FINAL Report

A Collaborative Study to Determine the Critical Chloride Threshold Test, OC_{crit}, Variability due to Material Sources

Prepared for

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by

PI: Ceki Halmen, PhD, PE (halmenc@umkc.edu) 816-235-1286

Associate Professor, Natural and Built Environment Division

University of Missouri – Kansas City, Kansas City, MO 64110

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I. Introduction

The critical chloride content, Cl_{crit}, is the quantity of chlorides at the reinforcement steel surface in concrete necessary to initiate corrosion. Knowing the value of Cl_{crit} is critical to determine the time to corrosion initiation and the service life of reinforced concrete structures. The Cl_{crit} data published in the literature exhibits significant scatter and varies between ~0.1 and 3.1% by weight of binder.[1] The variability in the published Cl_{crit} values has four sources:

1. Cl_{crit} test method used (geometry, steel condition, alkaline medium, exposure condition, monitoring and initiation criteria, sampling and chloride measurement, and other factors)
2. Physical and chemical properties of materials
3. Inter-laboratory variability
4. Random variability

Until recently, it was not possible to understand how much each of these sources in variability contribute to the reported variability of Cl_{crit} values in the literature because researchers were using different test methods in their labs with materials from different sources. To address this issue, the ACI 222 committee established a task group, TG1, to develop a standard Cl_{crit} laboratory test method. TG1 developed a framework for a standard Cl_{crit} test method and performed a CRC funded study to evaluate different proposed Cl_{crit} test methods satisfying this framework. Proposed test methods were performed in a round-robin test by different labs using materials from the same source. Results were evaluated for variability and one of the tested methods, the OC_{crit} method, was recommended by TG1 to the ACI 222 committee for further development as a standard Cl_{crit} test method.[2] This study, referred to as the first phase study in the remainder of this report, established the expected random variability and intra/inter-laboratory variability of the OC_{crit} method for a set of control materials obtained from a single source. This resolves three of the four listed sources of variability. However, for the OC_{crit} test to become a useful standard test method that can act as a reference for different researchers, the variability due to material differences needs to be evaluated (fourth source of variability). Physical and chemical properties of materials procured from different sources may have a significant effect on the test results, even if these materials satisfy the requirements of the same ASTM classification. A study that evaluated the effect of different cement sources on 28-day compressive strength of gravel aggregate concrete for example reported that a change in cement source could change the strength by up to 31.8 percent when comparing all samples from each cement source. [3]

a. Objective of the Study

The objective of this study was to measure the variability of OC_{crit} data due to different sources of materials that meet the requirements of the same ASTM specifications. Cement and steel, each procured from three different manufacturers in different parts of the country, was tested in different academic and commercial laboratories using the OC_{crit} test method to observe the variability in the results. The data presented in this report supplements the results of the first phase study and will allow the researchers to interpret the variances of OC_{crit} data obtained by different researchers in different laboratories.

b. Research Significance

The large variability of published Cl_{crit} data and lack of a standard Cl_{crit} test method creates significant difficulties for the concrete industry. Researchers cannot compare Cl_{crit} values obtained from their research to a standard value. Practitioners evaluating condition of reinforced concrete structures for maintenance and rehabilitation cannot make a reliable assessment based on measured chloride contents. Also, designers need a reliable estimate of the Cl_{crit} distribution to do a probabilistic analysis using available service life models. The data presented in this report combined with the results of the first phase study will allow researchers to collaborate with ASTM G01.14 committee to develop the OC_{crit} test as an ASTM standard.

II. OC_{crit} Test Method and Summary of First Phase Results

This section will provide a brief description of the OC_{crit} Test Method and a summary of first phase study results. More detailed information about the test method and results can be found in the first phase study report available on the ACI foundation website.[4] The OC_{crit} test is a macro-cell setup with separated anodes and cathodes made from mortar with embedded reinforcing steel samples as shown in Figure 1. The mortar mixture has a water-cement ratio of 0.42 and sand-cement ratio of 1.375. The anode of the OC_{crit} test is shaped like a dog-bone with a fully embedded 140 mm long No 5 (16 mm) reinforcing steel bar. The reinforcing steel bar located between the thicker ends of the specimen has a thin, uniform mortar cover with a radial cover thickness of 4.75 mm. The cathode is a mortar prism with five completely embedded 280 mm long No 5 (16 mm) steel bars. The mortar cover around the reinforcing steel bars and the horizontal distance between the steel bars is 25.4 mm. By connecting five anodes to each cathode, the OC_{crit} test setup provides a minimum 2:1 cathode to anode surface ratio. The reinforcing steel bars in anode and cathode specimens are used in as-received conditions without any surface treatment except degreasing through cleaning in xylene prior to fabrication of specimens. Anodes are placed in a saturated lime solution with 3.3% by weight NaCl solution and the cathodes are placed in saturated $Ca(OH)_2$ solution. The anodes and cathodes are electrically connected with a wire and the solutions are connected through a salt bridge to complete the macro-cell. The open circuit potential of steel embedded in the anodes are monitored daily using a Cu-$CuSO_4$ electrode and a high accuracy multimeter.

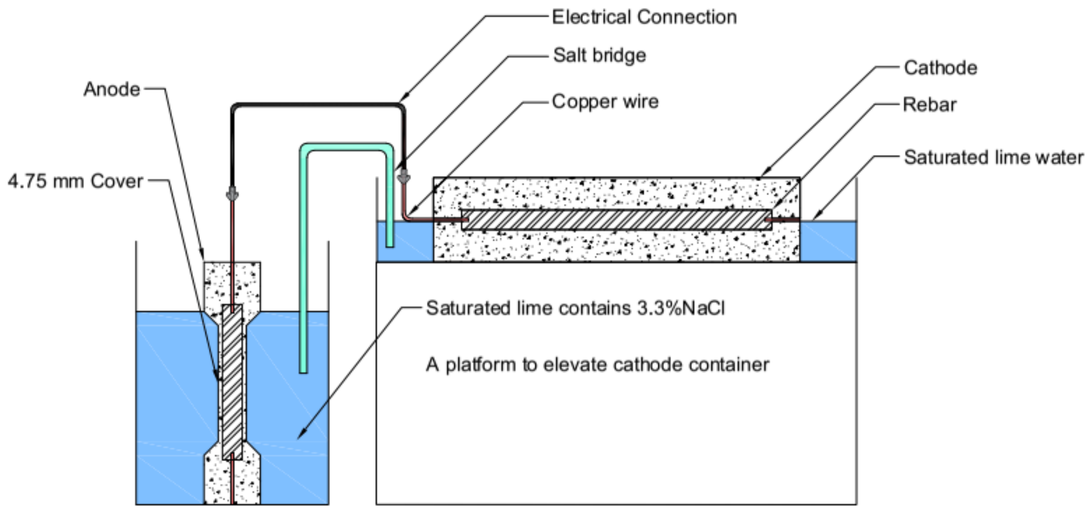


Figure 1 OC_{crit} Test Setup

Testing is terminated when a sample exhibits a steel potential less than -350 mV for two consecutive readings. After termination of testing (initiation of corrosion), the mortar with uniform thickness around the stem of the anodes is crushed and ground. The total acid soluble chloride content as percent by weight of the mortar powder (Cl % mortar) is determined following the ASTM C1152. The determined chloride content is then calculated as a percent by weight of cement in the mixture (Cl % cement) based on the mixture design. During the development of the OC_{crit} test, a statistical relationship between this average sample chloride content of the anode stem section, Cl % cement, and the critical chloride content at the steel mortar interface, Cl_{crit}, was established for mortar systems fabricated using ordinary portland cements (OPC) as shown in Eq. 1.[5] The critical chloride content of samples, Cl_{crit}, as percent by weight of cement is calculated using this relationship.

Eq. 1 $$Cl_{crit} = (0.492 + 0.004 \times TTA) \times Cl(\%\ cement)$$
Where: TTA is the time to activation in days.

a. Summary of the First Phase Study Results

As part of the first phase study, OC_{crit} test was performed in 3 laboratories: University of Missouri – Kansas City (UMKC), Oregon State University (OSU), and CTLGroup. The test was conducted twice at UMKC to assess the intra-lab variability. ASTM C 150 Type I/II Cement from a single source and ASTM A 615 Grade 60 Steel from a single source were used in all the laboratories. Processing of steel samples, such as cutting, drilling, and tapping were all performed at UMKC, and materials were shipped to the other labs ready to be tested. Figure 2 shows the mean Cl_{crit} values and distributions calculated using Eq. 1 for the four data sets obtained in the first phase study at 3 laboratories. Statistical analysis indicated that the differences between the mean Cl_{crit} values were statistically not significant at 95% confidence level. Combined data from all the test sets showed that the mean Cl_{crit} value of the tested steel in the mortar mixture was 0.423% by weight of cement with a standard deviation of 0.113.

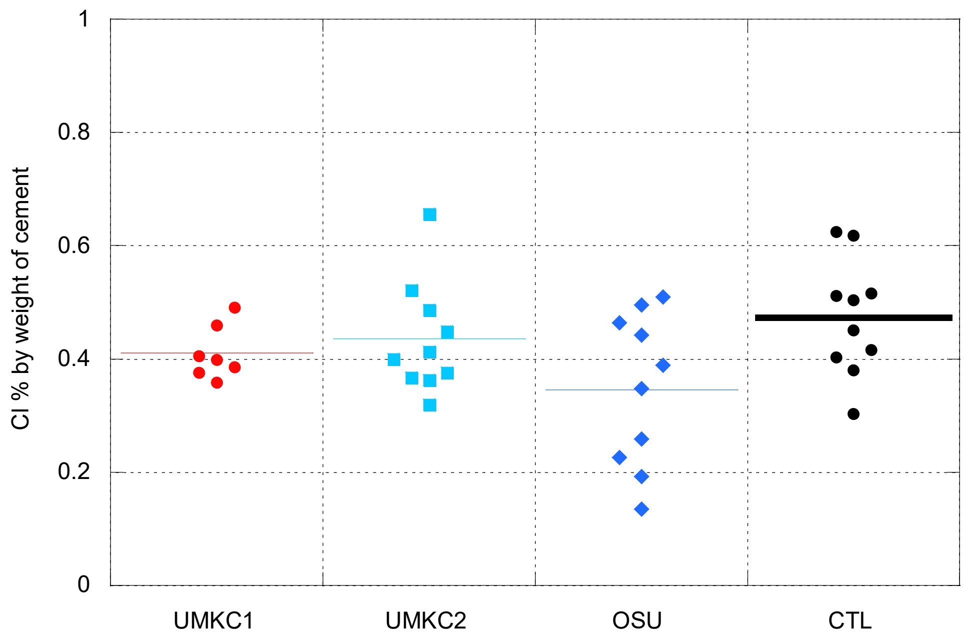


Figure 2 OC_{crit} First Phase Study Cl_{crit} Distributions

III. Experimental Design and Methodology

Although the initial proposal for this study planned for testing of 120 samples with participation of four laboratories, due to interest during the project kick-off meeting, the number of participating laboratories was increased to five for testing a total of 160 samples. The participating laboratories were a mix of academic and commercial labs, including University of Missouri – Kansas City (UMKC), Oregon State University (OSU), Colorado State University (CSU), CTLGroup, and Wiss, Janey, Elstner Associates (WJE). Three ASTM C150 Type I/II cements were procured from local sources by UMKC (C1), CSU (C2), and OSU (C3). Standard sand meeting the specifications of ASTM C 778 was procured from sole source and distributed to all participants for mortar production. Concrete Reinforcing Steel Institute (CRSI) was contacted to procure and supply the ASTM A615 Grade 60 steel samples from three different steel mills. Project team identified three mills, serving the Midwest, South, and West of US, based on differences in the scrap steel they use for production and based on different processes of production. Procurement of steel from two of the three mills was completed, identified as S1 and S2 in this report. The third mill was closed to production temporarily and was delayed in shipment of the samples, therefore a third commercial source was used to obtain the third steel sample, identified as S3 in this report. Steel samples S1 and S2 were processed (cut, drilled, tapped) at a machine shop arranged by CRSI and shipped separate samples to UMKC and OSU laboratories from where steel samples were distributed to the other participating labs. Steel samples from the third source, S3, were processed at UMKC machine shop and shipped to all the participating labs. Table 1 shows the chemical compositions of the steel samples as reported on the mill certifications.

Table 1 Chemical Compositions of Steel Samples

Steel	Components (%)												
	C	Mn	P	S	Si	Ni	Cr	Mo	Cu	V	Nb	Cb	Sn
S1	0.37	0.91	0.021	0.047	0.174	0.2	0.17	0.07	0.37	0.011	0.002		
S2	0.3	0.74	0.017	0.016	0.24	0.12	0.16	0.028	0.34	0.006		0.002	0.011
S3	0.3	0.95	0.014	0.042	0.2	0.11	0.16	0.042	0.24	0.005			0.006

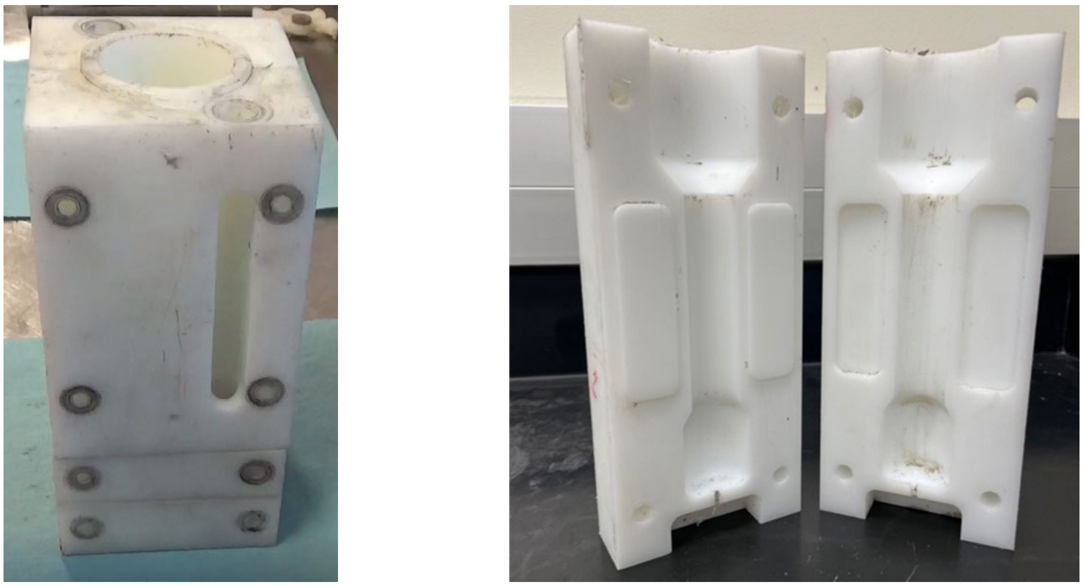
Table 2 show the modified experimental design, participating laboratories of the study, and the cement and steel combinations that were tested at each laboratory. This design allows comparison of effects of each steel and cement source on the determined mean Cl_{crit} results. Each steel-cement combination was tested with a set of 10 samples.

Table 2 Modified Experimental Design and Participating Laboratories

Laboratory	Cement	Steel		
		S1	S2	S3
UMKC	C1	☑	☑	
UMKC	C2	☑		☑
OSU	C1	☑		☑
OSU	C3	☑	☑	
CSU	C2	☑	☑	☑
CTL	C3	☑	☑	X
WJE	C1	☑		☑

X: Tests ongoing

In the first phase study, researchers used a three-part mold shown in Figure 3a to cast the OC_{crit} test samples which was taking a long time to assemble and cast. In this study, the mold design was modified to use a two-part mold as shown in Figure 3b to cast the anode samples. Using the steel and cement from the first phase study a set of 5 samples were tested to ensure that the new mold design did not affect the results. Similar to the first phase study, a shake table was used to consolidate the mortar during casting. The molds were made from Nylon 6/6 material. Additionally, the copper wires attached to the samples (Figure 1) were replaced with 18-8 stainless steel rods. These rods provided the electrical connection to the steel bars and were instrumental to keep the bars straight during casting. The molds were produced by an outside manufacturing company based on provided CAD drawings and shipped to UMKC. These molds were shipped to the participating laboratories together with the test materials. Unfortunately, this project was significantly affected by supply chain issues and difficulties with shipments. Several shipments containing molds and test materials were declared lost by shipment companies or delivered to the participating labs with missing components. Additional molds had to be ordered which were delayed by a shortage of Nylon 6/6 at the time. Coupled with temporary shutdown of a steel mill, shipment issues, material shortages, and delays in supply and manufacturing of steel samples caused significant delays in the project. The extended duration of the project with personnel changes in the laboratories also meant that testing was performed at different periods of time. Also, late delivery of S3 steel samples to the labs caused delays of testing of this steel by the CTLGroup that was moving their laboratories to a new location. At the time of writing of this report, testing of S3 samples at CTLGroup was not completed and the results of this last set will be later included in the analysis and submitted as a journal paper. The results of the last set are not expected to change the conclusions of this report.



a b

Figure 3 OC_{crit} Mold Designs in First and Second Phase Studies

b. Fresh and Hardened Mortar Tests

As a control measure to ensure consistency, participants were asked to measure the unit weight, flow, compressive strength, surface resistivity, and pH of the mortar mixtures. Flow was determined following the ASTM C1437 standard using a flow table and standard mold; however, the flow was measured after 10 drops following the ASTM C185 because the flow of the mortar was exceeding the size of the standard flow table at 25 drops. Compressive strength was measured using 52 mm cubes following the ASTM C109 procedure at 28 days. Surface resistivity of additionally cast 100 x 200 mm cylinders was also measured at 28 days following the procedure outlined in AASHTO T358 using surface resistivity meter. The pH of mortar mixtures was measured after 28 days of curing using an in situ-leaching method based on the procedure described by Sagues et al.[6]

IV. Results and discussion

a. Fresh and Hardened Properties of Mortar

Table 3 shows the average values calculated for fresh and hardened properties of mortar mixtures cast at participating laboratories. As stated earlier, all the laboratories used the cement and sand from the same sources to mix their mortar mixtures with their own source of water. These values were not reported for every mixture cast by the participants but for selected mixtures. The results were separated and averaged by cement type of the reported mixtures.

Table 3 Average Fresh and Hardened Properties of Mortar Mixtures

Lab	Cement	Unit weight (kg/m³)	Flow (% increase)	Strength 28 d (psi)	Surface Resistivity (kOhm-cm)	pH
UMKC	C1	2,192.9	103.0	11,257.7	6.5	13.5
	C2	2,155.3	106.5	10,532.0	5.3	13.1
OSU	C1	2,215.3	108.3	9,596.0	7.2	13.1
	C3	2,232.5	89.4	8,971.5	7.8	13.0
CSU	C2	2,169.0	103.4	8,672.5	5.8	12.6
CTL	C3	2,191.3	--	9,785.0	6.4	--
WJE	C1	2,187.0	129.5	9,886.5	7.0	12.9

"—" was not reported

b. OC_{crit} Test Results by University of Missouri Kansas City (UMKC)

As shown in Table 2, in the experimental design section, steel from three sources were tested at UMKC using cement from two different sources. Figure 4 shows the open circuit potential measurements over time for 10 steel samples from the first steel manufacturer, S1, in mortar made using cement from the first source, C1. The first corrosion initiation was observed after 13 days, and the last two samples activated after 55 days of testing.

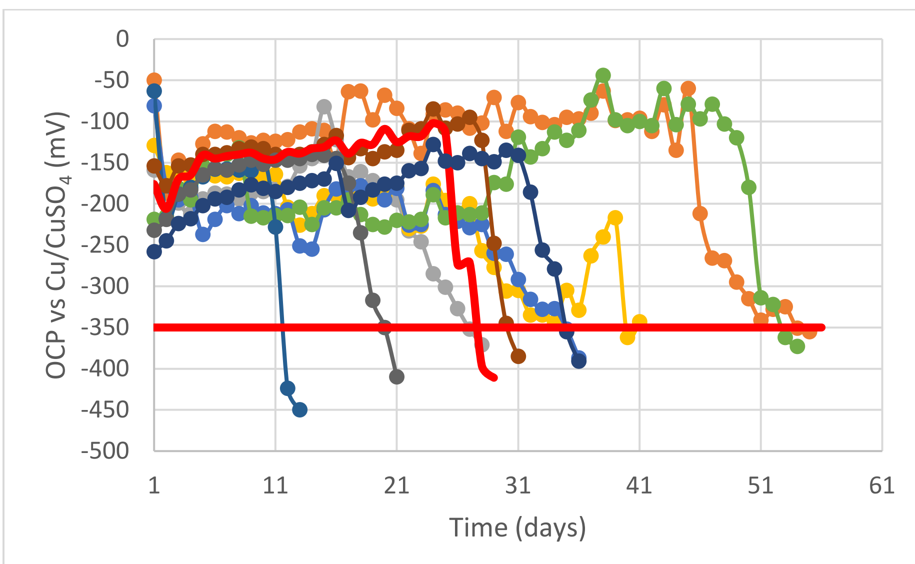


Figure 4 OCP vs $Cu/CuSO_4$ for S1 in C1 at UMKC

Table 4 shows the measured chloride content using titration as a percent by weight of the mortar and cement and the calculated critical chloride content using the empirical formula developed at Oregon State University shown in Eq. 1. The calculated average critical chloride content was 0.355 % by weight of cement with a standard deviation of 0.124.

Figure 5 shows the OCP vs time values for steel samples from the same source, S1, tested in mortar made using the cement from the second source, C2. These samples activated relatively quickly compared to the samples tested in mortar made using C1 with the latest activation at day 17, however the average critical chloride content of the samples was similar at 0.382 % by weight of cement with a standard deviation of 0.168 as shown in Table 5.

Table 4 Cl_{crit} for S1 Samples in C1 Mortar at UMKC

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
89	0.155	0.433	55	0.307
90	0.17	0.475	28	0.287
91	0.119	0.333	41	0.218
92	0.217	0.607	36	0.386
93	0.325	0.908	55	0.643
94	0.148	0.414	13	0.225
95	0.224	0.626	31	0.386
96	0.212	0.593	21	0.342
97	0.182	0.509	29	0.309
98	0.254	0.710	36	0.452
Average	0.201	0.561	34.300	0.355
Std	0.060	0.167	13.064	0.124

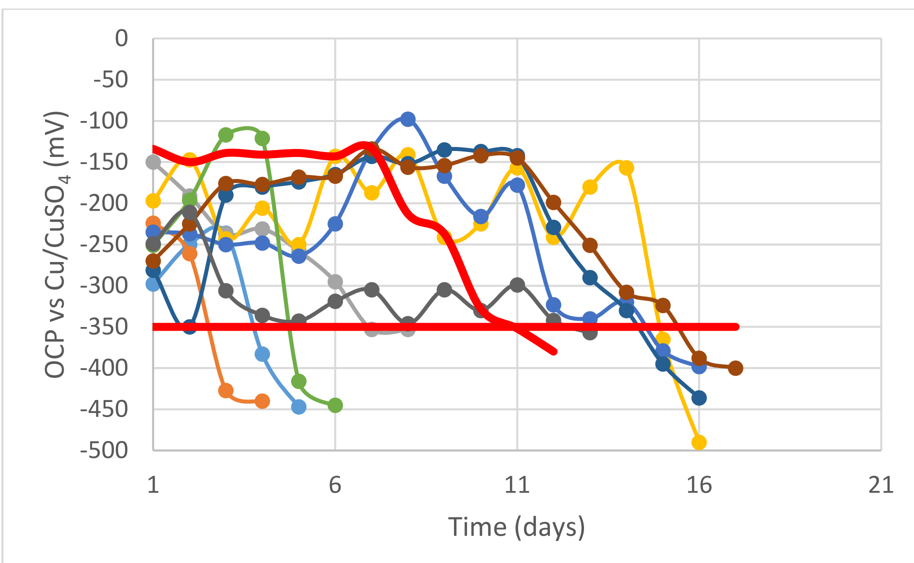


Figure 5 OCP vs $Cu/CuSO_4$ for S1 in C2 at UMKC

Figure 6 shows the OCP vs time values for steel samples from the second source, S2, tested in mortar made using the cement C1. Two of the samples (samples 13 and 22) in this group took significantly longer to activate compared to the rest of the samples and activated after 130 and 151 days of testing. A set of 11 samples was tested and Table 6 shows the critical chloride content of samples with an average of 0.447 % by weight of cement with a standard deviation of 0.283. The chloride contents of samples 13 and 22, that took significantly longer to activate compared to the rest of the samples, was much higher compared to the rest of the samples and were discarded from the analysis. Later it was found that the cathode bars attached to these samples exhibited an OCP value less than -350 mV and these cathode bars were not used again for testing.

Table 5 Cl_{crit} for S1 Samples in C2 Mortar at UMKC

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
109	0.186	0.520	5	0.266
110	0.198	0.553	4	0.281
111	0.138	0.386	8	0.202
112	0.367	1.026	16	0.570
113	0.343	0.959	16	0.533
114	0.219	0.612	6	0.316
115	0.366	1.023	16	0.569
116	0.324	0.906	17	0.507
117	0.067	0.187	13	0.102
118	0.312	0.872	12	0.471
Average	0.252	0.704	11.300	0.382
Std	0.105	0.293	5.100	0.168

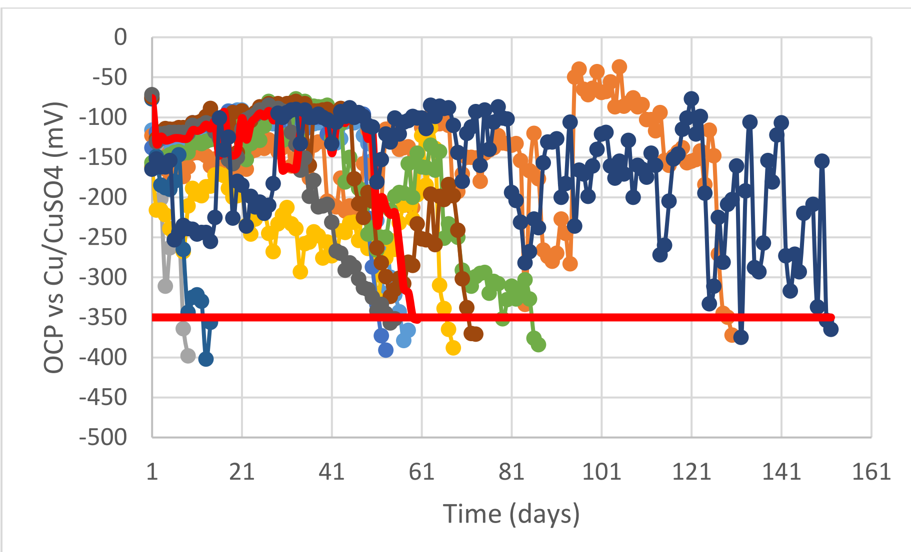


Figure 6 OCP vs $Cu/CuSO_4$ for S2 in C1 at UMKC

Figure 7 shows the OCP vs time values for steel samples from the third source, S3, tested in mortar made using the cement C2. All the samples in this group activated after 14 days of testing. Table 7 shows the critical chloride content of samples with an average of 0.626 % by weight of cement with a standard deviation of 0.130.

Table 6 Cl_{crit} for S2 Samples in C1 Mortar at UMKC

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
12	0.202	0.565	58	0.409
13*	0.348	0.973	130	0.984
14	0.035	0.098	9	0.052
15	0.191	0.534	68	0.408
16	0.25	0.699	53	0.492
17	0.117	0.327	87	0.275
18	0.068	0.190	14	0.104
19	0.21	0.587	73	0.460
20	0.195	0.545	55	0.388
21	0.219	0.612	60	0.448
22*	0.292	0.816	151	0.894
Average	0.193	0.540	68.909	0.447
Std	0.092	0.257	42.503	0.283

*Sample did not activate and is not included in the analysis

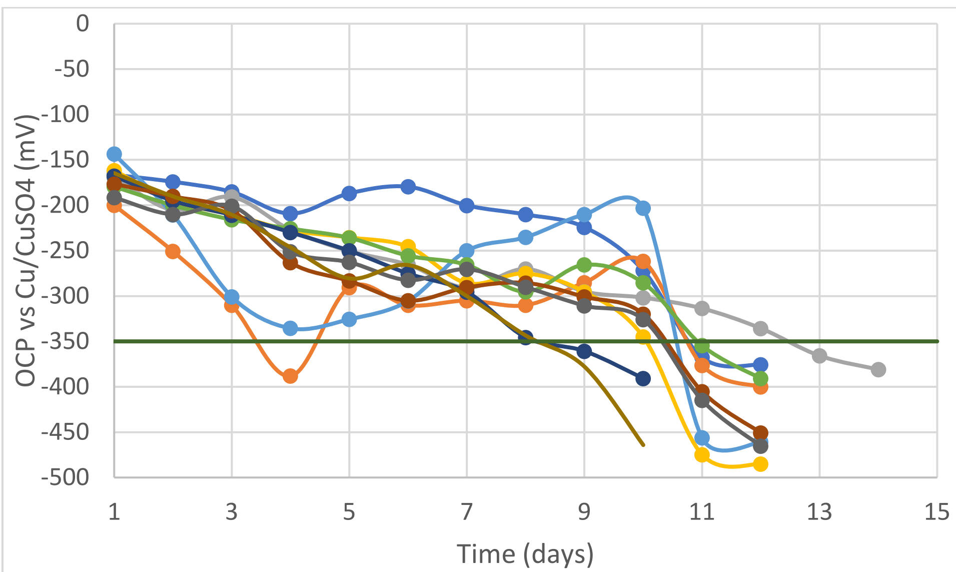


Figure 7 OCP vs $Cu/CuSO_4$ for S3 in C2 at UMKC

Although compared to the first phase study some longer activation times were observed, the measured critical chloride contents were not very different as will be further discussed later in the analysis section. Also different from the first phase study, some samples showed a repassivation behavior where OCP values showed some increases before going below -350 mV for two consecutive readings.

Table 7 Cl_{crit} for S3 Samples in C2 Mortar at UMKC

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
129	0.426	1.191	12	0.643
130	0.457	1.277	12	0.690
131	0.294	0.822	14	0.450
132	0.452	1.263	12	0.682
133	0.411	1.149	12	0.620
134	0.594	1.660	12	0.897
135	0.322	0.900	10	0.479
136	0.448	1.252	12	0.676
137	0.412	1.152	12	0.622
138	0.338	0.945	10	0.503
Average	0.415	1.161	11.800	0.626
Std	0.085	0.239	1.135	0.130

c. OC_{crit} Test Results by Oregon State University (OSU)

As shown in Table 2 Oregon State University (OSU) evaluated four different combinations of cement and steel samples. Only one of these combinations, S1 samples in mortar made with C1 cement, is a repetition of a test set performed at UMKC. Figure 8 shows the OCP vs. time values of S1 samples tested in mortar made using C1 and Table 8 shows the calculated critical chloride content values of the samples.

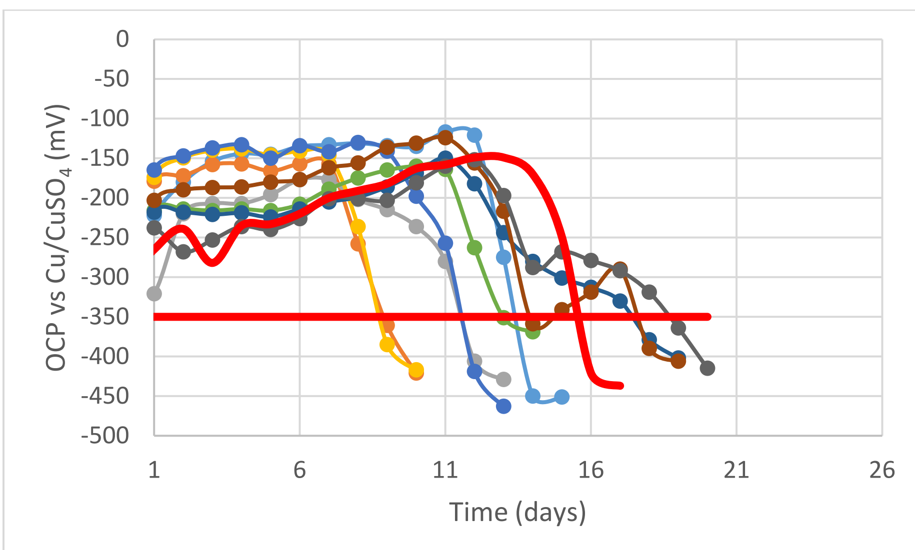


Figure 8 OCP vs $Cu/CuSO_4$ for S1 in C1 at OSU

Table 8 Cl_{crit} for S1 Samples in C1 Mortar at OSU

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.356	0.996	15	0.550
2	0.340	0.951	10	0.506
3	0.342	0.956	13	0.520
4	0.284	0.794	10	0.422
5	0.329	0.919	13	0.500
6	0.304	0.849	14	0.465
7	0.317	0.887	19	0.504
8	0.328	0.916	19	0.520
9	0.286	0.798	20	0.457
10	0.330	0.923	17	0.517
Average	0.322	0.899	15.0	0.496
Std	0.024	0.067	3.7	0.037

Unlike the first phase study where the differences between mean critical chloride values obtained in different labs for the same samples were statistically not significant, in this phase the difference between the mean Cl_{crit} values for the S1 samples between UMKC and OSU as shown in Figure 9 (0.355 and 0.496 % by weight of cement) were statistically significant at 95% confidence level mostly due to very low variability of results at OSU. However, the absolute value of the means were not as different as reported in the literature and were close to the mean value of 0.423% by weight of cement of the first phase study.

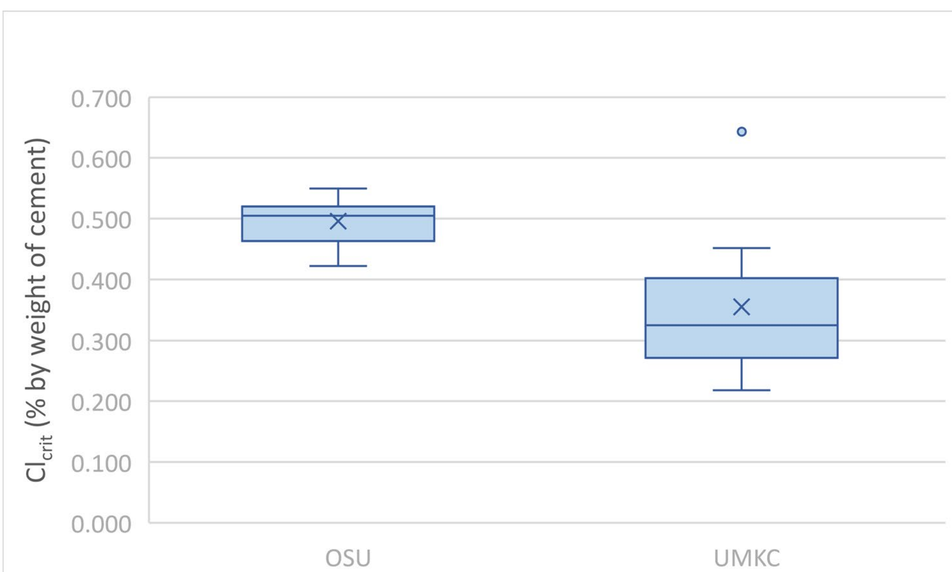


Figure 9 Cl_{crit} distributions for S1 samples in C1 at UMKC and OSU

Figure 10 shows the OCP vs. time data for S3 samples tested in mortar made with C1 cement and Table 9 shows the critical chloride contents of these samples.

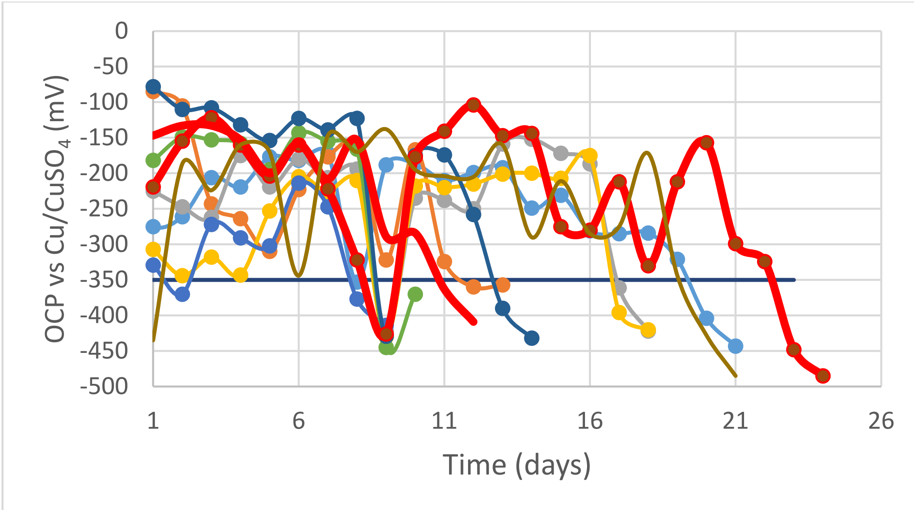


Figure 10 OCP vs Cu/CuSO$_4$ for S3 in C1 at OSU

Table 9 Cl_{crit} for S3 Samples in C1 Mortar at OSU

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.338	0.946	22	0.549
2	0.293	0.818	13	0.445
3	0.367	1.025	18	0.578
4	0.363	1.016	18	0.573
5	0.237	0.662	9	0.349
6	0.270	0.755	10	0.402
7	0.333	0.930	14	0.510
8	0.400	1.119	24	0.658
9	0.288	0.805	12	0.435
10	0.329	0.919	20	0.526
Average	0.322	0.899	16.0	0.502
Std	0.050	0.139	5.1	0.094

Figure 11 and Figure 12 shows the OCP vs time data for S1 and S2 samples tested in mortar made with cement C3. Table 10 and Table 11 show the critical chloride content values of these samples, respectively. One of the S1 samples and two of the S2 samples did not activate after 70 days of testing and OSU researchers decided to stop testing of these samples. These samples are not shown in OCP vs time figures and their measured chloride content was not included in the statistical analysis of data. All the samples tested in the first phase study activated within 30-40 days of exposure and not activation after 70 days of testing was only observed in this study.

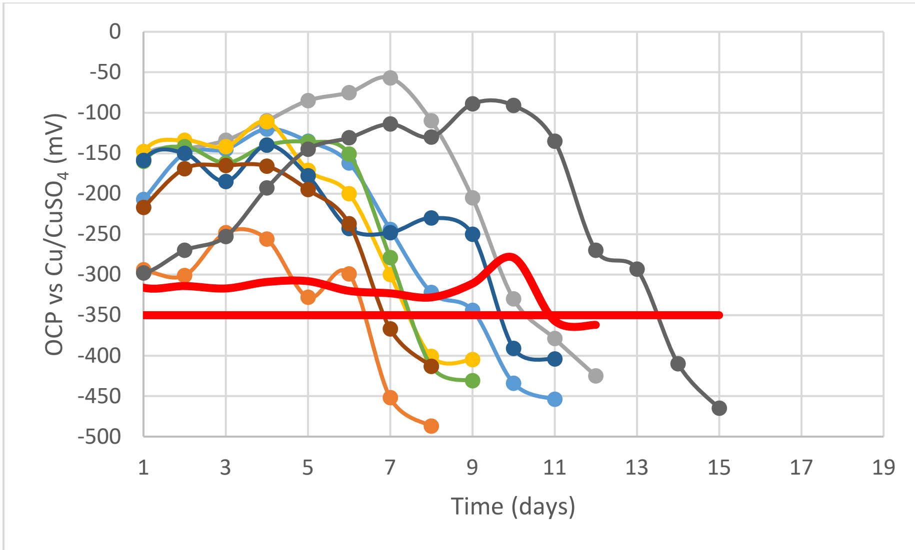


Figure 11 OCP vs $Cu/CuSO_4$ for S1 in C3 at OSU

Table 10 Cl_{crit} for S1 Samples in C3 Mortar at OSU

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.247	0.690	11	0.370
2	0.209	0.585	8	0.306
3	0.184	0.514	12	0.278
4	0.196	0.548	9	0.290
5*	0.516	1.443	70	1.114
6	0.251	0.701	9	0.370
7	0.256	0.716	11	0.384
8	0.188	0.526	8	0.276
9	0.175	0.488	15	0.270
10	0.255	0.713	12	0.385
Average	0.248	0.692	16.5	0.325
Std	0.100	0.278	18.9	0.051

*Sample did not activate and is not included in the analysis

Another complication in the testing program was OSU running out of the cement, C3, that was procured from a local supplier. OSU had to procure a new batch of C3 cement from the same supplier for testing of S1 and S2 samples. Therefore, the C3 cement used by the CTLGroup for testing of S1 and S2 samples did not come from the same batch of cement production. However, it will be shown later that the difference between the mean critical chloride contents of these samples between OSU and CTLGroup were not statistically significant and the different batches from the same manufacturer were providing similar results.

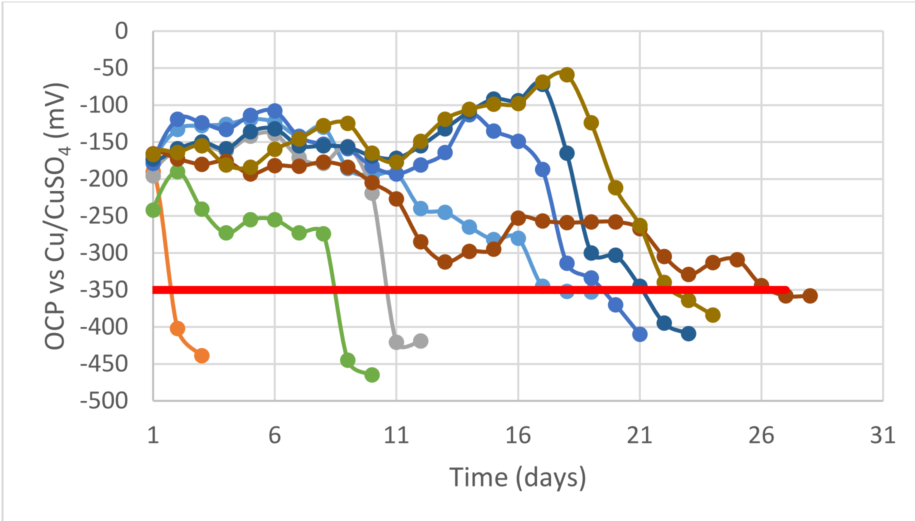


Figure 12 OCP vs Cu/CuSO$_4$ for S2 in C3 at OSU

Table 11 Cl_{crit} for S2 Samples in C3 Mortar at OSU

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.151	0.423	19	0.240
2	0.075	0.210	3	0.106
3	0.209	0.585	12	0.316
4*	0.223	0.624	70	0.482
5	0.119	0.331	21	0.191
6	0.196	0.548	10	0.292
7	0.227	0.636	23	0.371
8	0.236	0.658	28	0.398
9*	0.235	0.657	70	0.507
10	0.178	0.498	24	0.293
Average	0.174	0.486	17.500	0.276
Std	0.056	0.156	8.401	0.095

*Sample did not activate and is not included in the analysis

d. OC_{crit} Test Results by Colorado State University (CSU)

Colorado State University tested all steel samples from different sources, S1, S2, and S3 in mortar made with cement, C2, which they procured locally and supplied to some of the other participating labs. Figure 13, Figure 14, and Figure 15 show the OCP vs time data for steel samples S1, S2, and S3, respectively. Table 12, Table 13, and Table 14 show the measured critical chloride content data for these samples. In the whole testing program OSU observed the shortest activation durations but all the tested samples exhibited passive OCP values at the beginning of tests. Therefore, although some samples showed a very quick activation after 1 or 2 days of exposure, these samples were not discarded, and their chloride contents were determined.

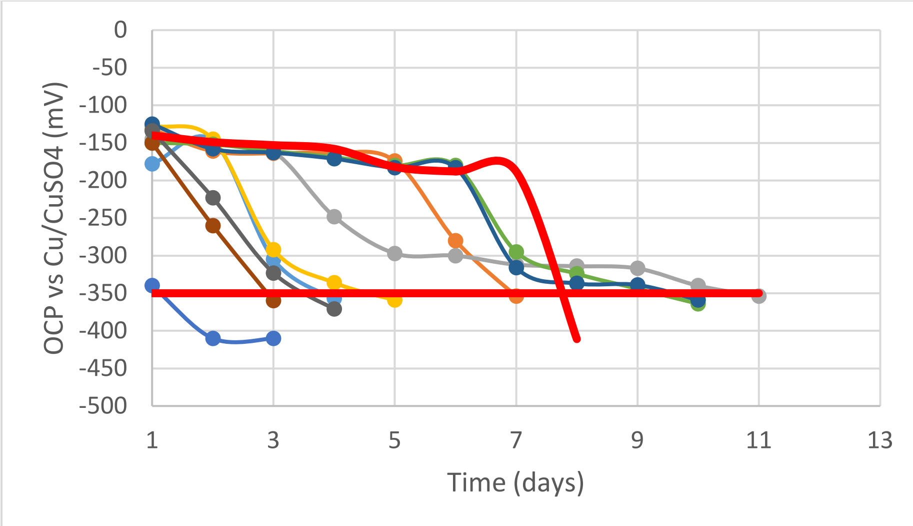


Figure 13 OCP vs $Cu/CuSO_4$ for S1 in C2 at CSU

Table 12 Cl_{crit} for S1 Samples in C2 Mortar at CSU

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.132	0.370	4	0.188
2	0.120	0.335	7	0.174
3	0.286	0.799	11	0.428
4	0.131	0.367	5	0.188
5	0.114	0.317	3	0.160
6	0.182	0.508	10	0.270
7	0.232	0.649	10	0.345
8	0.110	0.308	3	0.155
9	0.187	0.523	4	0.266
10	0.195	0.545	8	0.286
Average	0.169	0.472	6.500	0.246
Std	0.058	0.163	3.100	0.090

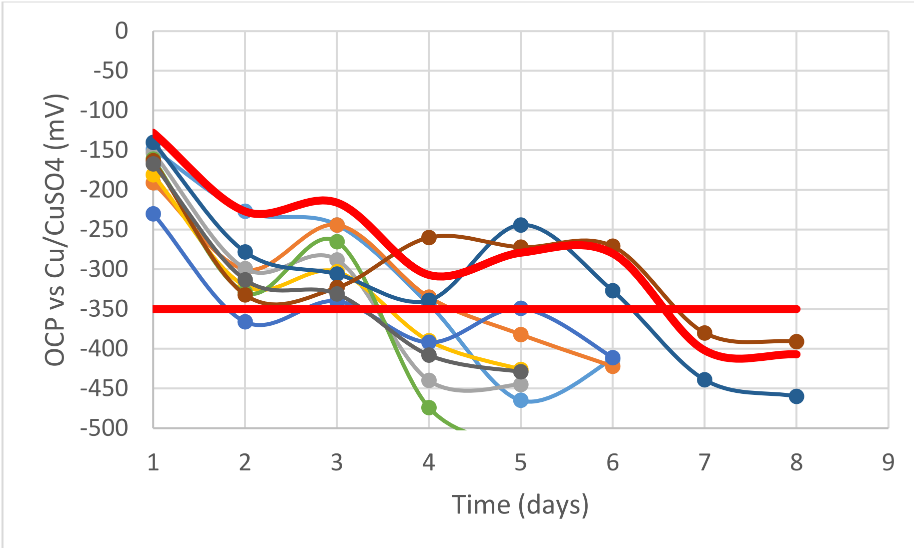


Figure 14 OCP vs $Cu/CuSO_4$ for S2 in C2 at CSU

Table 13 Cl_{crit} for S2 Samples in C2 Mortar at CSU

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.1983	0.554	6	0.288
2	0.1898	0.531	6	0.275
3	0.1722	0.481	5	0.248
4	0.1768	0.494	5	0.255
5	0.1563	0.437	6	0.227
6	0.1141	0.319	5	0.164
7	0.2619	0.732	8	0.386
8	0.1690	0.472	8	0.249
9	0.1862	0.520	5	0.268
10	0.1969	0.550	8	0.290
Average	0.182	0.509	6.200	0.265
Std	0.037	0.104	1.317	0.056

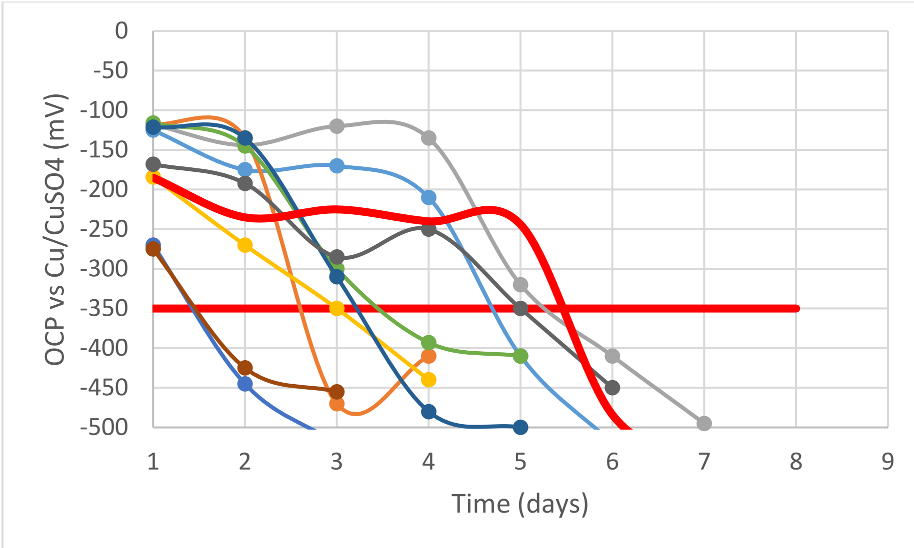


Figure 15 OCP vs Cu/$CuSO_4$ for S3 in C2 at CSU

Table 14 Cl_{crit} for S3 Samples in C2 Mortar at CSU

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.2840	0.794	6	0.410
2	0.1870	0.523	4	0.266
3	0.3138	0.877	7	0.456
4	0.2164	0.605	4	0.307
5	0.2015	0.563	3	0.284
6	0.1929	0.539	5	0.276
7	0.2047	0.572	5	0.293
8	0.1233	0.345	3	0.174
9	0.2579	0.721	6	0.372
10	0.2117	0.592	7	0.308
Average	0.219	0.613	5.000	0.314
Std	0.054	0.151	1.491	0.080

UMKC and CSU both tested steel samples, S1 and S3 in mortar made using Cement C2. Figure 16 shows critical chloride threshold values for S1 and S3 samples measured at CSU and UMKC laboratories. Both laboratory results indicate a similar trend with S3 samples showing higher mean values compared to S1 samples. However, the difference between the mean critical chloride values of steel samples S1 and S3 is statistically significant at 95% confidence level only for samples tested at UMKC. Additionally, the difference of mean Cl_{crit} values for S1 samples tested at CSU and UMKC were statistically not significant, i.e. both laboratories were providing similar results for the same combination. However, the difference between the mean Cl_{crit} values of S3 samples were statistically significantly different at 95% confidence level. Figure 17 shows the mean Cl_{crit} values and their 95% confidence intervals for all steel and laboratory combinations and only the mean of S3 samples tested UMKC were statistically significantly higher than the other 3 groups.

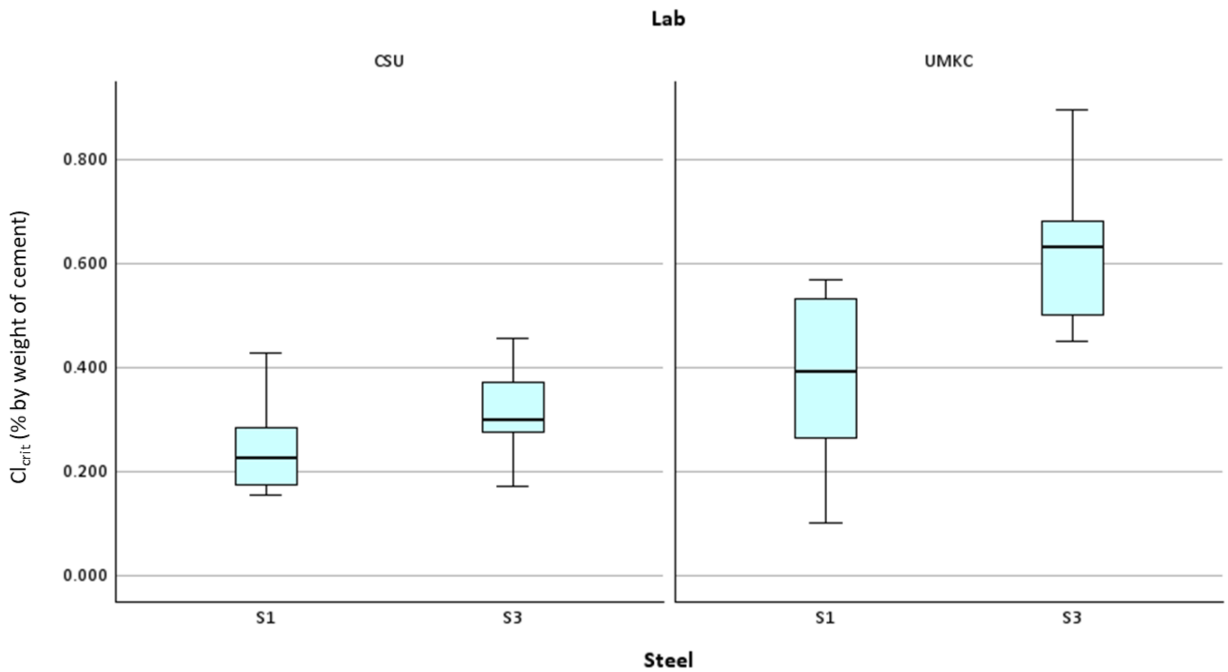


Figure 16 Cl_{crit} distributions for Steel Samples S1 and S3 measured at CSU and UMKC labs.

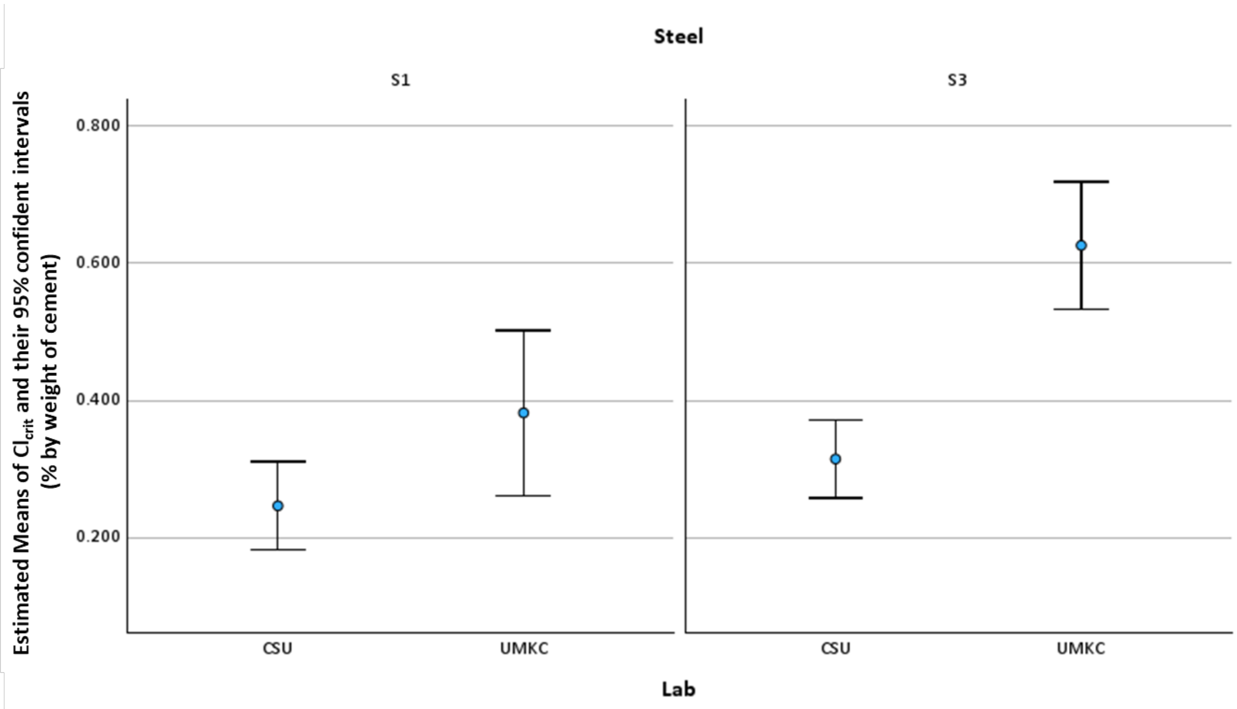


Figure 17 Mean Cl_{crit} values and 95% confidence intervals at CSU and UMKC for S1 and S3 samples

e. OC_{crit} Test Results by CTLGroup

According to the updated experimental design program, CTLGroup was going to test all steel samples in mortar made using cement C3. At the time of writing of this report testing of steel samples S1 and S2 were completed and S3 samples were still being tested. Testing of S3 samples were delayed due to moving of CTLGroup laboratories to a new location and these results will be included later in a journal publication submitted. Figure 15 and Figure 16 show the OCP vs. time values measured for S1 and S2 steel samples in mortar made with cement C3. Table 12 and Table 13 show the measured critical chloride threshold values of the samples. One sample in the S2 group had an OCP value lower than -350 mV from the beginning of the testing and it remained active for the next 15 measurements. This sample was discarded, and its chloride content was not measured.

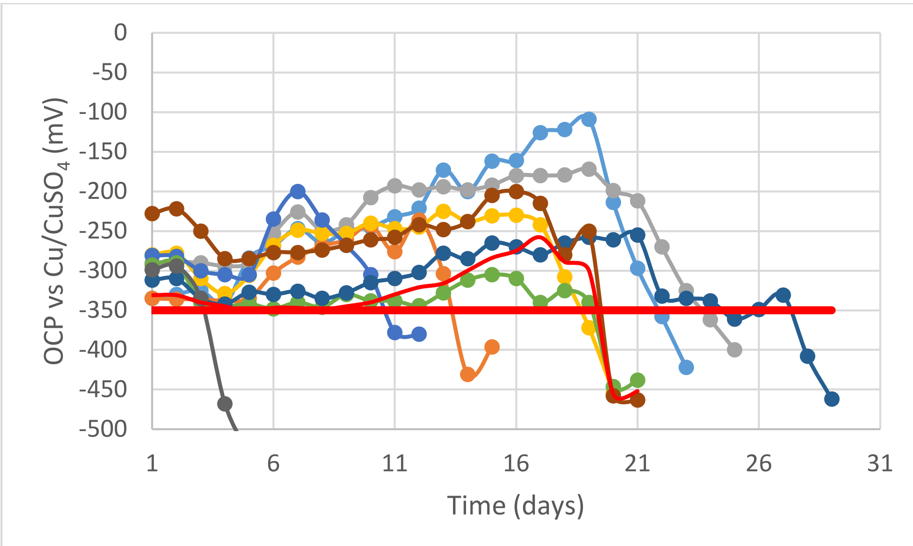


Figure 18 OCP vs Cu/$CuSO_4$ for S1 in C3 at CTLGroup

Table 15 Cl_{crit} for S1 Samples in C3 Mortar at CTLGroup

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.279	0.780	23	0.455
2	0.191	0.534	16	0.297
3	0.313	0.875	25	0.518
4	0.239	0.668	20	0.382
5	0.186	0.520	12	0.281
6	0.349	0.975	21	0.562
7	0.371	1.037	29	0.630
8	0.210	0.587	21	0.338
9	0.103	0.288	5	0.147
10	0.250	0.699	21	0.402
Average	0.249	0.696	19.300	0.401
Std	0.082	0.228	6.816	0.145

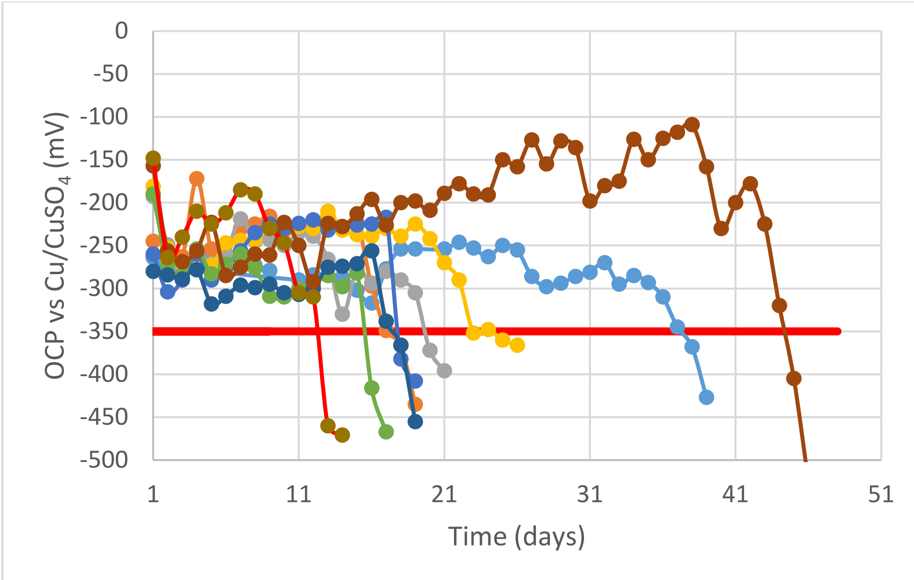


Figure 19 OCP vs Cu/$CuSO_4$ for S2 in C3 at CTLGroup

Table 16 Cl_{crit} for S2 Samples in C3 Mortar at CTLGroup

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.375	1.048	39	0.679
2	0.333	0.931	21	0.536
3	0.281	0.785	23	0.459
4	0.319	0.892	28	0.539
5	0.303	0.847	21	0.488
6	0.279	0.780	18	0.440
7	0.280	0.783	21	0.451
8	0.272	0.760	48	0.520
9*	0.226	0.632		
10	0.156	0.436	15	0.241
Average	0.289	0.807	26.0	0.484
Std	0.060	0.167	10.8	0.116

*Sample was active in the first cycle and is not included in the analysis

Testing of S1 and S2 samples in mortar made with cement C3 was repeated in OSU and CTLGroup laboratories. Figure 17 shows the critical chloride content distributions for these steel samples in these two laboratories. As shown in Figure 18 analysis indicates that for S1 samples the difference between the mean Cl_{crit} values obtained at CTL and OSU were not statistically significant but the difference between mean Cl_{crit} values of S2 samples were statistically significant at 95% confidence level. Measurements in both laboratories separately indicated that the difference between the mean Cl_{crit} values of steel samples S1 and S2 were not statistically significant.

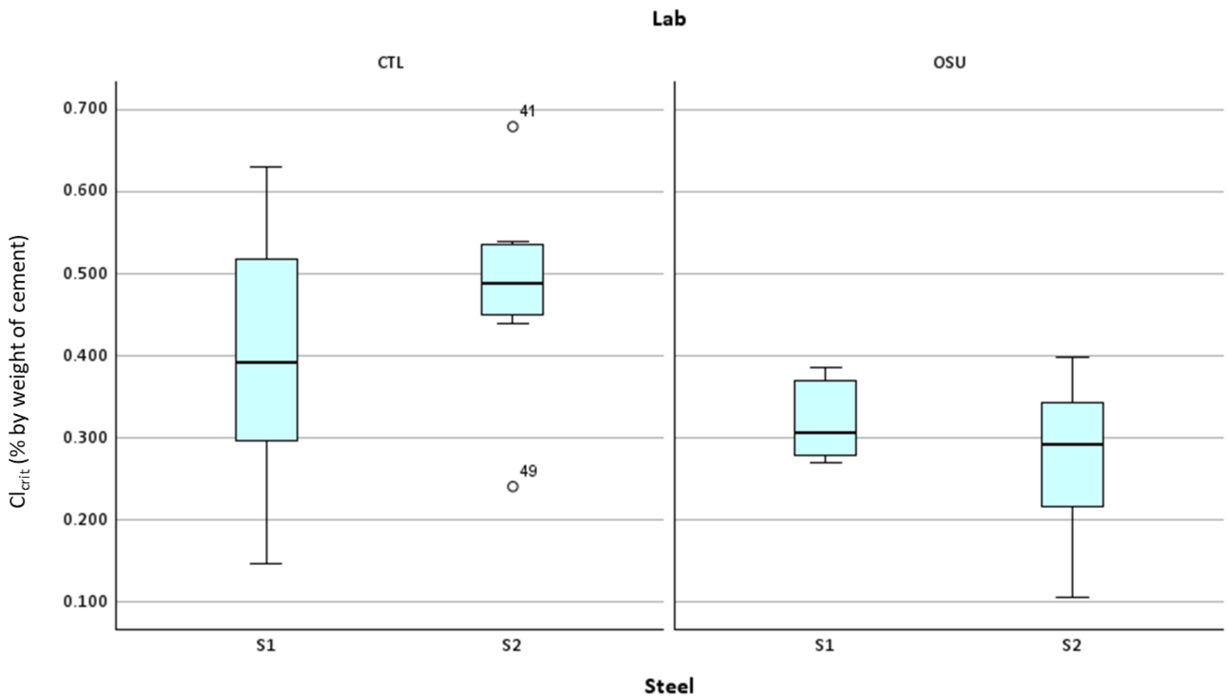


Figure 20 Cl_{crit} distributions for Steel Samples S1 and S2 measured at CTLGroup and OSU labs.

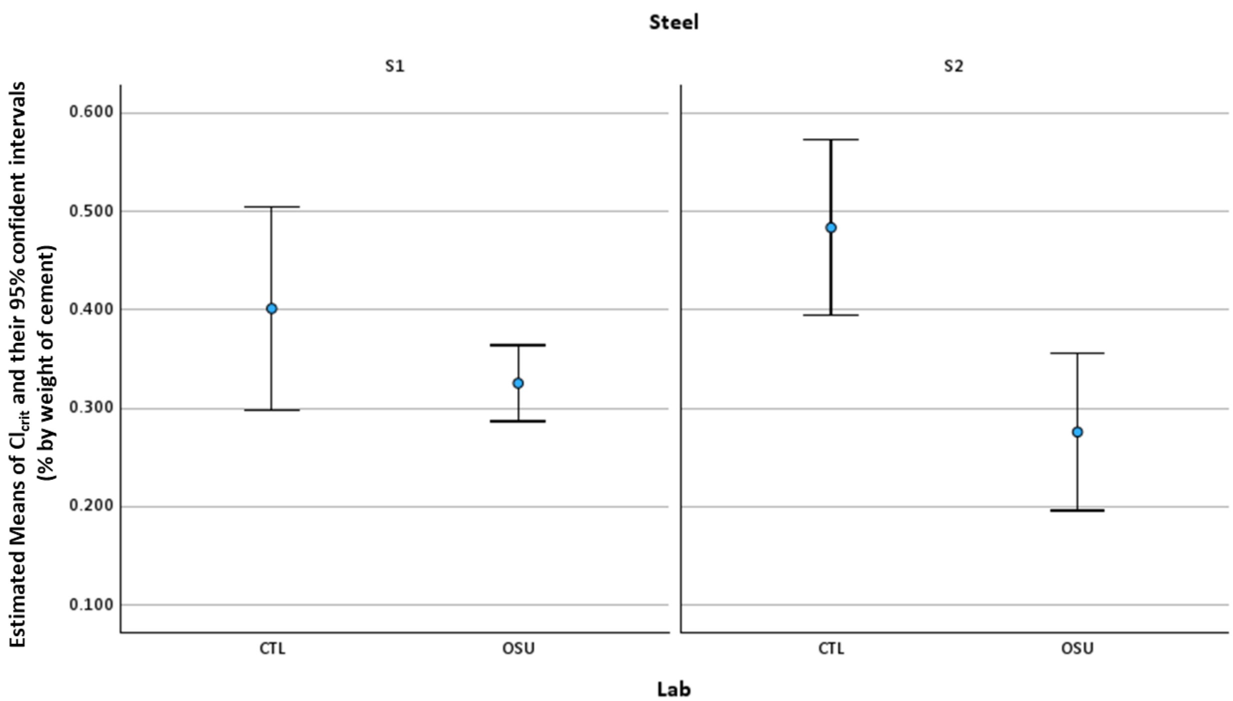


Figure 21 Mean Cl_{crit} values and 95% confidence intervals at CTLGroup and OSU for Steel S1 and S2

f. OC_{crit} Test Results by Wiss, Janey, Elstner Associates (WJE)

WJE tested S1 and S3 samples in mortar made using cement C1, supplied by UMKC. Figure 22 and Figure 24 show the OCP vs time data for these samples and Table 14 and Table 15 show their calculated critical chloride threshold values. The Cl_{crit} value of S1 steel samples in mortar made using cement C1 was tested at UMKC, OSU, and WJE. The mean Cl_{crit} values obtained for S1 samples at WJE was statistically significantly lower compared to the results obtained both at UMKC and OSU as shown in Figure 23. The two outliers of WJE results shown in Figure 23 are the two samples that took significantly longer to activate (Samples 1 and 3 in Table 17).

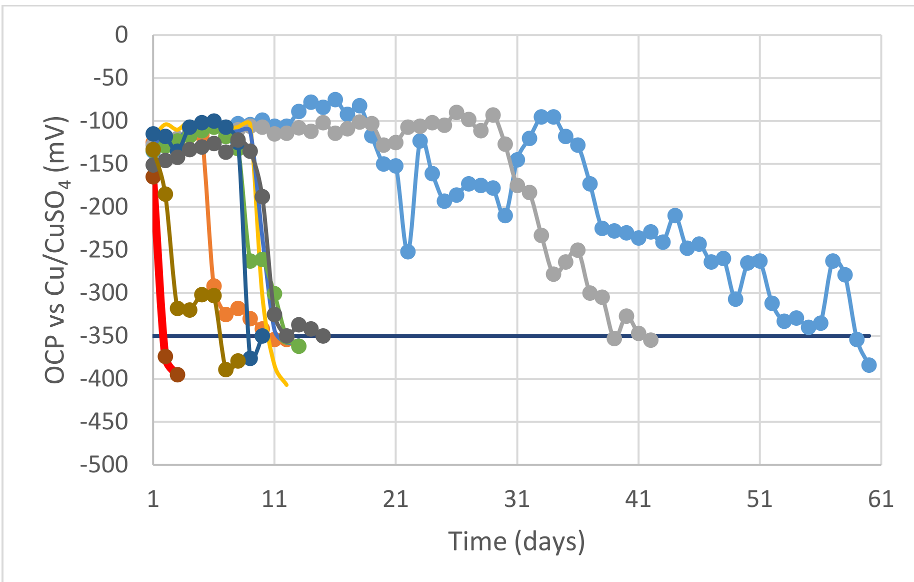


Figure 22 OCP vs $Cu/CuSO_4$ for S1 in C1 at WJE

Table 17 Cl_{crit} for S1 Samples in C1 Mortar at WJE

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.193	0.539	60	0.395
2	0.066	0.184	12	0.100
3	0.136	0.380	42	0.251
4	0.094	0.263	12	0.142
5	0.074	0.207	13	0.113
6	0.060	0.168	13	0.091
7	0.064	0.179	10	0.095
8*	0.039	0.109	3	0.055
9	0.074	0.207	15	0.114
10	0.078	0.218	8	0.114
Average	0.088	0.245	18.8	0.157
Std	0.043	0.119	16.9	0.096

*Sample was active in the first cycle and is not included in the analysis

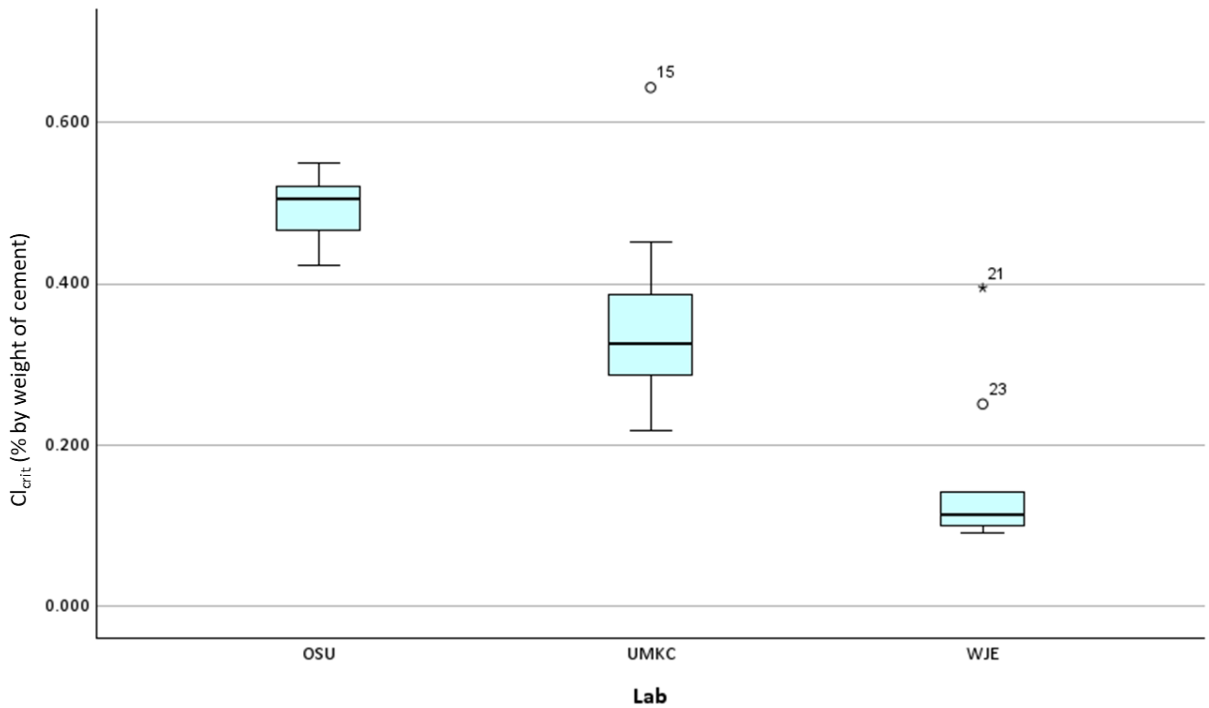


Figure 23 Cl_{crit} distributions for S1 samples in C1 cement measured at OSU, UMKC, and WJE labs.

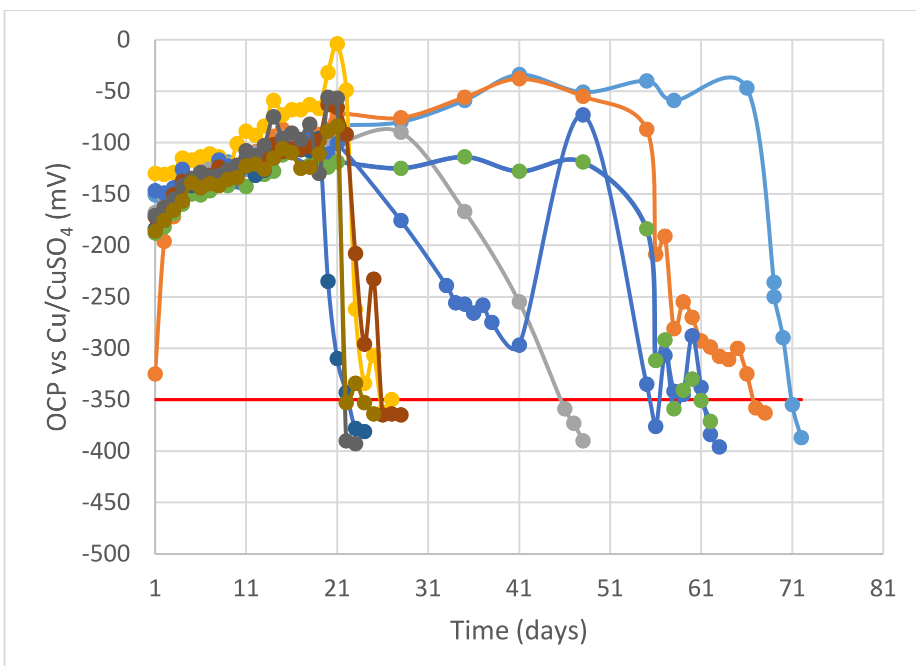


Figure 24 OCP vs $Cu/CuSO_4$ for S3 in C1 at WJE

Table 18 Cl_{crit} for S3 Samples in C1 Mortar at WJE

Sample #	Cl (% mortar)	Cl (% cement)	TTA (days)	Cl_{crit}
1	0.288	0.805	72	0.628
2	0.328	0.917	68	0.700
3	0.207	0.579	48	0.396
4	0.289	0.808	56	0.578
5	0.308	0.861	63	0.640
6	0.363	1.015	62	0.751
7	0.268	0.749	40	0.488
8	0.308	0.861	60	0.630
9	0.288	0.805	48	0.551
10	0.345	0.964	48	0.660
Average	0.299	0.836	56.5	0.602
Std	0.043	0.121	10.3	0.104

Figure 25 shows the Cl_{crit} distributions for S3 steel samples tested in mortar made with C1 cement at OSU and WJE laboratories. The difference in mean Cl_{crit} values were not statistically significant and results were in good agreement.

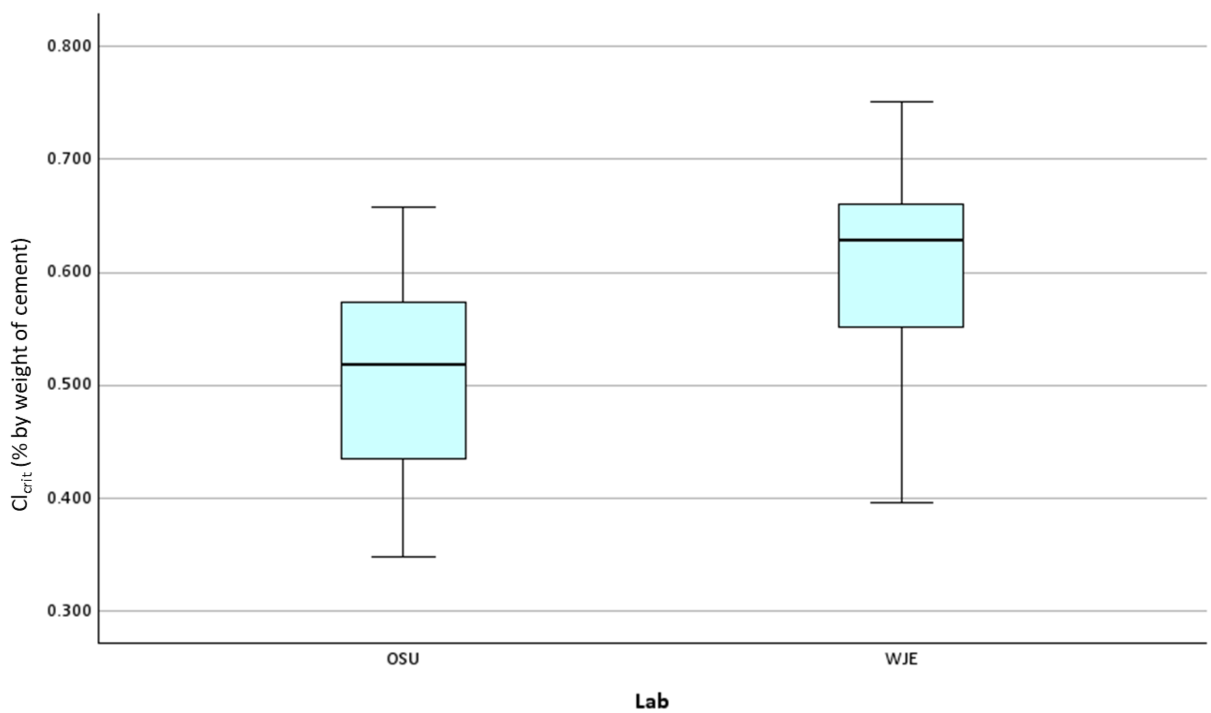


Figure 25 Cl_{crit} distributions for S3 samples in C1 cement measured at OSU and WJE labs

g. Analysis of Results and Discussion

Although the results showed higher variability compared to the first phase study, especially between the laboratories for the same steel-cement combinations, overall distributions are similar with mean values close to the mean Cl_{crit} value obtained in the first phase study. As explained earlier, in this study due to various reasons testing was performed in a much longer time period and at different time intervals by the participating laboratories. Production of steel samples were not controlled in one lab and the samples were exposed to different environments for various amounts of time before testing including during shipment before they were tested. Some laboratories reported observing larger amounts of surface corrosion products on received samples which may explain higher variability and differences in the observed mean Cl_{crit} values between the laboratories. The OC_{crit} test requires testing of steel samples in as received condition and no surface preparation was applied to the steel samples other than degreasing in xylene solution.

One of the main observations is that the cement source was not a significant factor in the variance of obtained Cl_{crit} values. Figure 26 shows the Cl_{crit} distributions and the mean values for all the S1 steel samples tested at all laboratories separated by the cement source. Figure 27 shows only the mean Cl_{crit} values of these distributions and their 95% confidence intervals. Both figures clearly indicate that the Cl_{crit} distributions were very similar for all the cement sources used in this study. It should be noted that the C3 cement includes two different production batches from the same manufacturer as explained earlier.

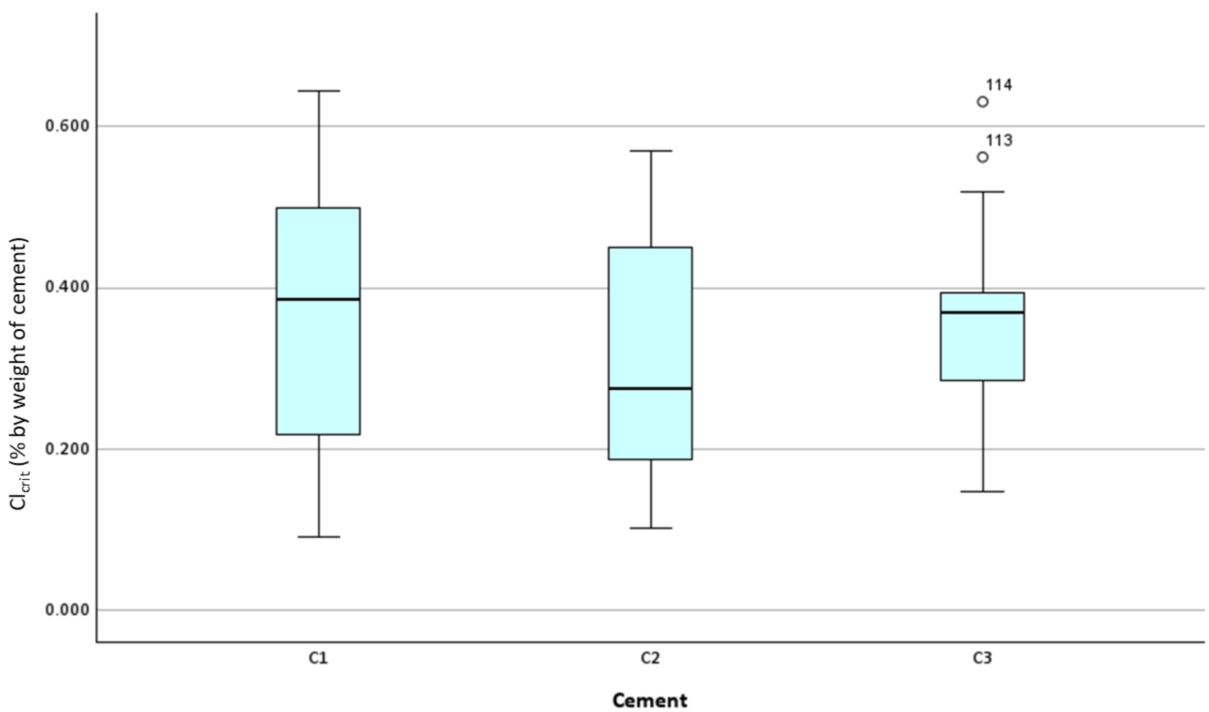


Figure 26 Cl_{crit} distributions for S1 samples in mortar made with C1, C2, and C3

Figure 28 shows the analysis of variance table for the S1 steel results obtained at all laboratories separated by the cement source. The significance value of 0.558 shown in the table indicates that the hypothesis of all groups having the same mean value cannot be rejected at 95% confidence level since this number is larger than 0.05. Comparison of groups using Tukey's HSD in Figure 29 also shows that all three groups were put into the same subset, i.e. the difference between them was statistically insignificant.

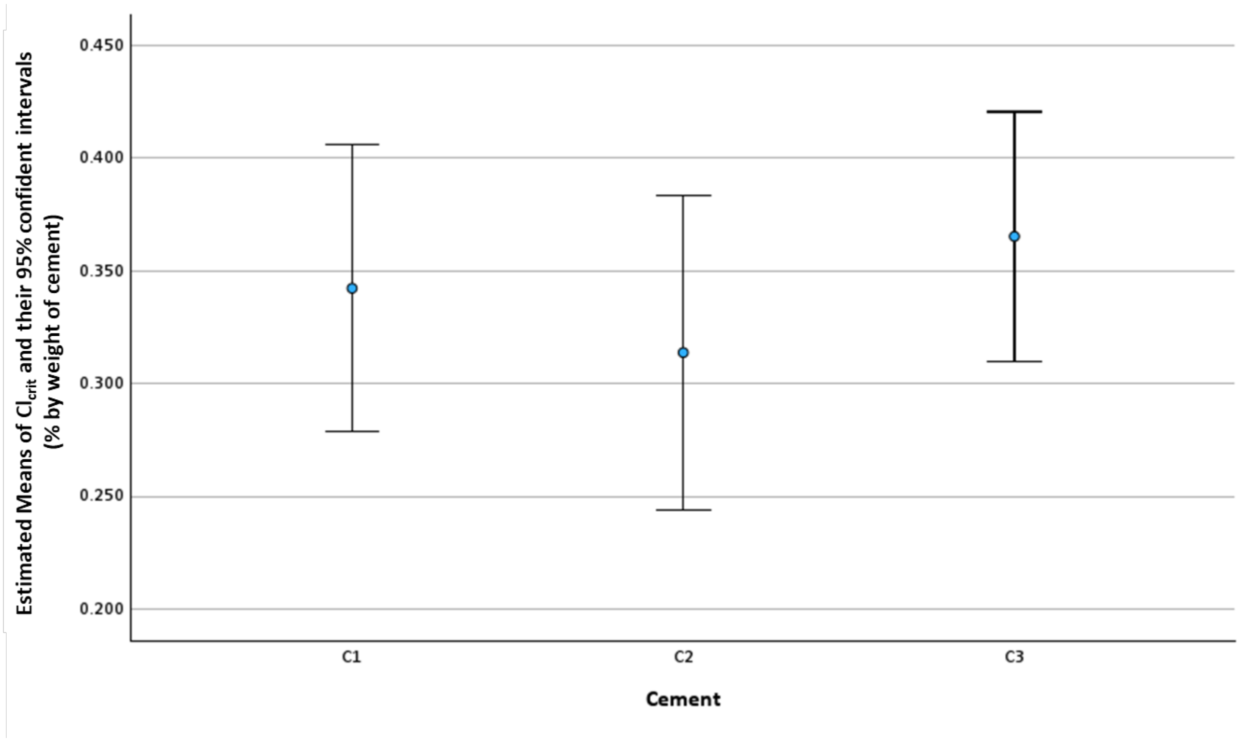


Figure 27 Mean Cl_{crit} values and 95% confidence intervals for S1 steel in C1, C2, and C3 cement mortar

ANOVA

Clcrit

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	.026	2	.013	.588	.558
Within Groups	1.438	65	.022		
Total	1.464	67			

Figure 28 Analysis of Varince for S1 steel tested with different cement sources

Figure 30 shows the Cl_{crit} distributions of S2 samples tested at all laboratories separated again by the source of cement and Figure 31 shows only the mean Cl_{crit} values and their 95% confidence intervals. For S2 steel samples, again the distributions look similar, although the S2 samples tested in cement C2 mortar seem to have a lower mean Cl_{crit} value compared to the other two groups. However, mainly due to the large within group variabilities the analysis of variance table shown in Figure 32 shows a significance value greater than 0.05 and all groups were placed in the same subgroup using Tukey's comparison in Figure 33. This indicates that the differences between the observed mean Cl_{crit} values of S2 samples tested in mortar made with different cement sources were statistically not significant at 95% confidence level similar to the S1 steel samples.

	CementType	N	Subset for alpha = 0.05 1
Tukey HSD[a,b]	2.00	20	.3138988537489
	1.00	29	.3423938627518
	3.00	19	.3653810463158
	Sig.		.490

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 21.880.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Figure 29 Tukey's comparison of S1 results grouped by cement source

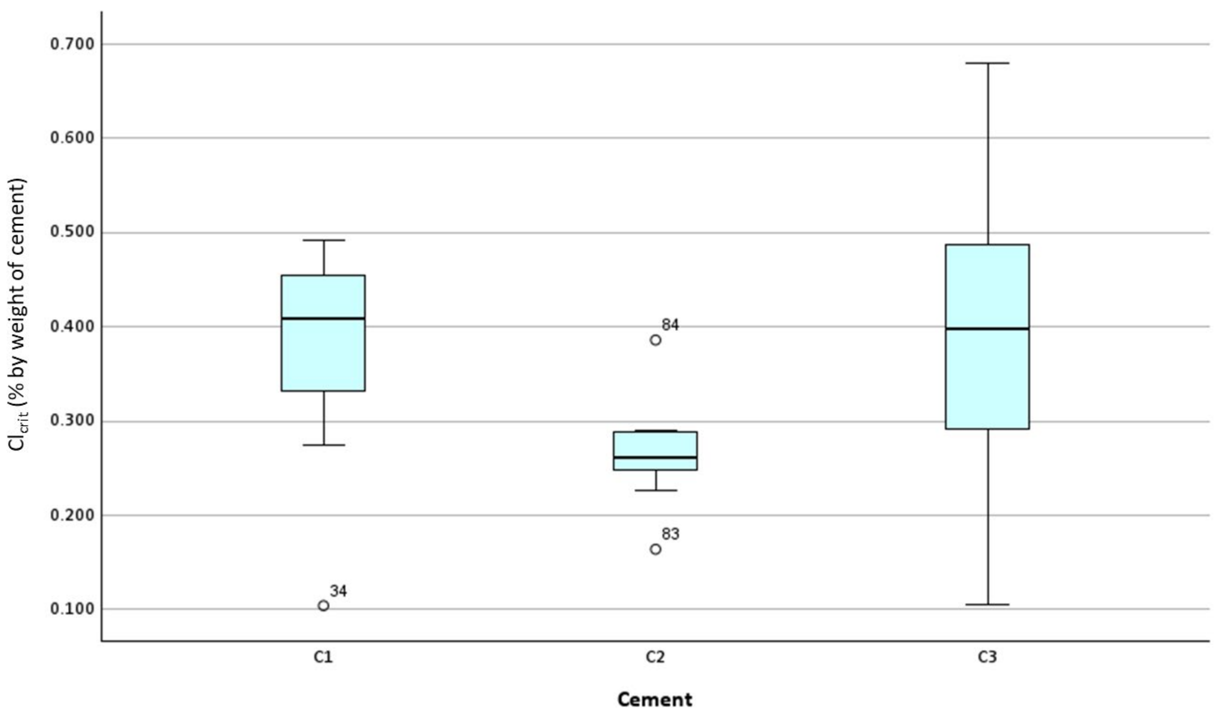


Figure 30 Cl_{crit} distributions for S2 samples in mortar made with C1, C2, and C3

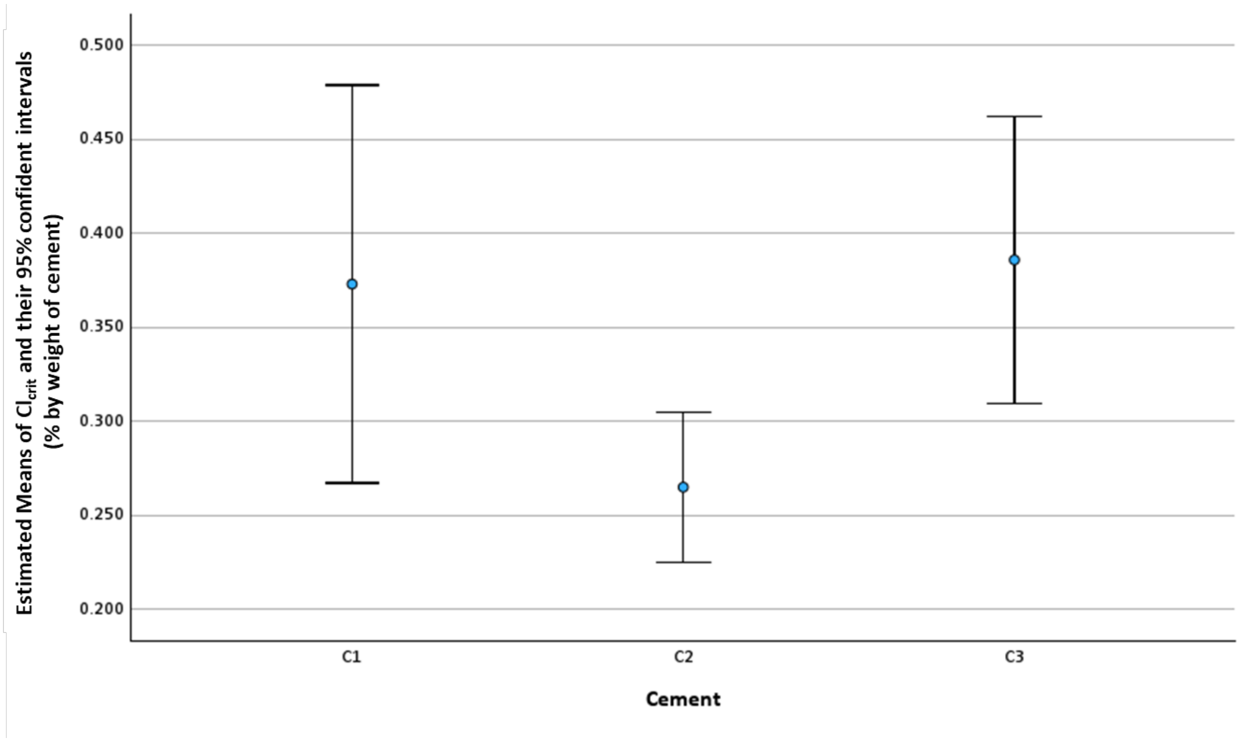


Figure 31 Mean Cl_{crit} values and 95% confidence intervals for S2 steel in C1, C2, and C3 cement mortar

ANOVA

Clcrit

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	.098	2	.049	3.177	.055
Within Groups	.494	32	.015		
Total	.592	34			

Figure 32 Analysis of Varince for S2 steel tested with different cement sources

Figure 34 shows the Cl_{crit} distributions and the mean values for all the S3 steel samples tested at all laboratories separated by the cement source. Unfortunately, due to the missing results of S3 steel samples testing in mortar with cement C3, results are grouped only into 2 groups for cements C1 and C2. Figure 35 shows only the mean Cl_{crit} values of these distributions and their 95% confidence intervals. Like the S1 and S2 steel sample results, the Cl_{crit} distributions of S3 samples grouped by the cement type seem to have similar distributions. Because there are only two cement groups for S3 steel samples, they were compared using an independent sample t-test and the results are shown in Figure 36. Comparison of variances of the two groups using Levene's test indicates that the variances were statistically significantly different. Comparison of the means of the two groups without equal variance assumption provides a significance value greater than 0.05 for both one- and two-sided p-tests, meaning that the difference between the mean Cl_{crit} values of the two groups was not statistically significant at 95% confidence level.

	CementType	N	Subset for alpha = 0.05 1
Tukey HSD[a,b]	2.00	10	.2650000000000
	1.00	8	.3729592100000
	3.00	17	.3857981788235
	Sig.		.081

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.570.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Figure 33 Tukey's comparison of S2 results grouped by cement source

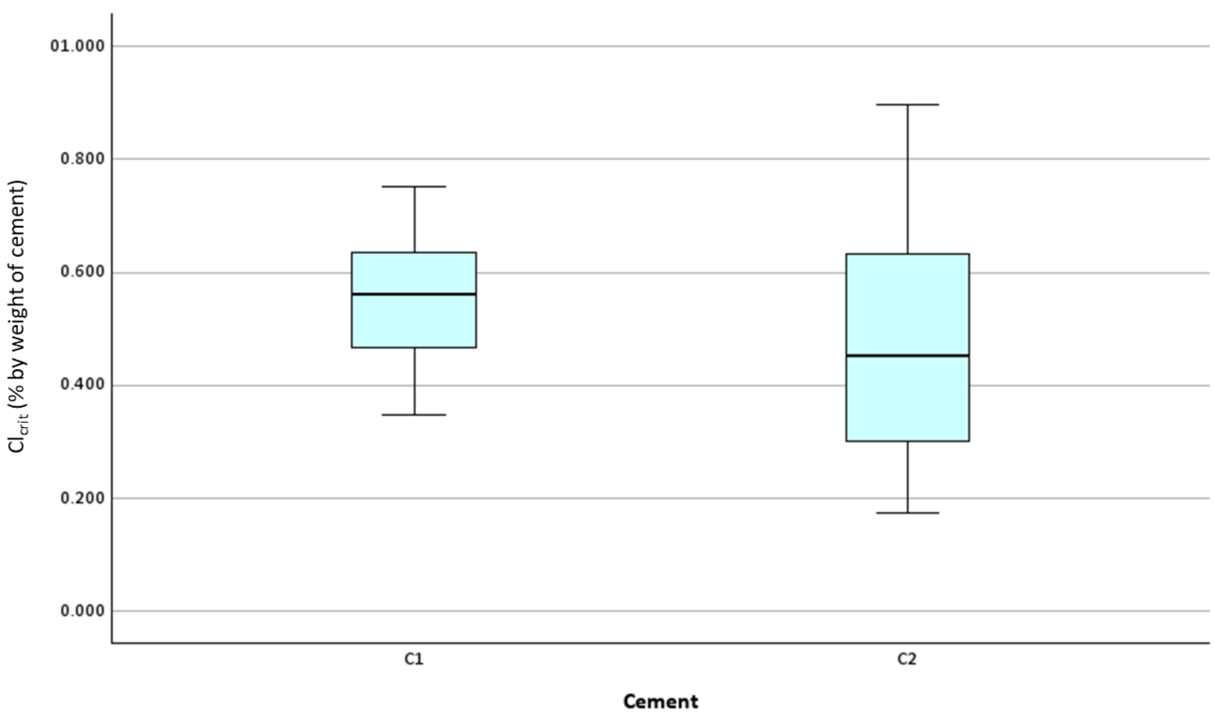


Figure 34 Cl_{crit} distributions for S3 samples in mortar made with C1 and C2

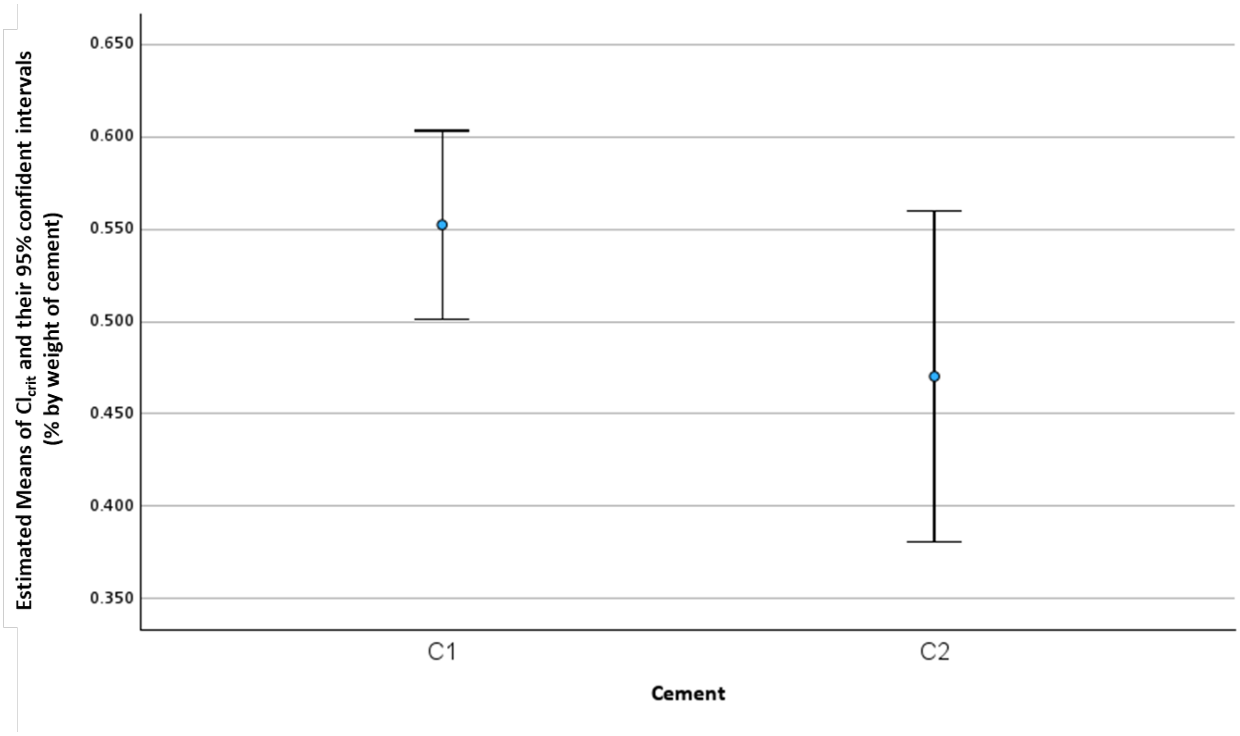


Figure 35 Mean Cl_{crit} values and 95% confidence intervals for S3 steel in C1 and C2 cement mortar

<table>
<tr><th colspan="12">Independent Samples Test</th></tr>
<tr><th colspan="2" rowspan="4"></th><th colspan="2">Levene's</th><th colspan="8">t-test for Equality of Means</th></tr>
<tr><th rowspan="3">F</th><th rowspan="3">Sig.</th><th rowspan="3">t</th><th rowspan="3">df</th><th colspan="2">Significance</th><th rowspan="3">Mean Difference</th><th rowspan="3">Std. Error Difference</th><th colspan="2">95% Confidence Interval</th></tr>
<tr><th rowspan="2">One-Sided p</th><th rowspan="2">Two-Sided p</th><th rowspan="2">Lower</th><th rowspan="2">Upper</th></tr>
<tr></tr>
<tr><td rowspan="2">Cl_{crit}</td><td>Equal variances assumed</td><td>7.030</td><td>0.012</td><td>1.668</td><td>38</td><td>0.052</td><td>0.104</td><td>0.082</td><td>0.049</td><td>-0.018</td><td>0.182</td></tr>
<tr><td>Equal variances not assumed</td><td></td><td></td><td>1.668</td><td>30.164</td><td>0.053</td><td>0.106</td><td>0.082</td><td>0.049</td><td>-0.018</td><td>0.183</td></tr>
</table>

Figure 36 Independent Sample t-test Comparison Results

Figure 37 shows the Cl_{crit} distributions from all laboratories using all cement sources separated by the steel source (groups S1, S2, and S3) and the first phase study results, identified as group S4. The Cl_{crit} distribution identified as group S4 is a combination of all the first phase study results that were shown in Figure 2. Figure 38 shows only the mean Cl_{crit} values of these groups and their 95% confidence intervals. Statistical analysis indicates that the steel type was a significant factor but as shown in Figure 39 Tukey's comparison of the groups, placed only S3 samples in a different subset, i.e. that the difference of the means between steel samples S1, S2, and the first phase steel samples (S4) were not statistically significant.

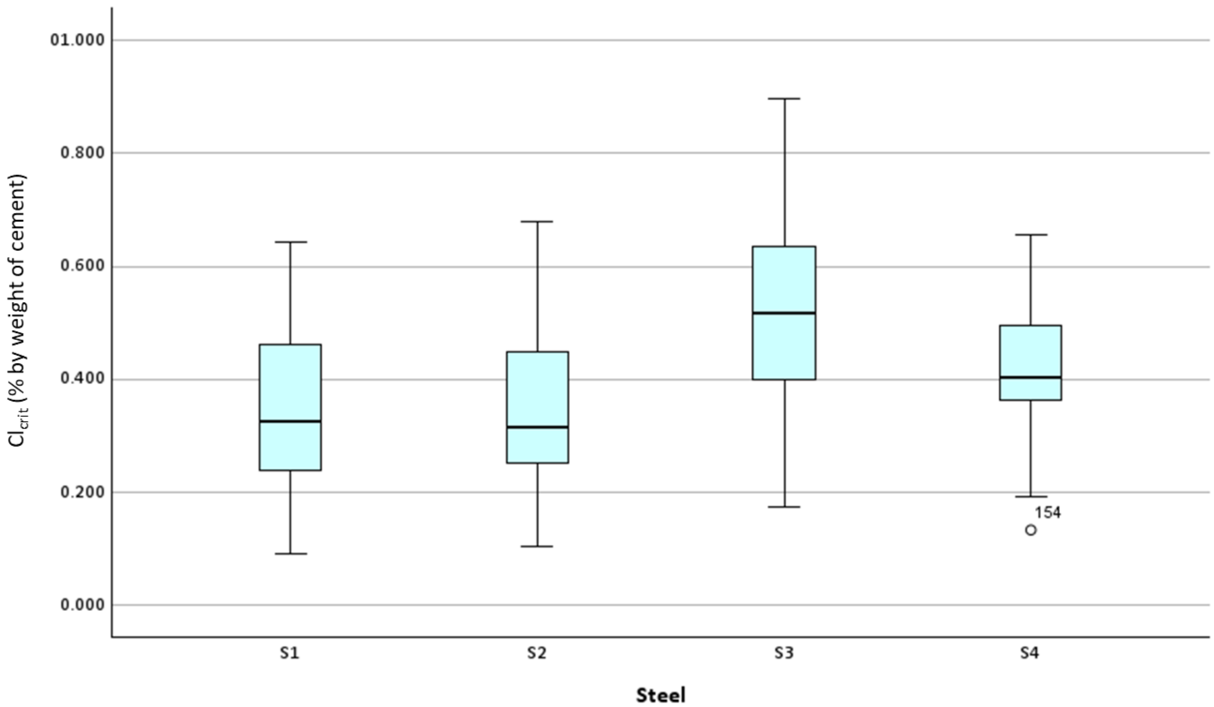


Figure 37 Cl$_{crit}$ distributions for all samples separated by steel source including the first phase study

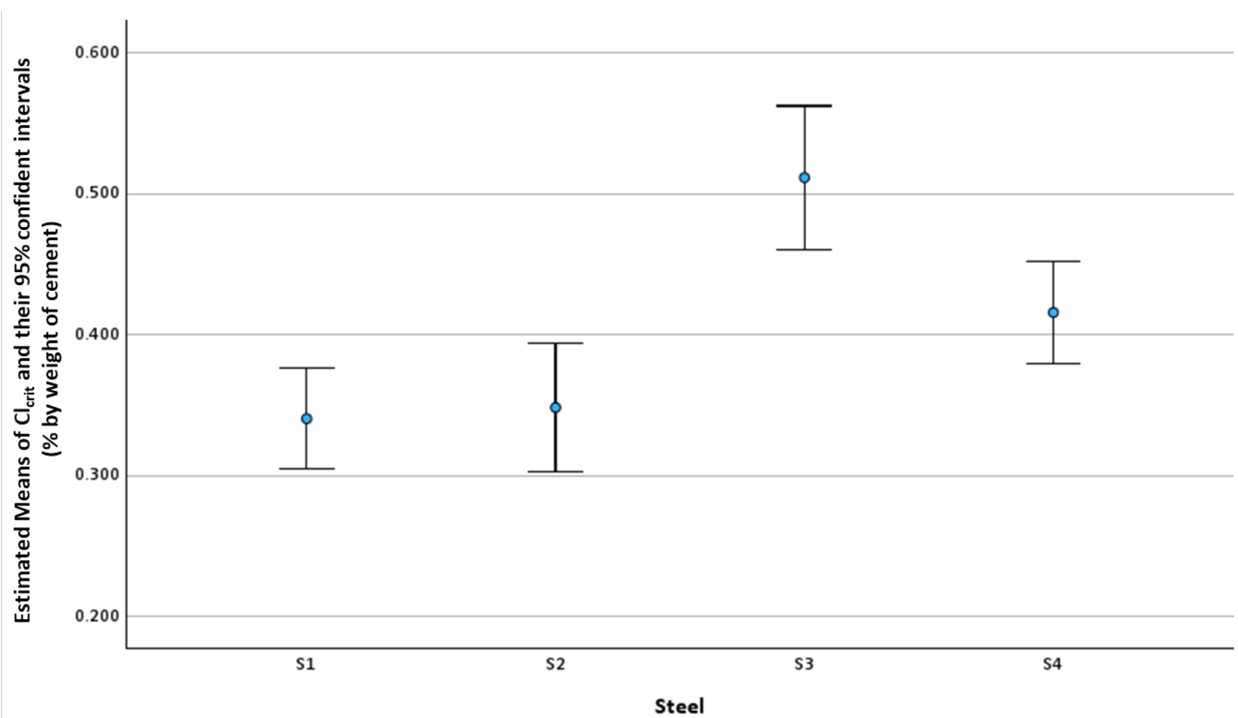


Figure 38 Mean Cl$_{crit}$ values and 95% confidence intervals for Phase I & II, separated by steel

	SteelType	N	Subset for alpha = 0.05 1	2
Tukey HSD[a,b]	1	68	.340	
	2	35	.348	
	4	42	.416	
	3	40		.511
	Sig.		.065	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 43.437.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

Figure 39 Tukey's comparison of all results (Phase I & II) grouped by steel

It should be noted again that all the steel samples shown in Figure 37 meet the ASTM A615 Grade 60 specifications and they were all tested in mortar made with ASTM C150 Type I/II cement using the OC_{crit} test method. Figure 40 shows the Cl_{crit} distributions for all the samples combined in Phase I and II studies and Figure 41 shows the analysis of variance table for their comparison which indicates that the difference between the means is not statistically significant. As expected with the introduction of different material sources for steel and cement in Phase II study, variability and range of Cl_{crit} results increased but the change in the mean value was not statistically significant. The standard deviation of data in Phase II was 0.165 compared to 0.113 in Phase I and the range was 0.806 compared to 0.522 in Phase I.

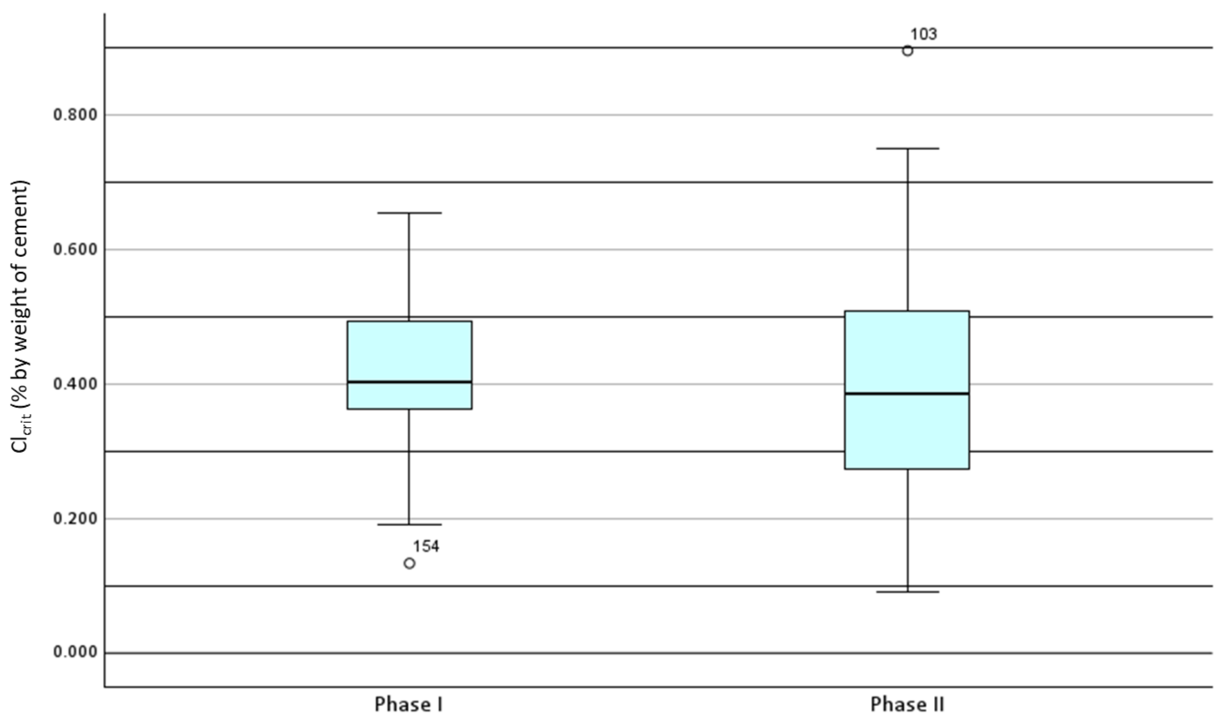


Figure 40 Cl_{crit} distributions for all samples in Phases I and II

ANOVA Table[a]

			Sum of Squares	df	Mean Square	F	Sig.
Clcrit * Phase	Between Groups	(Combined)	.021	1	.021	.880	.349
	Within Groups		4.413	183	.024		
	Total		4.435	184			

a. With fewer than three groups, linearity measures for Clcrit * Phase cannot be computed.

Figure 41 Analysis of Varince for Cl_{crit} values obtained in Phase I and II studies

h. All Cl_{crit} Results Combined

The Cl_{crit} distributions obtained in Phase I and II studies are combined for a general Cl_{crit} distribution for ASTM A615 Grade 60 steel samples in mortar made using ASTM C150 Type I/II cements since the difference between the means was not statistically significant. Figure 42 shows the histogram of the Cl_{crit} data that visually approximates a normal distribution and Figure 43 shows the QQ plot of the data that approximates a normal distribution with some deviation at the tails. Kolmogrov-Smirnov and Shapiro-Wilk tests for normality also showed that the hypothesis of normal distribution cannot be rejected as shown in Figure 44. The mean value of the combined data is 0.396 % by weight of cement with a standard deviation of 0.155 which translates into a 95% confidence interval between 0.373 and 0.418 % by weight of cement. The median Cl_{crit} value is 0.395 % by weight of cement between a minimum value of 0.091 and a maximum value of 0.897. The 25 and 75 percentile of the data are 0.279 and 0.506 % by weight of cement, respectively, defining an interquartile range of 0.227, i.e. the middle 50% of the data was located within this range around the mean.

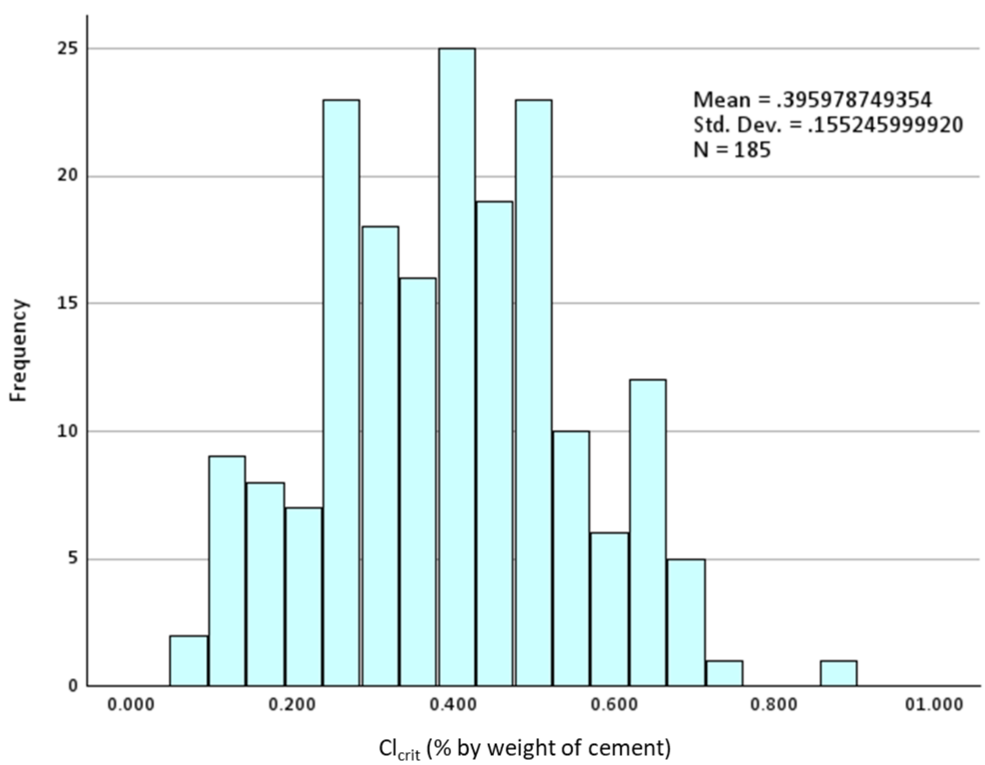


Figure 42 Histogram of the combined Cl_{crit} data of Phases I and II

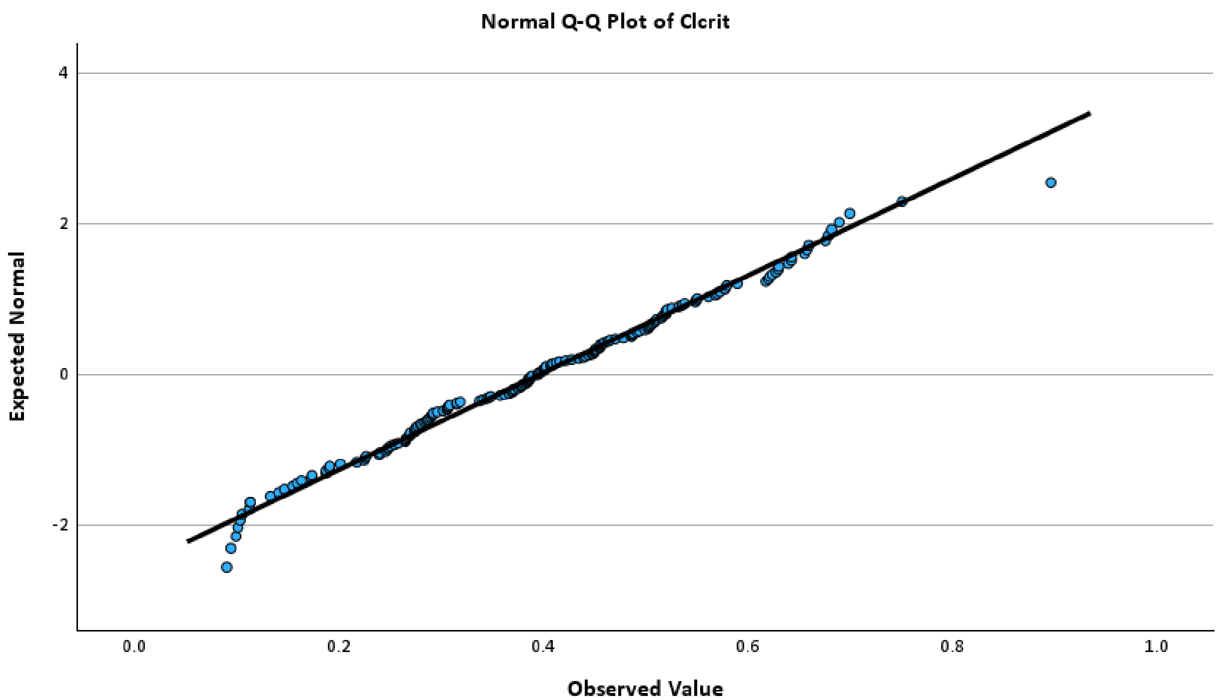


Figure 43 QQ Plot for the combined Cl_{crit} data of Phases I and II

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	Sig.	Statistic	df	Sig.
Clcrit	.058	185	.200*	.988	185	.124

*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Figure 44 Normality Test Results for Combined Data

V. Summary and Conclusions

The Cl_{crit} data published in the literature exhibits significant scatter and varies between ~0.1 and 3.1% by weight of cement. An important factor for this variability was believed to be due to the lack of a standard test method. To address this issue, the ACI 222 committee established a task group, TG1, to develop a standard Cl_{crit} laboratory test method. TG1 conducted a CRC funded research study (Phase I study) and evaluated two different test methods developed based on a general framework established by the task group. This study showed that the OC_{crit} test method developed at the Oregon State University was providing consistent results with good inter- and intra-laboratory variability and the task group recommended further evaluation of this test method which initiated the Phase II study described in this report. The objective of this study was to measure the variability of OC_{crit} data due to different sources of materials that meet the requirements of the same ASTM specifications. Cement meeting the specifications of ASTM C150 Type I/II cement was procured from three different suppliers and steel meeting the specifications of ASTM A615 Grade 60 was procured from three different suppliers. These materials were tested using the OC_{crit} method in five different laboratories consisting of academic and commercial labs to report the observed Cl_{crit} values. Important findings of this study are as follows:

- Testing in the Phase II study showed more variability in results for the same steel cement combinations tested at different laboratories. Delays in procurement and shipment of materials caused samples to be exposed to different environments for varying durations and may have caused more variability in the steel surface conditions before testing. Inclusion of new laboratories with less experience in performing the OC_{crit} test in the program and personnel changes due to the long duration of the testing program may have also added to the variability.
- The effect of source of cement was not statistically significant at 95% confidence level for the steel samples tested.
- The effect of source of steel was statistically significant but only steel from one source, S3, had a slightly higher mean Cl_{crit} value compared to the other steel samples.
- Comparison of Phase I and Phase II results indicated that the difference in mean Cl_{crit} values obtained in two phases was not statistically significant at 95% confidence level. As expected, varying the sources of cement and steel increased the variability of the results without changing the expected mean Cl_{crit} value although all the materials met the same respective ASTM standards. The combined Cl_{crit} data is normally distributed with a mean value of 0.396 % by weight of cement and a standard deviation of 0.155. The range of Cl_{crit} data increased from 0.522 in the first phase study to 0.805 for the combined Phase I and II data and the length of the interquartile range, where 50% of the data is located, was 0.227.

VI. References

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