

ORGANIC CORROSION INHIBITORS – NEW BUILD AND EXISTING STRUCTURES PERFORMANCE

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SUMMARY: Corrosion of embedded steel is a leading cause of deterioration in reinforced concrete structures. While concrete mix designs have become more durable, the possibility of cracking due to shrinkage, movement, or other forces is a consideration when designing for long service life. In addition, preserving integrity and extending the useful service life of existing structures is of paramount importance. Migrating, organic corrosion inhibitors have been used for more than 30 years in construction applications as an economical method of corrosion mitigation. This paper will give details on the chemistry of organic corrosion inhibitors and their performance in new construction, repair and specialty applications.

Keywords: Organic corrosion inhibitors, Migrating corrosion inhibitors, New construction, Repair, Post tensioning, Service life.

1. INTRODUCTION

High performance mix designs have significantly improved the quality of today's concrete. Unfortunately, cracking, whether due to shrinkage, settlement or other forces, is a reality that is never completely eliminated no matter how good your concrete is. The need for a corrosion inhibitor that can provide protection in the presence of minor cracks is very real. In addition, with aging infrastructure, preserving the integrity of structures and extending useful service life is of paramount importance. Topical corrosion inhibitor treatments must be capable of penetrating to the depth of embedded reinforcing steel.

Migrating corrosion inhibitors meet these challenges in a cost effective manner. They are available in many different forms for all types of construction – admixtures for new concrete; repair mortars and grouts; topical treatments; injectable products; and many specialty formats.

Organic corrosion inhibitors (OCI) are amine based chemicals that possess appreciable saturated vapour pressure under atmospheric conditions, thus allowing vapour transport of the inhibitive substance [1]. Concrete and other cementitious materials have pore structures that allow for liquid and vapour diffusion of these chemicals throughout the substrate. When organic inhibitors encounter embedded reinforcing, they have an attraction to it, adsorbing onto the metal surface forming a protective molecular layer.

OCI are a mixed (anodic/cathodic) inhibitor system. The inhibition of the cathodic process is achieved by the incorporation of one or more oxidising radicals in an organic molecule. Inhibitor molecules are hydrolysed in the electrolyte and then adsorbed on the metal surface. The nitrogen of the amine group is capable of entering into a coordinate bond with metal (including steel reinforcement) thus enhancing the adsorption process. Adsorption of amines and carboxylates increases the resistivity of metal to corrosion. This molecular layer serves as a buffer to hold the pH at the interface in the basic regime (above pH 9) [2].

Organic corrosion inhibitors migrate through the concrete depending on the OCI type and application method. OCI admixtures are readily dispersed in the mixing process and can also migrate to reinforcement by diffusion as both a liquid and a vapour. OCI topical treatments penetrate first as a liquid through capillary suction and then by diffusion [3]. OCI are effective against chloride induced corrosion, as well as carbonation and other corrosive elements (H₂S, SO₂, etc.).

The first generation of OCI was based on amine alcohol technology. The latest generation is based on amine carboxylates and was first introduced in the early 1990's. The amine carboxylate technology includes by-products of corn – a renewable resource.

Amine carboxylate based OCI can delay setting time of concrete 3-4 hours at 70°F (20°C). This delay is less pronounced at warmer temperatures. Set delay is a desired side effect in warm weather as less retarder will need to be added to the concrete mix when using OCI. Combining amine carboxylate based organic corrosion inhibitors with some set retarding

materials (admixtures) may create increased setting times, but generally no other concrete properties are affected. In cases where set delay is not desired, normal set (NS) versions of OCI have been developed, or non-chloride accelerators can be used.

2. CHEMISTRY OF ORGANIC CORROSION INHIBITORS

Corrosion inhibitors are classified based upon the part of the corrosion cell that they affect. Organic corrosion inhibitors are classified as mixed inhibitors, meaning they affect both anodic and cathodic portions of the corrosion cell. These inhibitors adsorb onto metal, forming a protective molecular layer on steel surfaces. This film prevents corrosive elements from further reacting with embedded reinforcement, and also reduces existing corrosion rates. Adsorption of the inhibitor takes place via its polar functional group anchored to the metal while the non-polar or hydrophobic chain is oriented perpendicularly to the metal surface. The hydrophobic chains not only repel aqueous corrosive fluids, but interact with each other to form aggregates thereby forming a tight film on the metal surface [2]. Figures 1 and 2 illustrate adsorption of amine alcohol and amine carboxylate organic inhibitors respectively.

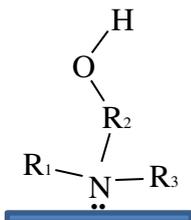


Figure 1. Illustration of Amine Alcohol Inhibitor, where R1,2,3 is H or alkyl group, OH tail end group

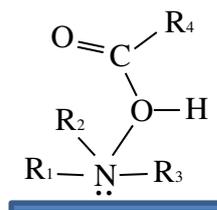


Figure 2. Illustration of Amine Carboxylate Inhibitor, where R1,2,3 is H or alkyl group, OH tail end group

Migrating corrosion inhibitors reach embedded reinforcement in several ways. First, the inhibitor is dispersed through the concrete with adequate mixing. The transportation of OCI within concrete then occurs via infiltration through concrete pore capillary networks. Thirdly, due to their relatively high vapour pressures, OCI move via diffusion in a gaseous state through the pore network and minor cracks. Finally, when the molecules come into contact with embedded metals, they have a specific attraction (as described above) to it. This process is illustrated in Figures 3, 4, and 5.

The molecules move randomly from areas of high concentration to areas of low concentration until equilibrium is reached (Fick's 2nd Law of Diffusion). Because of the final adsorption of OCI on embedded metal, the concentration gradient drives OCI to move towards the metal. X-Ray Photoelectron Spectroscopy (XPS) analysis has demonstrated the nitrogen rich layer (from amine moiety in OCI molecules) formed on rebar surfaces embedded in concrete that received a topical treatment of OCI. This protective, monomolecular layer also reduces the corrosion rate [4, 5].

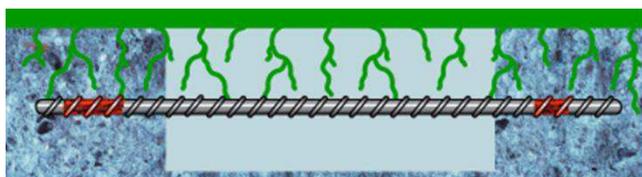


Figure 3. Liquid OCI infiltration through concrete capillary network

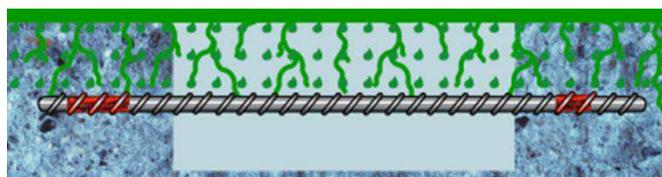


Figure 4. Vapour phase diffusion through concrete pores and minor cracks

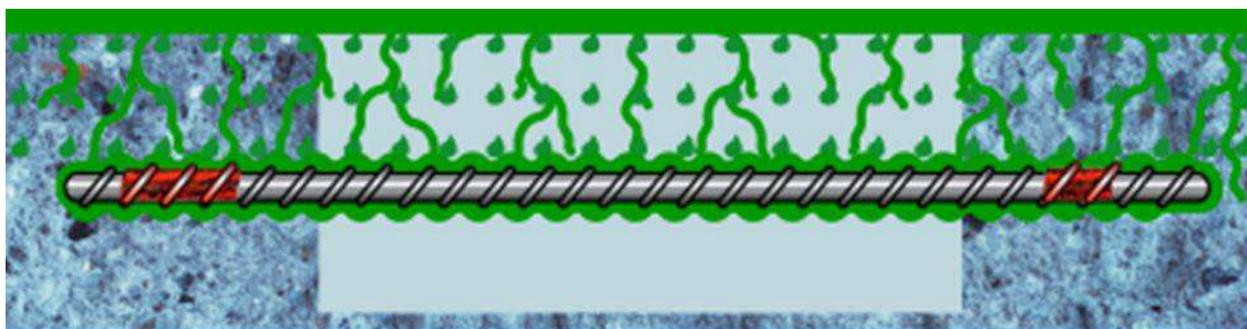


Figure 5. OCI Migrating Mechanisms – After liquid and vapour diffusion, showing final adsorption on metal to form a protective layer

Amine alcohols differ from amine carboxylates in their tail end group. As can be seen in Figures 1 and 2 above, the amine alcohols contain an OH terminal group at the tail end, whereas amine carboxylates have no such hydrophilic terminal group. The exposed OH group on amine alcohols makes it available to interact with water based electrolyte near the rebar. This means that the amine alcohol film could more easily desorb from the metal surface compared to the amine carboxylate film, and allow corrosive species (such as chlorides) to attack the metal. As a result the amine carboxylate based OCI by nature have a more tenacious bond to the metal surface [2].

The strength of the bond between the OCI molecule and the rebar is what delays the onset of corrosion and reduces corrosion rates compared to a control once initiated. With amine alcohols, we typically see a slight extension in time to corrosion initiation, and corrosion currents cut in half compared to a control following corrosion initiation. With amine carboxylates, we typically see a double to tripling in time to corrosion initiation, and once corrosion starts, rates are reduced by 5-15 times compared to a control [6].

Another small effect amine carboxylates have is that as they migrate through the concrete, some of them will react with calcium hydroxide to form calcium carboxylate/ $\text{Ca}(\text{COO})_2$, an insoluble salt, which effectively blocks some of the pores, making future ingress of chlorides and other contaminants more tortuous [7].

There are many test methods used to assess the efficacy of migrating corrosion inhibitors in concrete. It is important to note that these tests are described by category – corrosion protection, migrating ability, film forming ability, and compatibility with other materials.

2.1 Assessment of Corrosion Protection

The most widely used test method for evaluating concrete corrosion inhibiting admixtures is ASTM C1582 / C1582M – *Standard Specification for Admixtures to Inhibit Chloride-Induced Corrosion of Reinforcing Steel in Concrete* [8]. This standard consists of two components. The first is an evaluation of how the corrosion inhibitor affects concrete properties such as setting time, air entrainment, compressive and flexural strengths, freeze thaw resistance, and length change.

The second component involves corrosion testing according to ASTM G109 – *Standard Test Method for Determining Effects of Chemical Admixtures on Corrosion of Embedded Steel Reinforcement in Concrete Exposed to Chloride Environments* [9] or ASTM G180 – 13, *Standard Test Method for Corrosion Inhibiting Admixtures for Steel in Concrete by Polarization Resistance in Cementitious Slurries* [10].

ASTM G109 test samples can be seen in Figures 6 and 7 below. The test consists of casting concrete blocks containing a triangular arrangement of rebar embedded within them – one bar on top and two on the bottom. These samples are then exposed to cyclic wetting and drying cycles with salt water, and the macrocell current between the top bar and bottom two bars is monitored and recorded every 4 weeks (4 weeks = one cycle). The testing stops once the total integrated macrocell current reaches 150 Coulombs.



Figure 6. Example of ASTM G109 Test Blocks

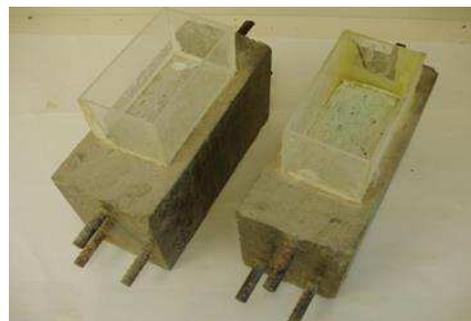


Figure 7. Example of ASTM G109 Test Blocks

The ASTM G180 – 13 [10] test setup can be seen in Figure 8. This test method provides a means for assessing corrosion-inhibiting concrete admixtures in a filtered cementitious liquid. Compared with a testing period of up to a few years required by ASTM G 109, this test method provides results within a few days. In this test, the polarization resistance of a steel sample is measured by applying a current through the test solution while varying the voltage. According to ASTM C1582, an admixture is behaving as a corrosion inhibitor when using the G180 Test Method (using 0.5 M NaCl with four control and four test specimens) if the mean $1/R_p$ value is less than or equal to $1/8$ the control average. Poor performance in G180 requires additional testing to determine if the admixture improves corrosion resistance.

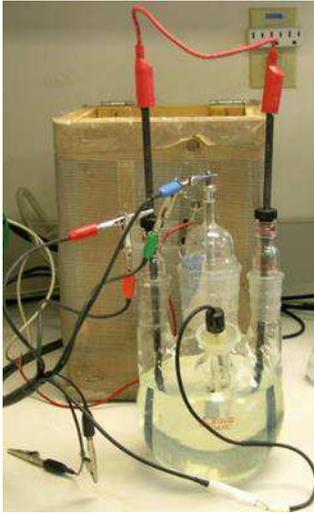


Figure 8. ASTM G180 Test Set-Up

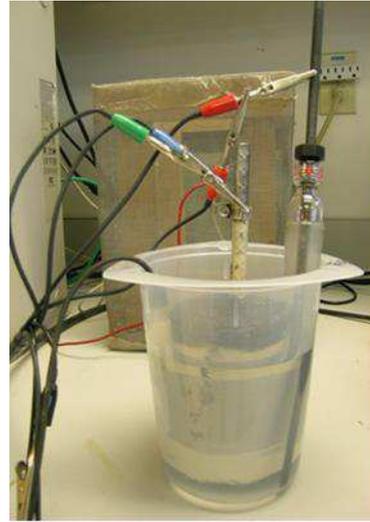


Figure 9. EIS Test Set-Up

Electrochemical Impedance Spectroscopy (EIS), Figure 9, is a powerful, rapid and accurate non-destructive method for the evaluation of a wide range of materials including coatings, anodised films and organic corrosion inhibitors on metal surfaces [11]. During EIS experiments, a small amplitude AC signal is applied to the metal system being studied. Data from EIS can provide detailed information on corrosion rate, electrochemical mechanisms, reaction kinetics, and detection of localised corrosion in a system.

In addition to the above standard tests, ASTM G109 is frequently modified to make it more aggressive so results can be seen faster. These tests are generally referred to as a Modified ASTM G109 or a cracked beam test. In a cracked beam test, test samples are created in the same manner as the ASTM G109 standard shown above, but the beams are cracked in a controlled way (saw cut and application of flexural stress) prior to salt water cycling to make the test more aggressive. Other modifications include changing the NaCl (salt) concentration (increasing from 3.5% to 6%) as well as the cycle times (adjusting from 4 weeks down to as little as 1 week cycles). Another variation on ASTM G109 is permanent immersion of concrete beams in salt water instead of using ponding cycles.

The most recent test for evaluating organic corrosion inhibitors in repair applications is the U.S. Bureau of Reclamation M-82 - *Standard Protocol to Evaluate the Performance of Corrosion Mitigation Technologies in Concrete Repairs* (USBR M-82) [12]. USBR M-82, Figure 10, is the outcome of a pilot program which was conducted by the Bureau of Reclamation under a cooperative agreement with the Strategic Development Council. The contract to develop the protocol was awarded to Tourney Consulting Group in the US and was funded by the US Bureau of Reclamation with funds from the American Concrete Institute (ACI) Foundation. The protocol covers two methods for measuring and differentiating the performance of reinforcing steel corrosion mitigating techniques for concrete repairs caused by damage due to chloride ingress. This protocol doesn't differentiate between active and passive corrosion, but it has all of the positive aspects of impedance testing (described in EIS above) but on a larger scale which is more representative of slabs in the field [13].

M-82 involves the production of 101.6cm long by 101.6 cm wide x 13.97cm thick (all dimensions +/- 1.27cm) concrete slabs with discrete steel reinforcing bars near the top of the slab (~2.54cm from surface) and welded-wire reinforcing (WWR) near the bottom of the slab (~2.54cm from bottom), Figure 10. The steel bars are connected to the WWR through 1 ohm (Ω) resistors through switches in a junction box attached to the side of the slab. Concrete slabs are cyclically ponded with a 5% by mass sodium chloride (NaCl) solution for two weeks and then the solution is removed for two weeks (one test cycle). Macrocell current between each bar and the WWR is determined by measuring the voltage drop across the resistor within 24 hours of the start and immediately prior to the end of each ponding cycle. The positive macrocell currents (reinforcing steel bar is corroding) are integrated over time to have total macrocell corrosion for each bar. The total for all of the bars is also determined. The switches in the junction box are set so that all the bars and the WWR can be electrically connected through the resistors. For potential mapping the bars are connected.

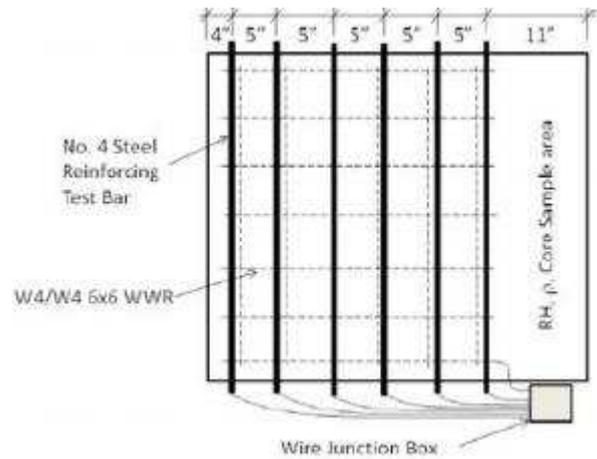


Figure 10. Photo and Bar Layout Diagram of M-82 Test blocks

2.2 Assessment of Migrating Ability

Migration of OCI materials into concrete can be evaluated using UV Spectroscopy [14]. This method allows us to detect OCI presence at various depths of concrete by analysing sliced core samples. OCIs are visible in the UV range. By extracting powdered concrete in deionised water, and measuring the extract using UV spectroscopy, the presence of OCI in concrete can be detected. This is done by comparing the absorbance values from treated concrete against the absorbance values of untreated concrete at specific wavelengths. While initial results are only qualitative, if a calibration curve is created with known doses of OCI, the actual concentration can be more accurately determined.

2.3 Assessment of Film Forming Ability

The film forming ability of OCIs can be measured using Scanning Electron Microscopy (SEM) and X-ray Photoelectron Spectroscopy (XPS). SEM is microscopy that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected. SEM coupled with EDAX Energy Dispersive Spectroscopy (EDS) Detector can provide enriched information about the sample's surface composition and topology. The presence of OCI, or the lack of it, on metal surfaces can thus be detected [15].

XPS is a spectroscopic technique in which a beam of x-rays is focused onto the surface of a material and as the beam bounces off the surface it is received by an electron collection device. The data received is then analysed to determine the elemental composition of the material which reflected the x-ray beam. A diagram showing how XPS works can be seen in Figure 11.

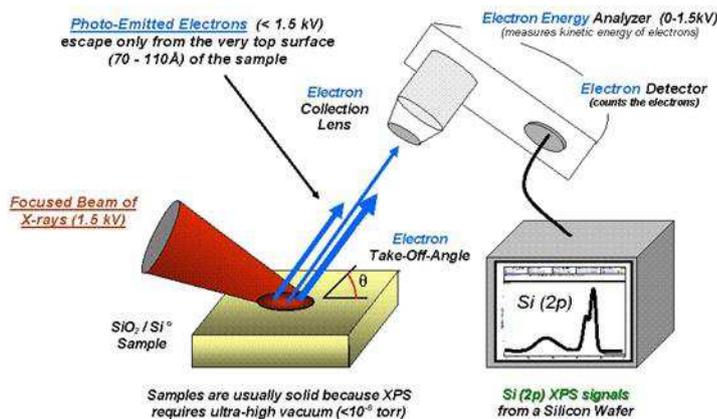


Figure 11. Diagram of XPS Process

In terms of organic, migrating corrosion inhibitors XPS detection can be used to analyse a sample of rebar to determine if there is inhibitor on the surface. Both amine alcohols and amine carboxylates are nitrogen containing compounds which is not found in abundance in the concrete matrix. Therefore, nitrogen levels (relative concentration when comparing two plots, i.e. control vs. treated) recorded on the XPS spectrogram can be used to determine if corrosion inhibitor has migrated to the depth of the rebar. XPS also has the capability to look at the depth at which an element is from the surface of the

steel so the spectrogram can be used to determine depth at which the corrosion inhibitor adsorbed into the rebar in comparison to other elements like chloride.

3. NEW CONSTRUCTION APPLICATIONS

In new construction, organic corrosion inhibitors are incorporated into concrete as an admixture, either by adding to the mix water at a ready mix plant, or by adding on the jobsite to the ready mix truck prior to pouring. Their dosage rate is independent of the expected chloride levels. The recommended dosage rate is a sufficient concentration of inhibitor to form a monomolecular film on embedded metals and is independent of chloride concentrations. Unlike anodic inhibitors such as calcium nitrite, OCI do not have a “dangerous” concentration level – situations where a lower than required dosage rate relative to chloride content concentration would promote pitting corrosion [16].

The latest generation of OCI admixtures, based on amine carboxylates, are certified to meet NSF/ANSI 61 – 2016 *Drinking Water System Components -Health Effects* requirements, meaning they are safe for use at their recommended dosage rates in structures containing potable water [17]. These corrosion inhibitors also contain renewable resources as raw materials – one of the corrosion inhibiting materials in the amine carboxylate admixtures is derived from corn. They have been tested to ASTM C1582 / C1582M – *Standard Specification for Admixtures to Inhibit Chloride-Induced Corrosion of Reinforcing Steel in Concrete* [18] and pass the requirements for both physical property effects on concrete as well as for corrosion inhibition capability.

3.1 Randolph Avenue Bridge

One of the oldest applications of OCI admixtures was a bridge deck overlay done in 1986 on the Randolph Avenue Bridge which spans Interstate 35E in St. Paul, MN. The bridge is located in an area where winters are severe and de-icing salts are heavily used to combat icy road conditions. Originally built in 1963, the deck underwent repair in 1986 to mitigate chloride induced corrosion of the reinforcing steel and spalling that was occurring. Repair involved removing deteriorated concrete, and milling to within 13 mm of embedded reinforcement. Deeper areas of unsound or damaged concrete were also removed, resulting in varying depths of extraction according to the degree of deterioration. The deck was then replaced with a low slump, dense concrete (LSDC) overlay. Depth of the overlay varied from 58 to 107 mm, with the top layer of reinforcing steel located at 76 mm depth. Mix design for the overlay is shown in Table 1. An amine alcohol based OCI admixture was added to the overlay for the two westbound traffic lanes at a rate of 0.6 l/m³. The two eastbound traffic lanes used the same mix design without the corrosion inhibitor, to act as a control. The deck of the bridge slants towards the northeast for water runoff. Due to the exposure conditions, the westbound lanes were experiencing more damage than the eastbound lanes, resulting in a slightly thicker mean depth (8 mm thicker) of the overlay on the westbound lanes vs. the eastbound lanes (according to measurements taken in 1991) [19].

Table 1. Mix Design of LSDC Overlay – Randolph Avenue Bridge

Component	Control (kg/m ³) – Eastbound Lanes	Treated (kg/m ³) – Westbound Lanes
Type I Cement	496	496
Water	160	160
W/C Ratio	0.32	0.32
Coarse Aggregate	821.69	821.69
Fine Aggregate	815.16	815.16
Water-Reducing Admixture	0.148	0.148
Air-Entraining Agent	0.043	0.043
Organic Corrosion Inhibitor	--	0.564

The project was sponsored by the Minnesota Department of Transportation (MNDOT) and a part of a corrosion study with the Federal Highway Administration (FHWA) from 1986-1990. In June 1991 and August 1992, researchers from Virginia Tech conducted corrosion assessments, including visual inspections, chloride content as a function of depth, electrode (half-cell) potentials, and estimates of corrosion current densities (i_{corr}), using a 3LP (“3 electrode, linear polarisation”) meter.

In November 2000, June 2007 and July 2011, local technicians returned to the bridge and took new measurements. Measurements included linear polarisation resistance (using Gecor 6 [20, 21] and GalvaPulse [22, 23] devices), as well as alkalinity, half-cell potentials and chloride content (profile) testing.

Based on the service life prediction model, LIFE-365 [24], the chloride threshold of concrete is 0.05% by weight of the concrete. This converts to 0.4% by weight of cement, or to 1.98 kg/m³ (assuming a concrete bulk density of 3960 kg/m³). Chloride contents shown in Table 2 indicate that the chloride levels in the control side were slightly higher than the treated side and that they have continued to rise at the depth of the steel over time.

Table 2. Average Chloride Levels – Randolph Avenue Bridge

Year	Treated (kg/m ³)			Control (kg/m ³)		
	0-2.54 cm	2.54-5.08 cm	5.08-7.62 cm	0-2.54 cm	2.54-5.08 cm	5.08-7.62 cm
1991	2.08	0	0.415	4.57	1.48	1.13
1992	3.86	0.65	1.13	5.64	2.08	1.48
2000	6.94	0.95	0.77	10.2	3.67	1.42
2007	6.94	0.59	1.54	11.87	4.39	1.36
2011	7.30	2.91	1.07	8.72	3.92	2.08

Corrosion current readings, Figure 11, have increased on both sides of the bridge over time. Readings taken in July of 2011 showed that the control side had entered into active corrosion levels (corrosion rate readings above 0.5µA/cm²). All three sections tested indicated readings in the active corrosion range with the highest rate of corrosion recorded in the centre section of the control side, with an average reading of 1.27 µA/cm². In comparison, all three sections of the control side had readings in the passive range (corrosion rate readings less than 0.5µA/cm²), with the highest being the centre section with an average reading of 0.42µA/cm², indicating the treated side had roughly 67% lower rates than the untreated areas.

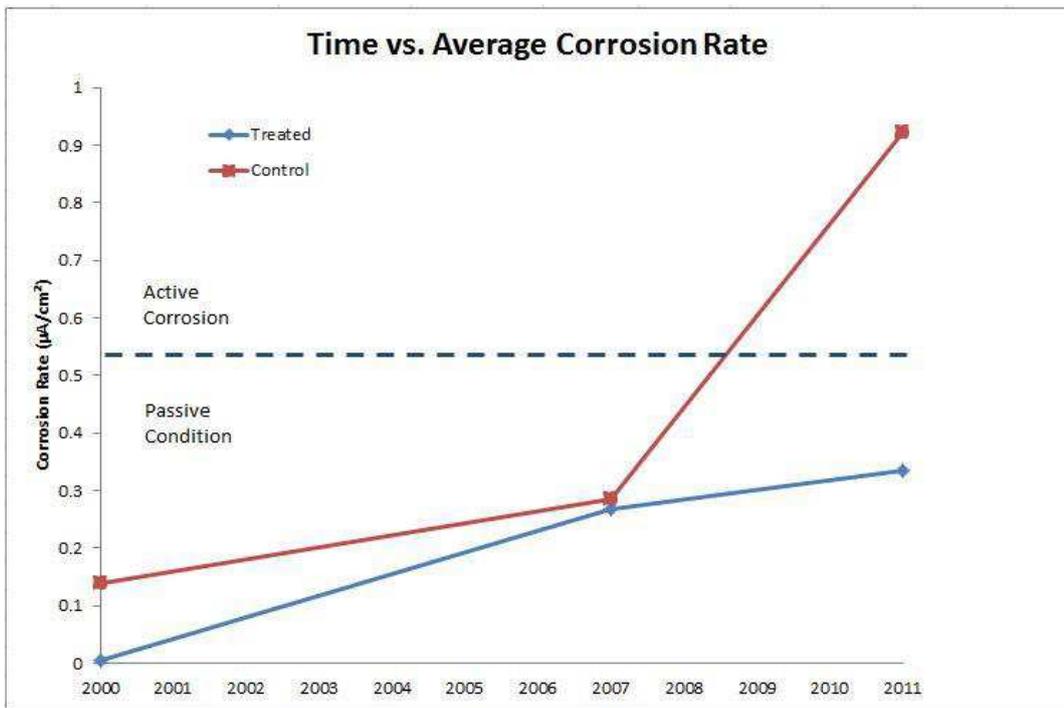


Figure 12. Randolph Avenue Bridge Average Corrosion Rates

Half-cell (electrode) potential readings can be seen in Figure 13. This figure shows that the electrode potential is more negative on the control side and has been for 20 years suggesting increased corrosion activity. This data along with the rest of the supporting information indicate that the level of corrosion on the treated side of the bridge is lower than the control areas.

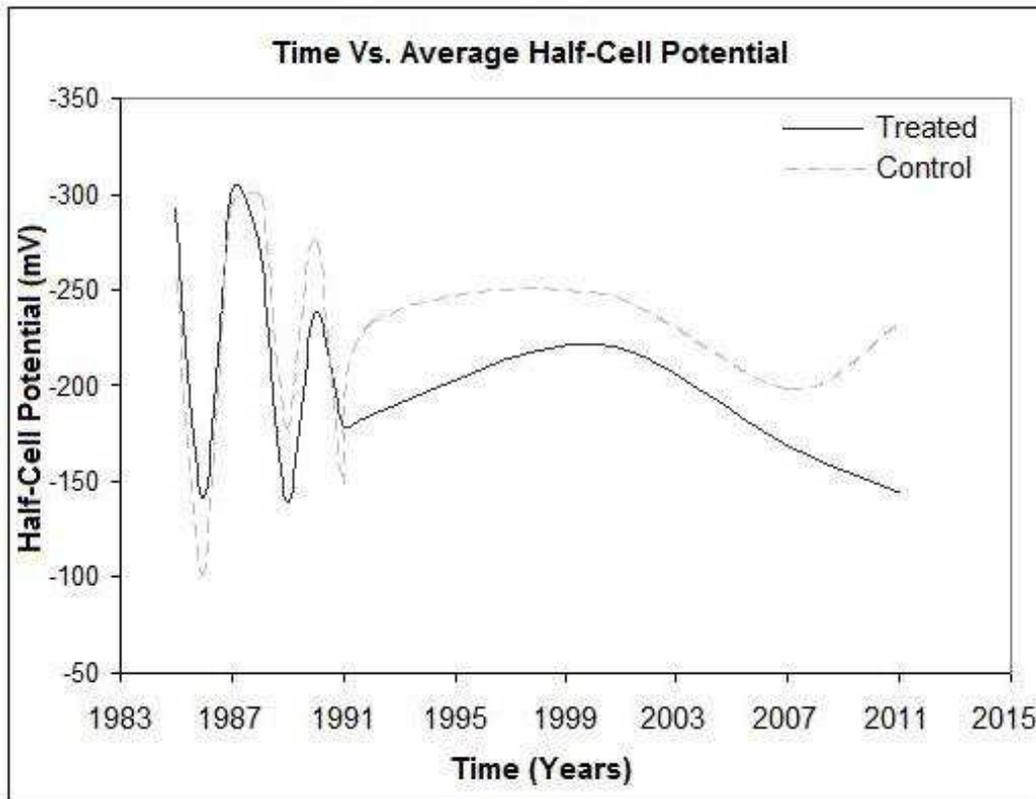


Figure 13. Randolph Avenue Bridge Average Corrosion Rates

3.2 Princess Tower Building, Dubai, UAE

The Princess Tower is a 101 story, 413.4 m [25] tall, residential-only skyscraper located in the Marina district of Dubai, UAE. When constructed, Princess tower was the tallest residential building in the world, but lost its top ranking in 2015 to 432 Park Avenue in New York City. Princess Tower remains the second tallest building in Dubai after Burj Khalifa, and the 20th tallest building in the world [26].

The building was completed and delivered by its developer, Tameer Holdings, in September 2012 and consists of 107 floors (6 basement levels, ground floor + 100 floors height above). It holds 763 residential units consisting of 1, 2, and 3 bedroom apartments, duplexes, and penthouses, together with 8 retail shops. It also houses 957 parking spaces.

In many areas of Dubai there is a high water table, as well as extremely high levels of chlorides and other contaminants in the soils where superstructures are being built. The level of chlorides in the ground water in this area has been shown to reach 90,000 mg/L in some cases. This aggressive environment leads to increased concern over the potential for corrosion and the impact on service life expectations. Service life analysis using LIFE-365 [24] software was utilised by the design engineers to see if the mix design proposed could meet the desired 100 year service life. Addition of an OCI admixture increases chloride threshold and slows corrosion rates once initiated. The chloride threshold increases from 0.05% (control) to 0.18% (OCI), and the propagation time increases from 6 years (control) to 30 years (OCI) to represent the reduced corrosion rate compared to a control. Table 3 shows the estimated construction cost of the building, the cost to add an amine carboxylate based organic corrosion inhibitor to the substructure of the building, as well as the expected service life of the substructure with and without the inhibitor addition. In this scenario, service life is defined as the time to first repair for the structure.

Table 3. Service Life & Cost Estimates – Princess Tower

Item	Cost (USD)
Construction Cost	\$188,000,000
Additional Cost OCI in substructure	\$136,000 (0.07% of total cost)
LIFE-365 Service Life no OCI	48 Years
LIFE-365 Service Life with OCI	103 Years

Further analysis was done by the supplier to estimate the return on investment (ROI) that could be gained by delaying costly repairs/maintenance on the substructure. Given that the building contains 763 rental units, calculation estimates

were done to consider anticipated revenue, minus building management, licensing, insurance, maintenance, and other costs over the design life of the structure. These calculations can be seen in Table 4.

Table 4. ROI Estimates (USD) – Princess Tower

Year	1	10	48	100
Revenue	\$28,066,757	\$33,542,373	\$71,186,938	\$199,346,790
Building Management	\$2,079,022	\$2,712,653	\$8,340,820	\$38,792,200
Licensing & Insurance	\$3,754,768	\$4,106,535	\$5,993,600	\$10,055,396
Others	\$3,754,768	\$4,106,535	\$5,993,600	\$10,055,396
Total Cost	\$9,588,559	\$10,925,722	\$20,328,019	\$58,902,991
Net Profit	\$18,478,198	\$22,616,651	\$50,858,920	\$140,443,799
Return on Investment	9.8%	12.0%	27.1%	74.8%
Present Value @ 3% discount rate	\$18,478,198	\$16,828,912	\$12,307,798	\$7,307,690
ROI	\$18,478,198	\$174,387,898	\$724,175,783	\$1,224,731,446

One can see that the ROI breaks even approximately 10.5 years into the life of the building. At 48 years, the property would have generated an additional \$536,175,783 in profit over the initial building cost. Extending the life to 100 years with an OCI inhibitor, generates an additional \$1,036,731,446 in profit – 93% more profit for an additional 0.07% in construction costs.

3.3 Vachon Bridge, Montreal, QB, Canada

The Vachon Bridge is a structure that is north of Montreal that carries Highway 13 over the Mille-Îles River [27]. The project was a study to determine the performance and factors affecting the effectiveness of corrosion inhibitors in bridge deck repairs. Corrosion inhibiting systems studied consisted of concrete admixtures, epoxy coated reinforcement, and/or concrete sealers/coatings. The consortium involved was formed by the National Research Council of Canada with other interested groups including owners and manufacturers.

At the end of the ten year study, an inorganic admixture, calcium nitrite (CNI), dosed at 20 L/m³ was ranked as best performing overall, followed by an organic corrosion inhibitor, dosed at 0.6 L/m³. It was noted that all systems had higher corrosion rate readings in areas of the structure that experienced vertical shrinkage cracks shortly after construction. The five year interim report presented results of field testing that included corrosion rate information. Where there was cracking, the OCI admixture (Span F) had the lowest corrosion rate of all methods tested, while the CNI (Span H) admixture performed worse than the control span (Span 21), Figure 14 (Corrosion Rate of Reinforcement, Cracks) [28].

Field Corrosion Tests \ SPANS	21*	12	A	B	C	D	E	F	G	H	Units
Half-cell potential of reinforcement (median value, June 2001)	-415 (4)	-453 (8)	-374 (1)	-420 (5)	-405 (3)	-420 (5)	-453 (8)	-487 (10)	-380 (2)	-428 (7)	mV
Corrosion rate of reinforcement, no cracks (average value, June 2001)	0.200 (5)	0.005 (1)	0.125 (2)	0.230 (8)	0.145 (3)	0.260 (10)	0.185 (4)	0.200 (5)	0.240 (9)	0.220 (7)	$\mu\text{A}/\text{cm}^2$
Corrosion rate of reinforcement, cracks (average value, June 2001)	0.260 (5)	0.195 (2)	0.200 (3)	0.375 (8)	0.270 (7)	0.410 (9)	0.230 (4)	0.180 (1)	0.500 (10)	0.263 (6)	$\mu\text{A}/\text{cm}^2$
Potential of rebar ladders, top bar (average value, June 2001)	-570 (4)	-630 (8)	-670 (1)	-620 (5)	-623 (3)	-515 (7)	-640 (9)	-600 (5)	-510 (2)	-360 (1)	mV
Potential of rebar ladders, 2nd bar (average value, June 2001)	-490 (5)	-460 (3)	-540 (8)	-520 (6)	-570 (9)	-607 (10)	-520 (6)	-480 (4)	-400 (2)	-333 (1)	mV
Corrosion rate of rebar ladders, top bar (average value, June 2001)	0.375 (5)	0.290 (3)	0.620 (7)	0.970 (9)	0.390 (6)	0.200 (2)	0.870 (8)	0.360 (4)	2.100 (10)	0.135 (1)	$\mu\text{A}/\text{cm}^2$
Corrosion rate of rebar ladders, 2nd bar (average value, June 2001)	0.250 (8)	0.200 (6)	0.400 (10)	0.120 (3)	0.100 (1)	0.240 (7)	0.163 (5)	0.110 (2)	0.270 (9)	0.130 (4)	$\mu\text{A}/\text{cm}^2$
Chloride content at 25-50 mm (average value, June 2001)	0.32 (10)	0.19 (4)	0.20 (6)	0.22 (7)	0.15 (2)	0.195 (5)	0.12 (1)	0.23 (8)	0.26 (9)	0.15 (3)	%
Chloride content at 50-75 mm (average value, June 2001)		0.03 (5)	0.04 (8)	0.03 (6)	0.03 (3)	0.037 (7)	0.02 (2)	0.02 (1)	0.05 (9)	0.03 (3)	%
OVERALL RANKING:	5.8	4.4	6.1	6.4	4.6	6.4	5.2	4.4	6.9	3.7	

Figure 14. Field Performance Ranking of Corrosion Inhibiting Systems Used on Vachon Bridge. Span 21 is Control, Span 12 is Epoxy Coated Reinforcing Steel, Span F is OCI Admixture, and Span H is Calcium Nitrite Admixture

4. REPAIR APPLICATIONS

Organic Corrosion inhibitors can also provide protection to existing concrete structures through topical coating or repair mortar application. Products in this category generally come in the forms of A) Pure inhibitor solution to be applied on to the concrete surface – the inhibitors will then be transported to embedded steel rebar via capillary infiltration and vapour phase diffusion; B) Hydrophobic sealer with OCI component – this approach protects by reducing ingress of corrosive species while simultaneously forming a barrier layer on the rebar surface though the action of the OCI molecules; and C) OCI containing repair mortar that supplies the rebar with the migrating corrosion inhibitors. When comparing surface treatments, generally the water based OCI solutions (Example A above, “pure inhibitor”) contain 2-3 times more inhibitor than the water repellent versions with OCI added in (Example B above, “hydrophobic sealer + inhibitor”), .

Several OCI topical treatments are certified to meet NSF/ANSI 61 – 2016 *Drinking Water System Components -Health Effects* [17] requirements, meaning they are safe for use at their recommended dosage rates in structures containing potable water.

OCI topical treatments have also been tested to USBR M-82 [12], EIS [11], modified ASTM G109 [29], XPS and SEM [15] to show their corrosion protection capabilities as well as their migrating capabilities.

USBR M-82 Protocol was run on three OCI topical treatments. Treatment A was a 20-25% OCI in water solution, followed by a 40% silane water repellent; Treatment B was a 40% silane water repellent containing 2.5-10% OCI; and, Treatment C was 100% silane water repellent containing 2.5-10% OCI. The testing was done according to Configuration A of the slabs in the Protocol (used when the repair treatment is to be surface applied only), and the OCI repair treatments were applied after 10,000 Coulombs of macrocell current had been reached. This variation of the test is used if the repair method does not depend upon the amount of chloride present at the reinforcing bars, or protection at a higher initial chloride content is to be demonstrated [30].

Table 5 shows chloride levels at time of repair and at the end of testing. All of the control slabs showed a statistically significant (95% CL) increase in chloride levels from the time of repair to the end of testing. However, for the treated slabs there was no a statistically significant change (less stringent 90% CL) in chloride levels from the time to repair to the end of testing. The topical treatments used were expected to decrease the ingress of chloride, and that is evident in the data shown in Table 5.

Table 5. Average Chloride Values (ppm) Before Repair and At Test End (USBR M-82 Protocol Configuration A)

	Control		Treatment A (20-25% OCI in water + 40% Silane)		Treatment B (40% Silane + 2.5-10% OCI)		Treatment C (100% + 2.5-10% OCI)	
	Repair	End	Repair	End	Repair	End	Repair	End
Average	2604	3520	2378	2620	2706	2600	2744	2540
SD	181	264	268	223	399	190	147	171
Sp	242							
95% CL	230							
90% CL	190							

Table 6 shows the total crack length and average width following the USBR M-82 Protocol Configuration A testing. Cracking is due to corrosion of the reinforcing steel. The 95% confidence limits in the table do not overlap, so there is a statistically significant reduction in crack length and area for the treated slabs versus the control slabs.

Table 6. Crack Length and Area by Treatment (USBR M-82 Protocol Configuration A)

	Length (mm)		Area (mm ²)	
	Average	SD	Average	SD
Control	2315	311	284	78.55
Treatment A (20-25% OCI in water + 40% Silane)	915	820	106.06	101.24
Treatment B (40% Silane + 2.5-10% OCI)	689	341	75.10	44.496
Treatment C (100% + 2.5-10% OCI)	465	527	51.92	61.63
Sp	539		74	
95% CL	511		71	

At the end of testing, slabs were excavated and bars visually inspected for corrosion damage. The galvanic corrosion between the top bars and the wire mesh (macrocell current) is measured and integrated over time. Based on macrocell data, the majority of the corrosion on embedded bars occurred before the OCI treatments were made to the slabs. Figure 15 shows the average integrated corrosion current (coulombs) after OCI repair treatments were applied.

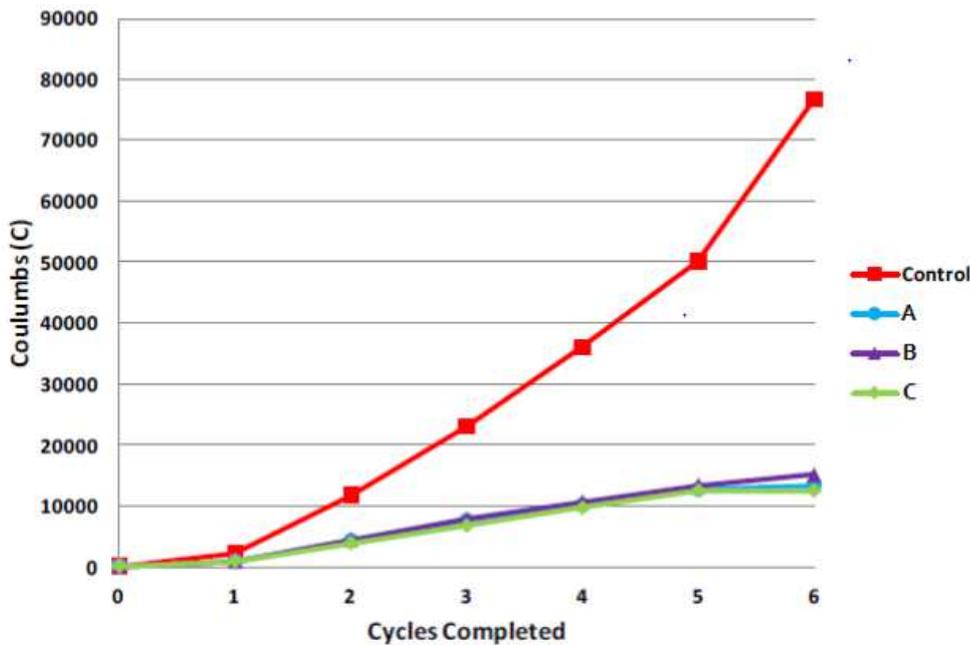


Figure 15. Average Integrated Current (After OCI Treatments) (USBR M-82 Protocol Configuration A Test)

Modified G109 testing was conducted on water based OCI inhibitors, Figure 16. The test lasted 1500 days and included over 200 cycles of 3.5%NaCl ponding/drying. It was found that the water based OCIs tested had increased polarisation resistance (R_p) (60-80 kohms*cm² with increasing trends) in the OCI coated concrete compared to controls, for both low density (2.08 g/cm³, 0.65 w/c) and high density (2.40 g/cm³, 0.35 w/c ratio) concretes. The untreated not only had lower R_p values, but also decreasing trends, refer to Figure 17.

Examination of the embedded steel rebar after corrosion tests showed no corrosion attack for the OCI treated concrete samples, while un-treated concrete showed corrosion. X-ray photoelectron spectroscopy (XPS) and depth profiling (XPS scan at different penetration depths from the rebar surface) confirmed that the inhibitor had reached the rebar surface in less than 150 days. Depth profiling showed an amine-rich compound on the rebar surface that corresponded with the increase in R_p and improved corrosion protection for the OCI treated steel rebar even in the presence of chloride ions, refer to Table 7. Based on the measured reinforcement corrosion rates in this laboratory study the life expectancy of a reinforced concrete structure can be improved by more than 40 years [31].



Figure 16. Modified ASTM G109 Test Set-up (continuous submersion)

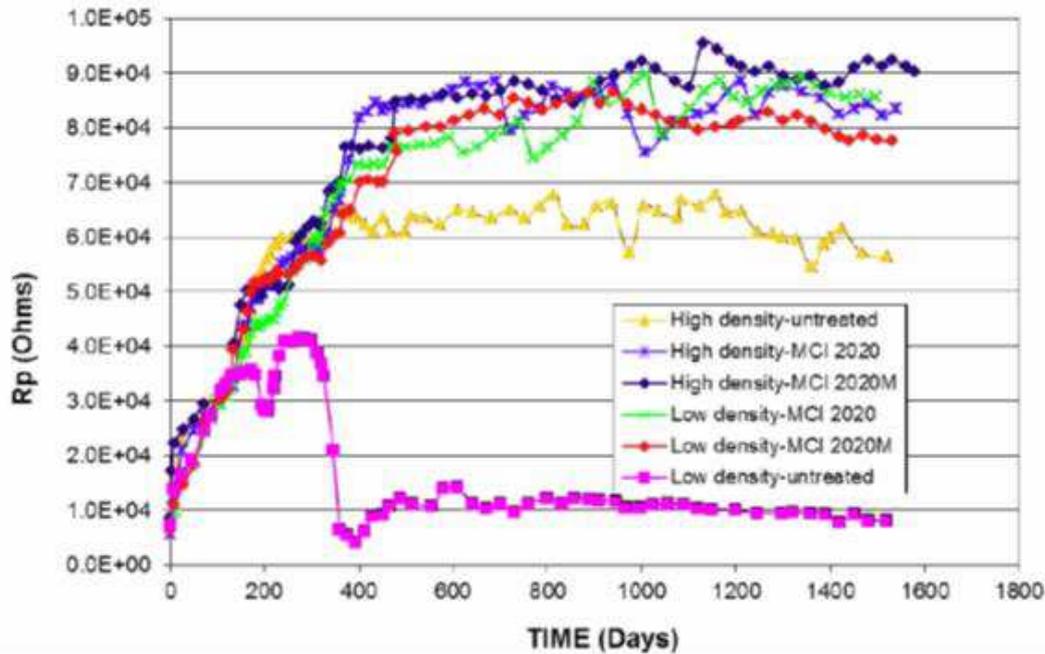


Figure 17. Comparison in Polarization Resistance (Rp) in Low & High Density Concretes – OCI Treated vs. Untreated

Table 7. XPS Analysis and Spectrum of the Rebar Surface, Untreated and OCI Treated Concrete after 1500 Days in 3.5% NaCl

Peak	Atomic Conc. (%)	Mass Conc. (%)	Atomic Conc. (%)	Mass Conc. (%)
	Untreated	Untreated	OCI	OCI
Fe 2p	0.87	3.32	0.08	0.3
O 1s	30.19	33.06	31.4	35.91
C 1s	62.48	51.37	59.43	48.12
Si 2p	4.72	9.08	1.26	4.14
Cl 2p	0.84	2.04	1.11	2.81
N 1s	0.74	0.71	5.64	5.71

4.1 Pentagon Renovation

The Pentagon is considered the world’s largest low-rise office building. It is a five-story concrete structure with five distinct rings. Above the third floor, each ring is separated by an exterior space known as a lightwell. The Pentagon Renovation was a 10 year renovation project to repair the exterior, concrete walls in the lightwell areas. The project began in 2002 and finished ahead of schedule in 2011. It is estimated that there was 23,225m² of spalled concrete repair, and almost 93,000m² treated with a topically applied OCI [32].

PENREN, the governing agency for the renovations, dictated that the repairs must last 50 years, which meant a detailed inspection was completed by the consulting engineer, and extensive repair methods were considered. Concrete testing completed during the inspection revealed that the walls were carbonated to a depth of 76-102 mm with the outer layer of reinforcing steel approximately 13 mm from the surface, refer to Figure 18. Furthermore, the surface of the concrete was very porous as a direct result of the rough cut lumber used for forms during original construction of the building. This environment caused corrosion on embedded reinforcing steel and deterioration of the concrete walls as can be seen in Figure 19.



Figure 18. Carbonation Testing on Lightwell Wall



Figure 19. Example of Corrosion on Lightwell Walls Prior to Repair

The repair program designed by the consulting engineers was to follow ICRI Technical Guidelines for Repair [33]. There was also a requirement that the new repairs not be visible from a distance of 9m. All delaminated, spalled and cracked concrete related to corrosion of the steel and any unsound concrete around rebar was removed to sound concrete. Exposed bar was mechanically cleaned to remove all existing rust and scale. Reinforcing steel was relocated to provide, at a minimum, 38 mm of concrete cover.

Once cleaned, exposed reinforcing steel was treated with a zinc rich, acrylic based, corrosion inhibiting coating and new proprietary polymer modified cementitious patching material was installed. Patch material and forms were designed to match the existing façade in colour and texture. When concrete repairs were completed, all surfaces of the building were cleaned and treated with a water based OCI formulated for vertical and overhead applications to help compensate for the loss of alkalinity around the steel (due to carbonation) and reduce corrosion rates. This treatment was then followed by a 100% silane sealer for water repellency, and then a mineral based, potassium silicate coating to create a uniform finish to the building exterior, Figure 20 and Figure 21.



Figure 20. Before and After Coating Application



Figure 21. Completed Lightwell area

During the repair years, linear polarisation readings were taken in selected areas of the lightwells using a GalvaPulse device [22], Figure 22, every spring and fall to ensure that corrosion was being mitigated with the repairs and that the design life would be achieved. A wire (cable) was installed on embedded reinforcing steel during the repairs and run outside of the wall to use to attach the GalvaPulse to for future readings. Since the silane and silicate surfaces changed the resistivity of the concrete surface, small test areas were left free of coating to allow for the taking of concrete resistivity readings in the future. An example can be seen in Figure 23.



Figure 22. GalvaPulse Readings Prior to Repair



Figure 23. Testing Site Lightwell Walls After Repair Completion

Estimated time to visible damage based on readings can be found in Table 8. This comes from the GalvaPulse Manual [22]. Table 9 shows a set of readings from before repair and one from the same area three months after repair. As can be seen, this area went from readings in the “low to moderate range” of corrosion (i.e. “5.8-174 $\mu\text{m}/\text{year}$) to the “passive range” (i.e. $<5.8 \mu\text{m}/\text{year}$) of corrosion. During the 10 years onsite, the readings never exceeded $5 \mu\text{A}/\text{cm}^2$ ($58 \mu\text{m}/\text{year}$), indicating no visible deterioration should be seen during the upcoming 10 years.

Table 8. GalvaPulse Time to Visible Damage

Corrosion Current, $\mu\text{A}/\text{cm}^2$	Corrosion Rate, $\mu\text{m}/\text{year}$	Corrosion Level	Time to Visible Deterioration
< 0.5	< 5.8	Passive	N/A
0.5 to 5	5.8 to 58	Low	> 10 years
5 to 15	58 to 174	Moderate	3 to 10 years
> 15	>174	High	< 2 years

Table 9. GalvaPulse Readings Before & After Repair

Before Repair – June 27, 2005, Temperature ~ 26C, Dew Point ~ 23C, Barometric Pressure~ 1023 hPa		After Repair – September 25, 2005, Temperature ~ 24C, Dew Point ~ 10C, Barometric Pressure~ 1015 hPa	
Corrosion Current, $\mu\text{A}/\text{cm}^2$	Corrosion Rate, $\mu\text{m}/\text{year}$	Corrosion Current, $\mu\text{A}/\text{cm}^2$	Corrosion Rate, $\mu\text{m}/\text{year}$
10.9000	126.44	0.0957	1.1
2.8133	32.63	0.0808	0.94
0.1552	1.80	0.0927	1.08
0.9165	10.63	0.1130	1.31
0.6977	8.09	--	--
June Average	32.41	September Average	1.11

4.2 C.A.T. Consorci D'Aigües De Tarragona Pipeline Repair

Consorci D'Aigües De Tarragona (C.A.T.) is the non-profit water consortium authority responsible for high pressure drinking water in the Tarragona region of Spain [34, 35]. C.A.T. had a 30 year old network of prefabricated, reinforced concrete pipes that were carbonated and experiencing corrosion problems. Restoration began in 2014 with damaged concrete removal and repairs completed using a cementitious bonding agent on exposed bar and repair mortars that contained OCI inhibitors certified for use on potable water structures. More than 7,000m² of repair were completed in this manner. After repairs were finalised, the entire length of the pipe was treated with a topically applied OCI (more than 13,000m²), Figures 24 and 25. Testing done after application showed that corrosion rates had dropped and the authorities were pleased with the results. Due to the success of the repair, C.A.T. has since implemented the use of OCI admixtures on the construction of their new potable water reservoirs.



Figure 24. Spraying OCI onto the Pipeline



Figure 25. View of Pipeline Treated in Region

5. SPECIALTY APPLICATIONS

OCI inhibitors have also been developed to help with corrosion issues in unique construction environments. Use of powder based OCI inhibitors on post tensioned structures has been occurring for more than 10 years in the United States to provide temporary protection to PT cables prior to grouting. Researchers at Penn State confirmed that the powder based OCI fogged into PT cable construction provided a significant reduction in corrosion compared to control samples in long term laboratory-based corrosion studies (one year of saltwater exposure) and that the OCI did not significantly affect the bond of grout injected after their application [36]. Based on this data and additional research, a novel use of OCI was created for the Severn Bridge Structure in the United Kingdom.

5.1 Severn Bridge, United Kingdom

The Severn Bridge is a 988 m span suspension bridge that carries the M48 motorway over the Severn River between Bristol and South Wales, Figure 27 [37]. It was built in 1966 and featured a number of unique innovations such as inclined hangers and use of streamlined box girder deck construction. Inspections completed in 2006-07 found that the PT cables were corroded and had reduced structural strength. Following this investigation, an acoustic monitoring system and dry air injection system were installed on both main suspension cables to control the deterioration. The purpose of the dry air injection system was to reduce the relative humidity (RH) within the cables to <40%, an accepted percentage to prevent corrosion of metals [37].

To provide additional corrosion protection to the PT cable wires during the initial period of moisture reduction—when corrosion rates could increase as oxygen became more available—and to provide a back-up in the event that the dehumidification system went out of service (i.e. for maintenance), an organic corrosion inhibitor system was developed for introduction into the dry air stream. Concerns over fogged OCI powders blocking air voids in the cables, water based systems being unsuitable when looking for moisture reduction to reduce corrosion rate, and solvent based systems being incompatible with the cable wrap and RH probes, meant a different approach had to be used in development of the OCI.

For these reasons, a powder version OCI, contained in a vapour permeable pouch, was developed to introduce pure inhibitor vapour into the system using the dehumidification air stream as the carrier. This ensured a sufficient level of inhibitor would be present within the air voids to protect exposed metal surfaces while avoiding the risks of blockage by solid or liquid material. Because the protective inhibitor layer is at a molecular level, it has no influence on clearances and had only a minimal effect on other physical properties. Testing showed that use of the inhibitor caused a small increase in wire-to-wire friction at low contact pressure and no significant frictional effect at higher loads. Further testing was carried

out to confirm the inhibitor would not adversely affect other components in the system including cable wraps, sealants, and probes, Figure 26.

The OCI system manufacturer also developed an inhibitor detection kit that is used to confirm the delivery system is effective in the field. The detection method involves a colour change of the indicator from blue to red, making it possible to confirm the presence of the inhibitor on the surface of the wires, even deep within the cable, by using swabs containing the indicator solution. This detection method is used in ongoing field inspections to ensure presence of the inhibitor.

The consulting engineers for the project [22] also developed monitoring criteria and systems for future management of the bridge to comply with the principals in the British BD79/13 Standard - *The Management of Sub-standard Highway Structures* [38]. This involves visual inspections by bridge operators who record any observations that may be relevant to the condition of the cables, including: damage, evidence of entrapment of water under the cable sleeve, and broken wrapping or main cable wire; installation of sensors that measure relative and absolute humidity, temperature, flow, pressure, and corrosion rates at the inlet and exhaust locations of the air drying system; and acoustic emission monitoring which determines a confirmed wire break based on the characteristics of wire break acoustic signatures.

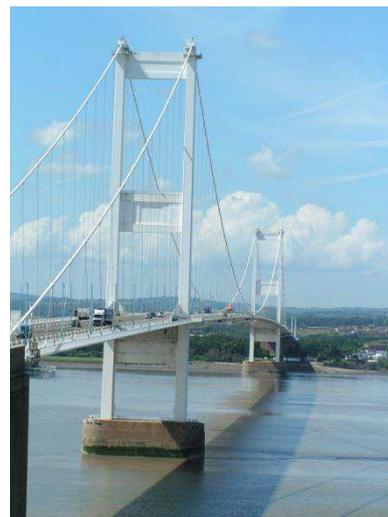
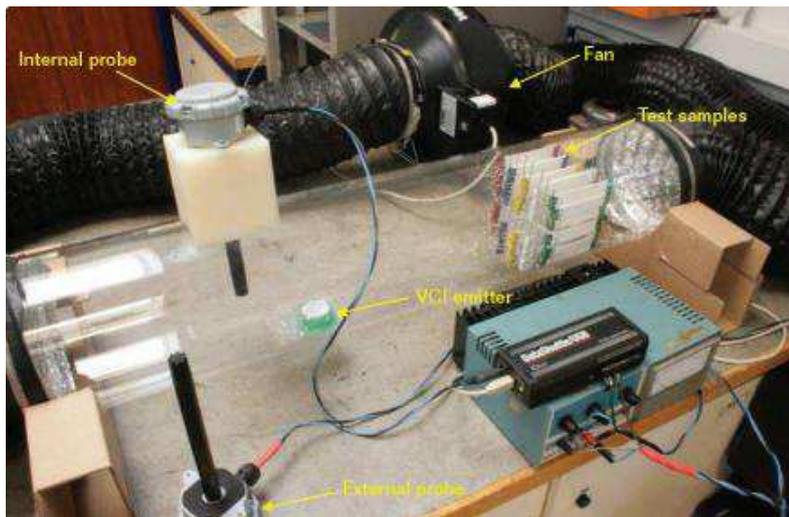


Figure 26. Laboratory Testing to Confirm OCI Compatibility with Dehumidification System **Figure 27. View of Severn Bridge**

The dry air injection system was installed during 2008 and the drying phase had been completed by September 2009, as demonstrated by the achievement of a RH of 40%. Acoustic emissions taken before installation of the dry air injection system represented wire breaks totalling 0.4% of the total number of wires in the cables. Acoustic emissions during the cable drying out period were reduced, indicating ~15 breaks per year or 0.1% per year, and level of acoustic emissions for wire breaks after drying out was reduced to less than 0.04%.

6. CONCLUSIONS

Organic corrosion inhibitors have been used in the construction industry for more than 30 years. Effectiveness has been demonstrated on many types and ages of construction.

OCI Admixtures have been tested to meet the requirements of ASTM C1582 Standard Specification for Admixtures to Inhibit Corrosion in Chloride Exposed Environments, and have demonstrated their ability to significantly increase expected service life of structures with a single application during construction.

OCI topical treatments used for repair have also been tested extensively and shown to reduce corrosion rates significantly even in the presence of existing chlorides and carbonation. Long term study on repair projects has demonstrated their ability to extend the useful life of existing structures.

Finally, use of OCI is not limited to concrete admixtures or topical treatments, but can also be done in novel applications as seen on the post tensioned tendons and cable ducts of the Severn Bridge rehabilitation.

7. ACKNOWLEDGMENTS

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9. AUTHOR DETAILS



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