

# Re-Evaluating Electrochemical Impedance Spectroscopy (EIS) for the Field Inspector's Toolbox: A First Approach

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## Abstract

Electrochemical Impedance Spectroscopy (EIS) has maintained a presence in protective coatings laboratories for decades. EIS is valuable in that it captures resistive and capacitive changes to a coating film as it is exposed to the environmental conditions that produce these changes, i.e. degradations. It also assists in ranking coating systems for their anticipated corrosion protection performance, complementing qualitative, visual observations. However, the EIS equipment has lacked the robustness to be a reliable field tool, and a Faraday cage is needed to minimize electromagnetic interferences during data collection.

An important step to reducing EIS measurements to field practice is to demonstrate a usefulness of the application. Facility and project owners of all infrastructure types commonly request the estimation of a coating's remaining service life. EIS, or some derivation, has the potential to answer these questions.

The Bureau of Reclamation's Materials Engineering and Research Laboratory has collected EIS data spanning more than five years. Data is organized according to generic polymer composition (i.e. epoxy, vinyl, etc.) and like materials are compared to identify basic trends. This is used as a first step to identify unique EIS signatures for each material's degradation process. The established lifecycle data is a starting point for inspectors to interpret field EIS data and quantitatively describe the coating condition to owners. The goal is to provide an estimated remaining service life based on this information. The influence of environmental factors, other variables, accuracy, and the logistics of applying this theory for coating life assessment during maintenance inspections are discussed.

# 1. Introduction

The Bureau of Reclamation (Reclamation) is part of the U.S. Department of Interior. Reclamation's infrastructure includes dams, power plants, pumping plants, canals, pipelines, storage tanks, fish hatcheries, desalination plants, and water treatment plants. The agency's mission is to provide water and hydropower to the 17 western United States. Reclamation produces 17% of the nation's hydropower, supplies irrigation water for 25% of fruits and 60% of vegetables produced in the U.S., and serves 31 million people for municipal, residential, and industrial water use [1].

This paper provides an analysis of ongoing electrochemical data collection. The original incorporation of EIS experimentation at Reclamation occurred in 2006. At that time, the researchers selected a series of commercial products specified by Reclamation for evaluation. In many cases, the EIS data spans 5 or 6 years, included in this paper are:

- Solvent-borne vinyl, VR-6—restricted from use
- Solvent-borne epoxy
- 100% solids epoxy
- 100% solids polyurethane

This list of coatings represents the main categories of coatings for infrastructure maintenance service used at Reclamation in order of emergence.

The vinyl coatings are very high solvent content—more than 70%—and, the inception of volatile organic compound (VOC) regulations diminished their use. They performed well in all service conditions, receiving use from the 1960's through the 1990's. Coal tar enamel coatings, AWWA C203-Type II, enjoyed widespread use during the construction of Reclamation projects from the 1930's through the 1970's and are also restricted from use due to environmental and human health impacts. EIS data for coal tar is in its first year of testing at Reclamation and is not included here.

Solvent-borne epoxy coatings are, in part, used as a replacement for the restricted of vinyl and coal tar enamel. Many research and development efforts moved toward 100% solids materials as the VOC regulations continue to tighten. The 100% solids epoxies, which contain no VOC's, are specified in situations with severe VOC restrictions as well as for applications in which shrinkage stresses must be avoided—such as spot repairs.

Additional 100% solids products, such as polyurethanes, appeared more recently as VOC-compliant alternatives. Some of these products also have the advantage of having high film build allowing the applicators to achieve the total film thickness in a single coat.

## 1.1. EIS Background

Researchers began evaluating organic coatings over metallic substrates using EIS several decades ago [2-10]. The method analyzes the frequency-dependent dielectric properties of a coating by applying small voltage perturbations and measuring the current response. This technique captures various processes over a broad range of frequencies. For instance, corrosion reactions at the coating/metal interface, and

the small currents released as a result, generally occur at long time scales (low frequencies). In contrast, the solution resistance between the coating surface and the counter electrode is the only process observed at high frequency measurements. EIS spectra measured at regular intervals throughout coating exposure can provide both the degree of coating degradation as well as the mode(s) of degradation. Therefore, applications of the EIS test method have advanced the understanding of coatings science by using this electrochemical data to describe physical phenomena. The typical degradation of an organic coating over metal proceeds by two key physical phenomena:

- Water and ions penetrate the coating system. As time passes, these water/ion pathways increase in width and decrease in tortuosity. (coating bulk)
- Water and ions participate in corrosion reactions at the coating/metal interface. The arrival of these species at the substrate may be the rate limiting reactants for the corrosion reactions. (coating/metal interface)

The laboratory exposure utilizes dilute electrolyte solutions to accelerate this deterioration, and the data characterizes each material's degradation process. The goal is to prepare Bode Plots for each coating system to serve as a library for comparison with field data.

## **1.2. EIS Field Applications**

EIS field application is not common; however, several field-applicable, portable EIS equipment prototypes exist [11, 12]. Furthermore, Sonco [13] demonstrated an EIS field coating evaluation technique. This paper helps to prepare for the increasing use of this technique on coated infrastructure within the United States.

The EIS equipment itself has largely limited the application of this method for field inspections. For instance, it typically requires a Faraday cage to minimize external noise and improve the accuracy of measured data. Specially designed, portable potentiostats are now available with USB connection to laptop computers as the sole power source and hard carrying cases for safe transportation. The instrument's durability requires further evaluation as does the experimental validation for these EIS field methods.

A second challenge for field EIS lies in establishing an electrochemical cell for measurement. Laboratory samples are typically thin, flat metal coupons. Other geometries, such as the curved surface of coated metal pipelines require creativity to set up the test cell(s). Laboratory tests connect the working electrode directly to the metal substrate to ensure that current flows perpendicularly through the coating film, and a clamp secure a glass cylinder to the surface. There are quick and non-destructive methods to attach two test cells to a coated surface in the absence of a substrate connection. This is known as non-substrate two electrode EIS—although it may not be applicable to all types of coating systems [14]. This data is a result of the instrument measuring the coating cross-section twice. Figure 1 illustrates this electrochemical test cell set-up on the exterior of cylindrical infrastructure such as an above-ground pipeline.

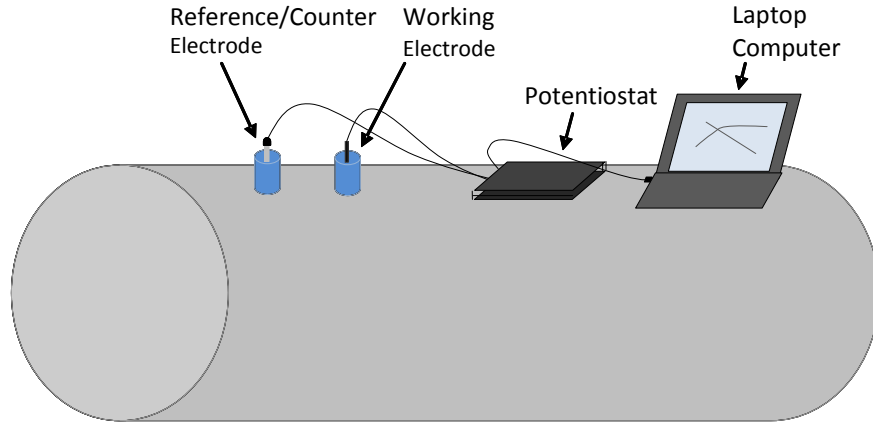


Figure 1. Theoretical field EIS set-up for non-substrate configuration.

## 2. Experimental Setup

Steel panels (3" x 6") received solvent cleaning and then abrasive blasting to SSPC-SP 10 [15] near-white metal prior to coating application. The applied coatings cured at ambient conditions in accordance with each product's application data sheet.

The samples were prepared for EIS measurement by clamping a glass cylinder to the coating surface using an *o*-ring. Dilute Harrison's solution (DHS) was added to the glass cylinder; this solution contains 0.35 wt. %  $(\text{NH}_4)_2\text{SO}_4$  and 0.05 wt. % NaCl. A saturated calomel electrode (SCE) was the reference electrode, platinum mesh served as the counter electrode, and the steel substrate was the working electrode. Figure 2a provides this test cell set-up.

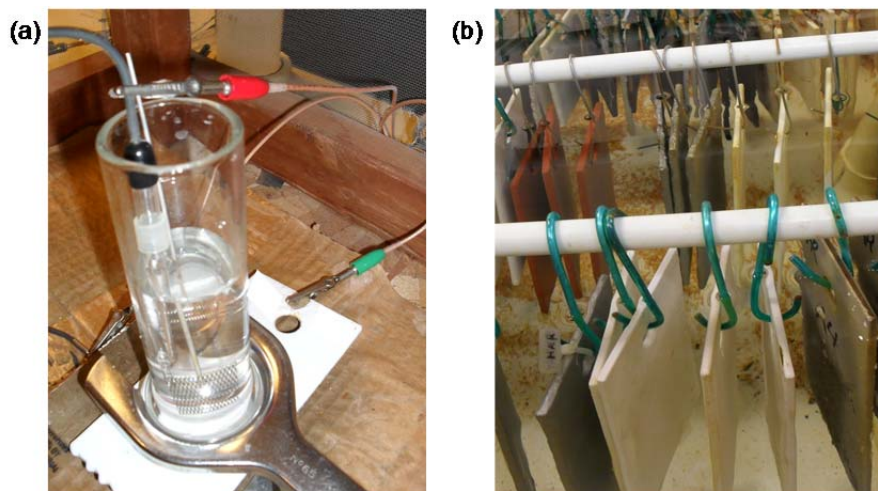


Figure 2. Traditional EIS experiment with coated metal coupons using (a) three electrode electrochemical cell and (b) exposure to immersion between measurements.

A Gamry Instruments FAS2 Femtostat was used to perform potentiostatic EIS experiments with EIS300 software; the test method is ASTM G106 [16]. The EIS test applied small, 10 mV perturbations across the coating film. The open circuit potential was the mean applied voltage. The test recorded the current required to satisfy this voltage pattern. The frequency ranged from  $10^5$  to  $10^{-2}$  Hz at ten per decade.

The measured surface area for these experiments was  $20 \text{ cm}^2$ , with no adjustments made for current density. A DHS water tank exposed the coatings to immersion between testing dates (Figure 2b). Significant changes occur to the polymer matrix as water enters the film. The solution ions accelerate the degradation of the coating matrix and encourage corrosion at the substrate. Samples were measured weekly at the beginning of the experiment. Following three months of immersion, testing occurs semi-annually and then annually, as coating changes become minor over the course of a year.

### **2.1. Demonstration of Portable EIS Feasibility**

An Ivium CompactStat performed the outdoor EIS testing. This instrument received power via laptop computer through the USB port. A 1.25" inner diameter (I.D.) polyvinyl chloride (PVC) pipe of three inch height was glued to an outdoor, coated structure using marine epoxy. The first structure is a known coating system exposed to Denver, CO weather for approximately 1.3 years. The second structure is an 18-inch I.D. coated metal pipe with two PVC test cells. The coating is unknown and the period of exposure to Denver, CO weather is unknown.

The experiments conducted outdoor did not use a Faraday cage to shield the test cell from electromagnetic noise. Therefore, the results contain surrounding electromagnetic noise in the data sets.

## **3. Laboratory Results and Discussion**

The organization of the EIS data is as Bode Plots. This representation displays both the impedance magnitude,  $|Z|$ , as well as the phase angle measured at each frequency tested. These properties appear on the left-side and right-side y-axis, respectively. Excellent barrier coatings maintain purely capacitive properties over long exposure periods, and the low frequency impedance is greater than or equal to  $(|Z|_{0.01 \text{ Hz}} \geq) 10^9 \Omega$ . Graphically, this is a diagonal line with a slope of -1 for  $|Z|$  and a horizontal line at  $-90^\circ$  for the phase angle. Coating breakdown is often indicated by a decrease in the low frequency region of these plots (frequencies  $< 10^0 \text{ Hz}$ ) as new resistive elements (corrosion) develop. The following sections describe changes in the electrochemical data for vinyl, solvent-borne epoxy, 100% solids epoxy, and 100% solids polyurethane as the exposure progresses over a period of 5-6 years.

### 3.1. Solvent-Borne Vinyl

The Bode Plot for Reclamation’s previously used vinyl coating appears in Figure 3. Each data series within the figure represents the results of a single test experiment. The series label provides the length of time, in years, for coating weathering by immersion in DHS.

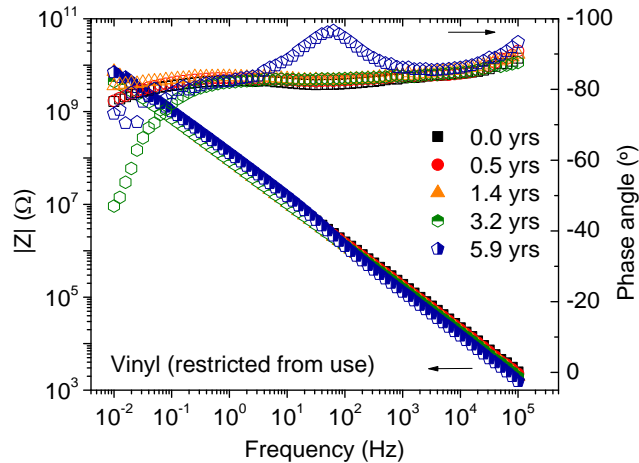


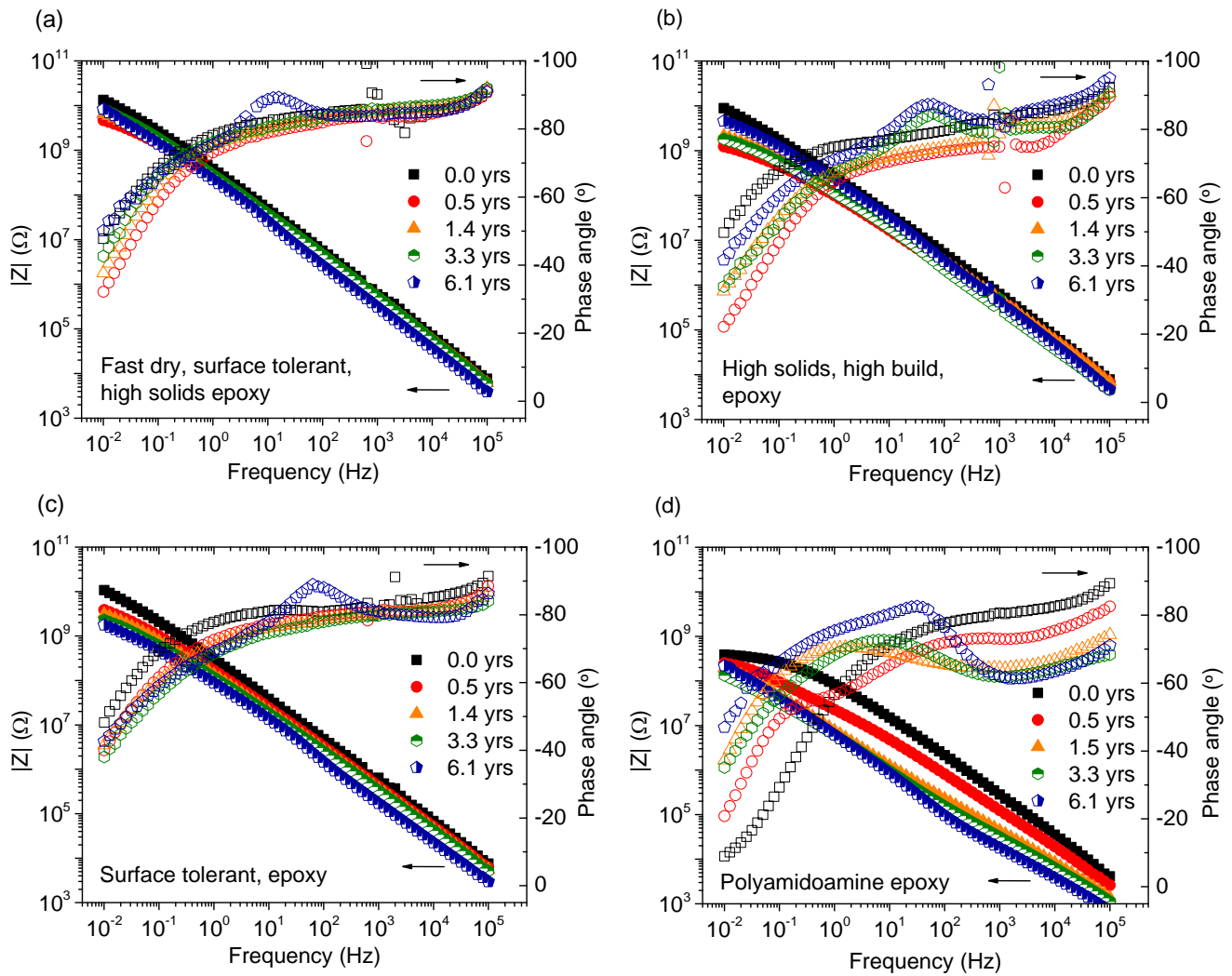
Figure 3. EIS data for Reclamation vinyl coating.

The vinyl polymer coating has outstanding field performance throughout Reclamation infrastructure for immersion and atmospheric exposure. The EIS data in Figure 3 supports the historical field performance of these vinyl coatings that provided 30+ years of service. Following approximately six years of laboratory testing, the data series are relatively unchanged:  $|Z|$  is diagonal with a slope of -1, and the phase angle is near  $-90^\circ$  at all frequencies. A few of the data series show some scatter at low frequencies. This may indicate the introduction of new resistive properties to the coating/metal interface. Further analysis and modeling can be used to determine whether this is attributed to charge transfer at the interface (corrosion), coating degradation, or otherwise.

### 3.2. Solvent-Borne Epoxy

Figure 4 provides Bode Plots for common solvent-borne epoxy coatings. They are in relative order of performance, based on the EIS results, in descending order. Figure 4(a) is similar to the vinyl data with slightly less “clean” appearance and some variation occurring at the low frequencies. Figures 4(b) and 4(c) are also similar in this regard.

The final data series in Figure 4(a) is nearly diagonal for  $|Z|$  and the phase angle has been steadily improving toward  $-90^\circ$  at the low frequencies. This behavior suggests a rebuilding of the barrier properties or corrosion inhibition by the coating, as opposed to a steady degradation. Figure 3(b) displays a similar resurgence in corrosion protection for  $|Z|$  and phase angle. In contrast, Figure 3(c) shows a steady degradation throughout the testing period for both  $|Z|$  and phase angle, which translates to a steady degradation of the coating over time. For all three products,  $|Z|_{0.01 \text{ Hz}}$  is greater than  $10^9 \Omega$  following 6 years in immersion testing. The coatings in Figure 4(a-c) provide good field performance as a corrosion-protective coating. One goal of this project is to develop the method for extrapolating this information to field performance and an expected service life.



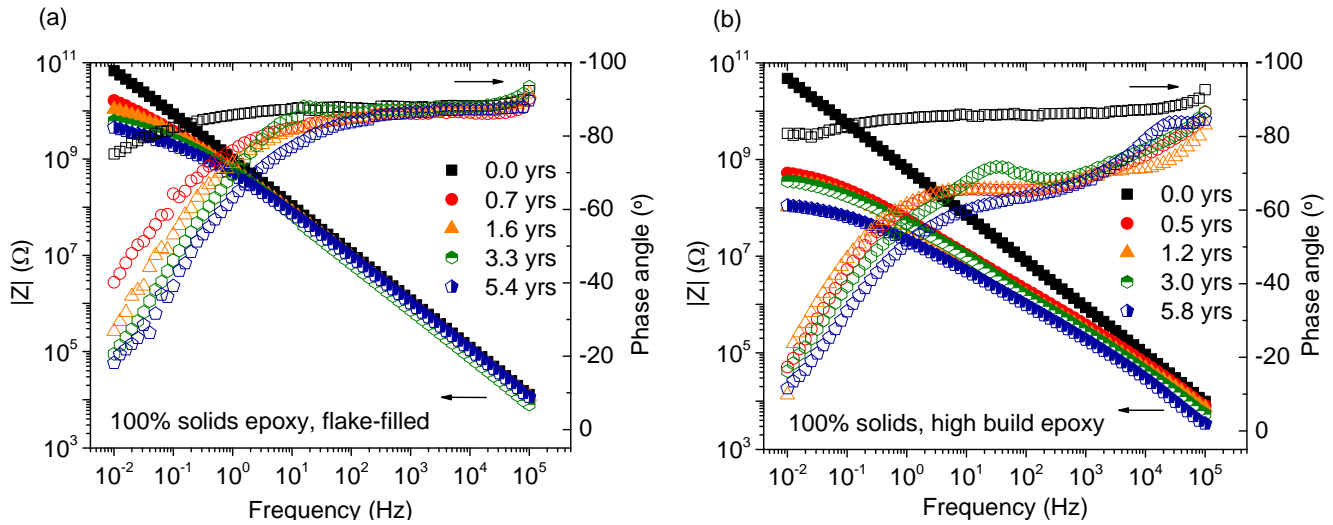
**Figure 4.** EIS data for solvent-borne epoxies: (a) fast dry, surface tolerant, high solids epoxy, (b) high solids, high build epoxy, (c) surface tolerant epoxy, and (d) polyamidoamine epoxy.

The  $|Z|$  in Figure 4(d) is lower than the other graphs in Figure 4, and the low frequency region is nearly diagonal following at the most recent data series. The phase angle is also changing with time; the low frequencies are approaching  $-90^\circ$  while the higher frequencies are decreasing. The low frequency shift is a reduction in the tendency of the material to allow corrosion to proceed. This behavior may be a material property of the polymer system. Alternatively, it may be due to corrosion-inhibitors that undergo a chemical or physical change to form solid precipitates that block coating pores, impeding the progression of water and ions, i.e., corrosion reactions.

### 3.3. 100% Solids Epoxy

Figure 5 provides EIS data for two no-VOC epoxy products evaluated. The first observation is that these coating materials experience greater change during the course of the evaluation than those shown above, especially Figure 5(b). This likely represents significant material changes and possibly degradations. In both cases, the initial data series at 0.0 years is very high, but after some exposure, the material provides reduced corrosion resistance compared to the solvent-borne materials shown above.

Figure 5(a) is a flaked-filled epoxy coating; the results suggest that the flakes succeed in increasing the tortuosity of water and ion pathways as compared to Figure 5(b).



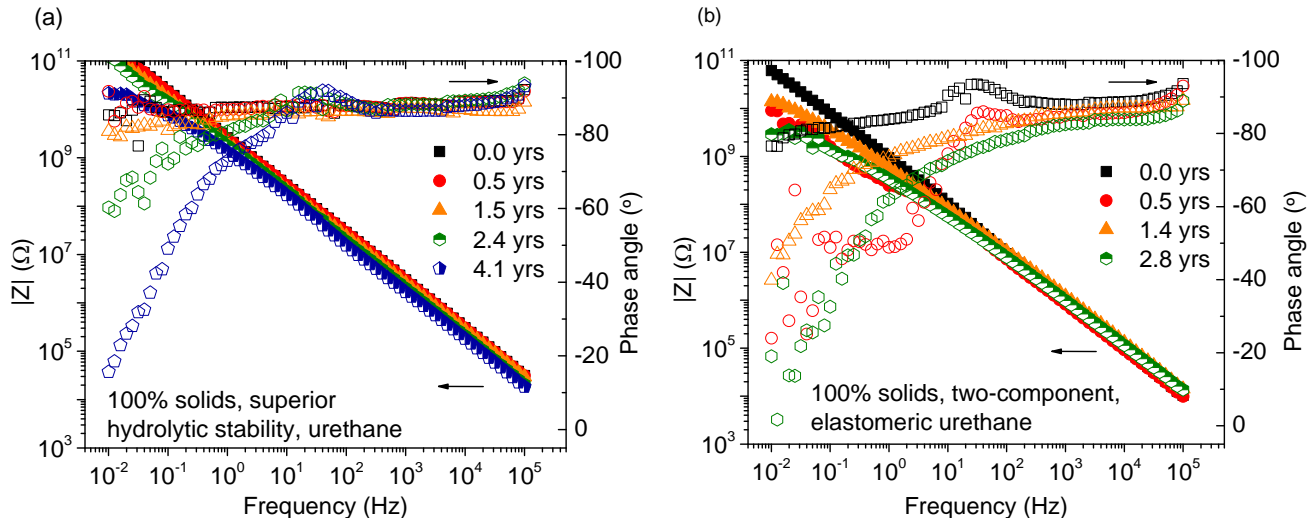
**Figure 5. EIS data for 100% solids epoxies (a) flake-filled epoxy and (b) high build epoxy represented as Bode Plots.**

Furthermore, significant degradation occurs in the initial year of exposure to DHS immersion. A potential cause for this decreased corrosion resistance is that 100% solids materials are typically higher viscosity, and they do not contain low-molecular weight solvents to assist the material in wetting the substrate. A partially wetted substrate presents opportunities for water and ions to initiate corrosion at the substrate/coating interface that may have migrated through the coating film. This may also explain why the degradation is apparent early in the experiment.

### 3.4. 100% Solids Polyurethane

Figure 6 provides 100% solids polyurethane coatings data. These plots are distinctly different from the epoxies shown above because of differences in basic material properties. Polyurethanes are more hydrophobic and generally have higher crosslink density. The  $|Z|$  is nearly diagonal for all measurements collected; however, it is also approximately one order of magnitude higher. The low frequency  $|Z|$  data for Figure 6(a) is greater than  $10^{11} \Omega$ . Figure 6(b) also has very high impedance values. The magnitude of these data is higher than the vinyl coating that has performed so well for Reclamation (Figure 3). However, the phase angle shifts significantly over time at low frequencies. This indicates the material is a very strong barrier to water and ions, but that they do penetrate over time. Typically, the polymer matrix reorganizes to form channels as these species migrate which is a permanent degradation to the material. Additional analysis and modeling of the results within Figure 6 may reveal a strong presence of corrosion reactions (charge transfer) at the coating/metal interface.





**Figure 6. EIS data for 100% solids polyurethanes (a) 100% solids, superior hydrolytic stability, urethane and (b) 100% solids, two-component, elastomeric urethane represented as Bode Plots.**

#### 4. Application to Coated Infrastructure

The Bode Plots shown here are unique in that they present degradation patterns for each commercial coating system. Similarities arise when organizing the results by polymer binder. The results are similar to those reported by others [17]. Solvent-borne epoxies have an initial  $|Z|_{0.01 \text{ Hz}}$  near  $10^{10} \Omega$  and experience a steady degradation with time. This impedance value is approximately one order of magnitude lower than those observed by polyurethane coatings. This is characteristic to the material's physical and chemical properties, including crosslink density and hydrophobicity. The vinyl coatings are also  $10^{10} \Omega$ ; however, there is no degradation, which indicates that water and ions do not easily penetrate this matrix.

Field results for each commercial system, when compared to these figures, indicate the material's stage of degradation based on where it falls between the initial and final laboratory test results. It may be feasible to provide an accurate prediction of the remaining service life at this time, and, as the application of EIS to coatings in service expands, useful theories are likely to follow.

##### 4.1. EIS Feasibility—Outdoor Test Versus Laboratory Data

EIS measurements made on the known coated structure used the standard three electrode EIS (3eEIS) set-up described in Section 2. The only variation was that the experimental frequency range recorded five points per decade, providing half the data points and reducing the time of experiment by half. The environmental conditions at the time of testing provided moderate wind gusts and a temperature of approximately 45 °F. Figure 7 provides the test set-up and results.

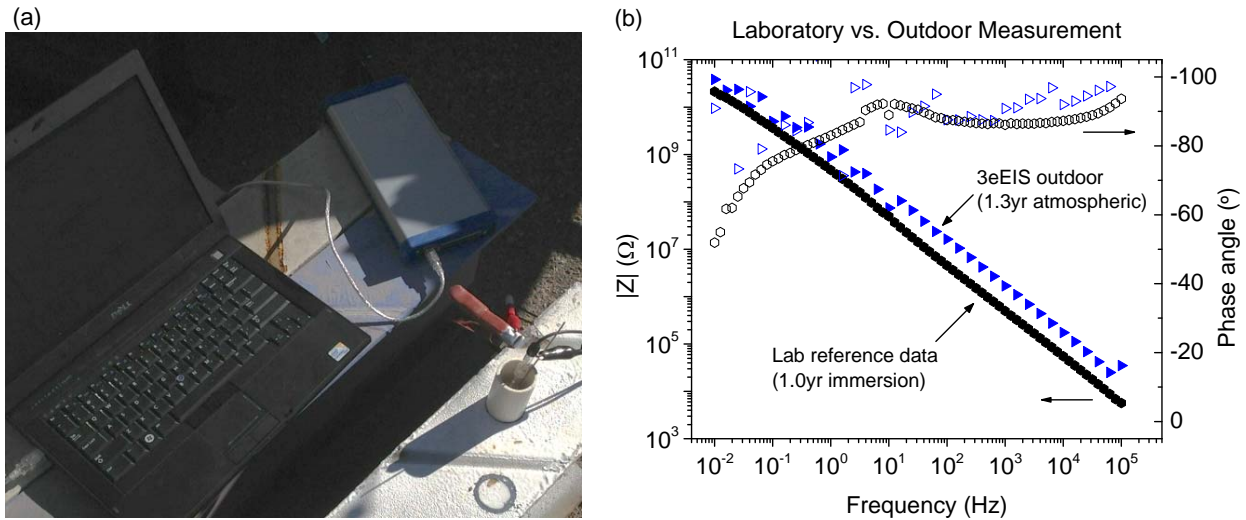


Figure 7. Outdoor 3eEIS test on known coating without Faraday cage: (a) test set-up and (b) EIS results.

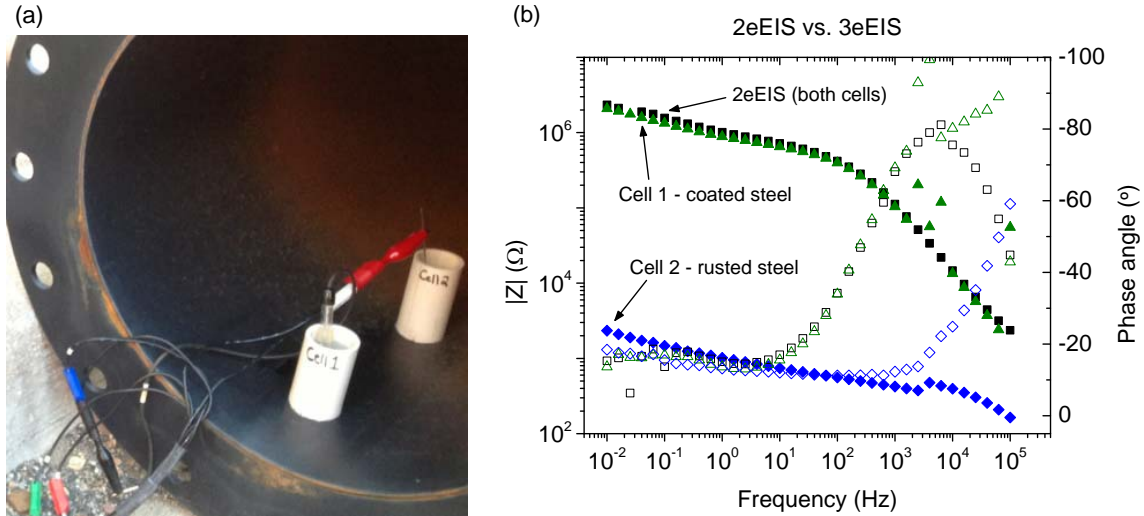
Figure 7 also provides available laboratory data on the same coating system as a reference, exposed to DHS immersion for one year. There is a slight offset between the two curves with the outdoor coating recording higher impedance. This is due to the smaller sample area—the test cell provides  $8 \text{ cm}^2$  for the outdoor coating versus  $20 \text{ cm}^2$  in laboratory. Possible additional variables could include:

- Less weathering/degradation—1.0 years DHS solution has reduced the laboratory coating impedance slightly. Atmospheric exposure has had minimal effect on the outdoor coating.
- Lower temperature—the outdoor coating may be more contracted than the laboratory sample. The temperature difference is approximately  $25 \text{ }^\circ\text{F}$ .
- Unsaturated coating—the outdoor coating received several hours' exposure to the ion solution prior to the measurement whereas the laboratory coating received one year of saturation.
- Higher film thickness—21.2 mils for outdoor versus 17.7 mils for laboratory.

This list suggests possible experimental variables that are likely to play a role in measurement data. However, for the purposes of determining the quality and remaining service life for a coating, the data curves in Figure 7 show insignificant difference. The  $|Z|_{0.01 \text{ Hz}}$  for both data sets is greater than  $10^{10} \Omega$ , indicating very high barrier protection and no or minimal degradation at this time. The phase angle is our second reference point for this conclusion; it is nearly  $-90^\circ$  for most frequencies and shows that the coating is still acting as a capacitor. Again, the outdoor exposure coating appears to have had slightly less weathering by this measure.

#### 4.2. EIS Feasibility—Standard Three-Electrode EIS versus Two-Electrode EIS

The unknown coating on steel pipe was measured by 3eEIS as well as two electrode EIS (2eEIS). Figure 8 provides the 2eEIS set-up and results for the three total measurements. Closed symbols are  $|Z|$  and open symbols are phase angle. As in Section 4.1., there are five points per decade; however, there was no wind, and the temperature was approximately  $60 \text{ }^\circ\text{F}$ .



**Figure 8. Outdoor EIS test for unknown coating on steel pipe: (a) 2eEIS test set-up and (b) 2eEIS versus Cell 1 and Cell 2 3eEIS results.**

Cell 2 has very low impedance values and is resistive (phase angle near 0°) for most frequencies because the steel surface is exposed within this test cell. Cell 1 provides the impedance data for a typical, thin coating. The  $|Z|_{0.01 \text{ Hz}}$  value of  $10^6 \Omega$  suggests that the coating provides a very low level of protection to the substrate. However, this value is adequate for short atmospheric exposure. The 2eEIS results look very similar to the Cell 1 results with slightly higher impedance. The phase angle also differs to show a significant deviation between Cell 1 and Cell 2 at frequencies greater than  $10^1 \text{ Hz}$ . The  $|Z|$  and phase angle for Cell 2 are consistent with that expected for a bare steel substrate.

The results demonstrate that the 2eEIS test is valid test set-up when no connection to the substrate is available and as a replacement for 3eEIS. In theory, the 2eEIS test measures film impedance additively as follows:

$$|Z|_{2eEIS} = |Z|_{\text{Cell 1}} + |Z|_{\text{Cell 2}} \quad \text{Eq. 1.}$$

With the several points deviating from the main curves removed, Eq. 1 above provides an average error of 11% for the data set, which is one tenth of an order of magnitude. This error is insignificant for the desired analyses purposes. These results show the 2eEIS test cell set-up applied outdoors and without the use of a Faraday cage to achieve useful EIS data.

### 4.3. Future Field Work

The next phase of this research is to evaluate field locations with these coating systems applied to metal infrastructure and to use a 2.0" I.D. PVC test cell to be consistent with the  $20 \text{ cm}^2$  laboratory data. By working with Reclamation facility owners and operators, our researchers have a unique opportunity to access and evaluate these coatings in the field. The goal is to develop an effective field EIS method and to begin collecting data. There is benefit in measuring Reclamation's oldest serving systems as well as in tracking newly applied coatings at regular intervals throughout their lifetime. Of greatest interest are coal tar enamel (> 60 years in service) and 100% solids polyurethane coatings (< 5 years in service).

## 5. Conclusions

The present paper focuses on using the EIS Bode Plot to characterize the coating degradation with exposure time. EIS data for vinyl, solvent-borne epoxy, 100% solids epoxy, and 100% solids polyurethane commercial coating systems spans an exposure period of 5-6 years in DHS immersion. The data analysis provided key (but expected) differences between the coating materials. The vinyl coating experienced almost no change within the Bode Plot over a 6-year period. Most solvent-borne epoxy coatings begin with  $|Z|_{0.01 \text{ Hz}}$  near  $10^{10} \Omega$ . There is a slow consistent decrease in this value as well as a small decrease in  $|Z|$  over the entire frequency range. In addition, the phase angle experiences dramatic shifts, most often toward  $0^\circ$  at the low frequencies. The 100% solids epoxy coatings begin with  $|Z|_{0.01 \text{ Hz}}$  near  $10^{11} \Omega$ ; however, this decreases dramatically within the first 6 months of exposure. The 100% solids polyurethane coatings have very high barrier resistance at the beginning of the test ( $|Z|_{0.01 \text{ Hz}} = 10^{11} \Omega$ ), and this decreases at a rate similar to some epoxies. These laboratory results provide standard degradation patterns for each commercial coating system.

A simple outdoor experiment demonstrated the feasibility of EIS measurement for field applications. Most importantly, this useful data did not require a Faraday cage. The experiments showed that: 1) laboratory reference data provides a good comparison tool to measurements of the same coating in the field and, 2) the 2eEIS results are within an acceptable accuracy of the standard 3eEIS.

The goal is to obtain field EIS results for the same coating systems and to compare the corresponding Bode Plots. This provides a quick, qualitative application of the EIS results, and coatings in immersion service provide a good application for the method. The EIS data could serve facility owners as a quantitative measure of the condition of their coated infrastructure, and its incorporation into the standard visual inspection and condition monitoring stands to improve coatings maintenance planning.

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