As a result, over 117 million tons of coal combustion byproducts are produced per year[1]. The American Coal Ash Association (ACAA) estimates that fly ash comprise 68 million tons. The 68 million tons is broken down into the following categories and tonnages[1]:

- Bottom ash is approximately 18.7 million tons;
- Boiler slag totals approximately 2.5 million tons; and
- Other byproducts are approximated at 24.8 million tons.

The ACAA states that fly ash use continually grows, but less than 32 percent of coal combustion byproducts are recycled each year leading to a sluice pond or landfill disposal practices [1]. Of the fly ash being recycled, the widest application is as a partial replacement of cement in Portland cement concrete.

ASTM C618 [Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete] defines fly ash as the fine residue produced from the burning of ground or powdered coal[2]. Fly ash is collected from the flu gas of coal-fired boilers by the means of an electrostatic precipitator or bag house. Fly ash color may vary from tan to gray[3]. Self-cementing fly ash is produced from the burning of low sulfur, subbituminous, and lignite coals. Fly ash particles are typically spherical in nature and contain some crystalline as well as carbonaceous matter[3, 4]. Misra noted that a large percentage of fly ash is in the form of silica, alumina, ferric oxide, and calcium oxide[3].

ASTM C618 states, "A pozzolan is a material rich in silica and alumina that has little or non self-cementing properties, but will, in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties[2]."

Research states that the pozzolinity of fly ash is mainly dependent upon the fineness of the ash, amounts of silica and alumina, and the presence of moisture and free lime[3, 4]. Winkerton and Pamukcu also state that density, amount of carbon, temperature, and age also affect the rate of pozzolanic reaction[5].

The objective of this research was to identify tools available for quality control (QC) of delivered class C fly ash, focusing primarily on penetration type devices and test procedures. The quick heat generation index test was also investigated, but is not the results are not included in this paper for length purposes. Sources with significant variation were then combined with portland cement and further tested to determine the effect of fly ash variability on the setting time characteristics of the mixture.

MATERIALS AND TEST METHODS

Materials

Eleven class C fly ash sources were included in this study. Each source is commercially available for use in Louisiana Department of Transportation and Development (LADOTD) construction projects. Each source was sampled twice weekly for a period of ten weeks totaling 20 samples per source, more or less. The non-combined 5-gallon bucket samples were taken from production by the ash companies and delivered to the concrete research laboratory at the Louisiana Transportation Research Center (LTRC) for testing. Each bucket was sampled and chemically characterized according to ASTM C618. The chemical characterization results can be found here[11].

Test Methods

The set time of each fly ash was determined by the following three different test methods. Each set time method was performed in triplicate and the results averaged.

- ASTM C191 [Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle][6]
- ASTM C266 [Standard Test Method for Time of Setting of Hydraulic-Cement Paste by Gillmore Needles][7]
- Iowa Set Time Test

The ASTM C191 and C266 test procedures were modified by changing the specified water content to a water content of 27.5 percent or a water/fly ash ration of 0.275. Without this change, the faster setting fly ash sources would have set to quick to determine the set time. Note that all set time tests were conducted to final set or an elapsed time of 240 minutes, whichever was greater.

The procedure for the Iowa Set Time Test is as follows:

1. Weigh out approximately 500 grams of fly ash.
2. Weigh the proper amount of water for 27.5 percent water content.
3. Mix with a mixer that conforms to ASTM C305 on speed one for 10 seconds, and then switch to speed two and mix for 50 seconds using a wire whip[8].
4. Spread mixture evenly in a suitable size container and determine the penetration resistance of the mixture about every 5 minutes using a pocket penetrometer.
5. Plot the elapsed time versus the penetration resistance. Initial set is determined to be the time at which the material exerts some penetration resistance, and the final set is determined to be when the penetration resistance is 4.5 tons per square foot.

RESULTS AND DISCUSSION

Chemical Characterization Results

The chemical characterization results (shown in Rupnow[11]) tend to show differences between sources. The standard deviations are very low for most measured properties as is expected. The coefficient of variation tends to be a little higher for the moisture content, LOI, and SO^3. This shows that, for the most part, the fly ashes tested are generally the same with little variation between each of the buckets for each source over about a 10 week period.

Set Time Results

After completing the set time testing, the initial and final set times were plotted for each bucket number to determine correlations. Figure 1 to Figure 3 shows the initial and final set times for the Gillmore needle, Vicat needle, and pocket penetrometer for Source 1, respectively. The remaining figures for Sources 2 to 11 can be found here[11].

Note the strong correlation between the initial and final set times for each test. Note that the Gillmore needle and the Vicat needle tend to give nearly the same set time results for each bucket. The pocket penetrometer, or Iowa set time test, tends to give shorter times to initial and final sets for each bucket. The author believes that this is due to the much increased surface area of the testing apparatus of the pocket penetrometer compared to those of the Vicat and Gillmore needles. These findings proved consistent throughout all sources tested.

Of particular interest are the results for bucket number 17. Note the increased set time at bucket number 17 for all set time tests showing that it is significantly different than all other buckets of material.

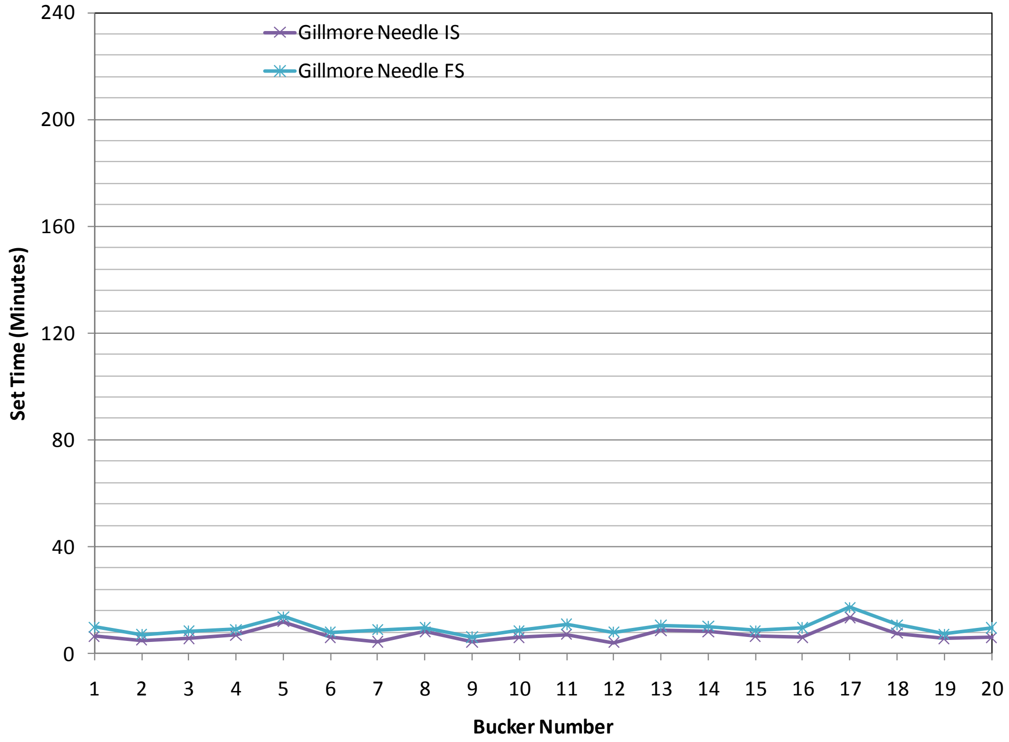

Figure 1. Gillmore needle initial and final set results versus bucket number for Source 1

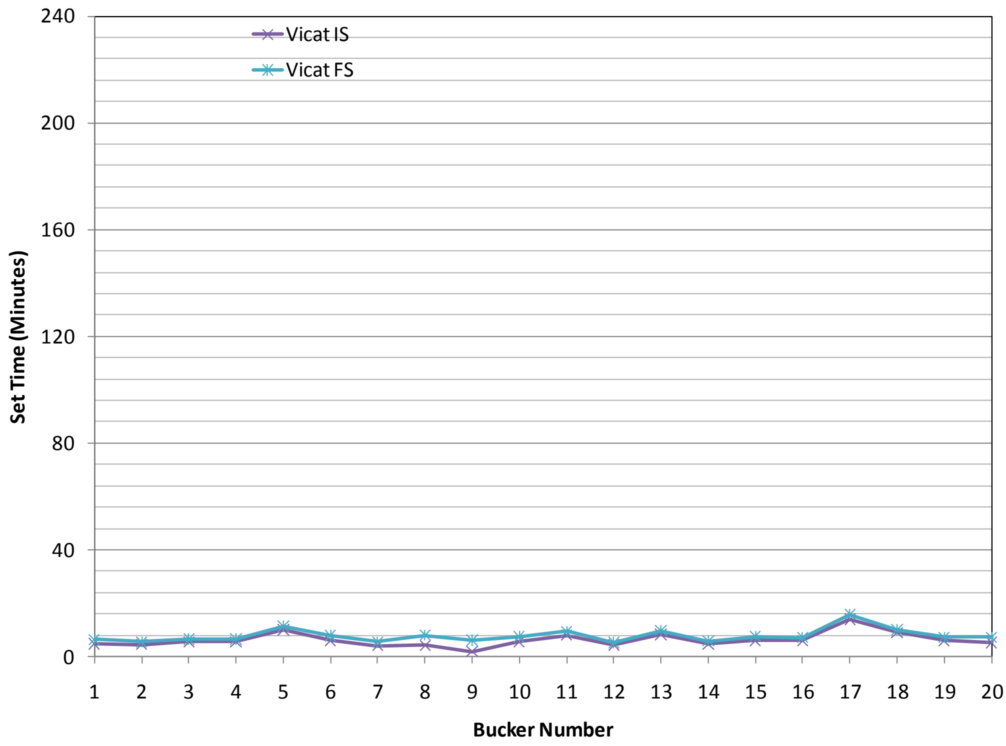

Figure 2. Vicat needle initial and final set results versus bucket number for Source 1

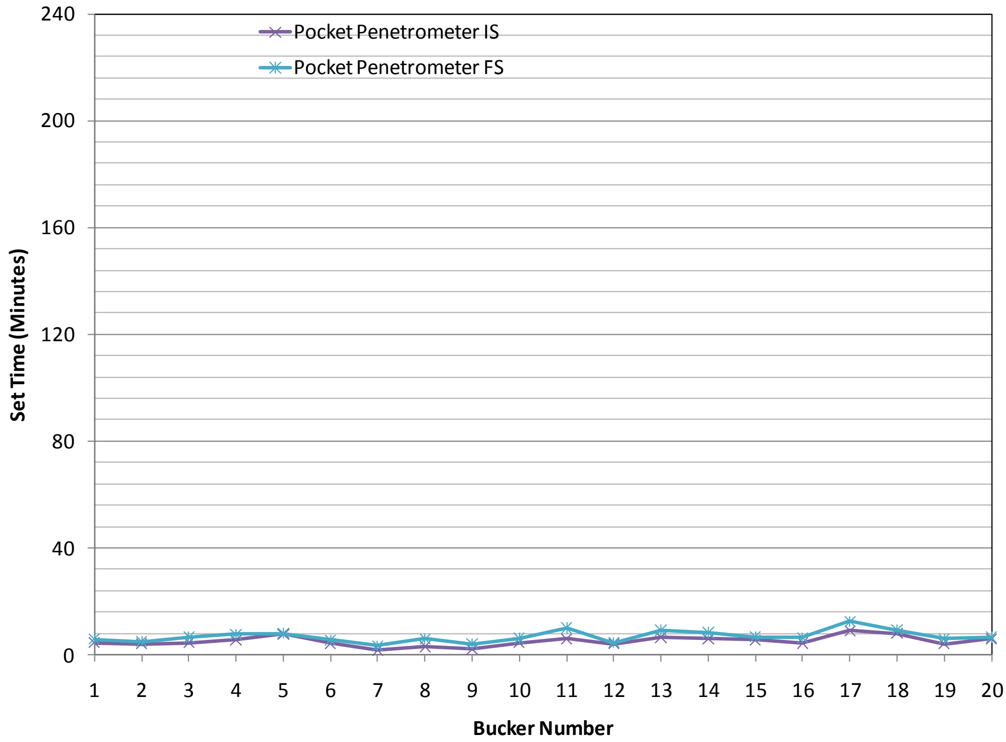

Figure 3. Pocket penetrometer initial and final set results versus bucket number for Source 1

Figure 4 to Figure 6 show the set time results for Source 8. Note the dramatic differences in set times between the different buckets for Source 8 indicating a widely variable material. Source 8 was then earmarked for further investigation as to the effects of source variability on portland cement systems.

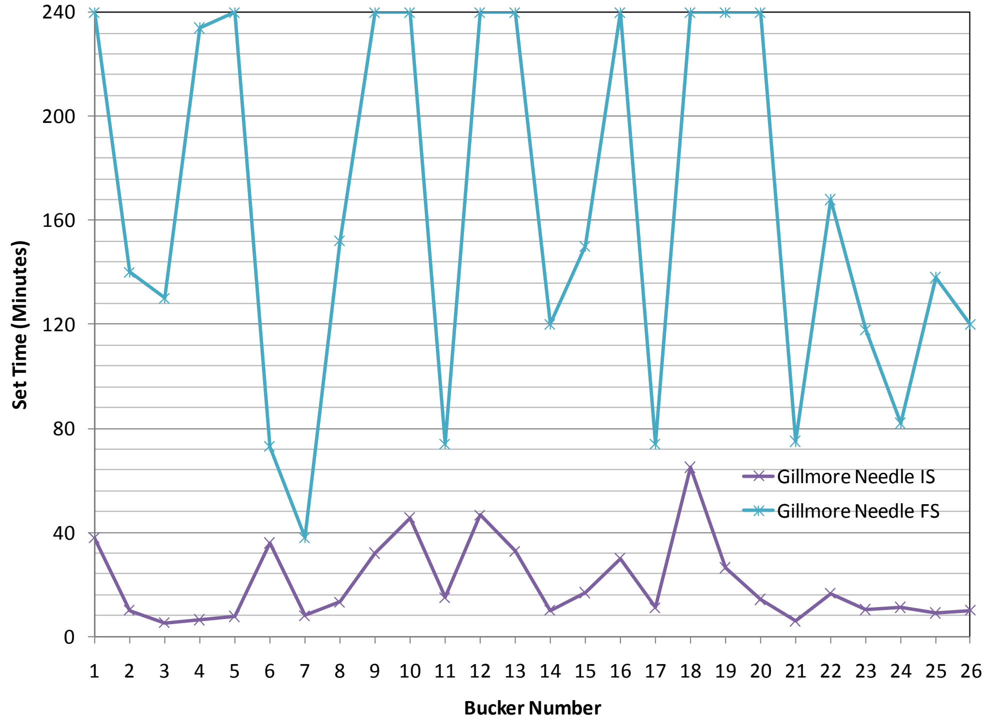

Figure 4. Gillmore needle initial and final set results versus bucket number for Source 8

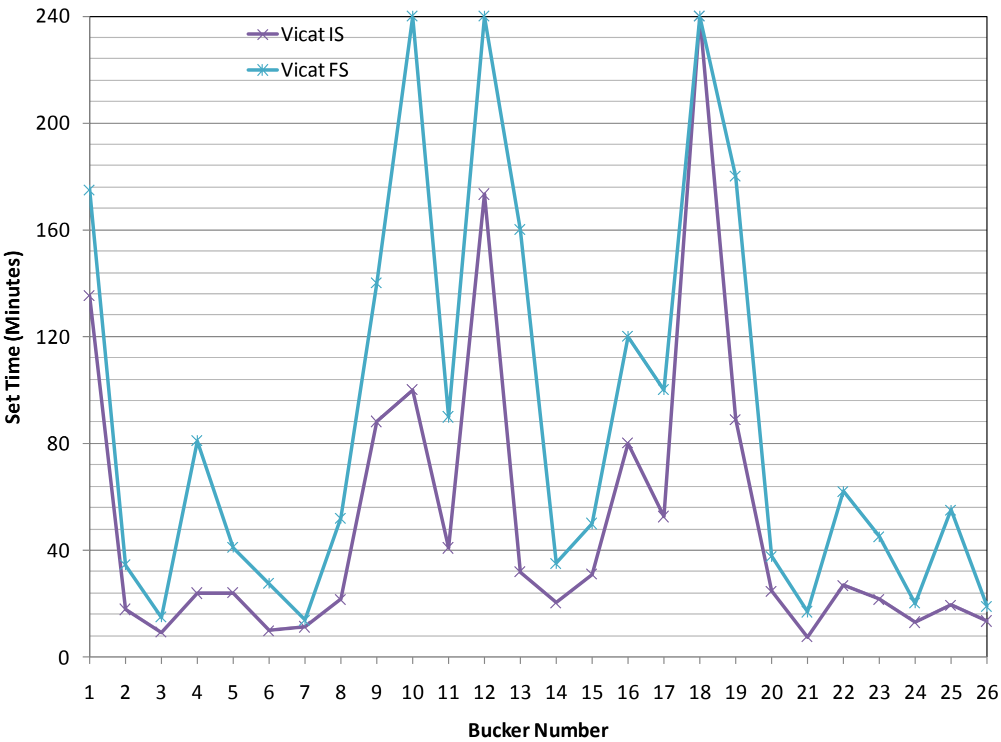

Figure 5. Vicat needle initial and final set results versus bucket number for Source 8

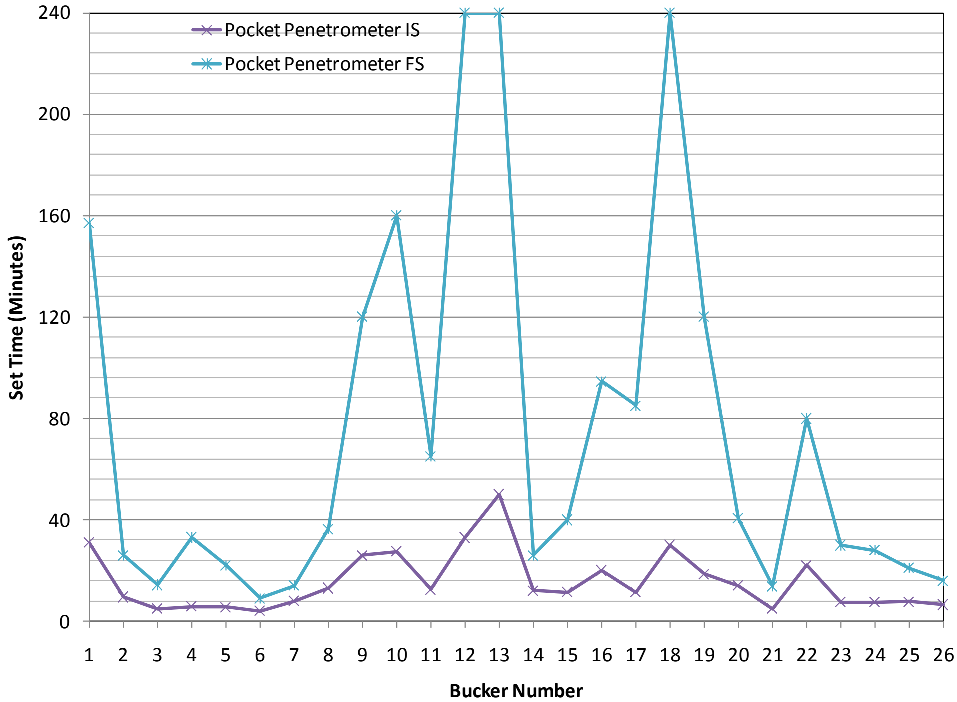

Figure 6. Pocket penetrometer initial and final set results versus bucket number for Source 8

The sources were selected for the portland cement systems portion of the study, Source 5 and Source 8. Source 5 was chosen as it was regarded as an average of the ashes tested. Source 8 was chosen as it represented the most variable ash of those selected and tested for this project.

The Vicat set time method was used and the mix design was produced as noted in the methodology section with the exception that 50 percent of the fly ash was replaced with type I/II portland cement by mass. The results for Source 5 and 8 are shown in Figure 7 and Figure 8, respectively.

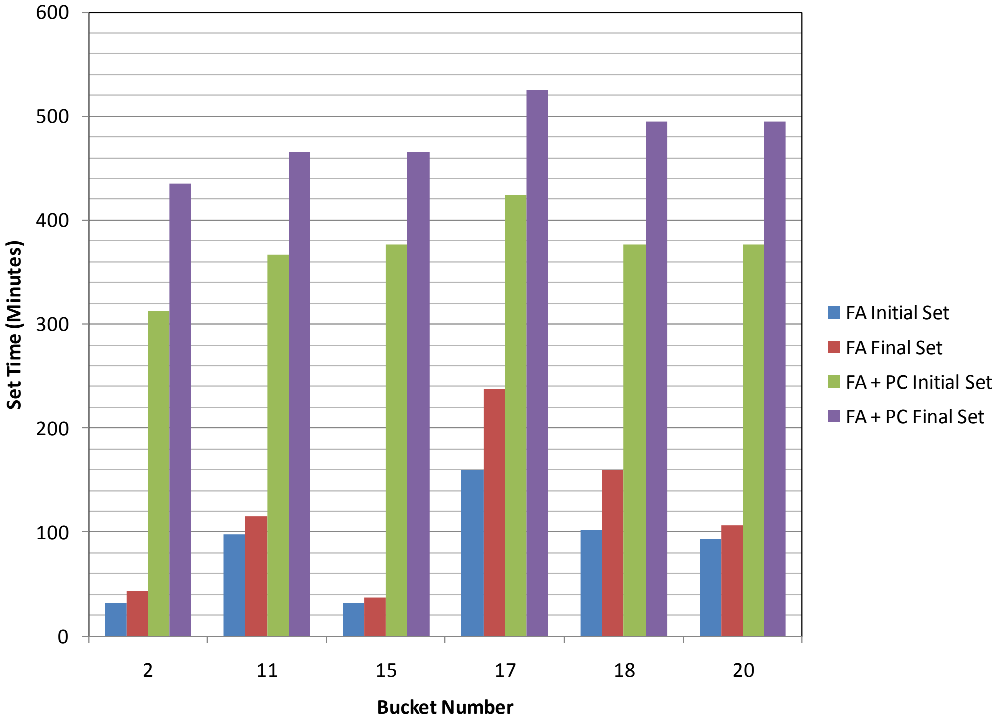

Figure 7. Comparison of Vicat initial and final set values for fly ash and 50 percent fly ash – 50 percent cement combinations from Source 5

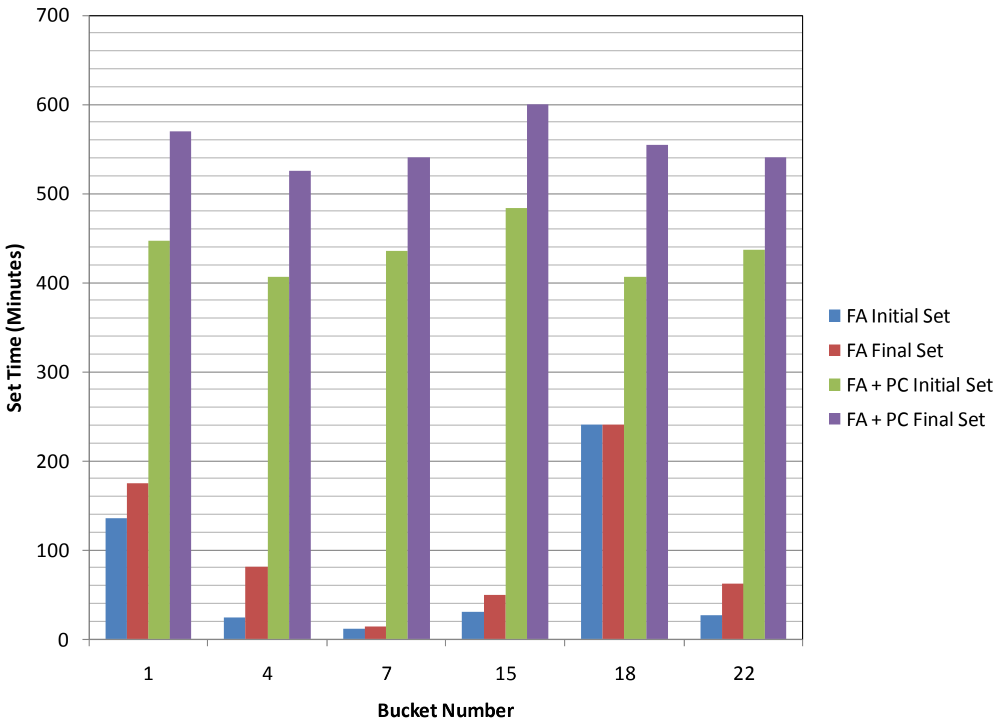

Figure 8. Comparison of Vicat initial and final set values for fly ash and 50 percent fly ash – 50 percent cement combinations from Source 8

The portland cement (PC) fly ash (FA) combined set time results showed that the perceived variability of the fly ash is not an issue. Note the results from Source 8 showed significant differences in the final set times between the buckets for the fly ash only samples. When comparing the results of the 50 percent fly ash / 50 percent cement samples, the final set values all fall within 80 minutes of each other. This indicates that at an increased replacement of fly ash, more than currently allowed under LADOTD specifications, does not lead to erratic set times of the portland cement concrete mixture. The same results are also validated when examining the results from Source 5.

The set time results of the fly ash only mixtures indicate that the test procedures may flag changes in chemistry or physical properties. The reality is that fly ash is a unique material with both a glassy and crystalline phase that can vary from particle to particle. The results shown in Figure 7 and Figure 8 indicate that this source variability is not a problem at a 50 percent portland cement replacement level, suggesting a lesser issue at LADOTD's currently accepted 20 percent replacement level.

Statistical Significance and Modeling Results

JMP 8.0 software was originally used to determine significance at an alpha level of 0.05[12]. The results of the t-test showed that each sample (i.e., bucket) was significantly different from the others within the same source. The research team examined the results more closely and determined that this was due to the very low variability in the measured set time results. The variability of the set time results was very low, on the order of less than 30 seconds to 3 minutes, between set time samples produced from the same mixing batch. This low variability produced little overlap between samples tested from each bucket.

The research team then used ASTM E178 to determine test results that were outliers[13]. A significance level of 0.05 was used in the analysis. The results were then much more realistic due to the methodology of comparing the average of the results for each bucket against the mean results for all samples of the source.

Table 1 to Table 11 show the results for the determination of outliers for all sources, the average, standard deviation, and the coefficient of variation for each source. Note that the results are an average of three tests and the outliers are shaded gray. Note the low number of outliers for Source 9 and Source 6. The results show that the class C fly ashes used in the State of Louisiana are generally uniform and the variations that occur are of no concern especially when being added to portland cement concrete.

Care should be exercised when interpreting the data from Source 11. Because of a very low number of actual samples exhibiting a set time and the removal of samples that did not exhibit a set time from the mean and standard deviation calculations, the statistical analysis showed all samples that did not set to be outliers. The author believes that the opposite is true and the samples that actually set are the outliers in this case.

The results also show varying degrees of variability of the individual sources when comparing the coefficients of variation (COV). The COVs range from 15% to 135% depending upon the source and the test method. Note that as the variability increases, the likelihood of producing an outlier becomes smaller.

Table 1. Average set time results for all set time tests for Source 1

	Gillmore Needle		Vicat Needle		Pocket Penetrometer	
Bucket Number	Initial Set (Minutes)	Final Set (Minutes)	Initial Set (Minutes)	Final Set (Minutes)	Initial Set (Minutes)	Final Set (Minutes)
1	6.33	9.82	4.72	6.33	4.50	5.83
2	4.90	7.05	4.53	5.50	4.00	5.00
3	5.68	8.43	5.52	6.50	4.50	6.58
4	6.83	8.98	5.50	6.50	5.50	7.67
5	11.88	13.73	9.95	11.33	7.67	8.00
6	6.00	8.00	5.97	7.83	4.25	5.50
7	4.42	8.90	4.06	5.50	1.75	3.33
8	8.19	9.66	4.17	8.00	3.00	6.00
9	4.39	6.08	1.85	6.05	2.00	4.00
10	6.00	8.50	5.57	7.33	4.50	6.17
11	7.18	10.83	8.00	9.45	6.00	10.00
12	4.12	8.00	4.31	5.25	4.00	4.50
13	8.52	10.43	8.28	9.66	6.50	9.13
14	8.06	10.18	4.65	5.84	6.00	8.25
15	6.33	8.60	5.91	7.50	5.50	6.67
16	5.92	9.57	5.91	7.17	4.34	6.33
17	13.50	17.25	13.97	15.75	9.00	12.50
18	7.58	10.75	8.98	10.00	8.00	9.33
19	5.50	7.25	5.90	7.25	4.00	6.00
20	6.00	9.50	5.28	7.25	6.00	6.50
Average	6.87	9.58	6.15	7.80	5.05	6.86
stdev	2.36	2.44	2.62	2.49	1.87	2.20
cov (%)	34.43	25.52	42.65	31.90	36.94	32.03

Table 2. Average set time results for all set time tests for Source 2

Bucket Number	Gillmore Needle Initial Set (Minutes)	Gillmore Needle Final Set (Minutes)	Vicat Needle Initial Set (Minutes)	Vicat Needle Final Set (Minutes)	Pocket Penetrometer Initial Set (Minutes)	Pocket Penetrometer Final Set (Minutes)
1	19.16	27.38	17.00	21.00	15.00	21.67
2	16.60	30.45	17.05	21.50	14.00	22.50
3	17.45	30.25	17.12	20.50	12.00	21.67
4	15.08	39.72	15.99	24.50	12.00	27.00
5	16.00	18.00	13.49	14.58	12.00	15.00
6	19.25	24.00	19.97	22.10	13.67	20.50
7	11.83	20.00	13.58	15.75	10.00	16.00
8	16.75	28.08	16.39	20.00	13.50	20.83
9	17.54	38.35	19.74	25.00	13.00	27.00
10	18.00	21.50	15.41	18.00	13.00	17.67
11	14.02	21.68	13.41	15.50	10.00	15.00
12	15.97	32.13	14.21	19.00	10.00	21.00
13	14.80	19.38	13.34	15.50	12.00	15.00
14	13.78	17.10	12.52	14.25	11.00	14.00
15	12.50	17.00	14.29	17.00	10.25	14.17
16	14.33	24.66	14.22	16.50	11.50	17.33
17	13.50	17.88	10.96	14.00	11.00	14.00
18	10.00	14.46	11.92	14.00	9.00	10.50
19	9.00	15.00	9.11	11.00	7.33	10.00
20	16.50	21.00	17.73	20.50	14.00	18.75
Average	15.10	23.90	14.87	18.01	11.71	17.98
stdev	2.80	7.36	2.79	3.80	1.93	4.77
cov (%)	18.54	30.78	18.77	21.10	16.44	26.51

Table 3. Average set time results for all set time tests for Source 3

Bucket Number	Gillmore Needle Initial Set (Minutes)	Gillmore Needle Final Set (Minutes)	Vicat Needle Initial Set (Minutes)	Vicat Needle Final Set (Minutes)	Pocket Penetrometer Initial Set (Minutes)	Pocket Penetrometer Final Set (Minutes)
1	8.80	98.80	19.69	60.00	9.00	36.00
2	10.00	99.00	16.27	55.00	2.50	28.00
3	5.92	95.83	16.04	44.00	5.00	23.00
4	10.08	114.00	21.58	50.00	9.50	35.00
5	12.72	240+	34.40	90.00	9.00	47.00
6	11.77	136.00	20.98	58.00	8.00	38.33
7	21.19	105.00	27.80	50.00	12.00	34.50
8	12.25	123.65	20.46	42.00	10.00	27.08
9	9.50	131.00	17.66	40.00	8.00	23.25
10	12.00	133.50	34.83	80.00	8.00	54.00
11	16.00	135.00	29.29	56.00	11.50	45.00
12	10.83	147.00	21.34	52.00	7.00	35.00
13	27.00	240+	28.99	95.00	13.00	98.00
14	9.25	124.00	16.52	50.00	7.50	32.00
15	13.22	240+	32.77	91.00	9.75	48.50
16	6.28	31.12	7.81	10.00	6.00	10.00
17	8.83	76.00	11.50	25.00	6.50	13.50
18	67.61	240+	73.92	100.00	41.00	100.00
19	9.03	17.63	14.41	38.00	5.00	19.33
20	19.50	93.00	46.90	69.00	13.00	75.00
Average	15.09	131.03	25.66	57.75	10.06	41.13
stdev	13.39	64.68	14.71	23.69	7.78	24.69
cov (%)	88.75	49.37	57.35	41.02	77.29	60.03

Table 4. Average set time results for all set time tests for Source 4

Bucket Number	Gillmore Needle Initial Set (Minutes)	Gillmore Needle Final Set (Minutes)	Vicat Needle Initial Set (Minutes)	Vicat Needle Final Set (Minutes)	Pocket Penetrometer Initial Set (Minutes)	Pocket Penetrometer Final Set (Minutes)
1	29.00	42.00	27.81	32.33	27.00	30.00
2	10.00	15.00	10.01	10.75	8.00	10.00
3	8.97	11.92	8.47	9.50	7.00	9.50
4	25.00	41.00	26.91	31.00	23.50	26.50
5	9.35	16.00	10.47	11.50	9.50	11.50
6	9.62	13.43	9.97	11.00	9.00	10.25
7	39.00	54.00	39.16	49.00	34.00	40.83
8	26.00	48.00	26.05	33.33	23.00	29.50
9	19.00	26.50	20.14	22.67	20.00	22.33
10	8.22	12.12	8.47	9.50	7.00	9.50
11	10.10	15.40	10.46	11.50	9.00	11.00
12	8.22	13.37	9.45	11.00	8.00	10.50
13	7.12	12.10	6.98	8.75	6.50	8.50
14	15.58	43.88	13.40	16.50	11.00	18.67
15	8.90	15.63	9.17	10.75	7.00	9.67
16	42.00	60.00	43.56	55.00	27.00	44.83
17	16.50	26.15	19.85	21.50	9.50	21.00
18	6.66	11.20	7.27	8.25	7.00	8.50
19	43.00	63.00	35.67	42.00	44.00	50.50
20	23.47	40.45	26.44	33.00	17.25	29.00
Average	18.29	29.06	18.49	21.94	15.71	20.60
stdev	12.11	18.03	11.61	14.65	10.81	13.23
cov (%)	66.24	62.05	62.79	66.78	68.81	64.21

Table 5. Average set time results for all set time tests for Source 5

Bucket Number	Gillmore Needle Initial Set (Minutes)	Gillmore Needle Final Set (Minutes)	Vicat Needle Initial Set (Minutes)	Vicat Needle Final Set (Minutes)	Pocket Penetrometer Initial Set (Minutes)	Pocket Penetrometer Final Set (Minutes)
1	37.05	85.28	48.16	60.00	31.00	54.00
2	26.27	50.75	31.61	43.00	15.00	37.50
3	35.05	71.08	46.15	54.00	31.00	50.00
4	32.88	69.50	39.75	45.00	26.50	46.50
5	29.06	33.55	35.87	43.00	25.00	43.00
6	38.15	56.05	45.08	51.00	34.25	52.00
7	38.36	105.00	54.00	71.16	32.00	51.00
8	54.60	121.00	44.30	85.00	44.00	82.00
9	43.00	83.50	53.41	62.50	42.50	63.00
10	40.17	76.00	51.11	60.00	37.00	62.00
11	78.00	146.00	97.89	115.00	65.00	111.00
12	48.00	94.00	57.30	63.83	45.00	68.58
13	42.55	60.56	52.20	64.67	45.00	78.00
14	60.28	120.37	61.06	68.67	60.00	95.00
15	25.92	46.35	31.87	36.50	22.00	36.00
16	70.50	118.50	78.62	91.00	60.50	85.00
17	163.00	240.00	159.83	238.00	105.00	185.00
18	87.00	180.27	102.12	160.00	70.00	130.00
19	240+	240+	240+	240+	240+	240+
20	86.50	163.00	93.56	106.00	72.00	100.00
Average	54.54	101.09	62.31	79.91	45.41	75.24
stdev	32.62	52.39	31.77	48.64	22.03	37.19
cov (%)	59.81	51.82	50.98	60.87	48.51	49.42

Table 6. Average set time results for all set time tests for Source 6

Bucket Number	Gillmore Needle Initial Set (Minutes)	Gillmore Needle Final Set (Minutes)	Vicat Needle Initial Set (Minutes)	Vicat Needle Final Set (Minutes)	Pocket Penetrometer Initial Set (Minutes)	Pocket Penetrometer Final Set (Minutes)
1	12.65	28.47	12.28	15.00	8.50	17.50
2	6.00	15.08	7.45	9.25	5.00	8.50
3	38.00	76.50	49.88	67.00	15.00	60.00
4	5.85	15.25	7.17	8.75	4.00	8.50
5	2.38	8.78	2.60	4.50	2.00	4.00
6	3.40	6.28	3.44	4.25	2.50	3.75
7	2.12	4.45	2.68	3.25	2.00	3.00
8	2.50	4.35	1.81	3.50	1.00	3.50
9	2.00	4.53	3.03	3.75	2.50	3.50
10	2.60	3.83	3.44	4.00	2.00	3.00
11	3.50	5.75	3.44	4.00	2.75	3.75
12	2.93	6.68	3.22	4.00	2.50	4.00
13	6.37	10.77	5.88	8.75	5.00	7.00
14	5.60	10.88	5.73	7.00	4.00	7.50
15	4.00	5.77	4.45	5.25	3.50	4.50
16	4.50	6.50	4.43	5.00	3.50	5.00
17	3.00	5.55	3.19	3.75	2.50	4.50
18	2.17	4.80	2.68	3.25	2.00	3.25
19	5.45	14.00	5.56	8.00	2.00	7.25
20	5.00	12.00	6.52	7.75	4.00	6.67
Average	6.00	12.51	6.94	9.00	3.81	8.43
stdev	7.91	16.16	10.39	13.97	3.10	12.59
cov (%)	131.89	129.20	149.65	155.18	81.32	149.23

Table 7. Average set time results for all set time tests for Source 7

	Gillmore Needle		Vicat Needle		Pocket Penetrometer	
Bucket Number	Initial Set (Minutes)	Final Set (Minutes)	Initial Set (Minutes)	Final Set (Minutes)	Initial Set (Minutes)	Final Set (Minutes)
1	6.75	15.00	6.84	8.00	6.00	9.17
2	21.35	21.92	11.49	13.25	9.75	15.00
3	9.90	14.00	10.91	11.75	9.00	12.00
4	8.78	19.67	9.46	10.25	8.00	10.50
5	20.72	31.58	20.26	23.00	16.00	22.00
6	11.00	24.62	12.31	13.25	10.00	15.00
7	12.70	21.35	11.42	13.00	10.00	12.67
8	8.88	23.43	8.27	10.00	7.00	9.67
9	24.00	35.90	26.40	31.00	20.00	40.67
10	16.90	35.77	14.63	16.50	14.00	17.33
11	11.92	73.50	14.03	17.50	9.00	14.50
12	12.50	42.50	10.40	13.00	10.00	13.00
13	4.83	7.50	5.20	7.00	4.00	5.50
14	15.97	45.15	19.38	25.00	13.00	26.00
15	12.00	133.50	8.38	80.00	8.00	54.00
16	11.67	24.62	13.32	15.00	9.50	14.50
17	8.25	16.62	7.12	9.25	6.00	8.00
18	14.60	29.05	14.12	17.50	11.00	16.00
19	8.00	20.15	7.40	10.00	7.00	9.50
Average	12.67	33.46	12.18	18.12	9.86	17.11
stdev	5.17	28.33	5.28	16.19	3.79	11.89
cov (%)	40.80	84.65	43.38	89.36	38.41	69.49

Table 8. Average set time results for all set time tests for Source 8

Bucket Number	Gillmore Needle Initial Set (Minutes)	Gillmore Needle Final Set (Minutes)	Vicat Needle Initial Set (Minutes)	Vicat Needle Final Set (Minutes)	Pocket Penetrometer Initial Set (Minutes)	Pocket Penetrometer Final Set (Minutes)
1	37.78	240+	135.23	175.00	31.00	157.00
2	10.00	140.00	17.85	34.50	9.50	26.00
3	5.28	130.00	9.26	15.00	5.00	14.33
4	6.35	234.00	23.87	81.00	5.75	33.00
5	7.68	240+	24.07	41.00	5.50	22.00
6	36.00	73.00	9.92	27.50	4.00	9.00
7	8.00	38.00	11.35	13.83	8.00	14.00
8	13.25	152.00	21.63	52.00	13.00	36.33
9	32.08	240+	88.18	140.00	26.00	120.00
10	45.63	240+	100.00	240+	27.50	160.00
11	15.00	74.00	40.78	90.00	12.50	65.00
12	46.70	240+	173.33	240+	33.00	240+
13	32.82	240.00	31.92	160.00	50.00	240+
14	10.00	120.00	20.29	35.00	12.00	26.00
15	16.83	150.00	31.00	50.00	11.50	40.00
16	30.00	240+	80.07	120.00	20.00	94.50
17	11.00	74.00	52.63	100.00	11.50	85.00
18	65.00	240+	240+	240+	30.00	240+
19	26.35	240+	88.95	180.00	18.50	120.00
20	14.25	240+	24.67	37.67	14.00	40.50
21	6.02	75.00	7.49	17.00	5.00	13.83
22	16.50	168.00	26.75	62.00	22.00	80.00
23	10.25	117.83	21.59	45.00	7.50	30.00
24	11.15	82.00	13.00	20.10	7.50	28.00
25	9.00	138.00	19.36	55.00	7.75	21.00
26	10.05	120.00	13.47	19.00	6.50	16.00
Average	20.50	125.05	43.47	68.29	15.56	54.41
stdev	15.63	55.14	43.23	53.03	11.39	46.84
cov (%)	76.24	44.10	99.45	77.66	73.21	86.09

Table 9. Average set time results for all set time tests for Source 9

	Gillmore Needle		Vicat Needle		Pocket Penetrometer	
Bucket Number	Initial Set (Minutes)	Final Set (Minutes)	Initial Set (Minutes)	Final Set (Minutes)	Initial Set (Minutes)	Final Set (Minutes)
1	3.08	6.03	3.36	4.25	2.50	3.67
2	3.17	5.38	3.34	4.17	2.00	3.00
3	3.75	7.28	3.98	4.67	3.00	4.08
4	2.70	4.95	2.93	3.50	2.50	3.25
5	2.75	4.43	3.15	3.67	2.50	3.25
6	2.46	4.87	2.94	3.50	2.50	3.25
7	3.88	5.38	3.40	3.92	3.00	4.50
8	2.58	4.48	3.17	3.75	2.50	3.25
9	2.97	4.40	3.13	3.75	2.50	3.00
10	3.75	5.67	3.97	4.75	3.25	4.50
11	4.92	6.93	3.70	4.50	3.50	5.17
12	4.22	7.62	4.87	5.75	3.00	5.00
13	2.62	6.67	3.14	3.75	2.25	3.50
14	4.00	8.08	3.64	4.50	3.25	4.50
15	3.50	6.00	3.94	4.50	2.50	4.50
16	2.78	4.07	3.11	3.75	2.50	3.50
17	3.00	6.08	2.85	3.50	2.50	3.50
18	6.35	7.50	6.36	7.00	5.92	6.62
19	3.93	17.00	5.78	11.00	3.25	8.50
20	2.93	5.42	3.24	3.75	2.50	3.50
Average	3.47	6.41	3.70	4.60	2.87	4.20
stdev	0.94	2.76	0.94	1.73	0.82	1.36
cov (%)	27.21	43.00	25.53	37.68	28.47	32.31

Table 10. Average set time results for all set time tests for Source 10

Bucket Number	Gillmore Needle Initial Set (Minutes)	Gillmore Needle Final Set (Minutes)	Vicat Needle Initial Set (Minutes)	Vicat Needle Final Set (Minutes)	Pocket Penetrometer Initial Set (Minutes)	Pocket Penetrometer Final Set (Minutes)
1	240+	240+	240+	240+	240+	240+
2	240+	240+	240+	240+	240+	240+
3	240+	240+	240+	240+	240+	240+
4	240+	240+	240+	240+	240+	240+
5	240+	240+	240+	240+	240+	240+
6	240+	240+	240+	240+	240+	240+
7	240+	240+	240+	240+	240+	240+
8	240+	240+	240+	240+	240+	240+
9	240+	240+	240+	240+	240+	240+
10	240+	240+	240+	240+	240+	240+
11	240+	240+	240+	240+	240+	240+
12	240+	240+	240+	240+	240+	240+
13	240+	240+	240+	240+	240+	240+
14	240+	240+	240+	240+	240+	240+
15	240+	240+	240+	240+	240+	240+
16	240+	240+	240+	240+	240+	240+
17	240+	240+	240+	240+	240+	240+
18	240+	240+	240+	240+	240+	240+
19	240+	240+	240+	240+	240+	240+
20	240+	240+	240+	240+	240+	240+
Average	N/A	N/A	N/A	N/A	N/A	N/A
stdev	N/A	N/A	N/A	N/A	N/A	N/A
cov (%)	N/A	N/A	N/A	N/A	N/A	N/A

Table 11. Average set time results for all set time tests for Source 11

Bucket Number	Gillmore Needle Initial Set (Minutes)	Gillmore Needle Final Set (Minutes)	Vicat Needle Initial Set (Minutes)	Vicat Needle Final Set (Minutes)	Pocket Penetrometer Initial Set (Minutes)	Pocket Penetrometer Final Set (Minutes)
1	240+	240+	240+	240+	66.00	240+
2	130.00	240+	214.86	240+	30.00	240+
3	240+	240+	135.00	160.00	71.00	230.00
4	89.00	240+	240+	240+	17.00	210.00
5	170.00	240+	240+	240+	55.00	240+
6	178.00	240+	240+	240+	25.00	240+
7	240+	240+	240+	240+	51.00	240+
8	240+	240+	240+	240+	38.00	240+
9	240+	240+	240+	240+	98.00	240+
10	240+	240+	240+	240+	240+	240+
11	240+	240+	240+	240+	240+	240+
12	240+	240+	240+	240+	240+	240+
Average	141.75	N/A	174.93	160.00	50.11	220.00
stdev	40.96	N/A	56.47	N/A	25.70	14.14
cov (%)	28.89	N/A	32.28	N/A	51.29	6.43

JMP 8.0 software was used in determining suitable models[12]. Input variables and their respective units are shown in Table 12. Response variables and their respective units are shown in Table 13. Note that attempts at simple linear regression modeling using one variable produced very low correlation values. To obtain a statistically significant model with the least number of variables, stepwise regression techniques were used alongside their respective residual plots to determine significance of the various input variables.

Table 12. Input variables and units for statistical modeling

Variable	Unit(s)
Silicon Dioxide	%
Ammonium Hydroxide Group	%
Sulfur Trioxide	%
Magnesium Oxide	%
Calcium Oxide	%
Fineness	% Retained on the 325 Sieve

Table 13. Response variables and units for statistical modeling

Response Variable	Unit(s)
Gillmore Initial Set	Minutes
Gillmore Final Set	Minutes
Vicat Initial Set	Minutes
Vicat Final Set	Minutes
Pocket Penetrometer Initial Set	Minutes
Pocket Penetrometer Final Set	Minutes
Maximum Temperature	°C
Time to Maximum Temperature	Minutes

Numerous attempts to obtain suitable models were attempted. The author was unable to find suitable models (i.e., R2 values were less than 0.5) to describe the initial and final set behavior and the time to maximum temperature for the data set using the input parameters defined in Table 12.

Equation 1 shows the regression equation for the maximum temperature. Note the correlation coefficient of 0.6255.

$$\text{Maximum Temperature} = -.406SiO2 + 0.236\text{Ammonium Hydroxide} + 2.56SO3 + .941MgO + 1.162CaO - .209\text{Fineness} + 4.015$$

Equation 1. Least squares regression analysis equation for maximum temperature

Equation 2 shows the stepwise regression analysis results for the maximum temperature. Note the slight reduction of the correlation coefficient from 0.6255 to 0.5798. Although the correlation coefficient has been reduced, the model makes sense due to the parameters of calcium oxide and sulfur trioxide. The addition of water to both of these compounds creates an exothermic chemical reaction giving off measurable heat.

$$\text{Maximum Temperature} = 3.612SO3 + 1.159CaO - 4.006$$

Equation 2. Stepwise Regression Analysis Equation for Maximum Temperature

The author believes the lack of modeling results show that hydration of self-cementing fly ash is a complex, poorly understood problem that can be addressed by further research. That being said, the author believes that the tricalcium aluminate content of the fly ash may be able to better define the set time and hydration characteristics. The drawback of using the tricalcium aluminate content is accurately quantifying or determining it. The tricalcium aluminate of fly ash can only be determined through semi-quantitative x-ray diffraction techniques that can be costly and very time consuming.

CONCLUSIONS

The results of this study warrant the following conclusions. The Gillmore Needle, Vicat Needle, and the pocket penetrometer yielded similar results when observing the times to initial and final set across the three test methods; therefore, any of these devices may be used to determine set time. Although all test methods pointed out significant differences in set times between buckets within a source, those differences were mitigated when incorporating portland cement into the sample. In other words, blending fly ash with portland cement normalized the set time of the fly ash, even from a source exhibiting high variability in set times when incorporated at 50 percent.

The statistical analysis results showed outliers within the sources, but further testing when incorporating portland cement showed these differences to be negligible.

A suitable correlation was found to exist between the calcium oxide and sulfur trioxide content and the maximum temperature of the fly ash temperature results.

RECOMMENDATIONS

The results of this study indicate that the hydration of class C fly ash is a complex phenomenon that cannot be fully described by the tests used in this study. To further define the relationship between fly ash chemistry and fly ash set time, another study must be undertaken to look at the tricalcium aluminate content and its role in hydration characteristics of class C fly ash.

The results of this study also show that the current practice of requiring set time tests to be conducted on fresh concrete mortar in the field are adequate and should be continued in quantifying field variation of set time.

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