The Efficacy of using Migrating Corrosion Inhibitors (MCI 2020 & MCI 2020M) for Reinforced Concrete

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Significant efforts have been made to develop a corrosion inhibition process that increases the life of existing structures while minimizing corrosion damages in new structures. In this investigation, a series of corrosion tests (potentiostatic and potentiodynamic) were conducted on bare rebar prior to casting samples for evaluating Cortec's MCI 2020 and MCI 2020M, a mixture of amine carboxylates and amino alcohols. Dramatic improvement in the corrosion rate was seen for rebar tested in MCI treated solutions. Varying density concrete with reinforcement at 2.5 cm (1 inch) concrete coverage was subsequently surface coated with the two inhibitors and immersed in 3.5% NaCl solution at ambient temperatures for a period of 500 days. The corrosion behavior of the six (6) concrete specimens was monitored on a biweekly basis using AC electrochemical impedance spectroscopy (EIS). Comparison of the open circuit potentials, Bode and Nyquist plots showed high polarization resistance values (low corrosion rates) for all specimen except an untreated low density concrete sample. XPS analysis demonstrated the presence of inhibitor on the steel rebar surface with nitrogen detection at levels 85 nm below the unetched surface (MCI 2020M sample) and as far down as 75 nm for the MCI 2020 sample. The XPS results showed similar diffusion rates for MCI and the corrosive species. The MCI inhibitors were able to provide a protective film on the rebar surface, whereas the untreated samples were subjected to localized corrosion attack.

Introduction

Extensive experimentation, typically designed to reproduce the most extreme conditions in a system, has been used to improve inhibitor capabilities [1]. Much time and effort has gone into the development of the corrosion inhibition process. Corrosion undermines the physical integrity of structures and can lead to destruction of property and loss of life. Because carbon steel represents the largest single class of alloys used [2], corrosion is a huge concern. Billions of dollars are spent on protective systems for iron and steel. Migrating corrosion inhibitors (MCIs) are one means of protection for reinforced concrete structures. Previous studies have established the benefits of using migrating corrosion inhibitors, the importance of good concrete, and the significance of the constituents used to make the concrete [3-8]. Reinforcing steel embedded in concrete shows a high amount of resistance to corrosion. The cement paste in the concrete creates an alkaline environment that protects the steel from corrosion by forming a ferric oxide film. The corrosion rate of steel in this state is negligible. Factors influencing the ability of the rebar to remain passivated are the water to cement ratio, permeability and electrical resistance of concrete. These factors determine whether corrosive species like carbonation and chloride ions can penetrate through the concrete pores to reach the rebar oxide layer. In highly corrosive environments (coastal beaches and areas where deicing salts are common), the passive layer will deteriorate, leaving the rebar vulnerable to chloride attack, thereby requiring additional protection.

Migrating Corrosion Inhibitor (MCI) technology was developed to protect the embedded steel rebar/concrete structure. Recent MCIs are based on amino carboxylate chemistry and the most effective types of inhibitor interact at the anode and cathode simultaneously [3, 4]. Organic inhibitors utilize compounds that work by forming a monomolecular film between the metal and the water. In the case of film forming amines, one end of the molecule is hydrophilic and the other hydrophobic. These molecules will arrange themselves parallel to one another and perpendicular to the reinforcement forming a barrier [6]. Migrating corrosion inhibitors are able to penetrate into existing concrete to protect steel from chloride attack. The inhibitor migrates through the concrete capillary structure, first by liquid diffusion via the moisture that is normally present in concrete, then by its high vapor pressure and finally by following hairlines and microcracks. The diffusion process requires roughly 120 days to reach the rebar surface and to form a protective layer.

MCIs can be incorporated as an admixture or can be surface impregnated on existing concrete structures. With surface impregnation, diffusion transports the inhibitor into the concrete where it can inhibit the onset of steel corrosion. The effectiveness of these migrating corrosion inhibitors has been demonstrated over five years of continuous testing [3, 4]. It has also been shown that the migrating amine-based corrosion inhibiting admixture can be effective when incorporated in the repair process of concrete structures [3]. This method is currently being used to rehabilitate the exterior Pentagon walls. Furthermore, laboratory testing and analysis have proven that MCI corrosion inhibitors migrate through the concrete pores to protect the rebar against corrosion even in the presence of chlorides [7, 8].

Experimental Procedures

Prior to investigating the performance of two inhibitors, MCI 2020 and MCI 2020M in reinforced concrete, their potentiodynamic behavior was assessed on bare rebar. Studies were conducted in a saturated $Ca(OH)_2$ solution with and without chloride ions using EG&G M352 DC corrosion test software. Comparisons of the polarization behavior were made for the steel rebar in solutions with varying concentrations of inhibitor and the introduction of a corrosive species (2000 ppm NaCl). The effects of the mixed inhibitor in an alkaline environment similar to the concrete medium were observed.

For purposes of this study, the steel rebar/concrete combination is treated as a porous solution and modeled by a Randles electrical circuit. EIS tests performed on a dummy cell containing a circuit with a capacitor and two resistors indicate that this model provides an accurate representation of a corroding specimen. EIS tests using a small amplitude signal of varying frequency give fundamental parameters relating to the electrochemical kinetics of the corroding system. Two important parameters for this study are R_p and R_Ω. The R_p value is a measure of the polarization resistance or the resistance of the surface of the material to corrosion. R_Ω is a measure of the solution resistance to the flow of the corrosion current. By monitoring the R_p value over time, the relative effectiveness of the sample against corrosion can be determined. If the specimen maintains a high R_p value in the presence of chloride, it is considered to be passivated or immune to the effects of corrosion. If the specimen displays a decreasing R_p value over time, it is corroding and the inhibitor is not providing corrosion resistance.

The experiments were conducted using an EG&G Potentiostat/Galvanostat (Model 273A with a 5210 Lock-in amplifier), EG&G Powersine, Power Suite Electrochemical Impedance Software, a Gamry PC4-750 Potentiostat with EIS300 software and Echem Analyst. Bode and Nyquist plots were created from the data obtained using the single sine technique. Potential values were recorded and plotted with respect to time. By comparing the bode plots, changes in the slopes of the curves were monitored as a means of establishing a trend for the R_p value over time. To verify this analysis, the R_p values were also estimated by using a curve fit algorithm on the Nyquist plots (available in the software). In these plots, the R_p and R_Ω combined values are displayed in the low frequency range of the bode plot and the R_{Ω} value can be seen in the high frequency range of the bode plot. The diameter of the Nyquist plot is a measure of the R_p value.

Concrete samples with dimensions 20 cm x 10 cm x 10 cm were prepared using a 20 cm steel rebar (class 60, 1.27 cm diameter) and a 20 cm Inconel 800 metal strip (for the counter electrode). A concrete mixture containing commercial grade-silica, Portland cement, fly ash, and limestone (concrete mixture ratio: 1 cement/2 fine aggregate/4 coarse aggregate) were combined with one-half gallon water per 27.2 Kg (60-lb) bag in a mechanical mixer. The water to cement ratio was varied to achieve the two densities and the coverage layer was maintained at 2.5 cm (1 inch) concrete for all samples. Compressive strengths were roughly 27.6 MPa (4000 psi) for the low density and 41.4 MPa (6000 psi) for the high density concrete cured for 28 days per ASTM C387 [9]. All samples were sandblasted to remove loose particles and provide surface uniformity.

Table 1 - Sample specifications.

Number of samples	Concrete Surface Coating	Density	Water to cement ratio
1	No treatment-control sample	Low = 2.08 g/cm^3	0.65
1	No treatment-control sample	High = 2.40 g/cm^3	0.35
1	MCI 2020	Low = 2.08 g/cm^3	0.65
1	MCI 2020	High = 2.40 g/cm^3	0.35
1	MCI 2020M	Low = 2.08 g/cm^3	0.65
1	MCI 2020M	High = 2.40 g/cm^3	0.35

As outlined in Table 1, there were six (6) concrete samples in total, two were surface impregnated with several coats of MCI 2020 and two were coated with MCI 2020M. The inhibitor was applied to the surface of the concrete with a paint brush while partially immersed in a shallow container of inhibitor. The remaining two samples were left untreated and used as standards for comparison. An additional coat of sealer was used to prevent the inhibitor from sloughing off in solution. Clear silicon was applied to the concrete/metal interface to prevent easy access for ions. The testing environment was a mixture of 3.5% NaCl and water with roughly 175 mm (7 inches) of each sample continuously immersed for 500 days (Figure 1). A $Cu/CuS0_4$ electrode was used as the reference and each sample was tested once every two weeks.

Figure 1 – The concrete samples partially immersed in a 3.5% NaCl solution.

Results & Discussion

Electrochemical measurements can be performed nondestructively on the embedded reinforcing steel. The data can provide early warning of structural distress and be used to evaluate the effectiveness of corrosion control systems. Once rebar corrosion has proceeded to an advanced state where its effects are apparent on the external concrete surface, it is too late to implement effective remedial measures. There are a number of electrochemical techniques for measuring the severity of rebar corrosion, each with certain advantages and limitations. Measuring the open circuit potential is easy and inexpensive, but not considered very reliable since the potential provides no information about the kinetics of the corrosion process. Linear polarization resistance (LPR) measurements are influenced by IR effects from the concrete. A significant potential drop in the concrete makes it difficult to accurately determine the potential for the rebar surface. Electrochemical impedance spectroscopy (EIS) is able to overcome the difficulties of the concrete resistance, yet requires more testing time. The different analytical methods of electrochemical impedance spectroscopy are capable of giving more detailed information than LPR. The rebar potential, polarization resistance and current density data can provide information as to whether the rebar is in the active or passive corrosion state. Estimates made from these parameters for Tafel constants can be input into LPR analysis or can be used for corrosion rate measurement and cathodic protection criteria. For a more comprehensive approach to the corrosion process, several tests methods have been implemented in this investigation.

Electrochemical Polarization Behavior

Figure 2 shows a comparison of the polarization behavior from a potentiodynamic test of steel rebar in a saturated $Ca(OH)_2$ solution. This graph shows the effects of a mixed inhibitor in an alkaline environment similar to the concrete medium with minor reduction in the corrosion current upon addition of MCI. Figure 3 shows the polarization results from the steel rebar tested in a saturated $Ca(OH)_2$ solution with 2000 ppm NaCl. The effects of the inhibitor are far more noticeable in the presence of a corrosive species. The breakdown potential for the rebar tested with no inhibitor was around 350 mV_{SCE} as compared to 600 mV for the rebar tested with 2000 ppm MCI. Figure 4 shows the corresponding current density for various additions of MCI in column format. Consistent with the graph in Figure 3, the rebar tested in a saturated $Ca(OH)_2$ solution with 2000 ppm NaCl and 2000 ppm MCI had the lowest corrosion rate. According to the data in Table 2, where a level of corrosion severity has been associated with a given i_{corr} value, the sample tested with 2000 ppm MCI and having a current density of less than 0.4 µm will have "no expected corrosion damage." Whereas, for the untreated sample (represented by the **$Ca(OH)_2$ only** curve in Figure 3 and the **0 ppm MCI** bar column in Figure 4), it is estimated that corrosion damage will occur in 10-15 years.

Table 2 - Proposed relationship between corrosion rate and remaining service life.

Corrosion rate (uA/cm^2)	**Severity of Damage**
< 0.5	no corrosion damage expected
0.5-2.7	corrosion damage possible in 10 to 15 years
2.7-27	corrosion damage expected in 2 to 10 years
> 27	corrosion damage expected in 2 years or less

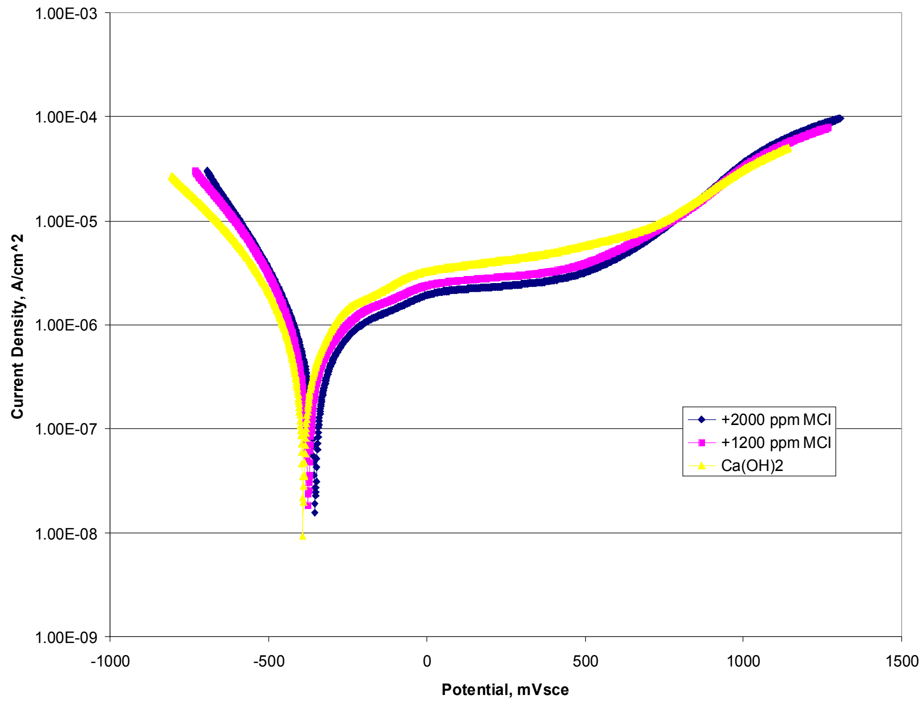


Figure 2 - Polarization behavior of steel rebar in a saturated $Ca(OH)_2$ solution with 2000 ppm NaCl and various ppm MCI 2020M additions, pH 12.4, 23 °C.

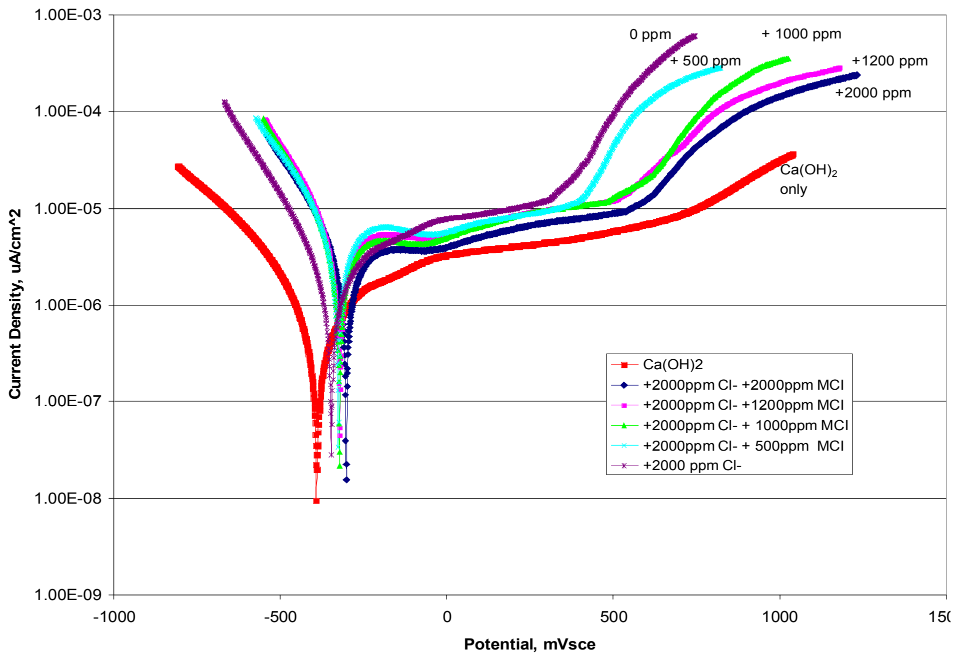


Figure 3 - Polarization behavior of steel rebar in a saturated $Ca(OH)_2$ solution with 2000 ppm NaCl and various ppm MCI 2020M additions, pH 12.4, 23 °C.

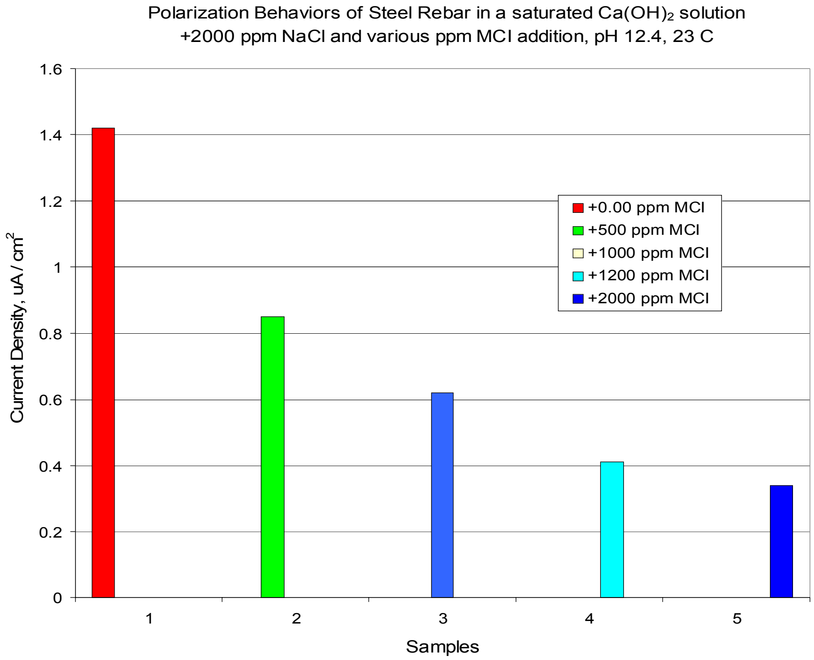


Figure 4 - Polarization behavior of steel rebar in a saturated $Ca(OH)_2$ solution with 2000 ppm NaCl and various ppm MCI 2020M additions, pH 12.4, 23 °C.

Corrosion Potentials

The corrosion inhibition for Cortec MCI 2020 has been investigated over a period of 450 days using AC electrochemical impedance spectroscopy (EIS). Throughout this investigation, changes in the corrosion potential of the rebar were monitored to determine the effects of this commercially available inhibitor. According to the ASTM (C876) standard [10], if the open circuit potential (corrosion potential) is -200 mV or higher, this indicates a 90% probability that no reinforcing steel has corroded. Corrosion potentials more negative than -350 mV are assumed to have a greater than 90% likelihood of corrosion. Figure 5 shows that the corrosion potentials for all samples were between the range of -10 mV to -100 mV after 450 days of immersion in NaCl, excluding the untreated low density sample.

Polarization Resistance

This electrochemical technique enables the measurement of the instantaneous corrosion rate. It quantifies the amount of metal per unit of area being corroded at a particular instant. The method is based on the observation of the linearity of the polarization curves near the potential E_{corr}. The slope expresses the value of the polarization resistance (R_P) if the increment diminishes to zero. This R_P value is related to the corrosion current I_{corr} by means of the expression:

$$R_p = \left(\frac{\Delta E}{\Delta I} \right)_{\Delta E \to 0} \qquad I_{corr} = \frac{B}{R_p \cdot A}$$

Where A is the area of the metal surface evenly polarized and B is a constant that may vary from 13 to 52 mV. For the case of steel embedded in concrete, the best fit with parallel gravimetric losses, results in B = 26 mV for actively corroding steel, and B = 52 mV for passivated steel [11]. Figure 6 shows increasing trends for the samples with polarization resistance values between 70 kohm and 85 kohm. The untreated low density sample, however, has been declining in value since day 326 of immersion. The R_P value after 450 days of immersion is roughly 9 kohm. The polarization resistance values seen in Figure 6 are somewhat better for the high density samples compared to the low density samples.

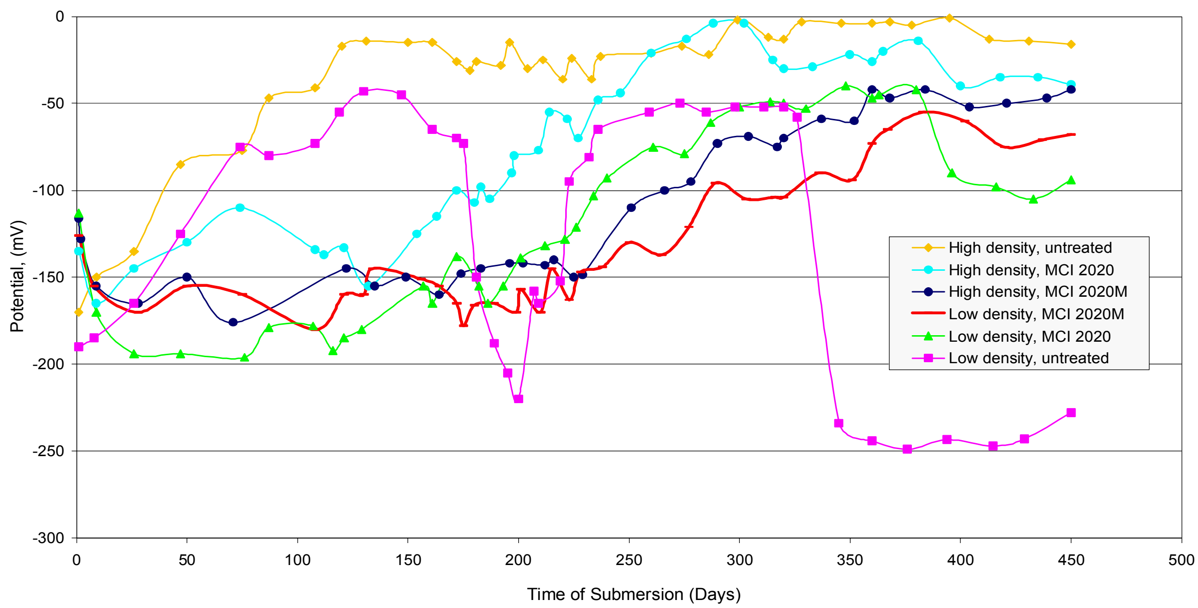


Figure 5 - Comparison of corrosion potential vs time for MCI treated and untreated samples.

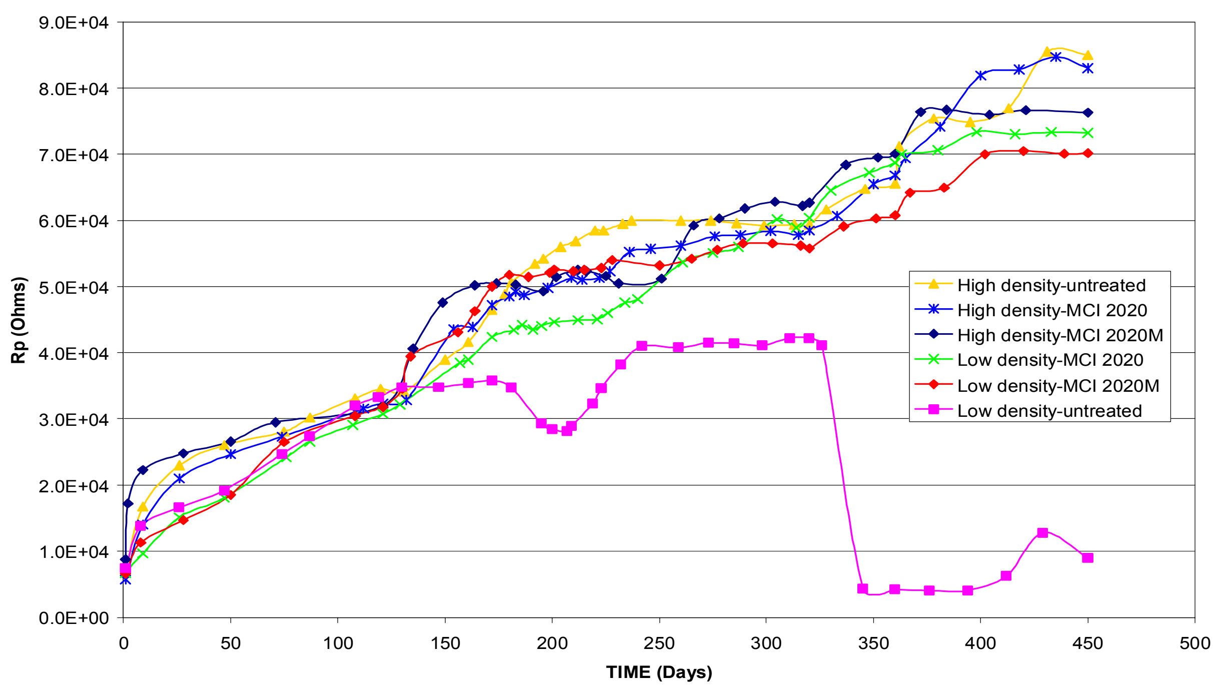


Figure 6 - Comparison of polarization resistance (R_P) for MCI treated & untreated concrete samples.

Bode Plots

Bode plots are not dependent on modeling the corroding system as are polarization resistance values. The electrochemical impedance spectroscopy data are obtained by applying a single sine wave over a range of frequencies while measuring the corresponding impedance. Since the results are independent of an assumed model, the technique is highly reliable. Figure 7 shows a comparison of the bode plots for the first day of testing and Figure 8 shows the sample data after 450 days of immersion. There is a noticeable contrast between the untreated low density sample and the others.

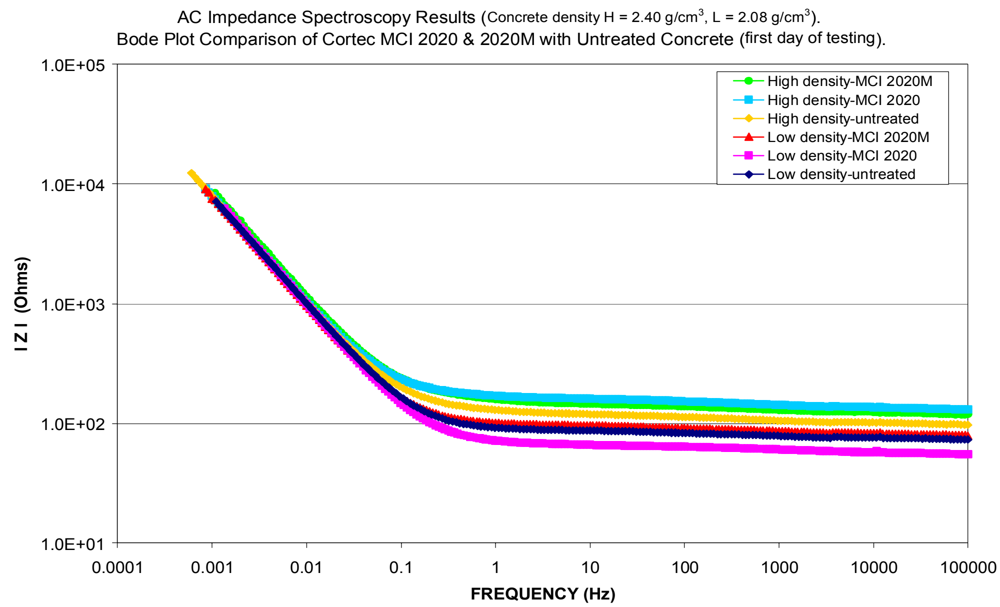


Figure 7 - EIS Bode plot for MCI 2020 treated & untreated concrete on day 1 of testing.

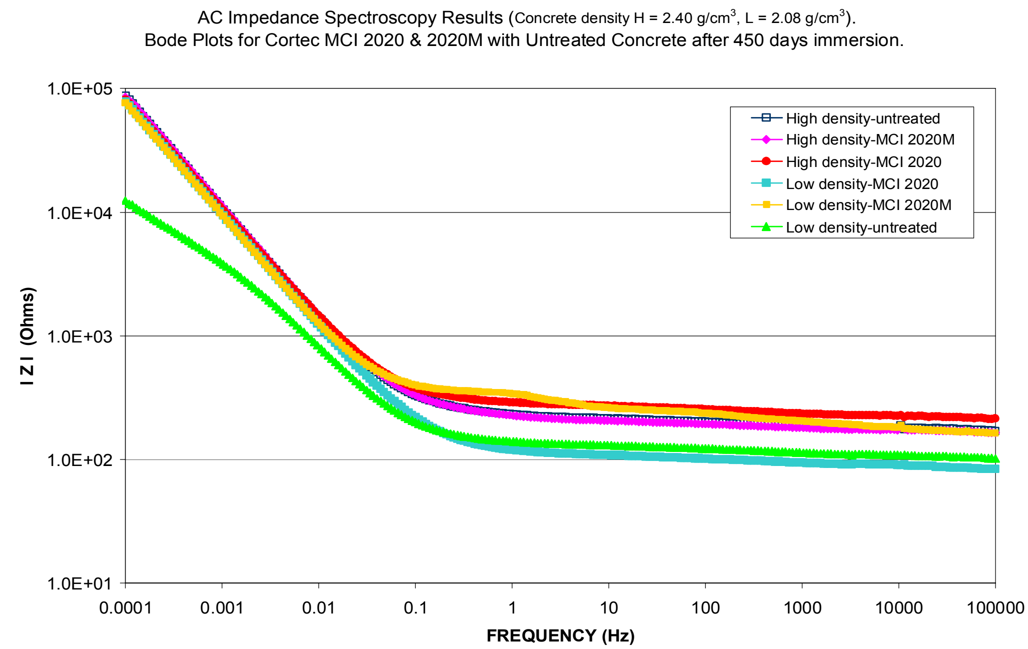


Figure 8 - EIS Bode plot for MCI 2020 treated & untreated concrete after 450 days of testing.

Potentiostatic Investigation

The potentiostatic data show a dramatic difference in the corrosion behavior of steel rebar in water with 150 ppm Cl^- compared to tests conducted in 2.5%, 5% and 10% MCI 2020M inhibitor solutions. The lowest corrosion rate occurred with a 10% solution of MCI 2020M (Figure 9).

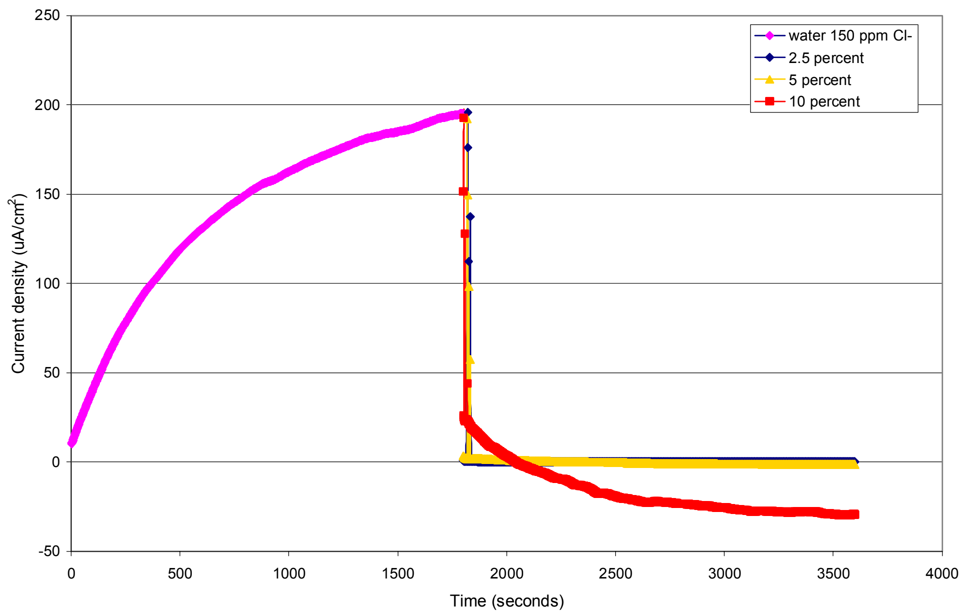


Figure 9 – Potentiostatic Corrosion Behavior of Steel Rebar in Water with Various Concentrations of Migrating Corrosion Inhibitor.

Visual Examination

After 500 days, partially immersed in a 3.5% NaCl solution at ambient temperatures, four of the six samples were cut open to remove the rebar. Visual examination of the untreated low density concrete sample showed corroborating evidence for the electrochemical data; there were obvious indications of corrosion rust products on the rebar and concrete surfaces (Figure 10). The samples treated with either MCI 2020 or MCI 2020M, as seen in Figures 11 and 12 showed no indication of corrosion.

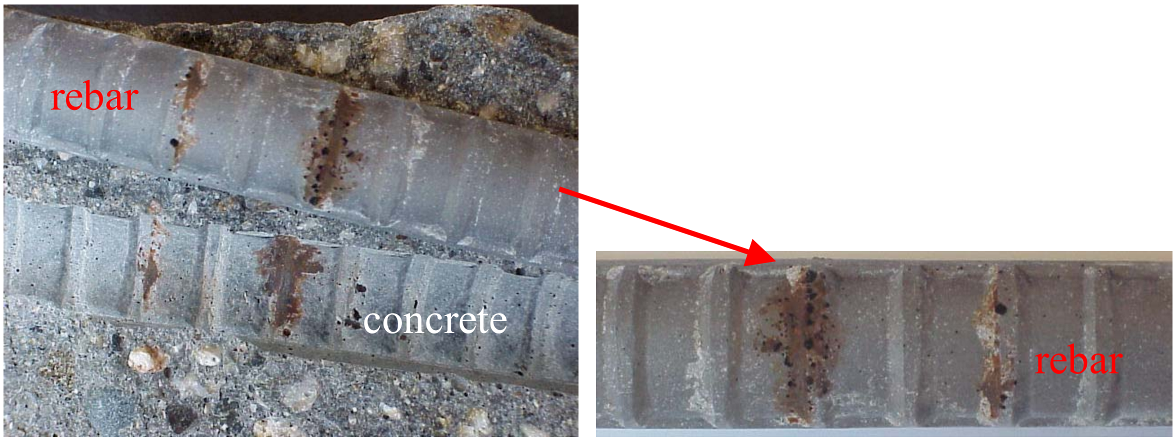


Figure 10 – Untreated low density concrete sample with corrosion rust products near the ribbing of the rebar and stains on the concrete. Photo on the right is an enlargement of the rebar.

Figure 11 – No corrosion is seen on the low density concrete sample surface treated with MCI 2020 inhibitor.

Figure 12 – No corrosion is seen on the low density concrete sample surface treated with MCI 2020M inhibitor.

Scanning Electron Microscopy Analysis

Scanning electron microscopy (SEM) and energy dispersive X-ray microanalysis (EDX) was performed on the rebar samples. Figure 13 shows an image of the rebar surface for the untreated concrete sample, its spectrum and the weight concentration percentage for elements typically found in concrete, corrosive species and rebar. For the untreated sample, nitrogen, the active component for MCI corrosion inhibitors is not detected. In Figure 14, the SEM/EDX analysis for the concrete sample (surface impregnated with MCI 2020) shows that nitrogen was detected and provides weight concentration percentages for two analysis points. Analysis for the concrete sample coated with MCI 2020M (Figure 15) also shows the presence of nitrogen on the surface of the rebar. The chemistry for the treated samples shows close similarity. The presence of nitrogen on the rebar surface is significant in that it confirms the inhibitors are able to migrate through the concrete to reach the surface of the rebar.

X-ray Photoelectron Spectroscopy (XPS)
Further examination of the rebar surface was conducted with a Kratos Axis Ultra XPS instrument. An XPS detector can analyze a much larger area than an SEM point analysis, providing a more comprehensive evaluation of surface chemistry. A comparison of the spectrums prior to etching with argon gas (X-ray parameters: Mg anode at 15 kV, current of 15 mA and pass energy of 80; ion etching with argon gas, 4 kV, current of 15 mA) are shown in Figure 16. Additional spectrums are shown for the etched samples after 120 seconds (Figure 17) and after 240 seconds (Figure 18). The XPS chemical quantification (Table 3) reveals organic compounds with carboxylate chemistry for the MCI 2020 & MCI 2020M sample. From the XPS depth profiling, chloride was detected at depths down to 60 nm from the analysis surface on the rebar and at a concentration of approximately 0.44 weight percent for the untreated sample and roughly 0.14 wt % for the treated samples (Figure 19). Nitrogen was detected at levels down to 75 nm on the MCI 2020 sample and as far down as 85 nm on the MCI 2020M sample. The XPS results demonstrate that MCI and the corrosive species have similar diffusion rates. The MCI inhibitors provided a protective film on the rebar surface, the untreated samples, however, were subjected to localized corrosion attack.

Untreated	N	O	Mg	Al	Si	S	Cl	Ca	Fe
Weight Conc%	0.00	16.29	1.24	0.83	9.08	1.54	0.97	67.03	3.03

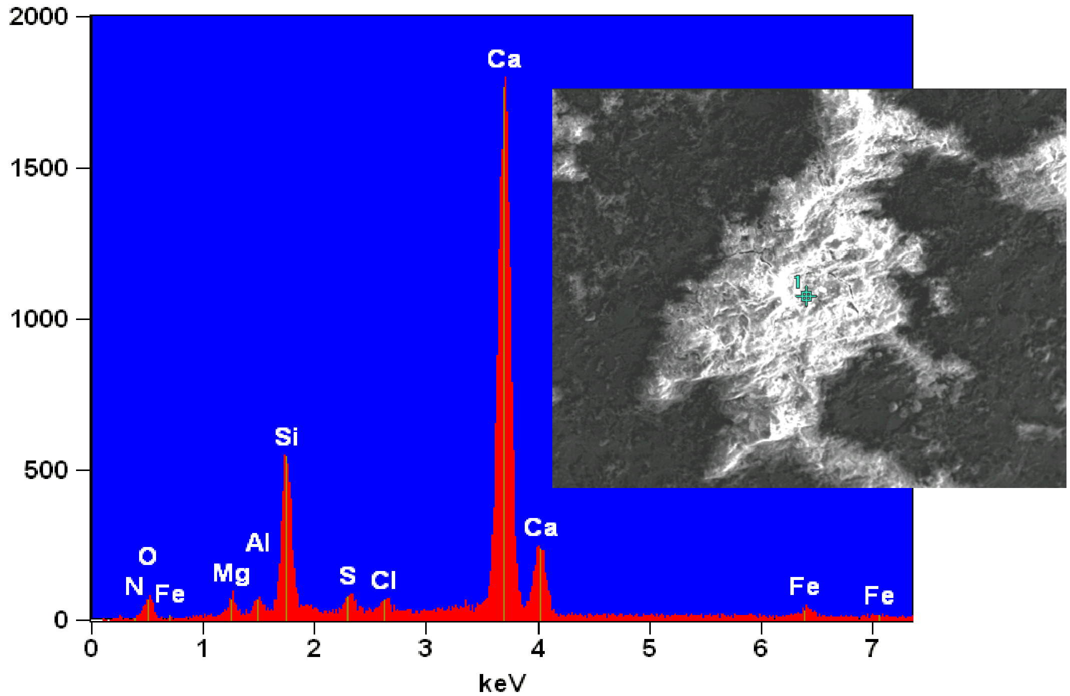
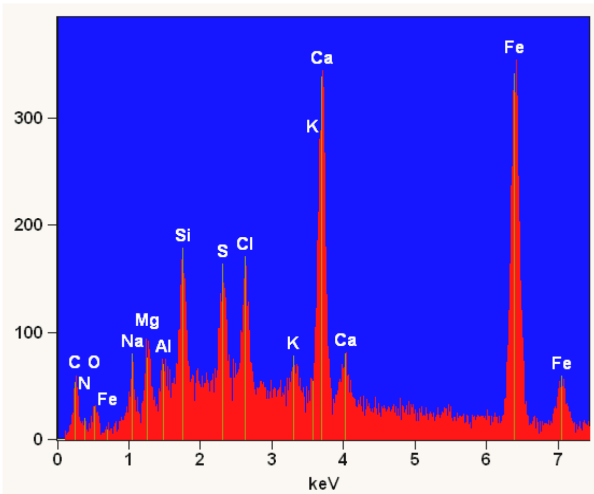


Figure 13 – SEM/EDX analysis of rebar surface of untreated sample after 500 days.

Weight Concentration %											
2020	N	O	Na	Mg	Al	Si	S	Cl	K	Ca	Fe
L2020_pt1	0.53	4.09	3.51	2.12	1.52	4.27	4.31	5.31	1.42	19.37	53.56
L2020_pt2	0.66	12.01		0.41	1.28	4.56	1.10	0.94		71.02	8.02



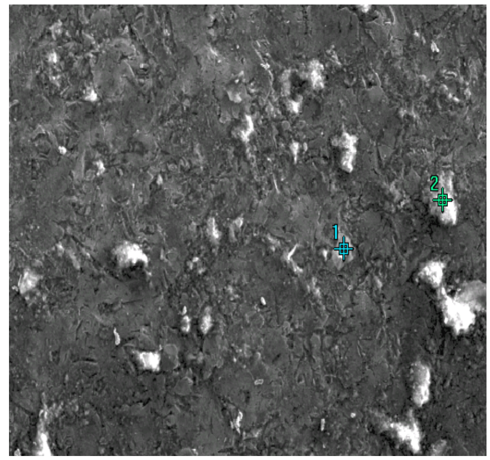
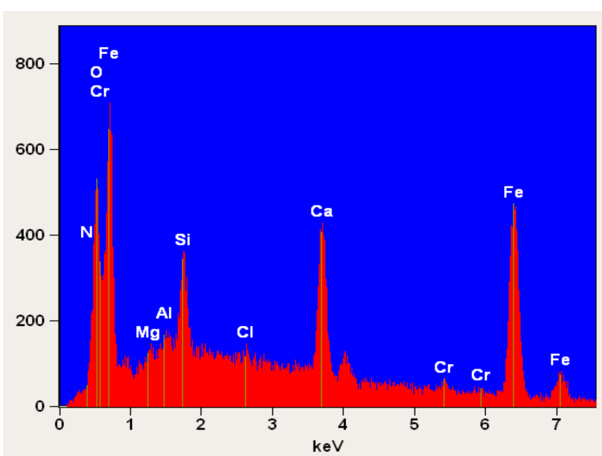


Figure 14 – SEM/EDX analysis of rebar surface of MCI 2020 low density concrete sample after 500 days.

2020 M	N	O	Al	Si	S	Cl	Ca	Mn	Fe
Weight Conc %	0.46	3.81	1.52	5.13	0.74	1.82	22.71	0.78	63.02
Atom Conc %	0.61	10.46	2.48	8.06	1.02	2.26	24.89	0.62	49.61



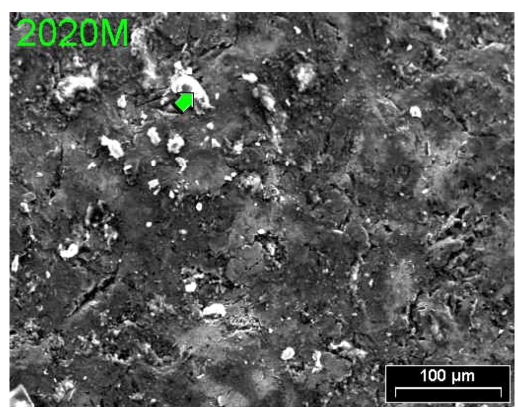


Figure 15 – SEM/EDX analysis of rebar surface of MCI 2020M low density concrete sample after 500 days.

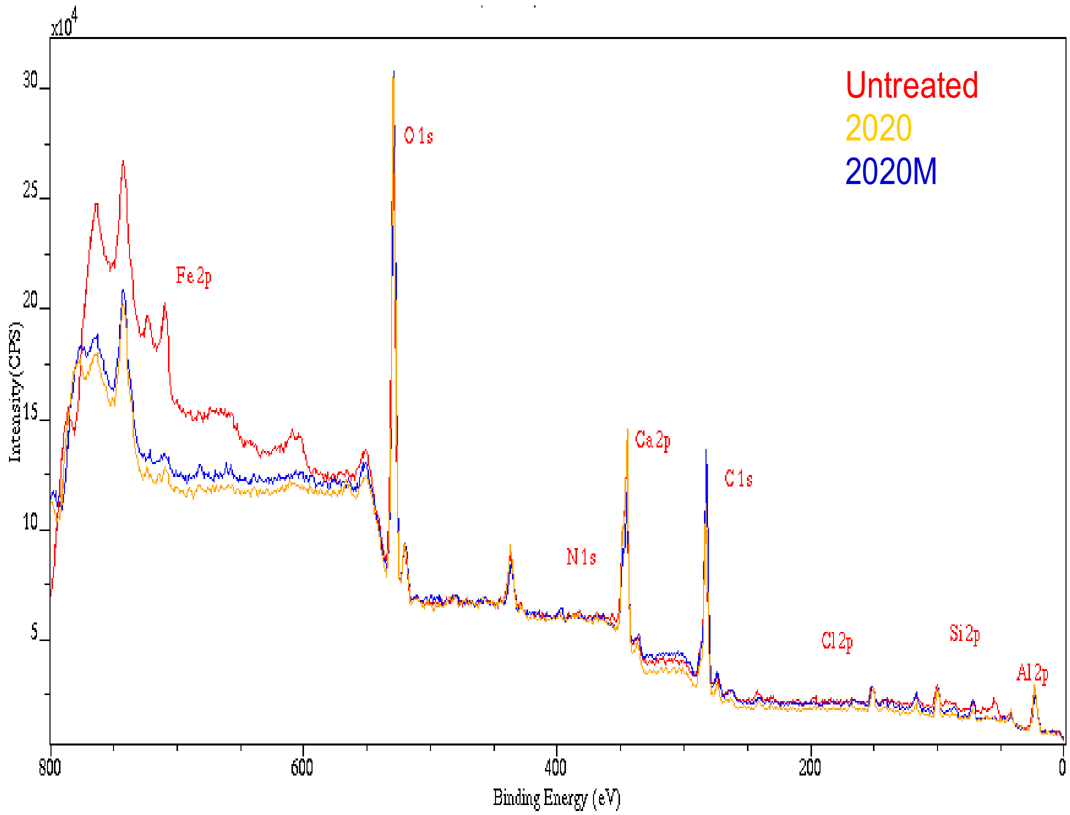


Figure 16 – XPS analysis on concrete samples after 500 days, before etching.

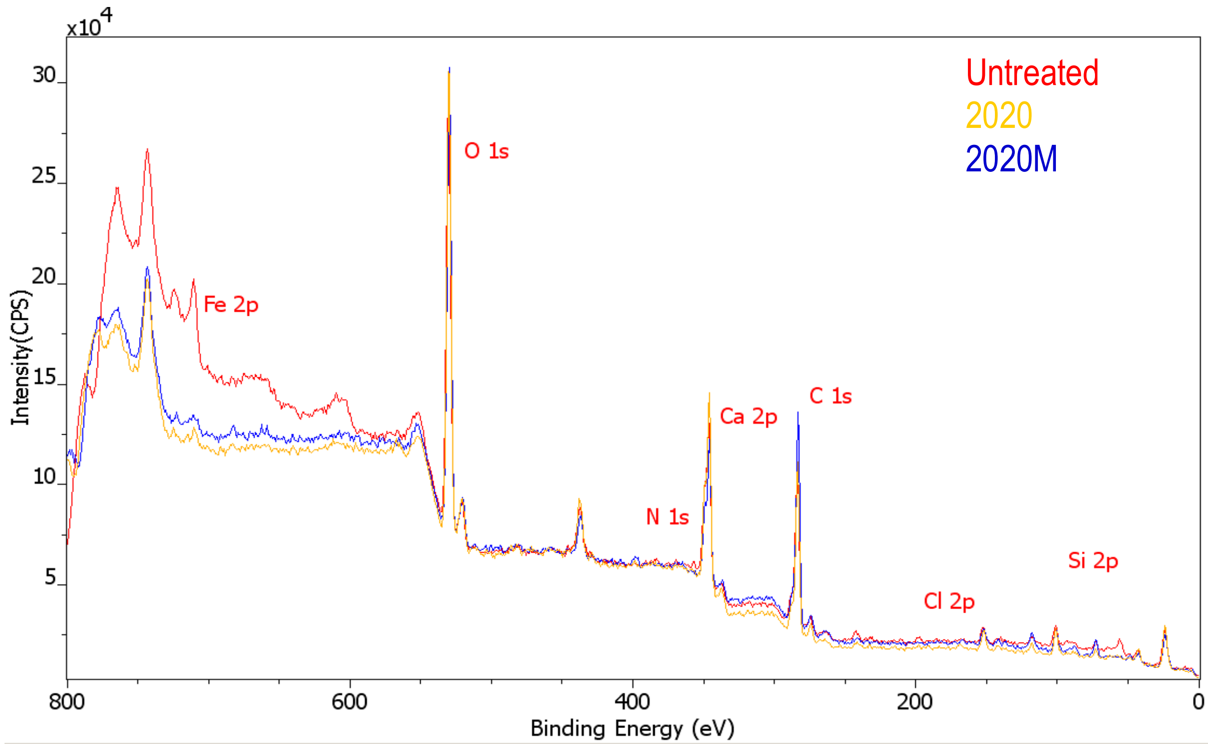


Figure 17 – XPS analysis on concrete samples after 500 days, 120 seconds etching.

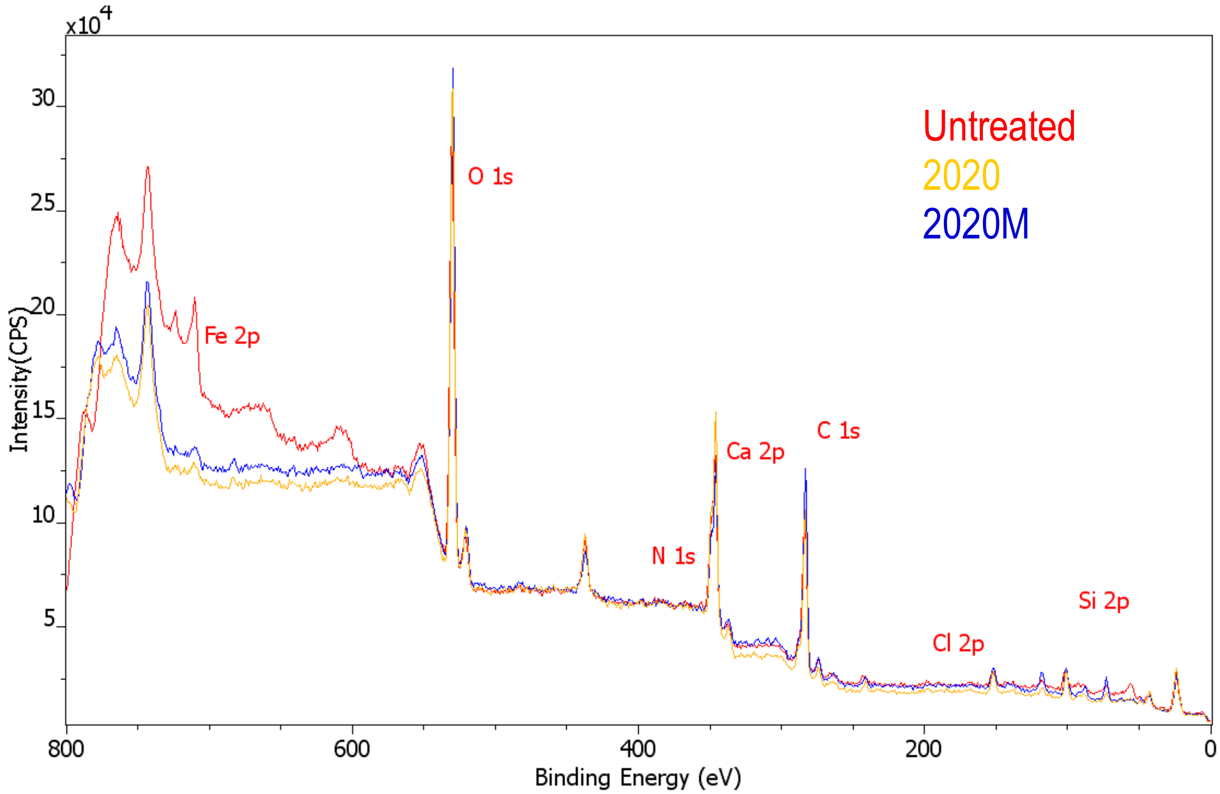


Figure 18 – XPS analysis on concrete samples after 500 days, 240 seconds etching.

Table 3 - XPS analysis on concrete samples after 500 days, showing the changes in chemistry with etch time.

		Mass Concentration %						
Sample	Etch Time (seconds)	**Fe 2p**	**O 1s**	**C 1s**	**N 1s**	**Cl 2p**	**Ca 2p**	**Si 2p**
Untreated	0	6.27	42.71	30.67	0.19	1.07	14.19	4.97
Untreated	120	13.60	39.43	23.08	0.14	1.06	17.59	5.19
Untreated	240	14.65	38.77	22.35	0.11	1.01	18.18	5.03
L2020	0	2.30	42.22	29.90	1.16	0.95	17.28	6.26
L2020	120	2.53	43.01	25.17	1.12	0.93	20.14	7.18
L2020	240	2.56	43.85	21.95	1.05	1.40	22.19	7.09
L2020M	0	2.02	40.20	38.55	1.32	0.87	11.54	5.53
L2020M	120	2.22	41.74	32.13	1.29	0.86	15.41	6.42
L2020M	240	2.82	43.61	28.99	1.15	0.83	15.92	6.68

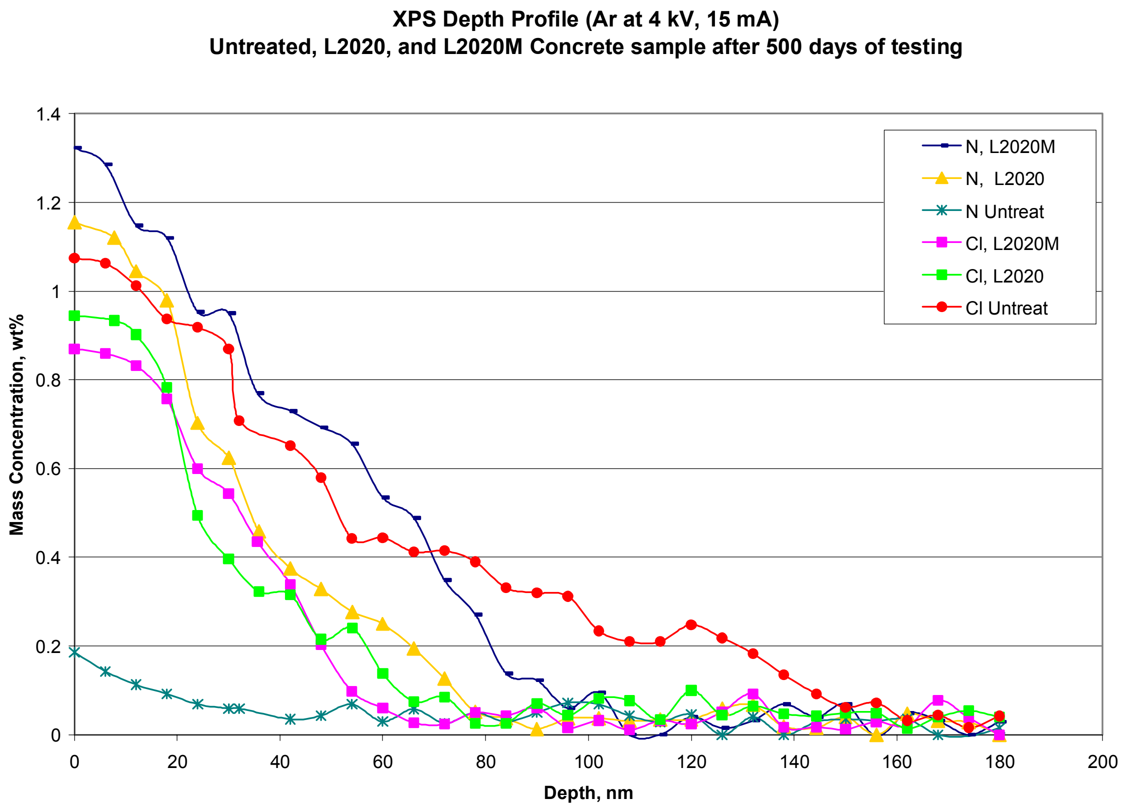


Figure 19 – XPS depth profiling showing mass concentration weight percentage of chloride and nitrogen for test samples.

Conclusion

Investigating the performance of two inhibitors, MCI 2020 and MCI 2020M began with tests on exposed rebar to determine the potentiodynamic behavior. Studies were conducted in a saturated $Ca(OH)_2$ solution with and without chloride ions. Comparisons of the polarization behavior were made for the steel rebar in solutions with varying concentrations of inhibitor. There was minor reduction in the corrosion current when MCI was added to the solution, demonstrating the effects of a mixed inhibitor in an alkaline environment similar to the concrete medium. The effects of the inhibitor were far more noticeable in the presence of a corrosive species. The breakdown potential for the rebar tested with no inhibitor was around +350 mV_{SCE} and improved to +600 mV for the rebar tested with 2000 ppm MCI.

Two concrete samples were surface impregnated with several coats of MCI 2020, two were coated with MCI 2020M, and two were untreated. The corrosion potentials for five of the samples were between the range of -10 mV to -100 mV after 450 days of immersion in NaCl. The untreated low density sample appears to have suffered from corrosion attack in this aggressive environment. The polarization resistance values were between 70 k-ohm and 85 k-ohm for all but the untreated low density sample. The R_P value for the untreated sample has been declining since day 326 of immersion. Overall, the polarization resistance values are somewhat better for the high density concrete samples compared to the low density samples. From the preliminary testing, MCI protected samples showed an average current density of 0.4

μA/cm^2 compared to untreated samples with 1.4 μA/cm^2. This behavior will increase the life expectancy by more than 15-20 years.

XPS analysis demonstrated the presence of inhibitor on the steel rebar surface with nitrogen detection at levels 85 nm below the unetched surface (MCI 2020M sample) and as far down as 75 nm for the MCI 2020 sample. The XPS results showed similar diffusion rates for MCI and the corrosive species. The MCI inhibitors were able to provide a protective film on the rebar surface, whereas the untreated samples were subjected to localized corrosion attack. From the XPS depth profiling, chloride was detected at depths down to 60 nm from the analysis surface on the rebar and at a concentration of approximately 0.44 weight percent for the untreated sample and roughly 0.14 wt % for the treated samples.

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