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Investigating Newly Formulated Polysiloxane Coating Systems with Improved Erosion and Impact Properties

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Peer Review

*Bureau of Reclamation
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Acronyms and Abbreviations

BOR	Bureau of Reclamation
DI	Deionized water
DTH	Dry to handle
DTM	Direct to metal
EIS	Electrochemical impedance spectroscopy
HAR	Dilute Harrison solution
Mil Spec	Military Specification
OCP	Open circuit potential
PDS	Product data sheet
PRO	Profession
PSI	Pounds per square inch
Reclamation	Bureau of Reclamation
SCE	Saturated calomel electrode
S&T	Science and Technology
TVA	Tennessee Valley Authority
USACE	United States Army Corps of Engineers
UV	Ultraviolet
VOC	Volatile Organic Compound

Measurements

RH	Relative humidity
°F	Degrees Fahrenheit
Hz	Hertz
PSI	Pounds per square inch
mV	Millivolt
g	Grams
in	Inches
in-lbs	Inch pounds
mg/hr	Milligrams per hour
ohms	Ohms

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Executive Summary

Vinyl has long been considered the benchmark coating system for immersion service for the Bureau of Reclamation (BOR) and the United States Army Corps of Engineers (USACE). Vinyl and other high performance historical coatings and linings used at Reclamation have been engineered to provide a minimum service life of 50 years, however, more stringent environmental regulations are causing these historical systems to be phased out. Many of the modern replacement coating systems exhibit reduced longevity. To address this, researchers from the USACE and Reclamation have investigated historical coatings, including vinyl resin options, to identify the properties that contribute to long-term corrosion resistance. Recent studies have highlighted several epoxy polysiloxane (polysiloxane) coatings that exhibit corrosion protection comparable to those of historical coatings, indicating that these modern formulations could potentially achieve a similar service life.

Vinyl coatings provide corrosion resistance through their barrier properties, have increased durability, and possess the desired minimum service life for Reclamation structures, but their volatile organic compound (VOC) content is a significant drawback, greatly limiting their application. Polysiloxane coatings systems displayed high barrier properties in prior laboratory testing and have reduced environmental safety factors, including low VOCs, however these coating systems displayed reduced erosion and impact resistance.

The goal of this study was to improve impact and erosion resistance of epoxy polysiloxanes while maintaining barrier properties for corrosion protection. Manufacturers provided modified commercial formulations or new formulations for polysiloxane coatings. A total of twelve polysiloxane coatings systems from three different manufacturers were evaluated in this study and directly compared against USACE vinyl system 4 and system 5EZ. The polysiloxanes were evaluated in conjunction with various primers including epoxy primers, zinc-rich primers, and direct-to-metal application. The evaluation included ASTM International and Reclamation-developed testing standards to assess the performance of the coatings.

This study found that only one coating system tested is considered a viable replacement system for vinyl system 4 and none of the systems tested were viable replacements for system 5-E-Z. Researchers recommend further investigation and field trials on this coating system.

The same manufacturer that formulated the promising replacement coating for system 4 also provided a coating with a zinc rich primer. This coating had promising erosion, impact, and EIS results, but had poor adhesion and blistered during testing. Researchers recommend that the manufacturer identifies a more compatible zinc rich primer, which could possibly mitigate the poor adhesion and blistering results, and retests the system as a potentially viable replacement for vinyl system 5-E-Z.

1. Introduction

Polysiloxane coating systems were originally developed for atmospheric exposure applications. Previous research projects, conducted by Reclamation's Materials and Corrosion Laboratory in collaboration with the USACE, evaluated various commercially available polysiloxane systems. Electrochemical impedance spectroscopy (EIS) testing was utilized to evaluate the coating's ability to resist electrolyte penetration. Findings from these previous projects demonstrated polysiloxanes can provide corrosion protection in water immersion and cyclical environments in addition to atmospheric exposure.

For decades, Reclamation has utilized vinyl coatings for immersion service applications with great success and used vinyl coatings as a benchmark and comparator for coatings in water immersion service. Some commercial polysiloxane products demonstrated comparable performance to vinyl coatings for corrosion protection, but under perform in erosion and impact resistance. While barrier properties are a critical performance metric for coatings used in immersion service, impact and erosion resistance are equally important properties in raw water environments.

Due to vinyl's superior properties in immersion, it is no surprise that vinyl coatings also provide long service lives. Examples include Unit 5 Penstock at Shasta Dam, where a vinyl coating system is still providing adequate corrosion protection after 70 years of service and Collbran Salt Creek Siphon after 63 years of service. While vinyl coatings display excellent barrier properties, corrosion protection, durability, and service life, they have one significant drawback. Vinyl coatings are applied using high levels of volatile organic compounds (VOCs). VOCs are chemicals that can vaporize, posing health, safety, and environmental risks. Regulatory restrictions on VOC emissions, which have become increasingly stringent in the past few decades, have led to limitations on the use of vinyl coatings on Reclamation infrastructure and in the coatings industry.

In response to the environmental concerns surrounding vinyl coatings, there has been growing interest in developing alternative coating technologies with lower VOC content and reduced environmental impact. Polysiloxane coatings are one potential alternative, offering comparable corrosion protection to vinyl coatings while being formulated with lower VOC content. By leveraging advancements in coating chemistry and formulation techniques, coatings manufacturers have attempted to develop more durable polysiloxane coating systems that retain their impressive barrier properties, while simultaneously improving their impact and erosion performance. If successful, this would allow polysiloxane coating systems to perform on par with vinyl in immersion service environments, while providing a low VOC product that is much better for the environment and the coating applicators working on job sites.

The goal of this study was to evaluate these advanced polysiloxane coating systems, to determine if they can be utilized as a suitable replacement to the vinyl coating systems currently used for immersion service at Reclamation facilities. The newly formulated systems were put through Reclamations performance evaluation protocols. This report illustrates the results of that testing.

2. Laboratory Experiments

2.1 Performance Evaluation of Commercially Available and Experimental Epoxy Polysiloxane Coatings

2.1.1 Surface Preparation and Coating Application

Surface preparation for all panels consisted of removing oil and contaminants by detergent cleaning following SSPC-SP1. Once panels were cleaned, they were abrasive blast cleaned to SSPC-SP 5/NACE 1 with an angular profile of 3.5 mils. The coatings were applied in accordance with manufacturers' instructions by Reclamation researchers. All specimens were applied to A36 carbon steel substrates. Conventional spray equipment was used for application and a stipe coat around edges was applied via brush for each coat. The polysiloxane systems, which require a minimum of 40 percent relative humidity (RH) to cure, were placed in a containment with a humidifier to obtain 60 percent RH during cure inside the spray booth. After coatings were cured dry to handle (DTH), they were moved to an environmentally controlled room held at 70 degrees Fahrenheit (°F) with 70 percent RH and remained there until fully cured. Each coating system had a sample set of twenty-four 3-inch x 6-inch x 1/8-inch steel coupons, two 4-inch diameter 1/8-inch-thick round disks, two 11-inch diameter 1/8-inch round disks, and one 3-inch diameter 1 foot section of pipe.

Several coating systems were manufacturer applied. In these cases, test panels were prepared with the above surface preparation method and shipped to the manufacturer for application. The coated test panels were then shipped back to the Reclamation labs for evaluation.

2.1.2 Coating Performance Evaluation

A version of Reclamation's standard testing protocol focusing on durability, specifically impact and erosion properties, was used (listed below) and the rating criteria can be found in Appendix A. The rating criteria allows for efficient and accurate comparisons between all of Reclamation's previously tested coatings.

- Rust Creep and Blistering Evaluation (ASTM D1654 and ASTM D714) following various exposures:
 - HAR - Immersion in a dilute Harrison solution (HAR), 0.5 g sodium chloride and 3.5 g ammonium sulfate per liter of deionized (DI) water (ASTM D870 Modified)
 - DI - Immersion in DI water (ASTM D870)
 - PRO - Prohesion cyclic testing (ASTM D5894)
 - BOR - Modified Prohesion cyclic test
 - FOG - Prohesion testing in a salt fog test cabinet (ASTM G85 Annex A4)
 - QUV - Condensation and UV cyclic test (ASTM D4587)
- Wet and Dry Pull-off Adhesion testing (ASTM D4541)
- Direct impact testing (ASTM D2794)
- Erosion resistance (USBR-5071-2015)

2.1.2.1 Rust Creep and Blistering Evaluation

The rust creep and blistering evaluation was performed on coated 3" x 6" x 1/8" steel test panels. Panels were suspended from hooks through pre-cut holes in immersion tanks containing either HAR or DI. The tank depth was sufficient to fully submerge the panels. To ensure homogeneity, the solutions were mixed before adding to the tank. Each tank was connected to a filtration system that circulated the solutions through the tank. The tanks were cleaned, and solutions replaced once a year.

The PRO test consists of panels rotated every week in the following order: QUV-FOG-QUV-FOG. This test intends to simulate the natural effects of alternating wet and dry exposure. The BOR test is a modified Prohesion test, which is intended to simulate the effects of a fluctuating immersion environment/splash zone. Panels were rotated every week in the following order: QUV-FOG-HAR-FOG.

Test panels were exposed for approximately 30 weeks (5040 hours) in accordance with industry standard practices. Test panels from Manufacturer 1 were removed from the rust creep and blistering evaluation test at 10 weeks (1680 hours) due to unacceptable erosion test results. This will be discussed in more detail later in the report.

After 30 weeks of exposure, panels were tested according to ASTM D1654, Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments (rust creep), and ASTM D714, Standard Test Method for Evaluating Degree of Blistering of Paints. Two panels each were prepared for HAR, DI immersion, BOR, and PRO, and one each for FOG and QUV tests. Panels were scribed down the center on one side with a Dremel® tool. The scribes were approximately 1 millimeter in width and 3 inches in length to expose the steel substrate. This scribe allows researchers to determine the degree of undercutting (rust creep) resistance the systems provide.

2.1.2.2 Wet and Dry Pull-Off Adhesion Testing

Pull Off Adhesion (ASTM D2794) utilizes a loading fixture (dolly) attached to a coating surface with an adhesive. A pull off adhesion tester (DeFelsko Positest AT) was used to gradually apply an increasing load on the dolly until it was "pulled" off. The force that was required to pull off the dolly is the yield strength and is measured in pounds per square inch (psi). Adhesion testing was performed on test panels that were not exposed to immersion (dry adhesion) and test panels that were exposed to at least 7 months of immersion (wet adhesion). For the wet adhesion test, the panels were removed from the Harrison's solution, dried, surface prepped, and dollies were glued down. The panels were then placed in Reclamation's 100 percent humidity room for 24 hours. After 24 hours the panels were removed from the 100 percent humidity room and tested. Adhesion pull-off tests that had more than 20 percent glue failure were excluded from results.

2.1.2.3 Impact Testing

Impact Testing was done following the ASTM D2794 test method. This test method exposes the coating systems to the effects of rapid deformation (impact) utilizing a hand operated impact testing machine that drops a pre-determine weight onto the coating surface. The test value is reported in inch-lbs and is a representation of the threshold of impact that a coating system can endure without cracking or developing a hole or void.

2.1.2.4 Erosion Testing

The slurry erosion test is a Reclamation standard following the USBR-5071-2015 testing procedure. Coated 11-inch diameter, 1/8-inch-thick steel discs were used. The test duration was 96 hours per sample. Test samples were removed at 24 hours intervals and weighed, then placed back in the test. Duplicate samples were run. The average weight loss was compared between samples and a control.

2.1.3 Electrochemical Impedance Spectroscopy Testing

Electrochemical impedance spectroscopy (EIS) was performed with a Gamry Instruments FAS2 Femtostat, with dedicated EIS300 software. All measurements had a 10-millivolt (mV) sinusoidal perturbation at the open circuit potential, a frequency range of 10^5 to 10^{-2} Hertz (Hz), and ten data points per decade. The EIS test cell was consistent with a three-electrode set-up; a saturated calomel electrode (SCE), platinum mesh electrode, and the steel substrate were connected to the instrument as the reference, counter, and working electrode, respectively. The EIS testing surface area, as defined by the test cell, is 23 centimeters squared (cm^2). No corrections were made to the raw data for surface area. EIS was performed periodically, at set intervals, throughout the 30-week exposure period (HAR, DI, PRO, BOR, FOG, QUV).

2.1.4 Coating Naming Convention

The coating systems in this report are given a product code using the following designations.

- Manufacturer: numbered 1-3
- Base coat type:
 - E = epoxy
 - Z = zinc rich epoxy
 - D = direct to metal
- Topcoat type:
 - P = polysiloxane
- Number of components:
 - 1K = one component
 - 2K = two component
- Total number of coats:
 - 2 = two coats
- Cure speed:
 - F = fast cure

- S = standard cure

For example, product code 1-E-P-2K-2-F, means manufacturer 1, epoxy basecoat, polysiloxane topcoat, two component, two total coats, and fast cure.

Table 1: Coating System Report Codes

Coating Manufacturer	Report Code	Primer	Topcoat	Components	Total Coats	Cure Time
	Vinyl Sys. 4	Vinyl	Vinyl	-	-	-
	Vinyl Sys. 5-E-Z	Zinc Rich Vinyl	Vinyl	-	-	-
1	1-E-P-2K-2-F	Epoxy	Polysiloxane	2K	2	F
	1-E-P-2K-2-S	Epoxy	Polysiloxane	2K	2	S
	1-Z-P-2K-2-F	Zinc Rich Epoxy	Polysiloxane	2K	2	F
	1-Z-P-2K-2-S	Zinc Rich Epoxy	Polysiloxane	2K	2	S
	1-D-P-2K-2-F	Direct To Metal	Polysiloxane	2K	2	F
	1-D-P-2K-2-S	Direct To Metal	Polysiloxane	2K	2	S
2	2-E-P-1K-2-S	Epoxy	Polysiloxane	1K	2	S
	2-E-P-2K-2-S	Epoxy	Polysiloxane	2K	2	S
	2-Z-P-1K-2-S	Zinc Rich Epoxy	Polysiloxane	1K	2	S
	2-Z-P-2K-2-S	Zinc Rich Epoxy	Polysiloxane	2K	2	S
3	3-Z-P-2K-2-S	Zinc Rich Epoxy	Polysiloxane	2K	2	S
	3-D-P-2K-2-S	Direct To Metal	Polysiloxane	2K	2	S

3. Results

Each coating system was evaluated on a qualitative and quantitative basis and assigned a score based on the ratings criteria found in Appendix A: Excellent (dark green), Good (light green), Fair (yellow), and Poor (red). This rating system is applied to both the corrosion and mechanical properties for each coating system.

The rust creep rating criteria is broken down into three categories based on the harshness of the exposure condition. The harshest exposure condition is cyclic weathering testing (PRO, BOR, FOG). The second harshest exposure condition is immersion weathering testing (HAR, DI). The least harsh exposure condition is UV weathering (QUV). The harsher the exposure condition, the more lenient the rating criteria is. Meaning a PRO panel can have more rust creep than a DI panel but still fall under the same rating category. In addition, a test panel can fall under one rating criteria based on the rust creep measurement, but ultimately be demoted to a lower category based on the visual assessment (blistering, delamination, and other visual defects).

Coating barrier properties (EIS) were evaluated based on the impedance magnitude and the phase angle at 0.01 Hz. EIS values are listed in the Summary of Corrosion Resistance Performance tables for each coating system.

It is well established in the coatings industry that the higher the impedance value the more corrosion resistant a coating is thought to be. Therefore, the higher the impedance value the better a coating will likely perform while in service.

Phase angle values illustrate how resistive the coating system is. Resistive behavior shows that a coating system has undergone some type of corrosion reaction and is indicative of poor coating performance. A pure resistor has a phase angle of 0 degrees, while a pure capacitor has a phase angle of -90 degrees. Anywhere in between is a combination of both behaviors and is what most coatings typically display. This report will utilize three categories to describe a coatings resistive behavior. A coating is considered resistive if the phase angle is between 0 and -30 degrees, mixed between -30 and -60 degrees, and capacitive between -60 and -90 degrees.

Coating systems with higher impedance magnitudes and more capacitive behavior, are thought to perform better in the field and have longer service lives than coatings with lower impedance magnitudes and more resistive behavior.

The two vinyl systems used for comparison in this study are System No. 4 (4), which is 5 coats of formula V-766E, and System No. 5-E-Z (5EZ), which consists of 1 coat of zinc rich primer VZ-108d and 4 coats V-766E. These two coating systems are the benchmark in performance for water immersion service and have been successfully used at Reclamation and USACE facilities. All test data used in this report was taken from a previous Reclamation study titled Polysiloxane and Vinyl Comparison and Field Trials, which can be found in the references section of this report. No vinyl coating systems were tested as a part of this study. Polysiloxane coating systems without a zinc rich primer (coating report code with D or E) will be compared to vinyl system 4, and polysiloxane coating systems with a zinc rich primer (coating report code Z) will be compared to vinyl system 5EZ.

3.1 Coating Manufacturer 1

Coating manufacturer 1 submitted six coating systems to Reclamation for evaluation. After 24 hours of erosion testing, the coating eroded down to bare steel. The primary purpose of this research was to improve erosion and impact resistance of polysiloxanes with the goal of having impact and erosion resistance equal to or exceed that of vinyl. Manufacturers were tasked with formulating a polysiloxane coating system with improved impact and erosion properties while retaining its corrosion properties. Results from erosion testing showed that the new formulation had not improved erosion resistance and in fact worsened it based on results from previous research projects. These formulations would not meet the requirements for service on Reclamation infrastructure. A discussion between Reclamation researchers and the coating manufacturer took place and it was decided all coating systems from manufacturer 1 would be removed from the study.

3.2 Coating Manufacturer 2

3.2.1 Coating System 2-E-P-1K-2-S

3.2.1.1 Corrosion Resistance and Mechanical Testing

Table 2 summarizes the corrosion resistance performance and Table 3 summarizes the mechanical properties results. The overall corrosion resistance performance rating is good. The overall mechanical rating is fair.

Table 2: Summary of Corrosion Resistance Performance.

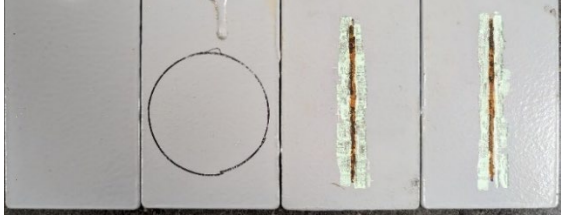
Exposure Type	Specimen Images and Visual Inspection Observations	Rust Creep Result (in)	Score
DI Immersion	 <p data-bbox="651 1593 850 1623">No visual defects</p>	0.02	Good

Table 2: Summary of Corrosion Resistance Performance.





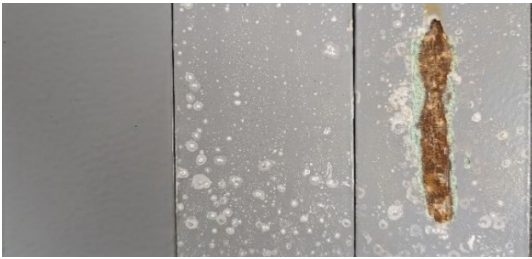
HAR Immersion	 <p data-bbox="647 520 854 548">No visual defects</p>	0.04	Good
PRO Cyclic	 <p data-bbox="647 819 854 846">No visual defects</p>	0.43	Fair
BOR Cyclic	 <p data-bbox="647 1125 854 1152">No visual defects</p>	0.46	Fair
QUV Cyclic	 <p data-bbox="647 1438 854 1465">No visual defects</p>	0.00	Excellent
FOG Cyclic	 <p data-bbox="480 1759 1024 1787">No visual defects (salt material seen on panel)</p>	0.17	Good

Table 2: Summary of Corrosion Resistance Performance.

Test	Result	Score
EIS (avg DI & HAR)	1.2×10^9 ohms @ 0.01 Hz	Excellent

Note: The images for DI immersion, HAR immersion, PRO cyclic, and BOR cyclic represent panels prepared pre-exposure and for EIS and rust creep testing from left to right. The images for QUV and FOG represent a panel prepared pre-exposure, a panel after exposure without a scribe, and a rust creep testing panel from left to right.

Table 3: Summary of Test Results for Mechanical testing.

Test	Result	Test Metric	Score
Impact	48 in-lbs	Threshold with no cracking or holidays, inch-lbs	Poor
Pull-off Adhesion	1854 psi g/adh/coh 15/5/80	Stress, psi Failure mode ¹	Good
Pull-off Adhesion (wet)	1325 psi g/adh/coh 15/0/85	Stress, psi Failure mode ¹	Good
Erosion	64 ± 10	mg/hour lost	Fair

¹ Adhesion (adh), cohesion (coh), intercoat (ic), glue (g). glue failure > 20% is not scored

3.2.1.2 Electrochemical Impedance Spectroscopy

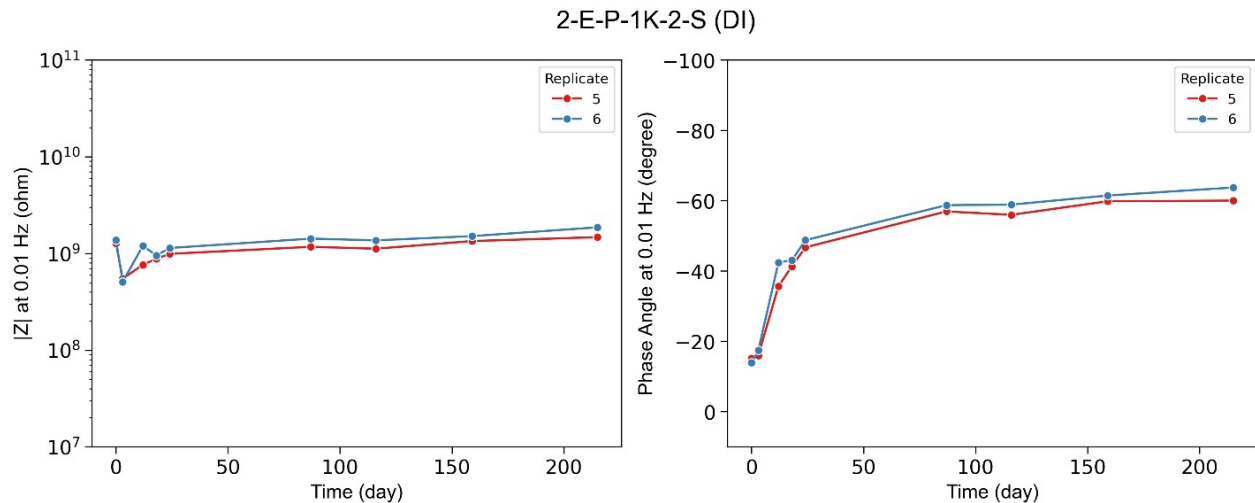


Figure 7: Impedance and phase angle over time (DI panels).

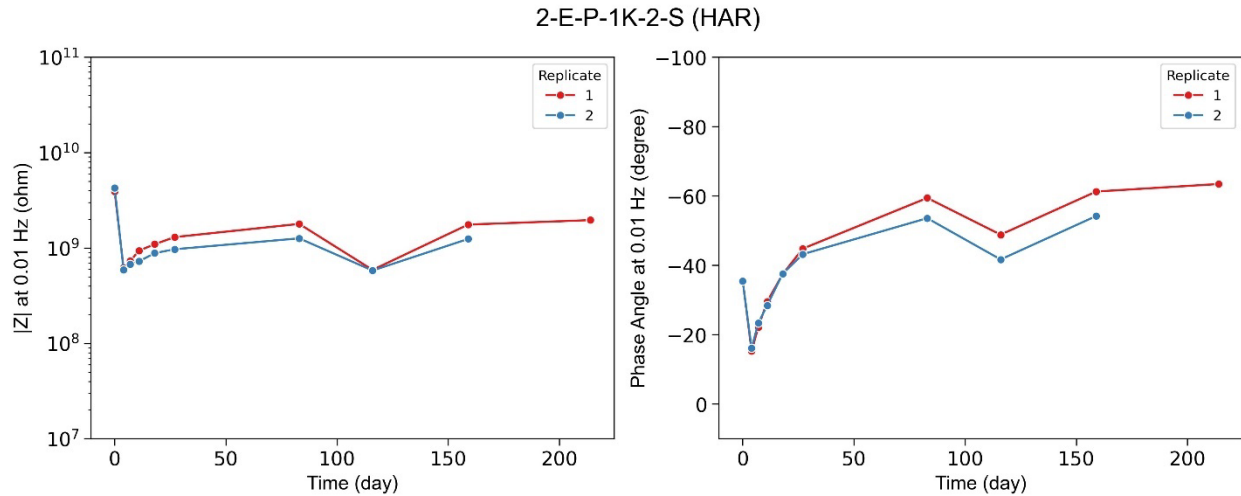


Figure 8: Impedance and phase angle over time (HAR panels).

3.2.1.3 Results and Discussion

Rust creep testing results for the PRO and BOR panels were fair. Slightly more rust creep on the PRO and BOR panels would have resulted in a poor rating. Rust creep results indicate satisfactory long term corrosion resistance performance.

The overall mechanical rating is fair. This coating system had particularly poor impact performance, good adhesion properties, and fair erosion performance, indicating unsatisfactory durability performance.

The average impedance value at 0.01 Hz (DI and HAR) was 1.2×10^9 ohms with less than one order of magnitude degradation at 0.01 Hz earning a rating of excellent. The average phase angle (DI and HAR) was -41 degrees earning a rating of mixed. EIS testing results indicate satisfactory long term corrosion performance.

3.2.2 Coating System 2-E-P-2K-2-S

3.2.2.1 Corrosion Resistance and Mechanical Testing

Table 4 summarizes the corrosion resistance performance and Table 5 summarizes the mechanical properties results. The overall corrosion resistance performance rating is fair. The overall mechanical rating is fair.

Table 4: Summary of Corrosion Resistance Performance.

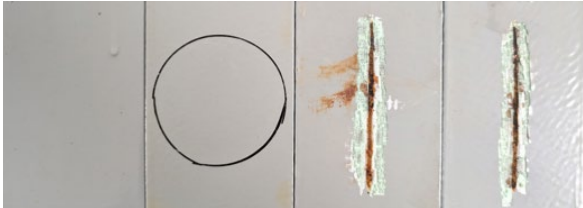




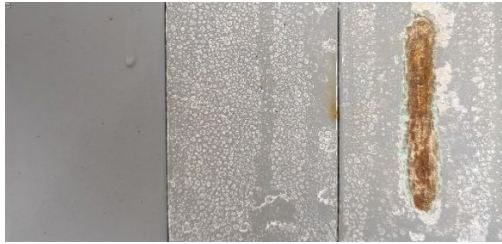
Exposure Type	Specimen Images and Visual Inspection Observations	Rust Creep Result (in)	Score
DI Immersion	 <p data-bbox="647 663 852 695">No visual defects</p>	0.08	Good
HAR Immersion	 <p data-bbox="647 947 852 978">No visual defects</p>	0.05	Good
PRO Cyclic	 <p data-bbox="647 1234 852 1266">No visual defects</p>	0.55	Poor
BOR Cyclic	 <p data-bbox="647 1522 852 1554">No visual defects</p>	0.39	Fair
QUV Cyclic	 <p data-bbox="647 1852 852 1883">No visual defects</p>	0.07	Fair

Table 4: Summary of Corrosion Resistance Performance.

FOG Cyclic	 <p>No visual defects (salt material seen on panel)</p>	0.21	Good
Test	Result	Score	
EIS (avg DI & HAR)	6.7 x10 ⁸ ohms @ 0.01 Hz	Good	

Note: The images for DI immersion, HAR immersion, PRO cyclic, and BOR cyclic represent panels prepared pre-exposure and for EIS and rust creep testing from left to right. The images for QUV and FOG represent a panel prepared pre-exposure, a panel after exposure without a scribe, and a rust creep testing panel from left to right.

Table 5: Summary of Test Results for Mechanical testing.

Test	Result	Test Metric	Score
Impact	36 in-lbs	Threshold with no cracking or holidays, inch-lbs	Poor
Pull-off Adhesion	1322 psi g/adh/coh 15/5/80	Stress, psi Failure mode ¹	Fair
Pull-off Adhesion (wet)	1291 psi g/adh/coh 20/0/80	Stress, psi Failure mode ¹	Good
Erosion	63 ± 10	mg/hour lost	Fair

¹ Adhesion (adh), cohesion (coh), intercoat (ic), glue (g). glue failure > 20% is not scored

3.2.2.2 Electrochemical Impedance Spectroscopy

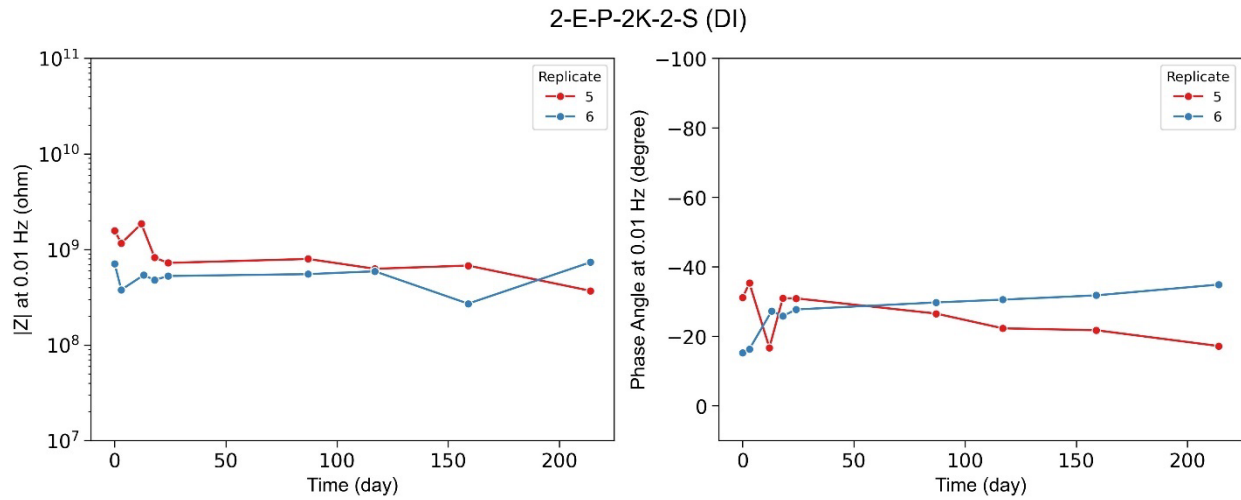


Figure 15: Impedance and phase angle over time (DI panels).

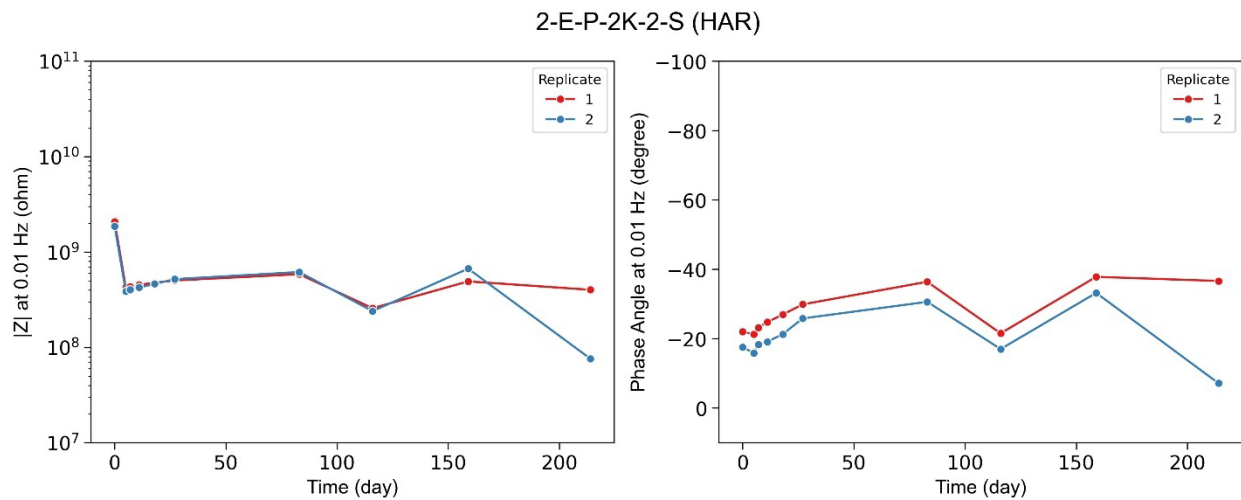


Figure 16: Impedance and phase angle over time (HAR panels).

3.2.2.3 Results and Discussion

Rust creep testing results for the BOR and QUV panels were fair. The rust creep results for the PRO panels were poor, displaying the highest rust creep number of any panel in the test. Rust creep results indicate unsatisfactory long term corrosion resistance performance.

The overall mechanical rating is fair. This coating system had particularly poor impact performance, fair adhesion properties, and fair erosion performance, indicating unsatisfactory durability performance.

The average impedance value at 0.01 Hz (DI and HAR) was 6.7×10^8 ohms with less than two orders of magnitude degradation at 0.01 Hz earning a rating of good. The average phase angle (DI

and HAR) was -25 degrees earning a rating of resistive. EIS testing results indicate unsatisfactory long term corrosion performance.

3.2.3 Coating System 2-Z-P-1K-2-S

3.2.3.1 Corrosion Resistance and Mechanical Testing

Table 6 summarizes the corrosion resistance performance and Table 7 summarizes the mechanical properties results. The overall corrosion resistance performance rating is good. The overall mechanical rating is fair to good.

Table 6: Summary of Corrosion Resistance Performance.

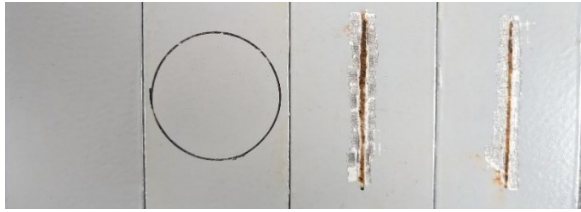





Exposure Type	Specimen Images and Visual Inspection Observations	Rust Creep Result (in)	Score
DI Immersion	 <p data-bbox="646 1060 852 1094">No visual defects</p>	0.01	Good
HAR Immersion	 <p data-bbox="646 1339 852 1373">No visual defects</p>	0.01	Good
PRO Cyclic	 <p data-bbox="646 1619 852 1652">No visual defects</p>	0.27	Fair

Table 6: Summary of Corrosion Resistance Performance.

BOR Cyclic	 No visual defects	0.23	Good
QUV Cyclic	 No visual defects	0.03	Fair
FOG Cyclic	 No visual defects (salt material seen on panel)	0.08	Excellent
Test	Result	Score	
EIS (avg DI & HAR)	1.9 x10 ⁹ ohms @ 0.01 Hz	Excellent	

Note: The images for DI immersion, HAR immersion, PRO cyclic, and BOR cyclic represent panels prepared pre-exposure and for EIS and rust creep testing from left to right. The images for QUV and FOG represent a panel prepared pre-exposure, a panel after exposure without a scribe, and a rust creep testing panel from left to right.

Table 7: Summary of Test Results for Mechanical testing.

Test	Result	Test Metric	Score
Impact	110 in-lbs	Threshold with no cracking or holidays, inch-lbs	Good
Pull-off Adhesion	1473 psi g/adh/coh 15/5/80	Stress, psi Failure mode ¹	Fair
Pull-off Adhesion (wet)	1385 psi g/adh/coh 10/5/85	Stress, psi Failure mode ¹	Good
Erosion	56 ± 8	mg/hour lost	Fair

¹ Adhesion (adh), cohesion (coh), intercoat (ic), glue (g). glue failure > 20% is not scored

3.2.3.2 Electrochemical Impedance Spectroscopy

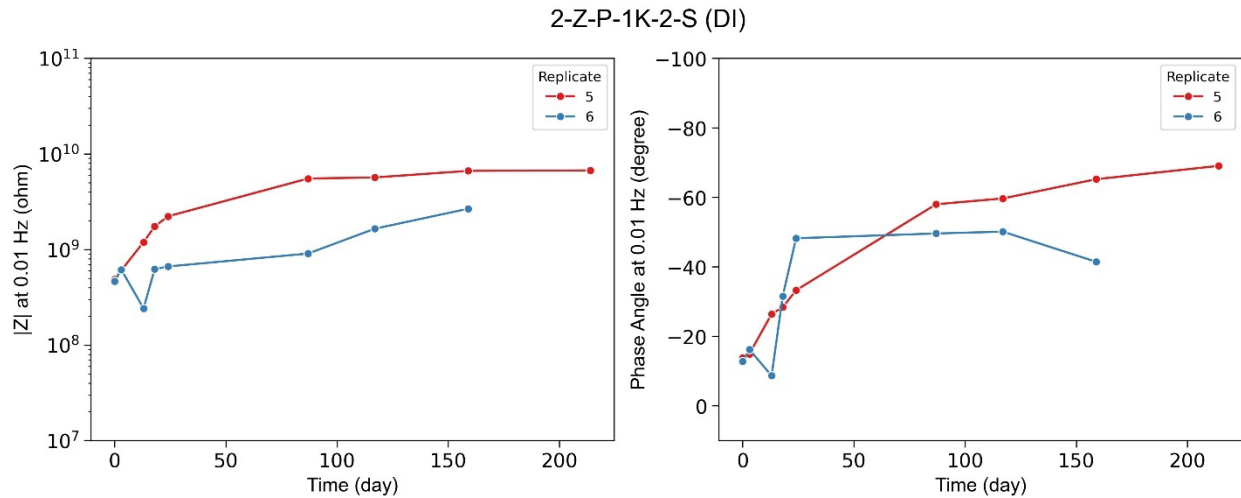


Figure 23: Impedance and phase angle over time (DI panels).

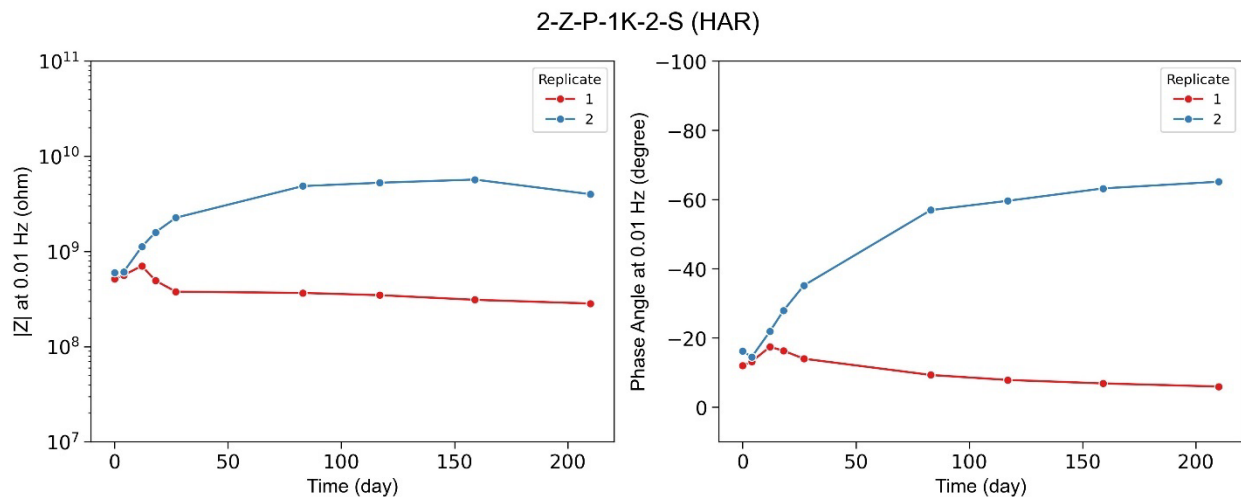


Figure 24: Impedance and phase angle over time (HAR panels).

3.2.3.3 Results and Discussion

Rust creep testing results for the PRO and QUV panels were fair, with an overall rating of good. Rust creep results indicate good long term corrosion resistance performance, but inferior to the excellent corrosion performance rating of vinyl system 5EZ. Rust creep was significantly higher for the PRO (3X increase) and BOR (1.5X increase) panels compared to vinyl system 5EZ.

The overall mechanical rating is fair to good. This coating system had good impact performance, fair to good adhesion properties, and fair erosion performance, indicating satisfactory durability performance.

The average impedance value at 0.01 Hz (DI and HAR) was 1.9×10^9 ohms with less than one order of magnitude degradation at 0.01 Hz earning a rating of excellent. The average phase angle (DI and HAR) was -30 degrees earning a rating of mixed. EIS testing results indicate unsatisfactory long term corrosion performance. EIS testing on the replicate 2 HAR panel was drastically worse than the replicate 1 HAR panel. Impedance was one degree of magnitude lower, and phase angle numbers were resistive compared to the mixed and in some cases capacitive numbers for replicate 1. Researchers believe this to be a result of poor coating application of the replicate 2 panel. Further testing would need to be conducted to verify this theory.

3.2.4 Coating System 2-Z-P-2K-2-S

3.2.4.1 Corrosion Resistance and Mechanical Testing

Table 8 summarizes the corrosion resistance performance and Table 9 summarizes the mechanical properties results. The overall corrosion resistance performance rating is good. The overall mechanical rating is fair to good.

Table 8: Summary of Corrosion Resistance Performance.







Exposure Type	Specimen Images and Visual Inspection Observations	Rust Creep Result (in)	Score
DI Immersion	 <p>No visual defects</p>	0.03	Good
HAR Immersion	 <p>No visual defects</p>	0.02	Good

Table 8: Summary of Corrosion Resistance Performance.

PRO Cyclic	 <p>No visual defects</p>	0.27	Fair
BOR Cyclic	 <p>No visual defects</p>	0.19	Fair
QUV Cyclic	 <p>No visual defects</p>	0.01	Fair
FOG Cyclic	 <p>No visual defects (salt material seen on panel)</p>	0.08	Excellent
Test	Result	Score	
EIS (avg DI & HAR)	1.6 x10 ⁹ ohms @ 0.01 Hz	Excellent	

Note: The images for DI immersion, HAR immersion, PRO cyclic, and BOR cyclic represent panels prepared pre-exposure and for EIS and rust creep testing from left to right. The images for QUV and FOG represent a panel prepared pre-exposure, a panel after exposure without a scribe, and a rust creep testing panel from left to right.

Table 9: Summary of Test Results for Mechanical testing.

Test	Result	Test Metric	Score
Impact	140 in-lbs	Threshold with no cracking or holidays, inch-lbs	Good
Pull-off Adhesion	1292 psi g/adh/coh 15/5/80	Stress, psi Failure mode ¹	Fair
Pull-off Adhesion (wet)	1239 psi g/adh/coh 20/5/75	Stress, psi Failure mode ¹	Good
Erosion	69 ± 13	mg/hour lost	Fair

1 Adhesion (adh), cohesion (coh), intercoat (ic), glue (g). glue failure > 20% is not scored

3.2.4.2 Electrochemical Impedance Spectroscopy

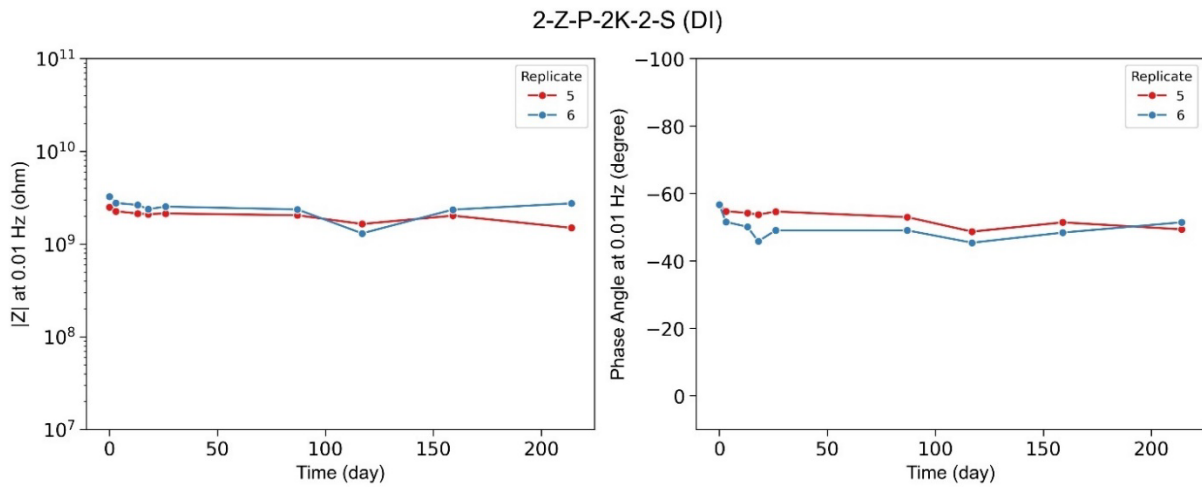


Figure 31: Impedance and phase angle over time (DI panels).

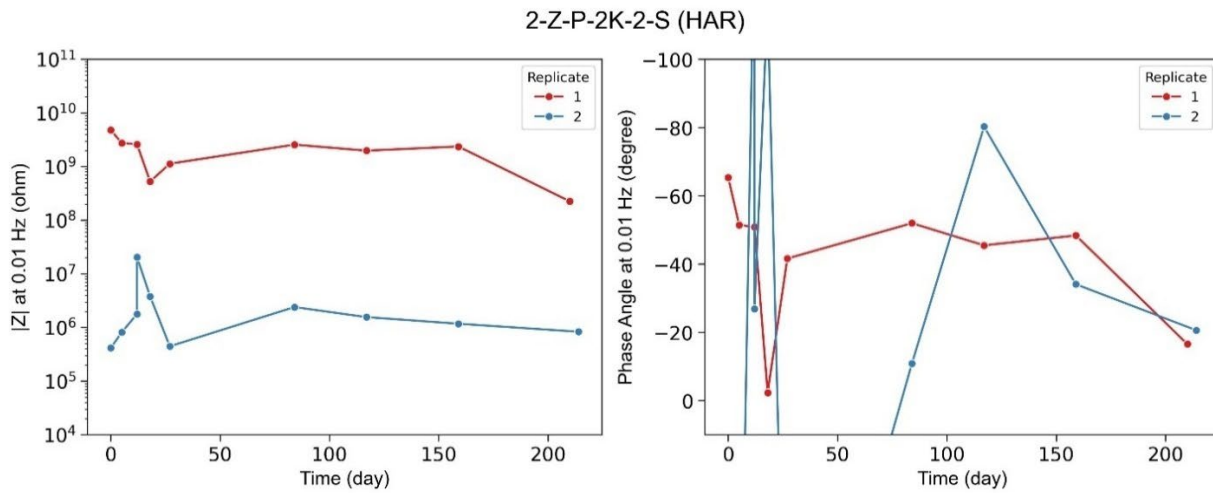


Figure 32: Impedance and phase angle over time (HAR panels).

3.2.4.3 Results and Discussion

Rust creep testing results for the PRO and QUV panels were fair, with an overall rating of good. Rust creep results indicate good long term corrosion resistance performance, but inferior to the excellent corrosion performance rating of vinyl system 5EZ. Rust creep was significantly higher for the PRO (3X increase) panels.

The overall mechanical rating is fair to good. This coating system had good impact performance, fair to good adhesion properties, and fair erosion performance, indicating satisfactory durability performance.

The average impedance value at 0.01 Hz (DI) was 2.3×10^9 ohms with less than two orders of magnitude degradation at 0.01 Hz earning a rating of excellent. The average phase angle (DI) was -51 degrees earning a rating of mixed. However, Impedance magnitude for HAR immersion at 0.01 Hz was highly variable, replicate 1 had an average value of 2.1×10^9 ohms and replicate 2 had an average value of 3.4×10^6 ohms. Phase angle values for HAR immersion were also highly variable. Replicate 1 had an average phase angle value of -41 degrees and replicate 2 had an average value of -4 degrees, with values ranging from +167 degrees to -142 degrees. The low impedance values, and erratic phase angle values, call into question the application quality of the replicate 2 HAR panel. Further research would need to be done to verify this assertion. With that said, the average impedance value at 0.01 Hz (DI and HAR) was 1.6×10^9 ohms earning a rating of excellent, and the average phase angle value was -45 degrees earning a rating of mixed, indicating satisfactory long term corrosion performance.

3.3 Coating Manufacturer 3

3.3.1 Coating System 3-Z-P-2K-2-S

3.3.1.1 Corrosion Resistance and Mechanical Testing

Table 10 summarizes the corrosion resistance performance and Table 11 summarizes the mechanical properties results. The coating system blistered and had poor adhesion in water immersion, therefore was scored poor, despite having minimal undercutting. The overall corrosion resistance performance rating is fair. The overall mechanical rating is fair.

Table 10: Summary of Corrosion Resistance Performance.

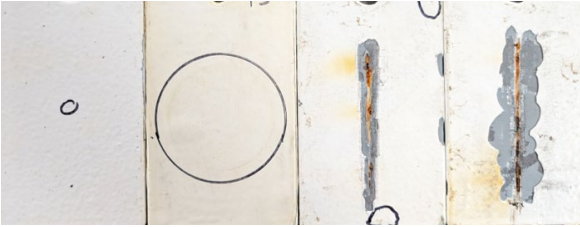

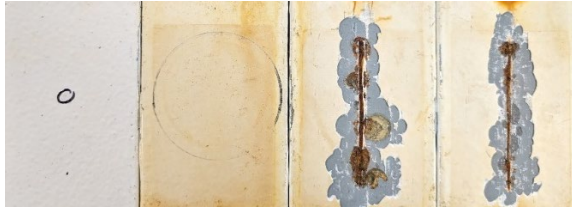



Exposure Type	Specimen Images and Visual Inspection Observations	Rust Creep Result (in)	Score
DI Immersion	 <p>Multiple blisters on coating surface</p>	0.01	Poor (Blisters)
HAR Immersion	 <p>Multiple blisters on coating surface</p>	0.01	Poor (Blisters)
PRO Cyclic	 <p>No visual defects</p>	0.12	Excellent
BOR Cyclic	 <p>No visual defects</p>	0.12	Excellent

Table 10: Summary of Corrosion Resistance Performance.

QUV Cyclic	 No visual defects	0.00	Excellent
FOG Cyclic	 No visual defects (salt material seen on panel)	0.00	Excellent
Test	Result	Score	
EIS (avg DI & HAR)	3.2 x10 ¹⁰ ohms @ 0.01 Hz	Excellent	

Note: The images for DI immersion, HAR immersion, PRO cyclic, and BOR cyclic represent panels prepared pre-exposure and for EIS and rust creep testing from left to right. The images for QUV and FOG represent a panel prepared pre-exposure, a panel after exposure without a scribe, and a rust creep testing panel from left to right.

Table 11: Summary of Test Results for Mechanical testing.

Test	Result	Test Metric	Score
Impact	87 in-lbs	Threshold with no cracking or holidays, inch-lbs	Fair
Pull-off Adhesion	1268 psi g/adh/coh 45/42/13	Stress, psi Failure mode ¹	Fair
Pull-off Adhesion (wet)	265 psi g/adh/coh 43/57/100	Stress, psi Failure mode ¹	Poor
Erosion	40 ± 6	mg/hour lost	Good

¹ Adhesion (adh), cohesion (coh), intercoat (ic), glue (g). glue failure > 20% is not scored

3.3.1.2 Electrochemical Impedance Spectroscopy

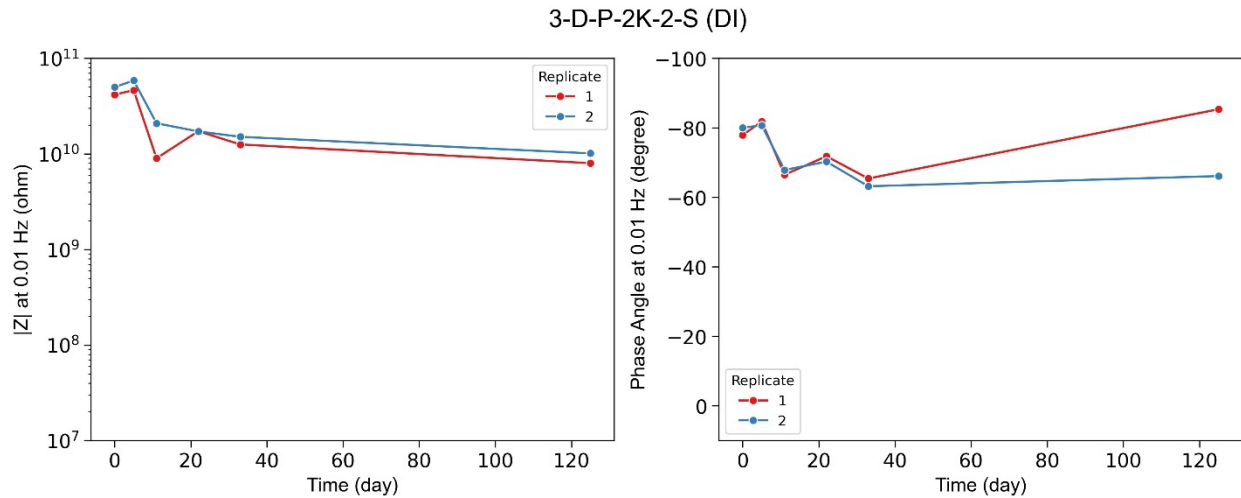


Figure 39: Impedance and phase angle over time (DI panels).

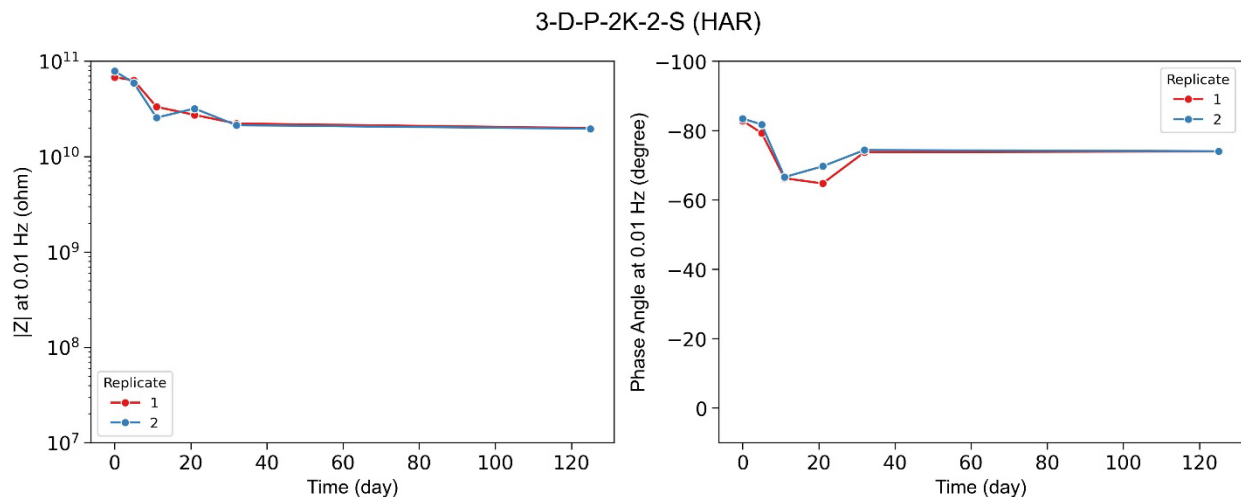


Figure 40: Impedance and phase angle over time (HAR panels).

3.3.1.3 Results and Discussion

Rust creep testing results for the DI and HAR panels were poor. Rust creep was measured at 0.01 inches for both the DI and HAR (immersion) panels, which would have resulted in a rating of good, however, blistering was present on the coating surface which resulted in a poor rating. Blistering was likely a result of an incompatible zinc rich primer formulation. The rust creep rating for PRO, BOR, QUV, and FOG panels was excellent. The overall corrosion resistance rating was fair. Rust creep results indicate below average long term corrosion resistance performance, however, if the manufacturer were to address the zinc rich primer issue, this coating system would likely display improved corrosion resistance, that is potentially on par with vinyl system 5EZ, or better, and have excellent long term corrosion resistance performance.

The overall mechanical rating is fair. This coating system had particularly poor adhesion performance, fair impact properties, and good erosion performance, indicating unsatisfactory durability properties. The poor adhesion performance is likely a result of an incompatible zinc rich primer formulation. Formulating a compatible zinc rich primer would likely result in better adhesion numbers, and better overall durability performance.

The average impedance value at 0.01 Hz (DI and HAR) was 3.2×10^{10} ohms with less than one order of magnitude degradation at 0.01 Hz earning a rating of excellent. The average phase angle (DI and HAR) was -74 degrees earning a rating of capacitive. EIS testing results indicate excellent long term corrosion performance.

3.3.2 Coating System 3-D-P-2K-2-S

3.3.2.1 Corrosion Resistance and Mechanical Testing

Table 12 summarizes the corrosion resistance performance and Table 13 summarizes the mechanical properties results. The overall corrosion resistance performance rating is fair to excellent. The overall mechanical rating is fair to excellent.

Table 12: Summary of Corrosion Resistance Performance.





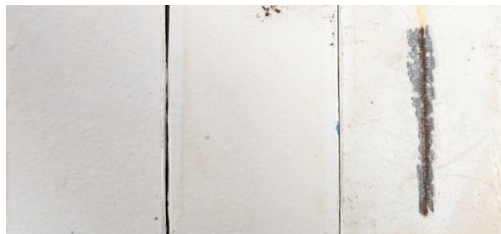
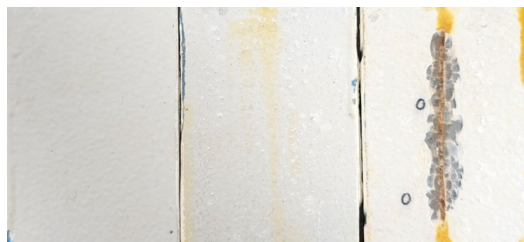
Exposure Type	Specimen Images and Visual Inspection Observations	Rust Creep Result (in)	Score
DI Immersion	 <p data-bbox="646 1371 854 1398">No visual defects</p>	0.02	Good
HAR Immersion	 <p data-bbox="646 1656 854 1684">No visual defects</p>	0.01	Good

Table 12: Summary of Corrosion Resistance Performance.

PRO Cyclic	 <p>No visual defects</p>	0.26	Fair
BOR Cyclic	 <p>No visual defects</p>	0.18	Good
QUV Cyclic	 <p>No visual defects</p>	0.00	Excellent
FOG Cyclic	 <p>Small imperfection on surface (no blistering)</p>	0.01	Excellent
Test	Result	Score	
EIS (avg DI & HAR)	4.4×10^{10} ohms @ 0.01 Hz	Excellent	

Note: The images for DI immersion, HAR immersion, PRO cyclic, and BOR cyclic represent panels prepared pre-exposure and for EIS and rust creep testing from left to right. The images for QUV and FOG represent a panel prepared pre-exposure, a panel after exposure without a scribe, and a rust creep testing panel from left to right.

Table 13: Summary of Test Results for Mechanical testing.

Test	Result	Test Metric	Score
Impact	70 in-lbs	Threshold with no cracking or holidays, inch-lbs	Fair
Pull-off Adhesion	2825 psi g/adh/coh 45/43/12	Stress, psi Failure mode ¹	Excellent
Pull-off Adhesion (wet)	1611 psi g/adh/coh 71/25/4	Stress, psi Failure mode ¹	Good
Erosion	38 ± 7	mg/hour lost	Good

¹ Adhesion (adh), cohesion (coh), intercoat (ic), glue (g). glue failure > 20% is not scored

3.3.2.2 Electrochemical Impedance Spectroscopy

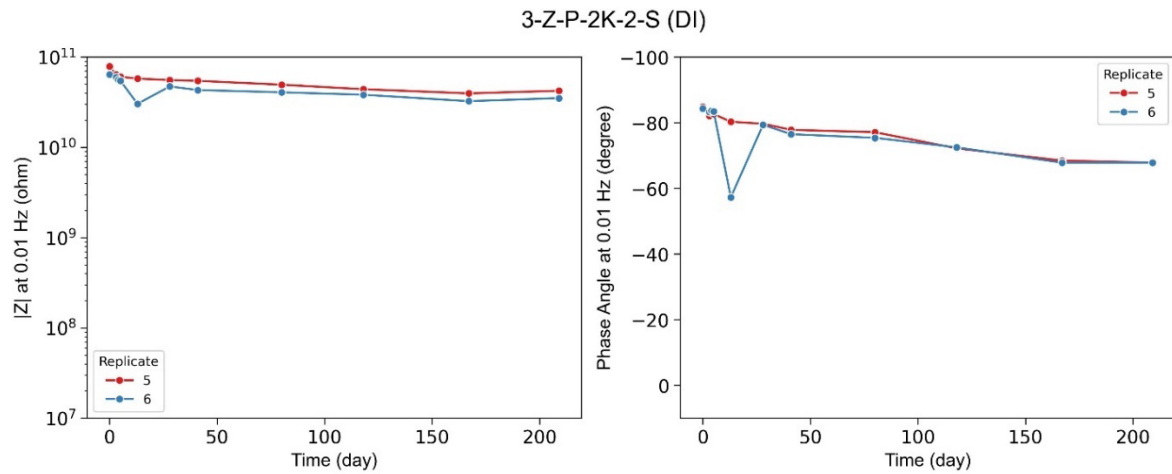


Figure 47: Impedance and phase angle over time (DI panels).

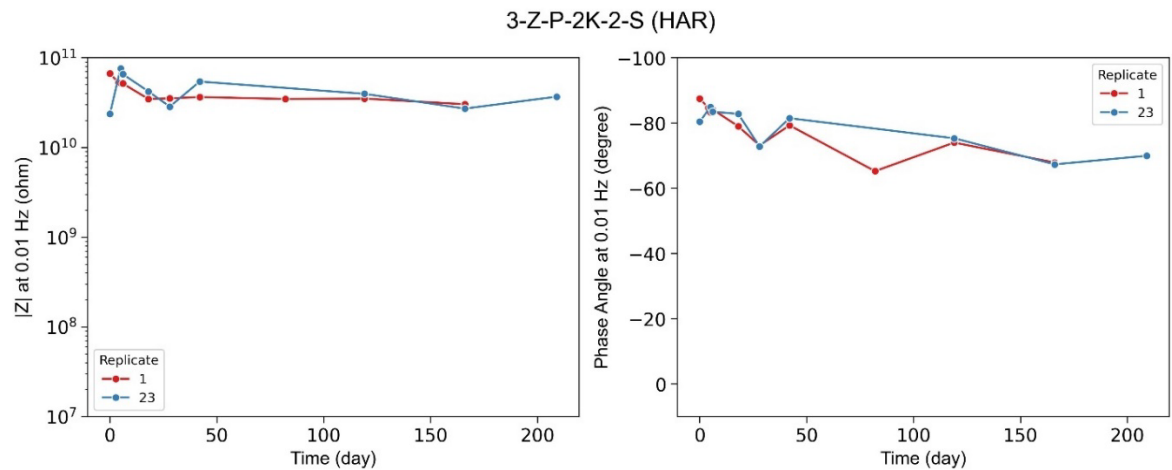


Figure 48: Impedance and phase angle over time (HAR panels).

3.3.2.3 Results and Discussion

Rust creep testing results for the PRO panels were fair. The rust creep results for the DI, HAR, BOR, QUV, and FOG panels were good to excellent. The overall corrosion resistance performance was fair to excellent which exceeded that of vinyl system 4, indicating satisfactory long term corrosion resistance performance.

The overall mechanical rating is fair to excellent. Impact results were fair, however, this coating system outperformed vinyl system 4 significantly. This coating system had particularly excellent adhesion performance, and erosion performance on par with vinyl system 4 indicating satisfactory long term durability performance.

The average impedance value at 0.01 Hz (DI and HAR) was 4.4×10^{10} ohms with less than one order of magnitude degradation at 0.01 Hz earning a rating of excellent. The average phase angle (DI and HAR) was -72 degrees earning a rating of capacitive. EIS testing results indicate excellent long term corrosion performance.

3.4 Comparison to Benchmark Vinyl Coating Systems

The goal of this research was to find an environmentally friendly replacement to vinyl by improving the impact and erosion properties of epoxy polysiloxane coating systems. For that reason, each coating system was compared to a U.S. Army Corps of Engineers (USACE) vinyl coating systems.

The two vinyl systems used for comparison are System No. 4, which is 5 coats of formula V-766E, and System No. 5-E-Z, which consists of 1 coat of zinc rich primer VZ-108d and 4 coats V-766E. These two coating systems are the benchmark in performance for water immersion service and have been successfully used at Reclamation and USACE facilities. The test data seen in the table below was gathered as part of the Polysiloxane and Vinyl Comparison and Field Trials S&T report found in the references section.

Coating systems that utilize zinc rich primers provide an increase in overall corrosion, adhesion, and durability (impact) performance compared to their non-zinc rich primer counterparts. For this reason, vinyl system 5EZ (zinc rich primer) is utilized in harsher impacted immersion environments. Vinyl system 4 still provides excellent corrosion resistance and good erosion properties and therefore can be utilized in immersion service where impacts are not expected, or harsh atmospheric environments.

Polysiloxane coating systems without a zinc rich primer (coating report code with D or E) will be compared to vinyl system 4, and polysiloxane coating systems with a zinc rich primer (coating report code Z) will be compared to vinyl system 5EZ.

Table 14: Comparison to USACE vinyl System Nos. 4 and 5-E-Z for Corrosion Resistance Performance

	HAR Immersion	DI Immersion	PRO	BOR	QUV	FOG	EIS
	Undercutting (in)						(Poor – Excellent)
USACE System No. 4	0.11	0.11	0.43	0.35	0.00	No Data	Excellent
2-E-P-1K-2-S	0.02	0.04	0.43	0.46	0.00	0.17	Excellent
2-E-P-2K-2-S	0.08	0.05	0.55	0.39	0.07	0.21	Good
3-D-P-2K-2-S	0.02	0.01	0.26	0.18	0.00	0.01	Excellent

USACE System No. 5-E-Z	0.00	0.00	0.09	0.15	0.00	No Data	Excellent
2-Z-P-1K-2-S	0.01	0.01	0.27	0.23	0.03	0.08	Excellent
2-Z-P-2K-2-S	0.03	0.02	0.27	0.19	0.01	0.08	Excellent
3-Z-P-2K-2-S	0.01 (blisters)	0.01 (blisters)	0.12	0.12	0.00	0.00	Excellent

Table 15: Comparison to USACE vinyl System Nos. 4 and 5-E-Z for Mechanical testing.

	Slurry Erosion	Impact	Pull-off Adhesion	Pull-off Adhesion (Wet)
	Stabilized weight loss rate (mg/hr)	(inch-lbs)	Stress (psi)	Stress (psi)
USACE System No. 4	39 +/- 3	20	1412	769
2-E-P-1K-2-S	64 +/- 10	48	1854	1325
2-E-P-2K-2-S	63 +/- 10	36	1322	1291
3-D-P-2K-2-S	38 +/- 7	70	2825	1611

Table 15: Comparison to USACE vinyl System Nos. 4 and 5-E-Z for Mechanical testing.

USACE System No. 5-E-Z	41+/- 4	100	1012	Glue
2-Z-P-1K-2-S	56+/- 8	110	1473	1385
2-Z-P-2K-2-S	69+/- 13	140	1292	1239
3-Z-P-2K-2-S	40+/- 6	87	1268	265

3.4.1 Coating Manufacturer 2

- 2-E-P-1K-2-S had an overall corrosion resistance performance rating of good. Undercutting results for PRO and BOR panels were fair. EIS testing resulted in an impedance ranking of excellent, with a phase angle rating of mixed. The overall mechanical and erosion rating was fair. This coating system had poor impact performance. Due to the decreased erosion, impedance, and phase angle results compared to vinyl system 4, this coating system would not be considered a suitable replacement for vinyl system 4.
- 2-E-P-2K-2-S had an overall corrosion resistance performance rating of fair. Undercutting results for PRO panels were particularly poor. EIS testing resulted in an impedance ranking of good, with a phase angle rating of resistive. The overall mechanical rating is fair, with decreased erosion resistance compared to vinyl system 4. Due to the poor corrosion protection results (PRO), fair erosion performance, lower impedance values, and resistive phase angle rating this coating system would not be considered a suitable replacement for vinyl system 4.
- 2-Z-P-1K-2-S had an overall corrosion resistance performance rating of good. Undercutting results for PRO and QUV panels were fair. EIS testing resulted in an impedance ranking of excellent, with a phase angle rating of mixed. The overall mechanical rating is fair to good. This coating system's corrosion resistance falls below vinyl system 5EZ. Undercutting is significantly higher for PRO (3X increase) and BOR (1.5X increase). This coating system also had an average phase angle of -30 degrees with mixed resistive and capacitive behavior, more resistive than vinyl system 5EZ, indicating poor long term corrosion protection. Therefore, this coating system is not considered a suitable replacement for vinyl system 5EZ.
- 2-Z-P-2K-2-S had an overall corrosion resistance performance rating of good. Undercutting results for PRO and QUV panels were fair. EIS testing resulted in an impedance ranking of excellent, with a phase angle rating of mixed. The overall mechanical rating is fair to good. Erosion testing resulted in an increase of approximately 60% compared to vinyl system 5EZ. This coating systems corrosion resistance, and erosion performance, falls below vinyl system 5EZ. Not only is undercutting significantly higher for PRO (3X increase) and BOR (0.25X increase), but this coating system had an average phase angle of 45 degrees with mixed resistive and capacitive behavior, more

resistive than vinyl system 5EZ, indicating poor long term corrosion protection. Therefore, this coating system is not considered a suitable replacement for vinyl system 5EZ.

3.4.2 Coating Manufacturer 3

- 3-Z-P-2K-2-S had an overall corrosion resistance performance rating of fair. This coating system had the best undercutting performance of any of the formulated systems and was on par with vinyl system 5EZ. However, blisters on the DI and HAR panels resulted in an automatic poor rating. EIS testing resulted in an impedance ranking of excellent, with a phase angle rating of capacitive. The overall mechanical rating is fair. This coating system had particularly poor wet adhesion performance. Although system 3-Z-P-2K-2-S had promising erosion, impact, and EIS results, it also had poor adhesion and blistered during testing. Researchers recommend that the manufacturer identifies a more compatible zinc rich primer, which could possibly mitigate the poor adhesion and blistering results, and retest the system as a potentially viable replacement for vinyl system 5-E-Z. However, the current formulation is not a suitable a replacement for vinyl system 5EZ for the above reasons.
- 3-D-P-2K-2-S had an overall corrosion resistance performance rating of good. Undercutting results for PRO panels were fair. EIS testing resulted in an impedance ranking of excellent, with a phase angle rating of capacitive. The overall mechanical rating is good. Overall corrosion resistance outperformed vinyl system 4. Erosion resistance was on par with vinyl system 4, and impact resistance was significantly better. Adhesion results were very strong. Taking into consideration all the test data, 3-D-P-2K-2-S is considered a viable replacement for vinyl system 4.

4. Conclusions & Recommendations

The Bureau of Reclamation operates in challenging service environments that demand highly durable coating systems. These coating systems must withstand impacts from debris and erosion caused by sediment in flowing water, all while providing effective corrosion protection. Previous research has indicated that polysiloxanes have the potential to replace the conventional vinyl coating systems commonly used in severe environments such as high-impact water immersion. Improvements to mechanical properties such as erosion and impact resistance are required prior to full approval as a vinyl replacement. In this study, comprehensive laboratory assessments were conducted on newly formulated polysiloxane coating systems. Manufacturers were asked to formulate new polysiloxane coating systems which retain their corrosion resistance properties and improve erosion and impact resistance. The following conclusions were made.

- No coating system from manufacturer one is a viable replacement for either vinyl system 4 or vinyl system 5EZ.
- No coating system from manufacturer two is a viable replacement for either vinyl system 4 or vinyl system 5EZ.
- One coating system from manufacturer three is considered a viable replacement system for vinyl system 4. No coating system from manufacturer three is considered a viable replacement for vinyl system 5EZ.

Laboratory testing results, comparing the newly formulated polysiloxanes and historical vinyl systems, provided insights into the durability and corrosion protection properties of the polysiloxane formulations. Manufacturers can use this information to improve upon future durable polysiloxane formulations.

Researchers recommend further investigation and field trials on coating system 3-D-P-2K-2S, as a potential viable replacement for vinyl system 4. Although system 3-Z-P-2K-2-S had promising erosion, impact, and EIS results, it also had poor adhesion and blistered during testing. Researchers recommend that the manufacturer identifies a more compatible zinc rich primer, which could possibly mitigate the poor adhesion and blistering results, and retest the system as a potentially viable replacement for vinyl system 5-E-Z.

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Appendix A – Coatings Rating Criteria

Immersion Testing (Dilute Harrison, Deionized):

Excellent: No visual defects

Good: No blistering, minor rust creep up to $\frac{1}{8}$ "

Fair: No blistering, moderate rust creep up to $\frac{1}{4}$ "

Poor: Blistering, delamination or rust creep over $\frac{1}{4}$ "

Cyclic Weathering Testing (BOR, Prohesion, FOG):

Excellent: No blistering, minor rust creep up to $\frac{1}{8}$ "

Good: No blistering, minor-moderate rust creep up to $\frac{1}{4}$ "

Fair: No blistering, moderate rust creep up to $\frac{1}{2}$ "

Poor: Blistering, delamination or rust creep $> \frac{1}{2}$ "

UV and Condensation Cabinet Cyclic Weathering Testing (QUV)

Excellent: No visual defects

Good: No blistering, no rust creep, minor color change

Fair: No blistering, moderate color/gloss change, chalking, or undercut up to $\frac{1}{8}$ "

Poor: Any of the following: blistering, delamination, rust creep $> \frac{1}{8}$ "

EIS (immersion):

Excellent: After 5000 hrs - Minimal degradation < 1 order of magnitude @ 0.01 Hz and $> 10^9$ ohms

Good: Minimal degradation after 5000 hrs ≤ 2 order of magnitude @ 0.01 Hz and $\geq 10^8$ ohms

Fair: Moderate degradation after 5000 hrs ≤ 3 orders of magnitude @ 0.01 Hz and $\geq 10^7$ ohms

Poor: Signification degradation after 5000 hrs > 3 orders of magnitude @ 0.01 Hz

Adhesion (initial, dry):

Excellent: $\geq 2,500$ psi

Good: $\geq 1,500$ psi

Fair: $\geq 1,000$ psi

Poor: $< 1,000$ psi

Wet Adhesion:

Excellent: $\geq 2,000$ psi

Good: $\geq 1,000$ psi

Fair: ≥ 500 psi

Poor: < 500 psi

Erosion:

Excellent: < 30 mg/hr average loss

Good: < 50 mg/hr average loss

Fair: < 100 mg/hr average loss

Poor: > 100 mg/hr average loss

Impact:

Excellent: ≥ 160 in-lbs

Good: ≥ 100 in-lbs

Fair: ≥ 50 in-lbs

Poor: < 50 in-lbs