

A New Method of Evaluating Corrosion-inhibiting Admixtures

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ABSTRACT

The quintessential standard for evaluating corrosion-inhibiting admixtures and their effects on a cement mix is ASTM⁽¹⁾ C1582. This standard relies on ASTM⁽¹⁾ G109 and ASTM⁽¹⁾ G180 to determine the admixture's impact on corrosion rate in concrete. Unfortunately, these tests come with several intrinsic issues that limit their use and consistency. Notably, ASTM⁽¹⁾ G109 runs for several years and ASTM⁽¹⁾ G180 has an experimental precision of one magnitude. To date, ASTM⁽¹⁾ G180 remains the most favorable test to perform regardless of its inaccuracy because it only requires roughly three days of experimentation.

This report will evaluate immersion testing run in similar conditions to ASTM⁽¹⁾ G180 over a longer time frame than standard to ASTM G180. Immersion testing typically yields precise results, and since ASTM⁽¹⁾ G180 already evaluates a metal's reactin to immersion in a test solution, a true immersion test in a similar slutin has the potential to afford comparable results that are easier to understand and utilize while obtaining higher precision.

Key words: Corrosion inhibitor, admixture, ASTM⁽¹⁾ G180, ASTM⁽¹⁾ G31, ASTM⁽¹⁾ C1582, concrete, cement, reinforcement, test method

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INTRODUCTION

Corrosion inhibiting chemical products have been developed for several decades with the purpose of reducing and preventing corrosion within reinforced structures. While some of these materials can be applied to the surface of hardened concrete¹, there are no major standardized methods or specifications which govern the use or performance of these materials. At this time, directly admixing chemical corrosion inhibitors into concrete is one of the most common methods of arresting corrosion in newly constructed buildings and infrastructure. Due to this, several standards have been developed to judge the effect of corrosion inhibiting admixtures on concrete as well as verify their performance. This paper will address issues with one such standardized test method, ASTM⁽¹⁾ G180, and evaluate the comparability of immersion testing in similar conditions.

Standard Test Methods for Corrosion Inhibiting Admixtures

At present, the major performance requirements of corrosion inhibiting admixes are defined by ASTM C1582². This specification references sixteen other standards and standard test methods to evaluate the effect a given corrosion inhibiting admixture has on the physical properties of a concrete mix design as well as to determine its performance as a corrosion inhibitor. This is intended to provide robust criteria for engineers to use when judging the merits of proposed corrosion inhibitor admixtures. The two test methods utilized in ASTM⁽¹⁾ C1582 to determine the corrosion inhibition performance of an admixture are ASTM⁽¹⁾ G180 and ASTM⁽¹⁾ G109.

ASTM⁽¹⁾ G109

ASTM⁽¹⁾ G109's procedure evaluates the inhibition performance by measuring the macrocell current of embedded steel bars in a concrete test block on a monthly cycle³. Once the average integrated macrocell current of the control blocks exceeds 150 Coulombs, the test is brought to a halt, the bars are exhumed and evaluated, and the acid soluble chloride content at the rebar depth is determined. This method allows for the empirical observation of corrosion inhibitor performance in an actual block of concrete, however the primary issue with this procedure which limits its use is the timeframe over which the test is conducted. As with almost any test performed on rebar in-situ, this method can take several years depending on the permeability of the concrete block¹. This time frame is undesirable when trying to formulate a product or otherwise meet customer requirements.

ASTM⁽¹⁾ G180

ASTM⁽¹⁾ G180 is commonly used in place of ASTM⁽¹⁾ G109 to meet the requirements of ASTM⁽¹⁾ C1582 due to its relatively short test duration. The procedure of ASTM⁽¹⁾ G180 only takes three days, allowing for two rounds of testing to be completed in a single week⁴. This test method utilizes a linear polarization resistance (LPR) method to determine the polarization resistance (R_p) of a metal sample in simulated concrete pore solution. To pass this test, an admixture must return an average polarization resistance greater than ten times the average value of the control. ASTM⁽¹⁾ C1582 lowers this requirement slightly by only requiring a corrosion-inhibiting admixture to return an average result greater than eight times that of the control.

Despite the rapid nature of this test, there are several conceptual issues which challenge the utility of the method. One large issue is described in the precision and bias statement of the method, namely that the acceptable single-operator precision is one magnitude and acceptable interlaboratory precision is even wider (1.24 magnitude). This causes the method's precision to be equal to or wider than the criteria used to judge whether an admixture operated as a corrosion inhibitor. Additionally, due to the wide interlaboratory precision, it is possible for two different laboratories to test the same admixture and return with conflicting results despite both performing the test correctly. This wide precision is not

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unexpected. LPR measurements assume uniform corrosion over a metal surface and localized corrosion can cause erratic corrosion potential fluctuations⁵. Such localized corrosion is expected both in existing infrastructure⁶ as well as in tests utilizing alkaline solutions with high chloride concentrations⁷. Ultimately, the solution's composition results in a predictably large inaccuracy during LPR measurements that cannot be avoided.

Another concern with the test is the metal used for the experiment. AISI⁽²⁾ 1215 steel is required by ASTM⁽¹⁾ G180 (section 5.5.1) and is metallurgically defined as resulfurized, rephosphatized machining steel containing 0.26 – 0.35 % sulfur and 0.04 – 0.09 % phosphorous⁸. The addition of these elements is specifically designed to make the act of machining the steel easier⁹, but this steel chemistry does not correspond to the steel used in rebar. For instance, sulfur has been demonstrated to increase the corrosion experienced by steel in concrete pore solution¹⁰, showing that sulfur is undesirable in rebar metallurgy. Furthermore, the only requirement of rebar metallurgy per ASTM⁽¹⁾ A615 (the standard specification for deformed and plain carbon-steel bars for concrete reinforcement) is that rebar contain no more than 0.06 % phosphorous¹¹. The range of phosphorous in AISI⁽²⁾ 1215 steel goes above this requirement meaning it is fundamentally unfit for use as rebar steel according to this specification.

Due to the importance of this test method, it is critical that the information it provides be applicable to the situation it informs upon and further is understandable by the engineers seeking to utilize the results. However, beyond the issues noted above, ASTM⁽¹⁾ G180 also requires an air purge (which is unnecessary and not representative of many concrete environments because concrete internals can remain consistently saturated with water¹²), and the results, reported in $\log_{10}(1/R_p)$ values, are unintuitive to those unfamiliar with the method and cannot be easily correlated to corrosion rate.

Immersion Testing

Immersion testing is fundamental methodology to corrosion science. By exposing a metal sample to a given solution, one can evaluate the corrosivity of a solution or the efficiency of a corrosion inhibitor. Testing has even shown immersion testing to be generally more precise than LPR measurements of the same corrosion system¹³.

Fundamentally, ASTM⁽¹⁾ G180 is an electrochemically-evaluated immersion test since it, in essence, exposes a metal sample to a given solution for a period of time and then assesses the metal's reaction. This suggests that performing an immersion test in the solution conditions experienced in ASTM⁽¹⁾ G180 could afford comparable results to ASTM⁽¹⁾ G180 and potentially offer a route to obtain data which is more precise and intuitive. The purpose of this report is to present the results of such immersion testing alongside results generated via ASTM⁽¹⁾ G180 to demonstrate that the answers sought through the ASTM⁽¹⁾ G180 procedure are attainable through simpler methods.

This paper will seek to demonstrate the efficacy of immersion testing as a replacement to the ASTM⁽¹⁾ G180 method by performing ASTM⁽¹⁾ G180 and immersion test methods on five products (A – E) and comparing the results. Products A – D are amine carboxylate admixtures while Product E is a calcium nitrite admixture. All products were used at concentrations typical to their chemistry as suggested by commercially available products and their corresponding dosage in ASTM⁽¹⁾ G180.

EXPERIMENTAL PROCEDURE

Creating an ASTM⁽¹⁾ G180 Solution⁽⁴⁾

The initial solution should consist of deionized water unless evaluating an admixture. When evaluating an admixture, water is replaced volumetrically with the admixture at a rate corresponding to the

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admixture's dose rate (35 ml/l is equivalent to a 5 l/m³ concrete dosage). 200 g of cement powder is added to the test solution and the solution is stirred for one hour. This slurry is filtered to retrieve the main test solution.

Calcium hydroxide is added to this solution at 4 g/l and stirred for an additional 30 minutes before the test metal is added. After 24 hours, sodium chloride (NaCl) is added to bring the solution to a 0.5 M concentration.

Procedure A - ASTM⁽¹⁾ G180

An ASTM⁽¹⁾ G180 solution is created as described above. The initial amount of solution should total 1.00 L, but due to losses in filtering, 900 ml of filtrate is measured and transferred to an ASTM⁽¹⁾ G5 test cell. The test cell is then purged with carbon dioxide-free air at a rate greater than 300 cc/min. The cell is stirred and purged with air throughout the test until directed otherwise.

Concurrently to the test cell preparation, the AISI⁽²⁾ 1215 steel plug is degreased with ultrasonic cleaning for two minutes while submerged in hexanes. Once the degreasing is complete, the plug is carefully removed from the hexanes and wiped with lint-free laboratory cloths. Care must be taken to avoid scratching the plug or contaminating the metal surface. The plug is mounted in an electrode holder and placed in the test cell.

Once the chloride is added as described above in the section on creating an ASTM⁽¹⁾ G180 solution, the solution is stirred and purged for an additional 4 hours at which point the stirring is stopped. After 20 hours of additional purging, the open circuit potential (OCP) is measured. Polarization resistance (R_p) is measured by plotting the potentiodynamic polarization curve between ± 20 mV from the OCP and plotting the tangent line at the point on the line where the current is zero. The polarization resistance (R_p) is the slope of this tangent, and the results of this method are reported as $\log_{10}(1/R_p)$ values. An admixture must have a $\log_{10}(1/R_p)$ value 1.0 or less than the $\log_{10}(1/R_p)$ value of the control to pass the test.

Procedure B - ASTM⁽¹⁾ G31 Immersion Test with ASTM⁽¹⁾ G180 Preparation

An ASTM⁽¹⁾ G180 solution is created as described above. Due to the size of glassware used, the total initial volume of test solution is 2.00 L. After the solution is filtered, it is placed into an appropriate graduated cylinder and the volume is noted before transferring the solution into the immersion flask. Calcium hydroxide is added to the flask at this time. Once the calcium hydroxide is added, the immersion solution is stirred and the containing flask is placed on a thermocouple-controlled hot plate set to 40 °C.

While the immersion solution warms, C1018 steel panels are degreased with ultrasonic cleaning for two minutes while submerged in hexanes. Once the degreasing is complete, the panels are removed from the hexanes, wiped dry with lint-free laboratory cloths, and weighed to the nearest 0.0001 g to obtain the initial sample mass (M_i). The panels are then hung in the immersion flask with fishing line at equivalent relative heights.

After 24 hours, enough NaCl to bring the solution to a 0.5 M concentration is added to a secondary flask. A qualitative amount of solution is removed from the immersion flask and added to the secondary flask until all the NaCl is dissolved. The NaCl solution is then warmed up to 40 °C via a hot bath before

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being added to the immersion solution. This finalized solution is kept on the hot plate for two weeks at a constant temperature of 40 °C. After two weeks, the metal samples are retrieved from solution, corrosion products are removed, and the final mass and mass loss of the panels is determined.

Procedure C - Rudimentary Immersion Method Utilizing ASTM⁽¹⁾ G180 Preparation

An ASTM⁽¹⁾ G180 solution is created as described⁽¹⁾ above. Due to the size of glassware used, the total initial volume of test solution is 1.00 L. Once the solution is filtered, 200 ml is added to each of three 8 oz. jars, and calcium hydroxide is added to each jar at 4 g/l. The jars are closed and upturned several times to incorporate the calcium hydroxide.

C1008/1010 steel panels are degreased with ultrasonic cleaning for two minutes while submerged in hexanes. Once the degreasing is complete, the panels are removed from the hexanes, wiped dry with lint-free laboratory cloths, and weighed to the nearest 0.0001 g to obtain the initial sample mass (M_i). The panels are then placed in the immersion jars and left to condition overnight in a 40 °C oven.

After 24 hours, the jars are removed from the oven and enough NaCl to bring the solution to a 0.5 M concentration is added to each jar. The jars are upturned several times over roughly 30 seconds to ensure proper dissolution of the NaCl in solution. The jars are then placed back into the 40 °C oven. After two weeks, the metal samples are retrieved from solution, corrosion products are removed, and the final mass and mass loss of the panels is determined.

Procedure D - Rudimentary Immersion Method Without ASTM⁽¹⁾ G180 Preparation

200 ml of deionized water is added to each of three 8 oz. jars, and calcium hydroxide is added to each jar at 4 g/l. The jars are closed and upturned several times to incorporate the calcium hydroxide.

C1008/1010 steel panels are degreased with ultrasonic cleaning for two minutes while submerged in hexanes. Once the degreasing is complete, the panels are removed from the hexanes, wiped dry with lint-free laboratory cloths, and weighed to the nearest 0.0001 g to obtain the initial sample mass (M_i). The panels are then placed in the immersion jars and left to condition overnight.

After 24 hours, the jars are removed from the oven and enough NaCl to bring the solution to a 0.5 M concentration is added to each jar. The jars are upturned several times over roughly 30 seconds to ensure proper dissolution of the NaCl in solution. The jars are then placed back into the 40 °C oven. After two weeks, the metal samples are retrieved from solution, corrosion products are removed, and the final mass and mass loss of the panels is determined.

Metal Cleaning and Mass loss analysis

The metal cleaning process of procedures B, C, and D utilize a modified ASTM⁽¹⁾ G1 procedure. ASTM⁽¹⁾ G1 recommends several iterations of the cleaning procedure with mass loss measurements taken on each iteration to determine when all corrosion products have been successfully removed. This step was not performed due to the large volume of samples being tested and because the only corrosion witnessed within these experiments was localized and therefore very simple to determine the degree of cleanliness via visual appraisal.

Once all corrosion products have been removed, the panels are weighed and the new mass is subtracted from the initial mass (M_i) to determine the mass lost due to corrosion and cleaning (M_{f+c}). All cleaning is duplicated on fresh, preweighed panels, and once complete, the average mass lost due to

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cleaning (M_c) is recorded and subtracted from the M_{f+c} of each sample to yield the final mass of the panels (M_f). Mass loss due to corrosion is calculated by subtracting the final mass (M_f) from the initial mass (M_i). Corrosion inhibitor efficiency (IE) is calculated via Equation 1.

$$\frac{(\text{Control } M_f - \text{Experimental } M_f)}{\text{Control } M_f} \times 100\% = IE \quad (1)$$

RESULTS

Procedure A

Products A-E were tested according to Procedure A. From this testing, only Product A and Product E possessed $\log_{10}(1/R_p)$ values less than 1.0 of the control and are therefore the only products that have passed this testing. In contrast, products B, C, and D do not have the required $\log_{10}(1/R_p)$ values, and have failed this test.

Table 1: Results from Procedure A

Treatment	Solution Concentration	Equivalent Concrete Dose	R_p (k Ω)	$\log_{10}(1/R_p)$	Avg. R_p (k Ω)	Average $\log_{10}(1/R_p)$	Std. Dev $\log_{10}(1/R_p)$	Diference from Control $\log_{10}(1/R_p)$
Control	N/A	N/A	3.897	-0.591	4.26	-0.589	0.18	N/A
			7.637	-0.883				
			2.740	-0.438				
			2.779	-0.444				
Product A	4.2 ml/l	1.0 pint/yd ³ (0.6 l/m ³)	44.62	-1.650	47.64	-1.677	0.03	-1.088
			50.65	-1.705				
Product B	7.0 ml/l	1.5 pint/yd ³ (1.0 l/m ³)	3.959	-0.598	3.13	-0.480	0.12	0.109
			2.300	-0.362				
Product C	4.2 g/l	1 lb/yd ³ (0.6 kg/m ³)	31.73	-1.501	23.96	-1.355	0.15	-0.767
			16.19	-1.209				
Product D	7.0 ml/l	1.0 pint/yd ³ (0.6 l/m ³)	1.327	-0.123	1.67	-0.213	0.09	0.376
			2.007	-0.303				
Product E	104 ml/l	3 gal/yd ³ (14.9 l/m ³)	125.9	-2.100	124.65	-2.096	0.00	-1.507
			123.4	-2.091				

Procedure B

From this testing, only Product A and Product E possessed inhibitor efficiencies over 90%. Product C offered limited protection, and Product B did not offer any protection. This procedure was not performed on Product D. Since this method is not designed to judge the corrosivity of an admixture, negative results are represented as 0% inhibitor efficiency as it is clear corrosion inhibition did not occur.

Table 2: Results from Procedure B

Panel	Treatment	Solution Concentration	Equivalent Concrete Dose	M _i (g)	M _{f+c} (g)	M _c (g)	M _f (g)	Mass Loss (g)	Avg. Mass Loss (g)	Standard Deviation	Inhibitor Efficiency (%)
1	Control	N/A	N/A	14.5037	14.4929	0.0031	14.4960	0.0077	0.0063	0.0016	N/A
2				14.8820	14.8719		14.8750	0.0070			
3				13.6408	13.6336		13.6367	0.0041			
4	Product A	4.2 ml/l	1.0 pint/yd ³ (0.6 l/m ³)	15.0532	15.0521	0.0008	15.0529	0.0003	0.0002	0.0000	96.1
5				15.0789	15.0779		15.0787	0.0002			
6				15.1972	15.1961		15.1969	0.0003			
7	Product B	7.0 ml/l	1.5 pint/yd ³ (1.0 l/m ³)	14.7918	14.7814	0.0031	14.7845	0.0073	0.0064	0.0007	0.0
8				14.5451	14.5357		14.5388	0.0063			
9				14.3602	14.3515		14.3546	0.0056			
10	Product C	4.2 g/l	1.0 lb/yd ³ (0.6 kg/m ³)	14.6559	14.6525	0.0008	14.6533	0.0026	0.0024	0.0004	61.8
11				14.5975	14.5940		14.5948	0.0019			
12				14.9533	14.9506		14.9514	0.0019			
13	Product E	104 ml/l	3 gal/yd ³ (14.9 l/m ³)	15.0109	15.0096	0.0008	15.0104	0.0005	0.0006	0.0004	90.3
14				14.0270	14.0260		14.0268	0.0002			
15				13.6507	13.6487		13.6495	0.0012			

Procedure C

Products A-E were tested according to Procedure C. From this testing, only Product A and Product E possessed positive corrosion inhibitor efficiencies with results that were greater than 95%. In contrast, products B, C, and D possessed negative values of corrosion inhibition efficiency from this testing. Since this method is not designed to judge the corrosivity of an admixture, negative results are represented as 0% inhibitor efficiency as it is clear corrosion inhibition did not occur.

Table 3: Results from Procedure C

Panel	Treatment	Solution Concentration	Equivalent Concrete Dose	M _i (g)	M _{f+c} (g)	M _f (g)	Mass Loss (g)	Avg. Mass Loss (g)	Standard Deviation	Inhibitor Efficiency (%)
1	Control	N/A	N/A	23.8061	23.7907	23.7956	0.0105	0.0099	0.0008	N/A
2				23.9073	23.8936	23.8985	0.0088			
3				23.8787	23.8634	23.8683	0.0104			
4	Product A	4.2 ml/l	1.0 pint/yd ³ (0.6 l/m ³)	23.9038	23.9001	23.9050	-0.0012	-0.0009	0.0004	100
5				23.6072	23.6035	23.6084	-0.0012			
6				23.8719	23.8674	23.8723	-0.0004			
7	Product B	7.0 ml/l	1.5 pint/yd ³ (1.0 l/m ³)	23.8207	23.7860	23.7909	0.0298	0.0250	0.0038	0
8				23.8132	23.7836	23.7885	0.0247			
9				23.8296	23.8043	23.8092	0.0204			
10	Product C	4.2 g/l	1 lb/yd ³ (0.6 kg/m ³)	23.8352	23.8156	23.8205	0.0147	0.0163	0.0017	0
11				23.7579	23.7343	23.7392	0.0187			
12				23.8320	23.8117	23.8166	0.0154			
13	Product D	4.2 ml/l	1.0 pint/yd ³ (0.6 l/m ³)	23.8513	23.8322	23.8371	0.0142	0.0129	0.0012	0
14				23.7667	23.7487	23.7536	0.0131			
15				23.8159	23.7996	23.8045	0.0114			
16	Product E	104 ml/l	3 gal/yd ³ (14.9 l/m ³)	24.3142	24.3094	24.3143	-0.0001	0.0003	0.0003	96.6
17				23.6230	23.6177	23.6226	0.0004			
18				23.7321	23.7266	23.7315	0.0006			

Procedure D

This procedure was not performed on Products A-E, but was instead performed on 28 different raw materials in triplicate for a total of 87 tests (including controls) within a two week period of time. Figure 1 presents the average mass losses from this testing along with the standard deviation of the data groups shown as error bars.

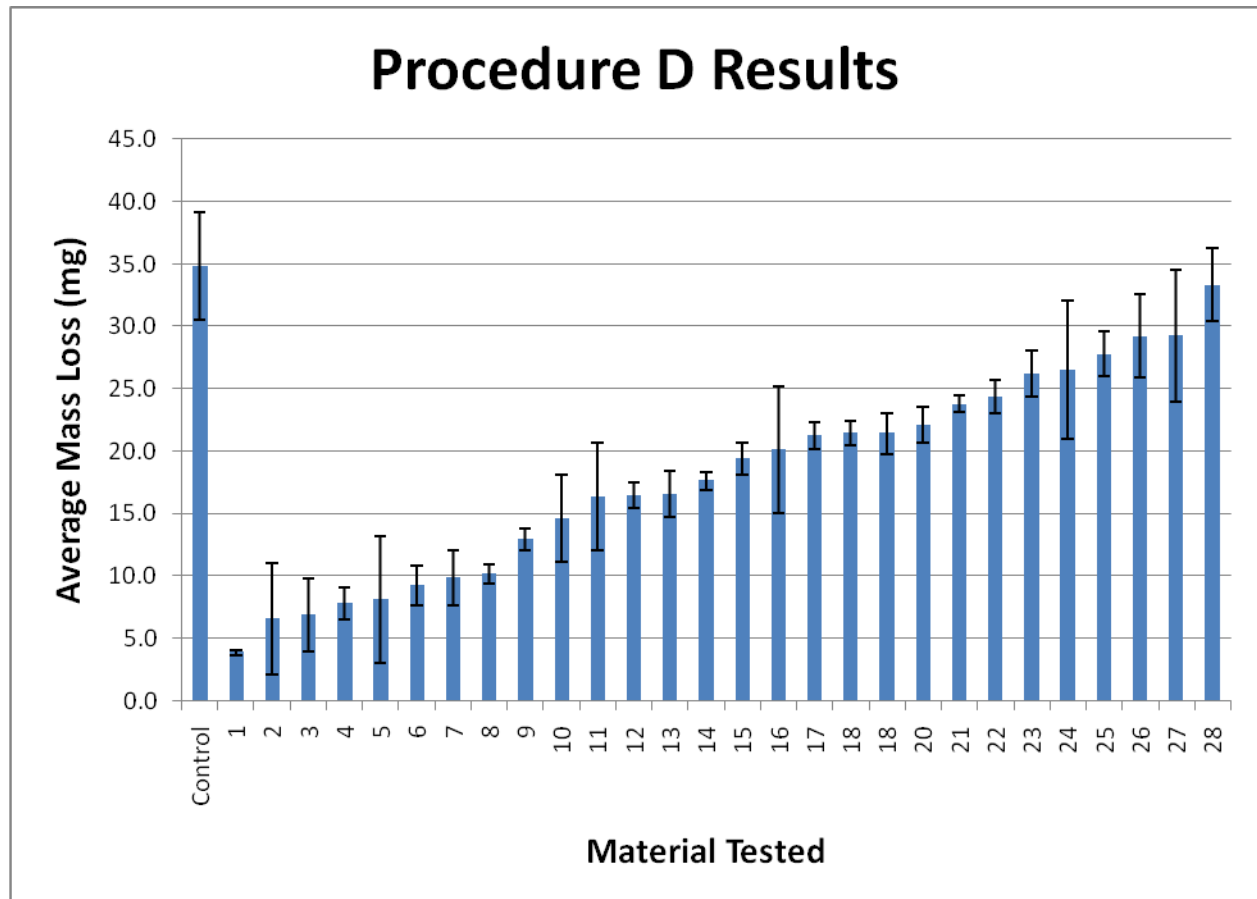


Figure 1: Graph of Procedure D results

Table 4: Aggregated results of Procedures A, B, and C

Treatment	Procedure A Result Pass/Fail	Procedure B Result (IE%)	Procedure C Result (IE%)
Control	N/A	N/A	N/A
Product A	Pass	96.1	100
Product B	Fail	0.0	0
Product C	Fail	61.8	0
Product D	Fail	N/A	0
Product E	Pass	90.3	96.6

CONCLUSIONS

Testing has shown that utilizing immersion methodology with the same solution preparation as ASTM⁽¹⁾ G180 yields similar results with a narrower precision. The results of Procedures A, B, and C are all in agreement. Product A and Product E were seen to pass ASTM⁽¹⁾ G180 testing while also maintaining high corrosion inhibitor efficiencies in immersion testing. All other tested products were seen to fail both ASTM⁽¹⁾ G180 testing and immersion testing.

Of particular interest in this testing is the performance of Product C because it did not outright pass or fail Procedure A or B. Not only do the results demonstrate the comparability between Procedures A and B, but it suggests Procedure C to be more aggressive than the other two methods. This is conceptually understandable because the Ca(OH)₂ required per the ASTM⁽¹⁾ G180 method is significantly more than can dissolve in the required volume of water. This caused the tested panels to sit in a powder bed of Ca(OH)₂ throughout the experiment which could in turn could have offered a more advantageous location for corrosion to begin due to the inhomogeneity of the metal's surroundings.

Scalability of rudimentary immersion testing in simulated concrete pore solution was evaluated via Procedure D. With this method, three operators were capable of gathering 87 data points in a two week period of time with the most time spent only during two days (initiating and concluding the test). The data of this method maintained a relatively narrow precision, characteristic of immersion testing. Attempting to perform ASTM⁽¹⁾ G180 a similar number of times would either require a great investment in glassware and gas or several months of continuous testing. However, while this method does allow for quick data acquisition, nuances of the proper ASTM⁽¹⁾ G180 solution preparation are lost, and the panels sit in the Ca(OH)₂ powder bed identified above as a potential to obscure the results. For these reasons, Procedure D works best as a method to rapidly screen inhibitor chemistries.

As identified in table 4, the immersion test methods performed maintain agreement with the results of ASTM⁽¹⁾ G180 while returning values with a greater degree of precision. Not only was greater precision achieved, immersion testing was demonstrated to be easily scalable while retaining said precision. Furthermore, the results of the immersion testing return values of mass loss, which is significantly easier to understand than the base ten logarithm of the inverse of the measured polarization resistance (returned by ASTM⁽¹⁾ G180).

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