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Natural marine exposure results for reinforced concrete slabs with corrosion inhibitors

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Abstract

Reinforced concrete slabs were cast with a concrete cover of 20 mm. The water-to-cement ratio was 0.40, and the concrete slabs were either uncracked or precracked. A simulated crack 0.2 or 0.4 mm wide was formed transverse to the axis of the reinforcing bar during the casting. Two types of commercial corrosion inhibitors were added to concrete mixtures for corrosion protection. Slabs were placed about 1 m below high tide at the Treat Island, Maine, USA, natural marine exposure site. The specimens were visually inspected and the corrosion rates were measured annually using the linear polarization technique. Some of the concrete slabs were broken open after 12 months of exposure and corrosion damage was evaluated. Water-soluble chloride content analysis was performed at this time. After three years of exposure, it was found, that both corrosion inhibitors were effective in reducing the corrosion rate for uncracked concrete slabs, but relatively ineffective in preventing localised corrosion of reinforcing steel in the crack area for precracked concrete slabs.

Keywords: Chlorides; Corrosion; Cracking; Inhibitors; Marine exposure; Reinforced concrete

1. Introduction

Due to the inherent durability of reinforced concrete, it is often used in severe marine and de-icing salt environments and it performs well until deterioration due to the corrosion of steel reinforcement begins. The corrosion of reinforcing steel in concrete is a major problem today, as an ageing infrastructure requires significant engineering maintenance efforts [1].

When steel in concrete corrodes, the cross section of the reinforcing bar becomes smaller, thus reducing the load carrying capacity of the reinforced concrete member. The volume of corrosion product exerts pressure on the concrete resulting in spalling of the concrete cover and directly exposing the steel to the corrosive agents. This leads to the loss of a structure's load carrying capacity and to the need for repairs.

Cracks form in the concrete during hardening process and other cracks develop when it is exposed to environmental gradients or service loads; these cracks have been shown to have a significant influence on the corrosion of the reinforcement.

The risk of cracking for modern concrete mixtures with a high content of normal or high-early strength Portland cement is heightened due to increases in drying shrinkage, thermal shrinkage, and elastic modulus along with a reduction in the creep coefficient, and, as a result, recently built concrete bridge decks often show transverse cracks even before the structures are a month old [2,3].

It was suggested, that crack width does not play an important role for significant corrosion to occur and corrosion protection must be assured by the use of good quality concrete and suitable cover depth [4,5]. On the other hand, crack width was reported to have a significant effect on corrosion in low w/c concrete [6].

Calcium nitrite can provide more than an order of magnitude reduction in corrosion rate for specimens with water-to-cement ratios higher than 0.50 [7]. In a recent study [8], the calcium nitrite based DCI corrosion inhibitor performed the best of several corrosion inhibitors tested and resisted active corrosion even when the concrete was above normal chloride threshold levels.

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The role of calcium nitrite is to prevent the continuation of the chloride-induced iron dissolution by reacting with ferrous ions. For concrete exposed to the chloride-laden environment, Cl^-, NO_2^-, and OH^- anions will compete to combine with ferrous cations, thus, the chloride/hydroxyl and chloride/nitrite ion ratios are important parameters that affect corrosion protection. As these ratios increase, as in the crack area, the probability of chloride ions forming complexes with iron ions also increases and, as a result, the initiation of localised corrosion can be expected [9,10]. Information on the performance of calcium nitrite corrosion inhibitor (CNI) in cracked concrete is contradictory. It was reported [11] that the addition of calcium nitrite to a good quality concrete ($w/c = 0.4$ and 38 mm cover) provided good corrosion resistance in the presence of cracks of 0.2 mm in width, but other studies [12,13] showed that calcium nitrite does not perform well in cracked concrete.

Organic-based corrosion inhibiting admixtures were introduced in an attempt to compensate for the limitations of anodic inhibitors. They are referred to as “adsorption corrosion inhibitors”, because they adsorb onto the surface of the steel and suppress both the cathodic and anodic reactions on the metal surface. It was reported that adsorption appears to be general over the entire surface rather than at specific anodic or cathodic sites, and both reactions tend to be retarded, thus providing corrosion protection even if the concrete cracks [14,15].

The economic loss and damage caused by corrosion of steel in concrete makes it the largest infrastructure problem for industrial countries. Repair costs for reinforced concrete structures run at values in excess of $200/m^2 of exposed surface area, not accounting for loss of use or traffic control [16]. Thus, the effective application of corrosion prevention measures in reinforced concrete structures is critical to protect public safety and avoid costly repairs and downtime.

The objective of this study was to evaluate the effectiveness of two corrosion inhibitors in uncracked and precracked reinforced concrete slabs with a water-to-cement ratio of 0.40, when placed in a natural marine environment.

2. Test program

2.1. Materials and specimen preparation procedures

Concrete with a maximum aggregate size of 12.5 mm, water-to-cement ratio of 0.40, and an air content of 6% was used. Canadian Standard Association Type 10 Portland cement was used for concrete mixtures and it’s physical properties and chemical composition are given in Table 1. The concrete mixture proportions are as listed in Table 2.

Table 1
Chemical and physical analysis of CSA Type 10 Portland cement

Chemical composition	
Silica (SiO_2)	20.6%
Alumina (Al_2O_3)	5.3%
Iron oxide (Fe_2O_2)	2.2%
Calcium oxide, total (TCaO)	63.1%
Magnesium oxide (MgO)	1.4%
Sulfur trioxide	4.1%
Loss on ignition	2.5%
Free lime (FCaO)	0.6%
Equivalent alkali (as Na_2O)	0.83%
C_3A	10.3%
Physical analysis	
Fineness 45 μm sieve	87.1% passing
Blaine	385 m^2/kg
Setting time—initial	155 min
Autoclave expansion	0.01%
Compressive strength at 3 days	26.0 MPa
Compressive strength at 7 days	31.1 MPa
Compressive strength at 28 days	38.4 MPa
Sulphate expansion	0.011%

The nominal dimensions of the concrete slabs were 55 × 230 × 300 mm. To accelerate corrosion, concrete cover was 20 mm. All concrete slabs contained two duplicate straight 15.9 mm diameter steel bars cast in concrete. For the purpose of electrochemical measurements, a stainless steel rod was cast in the concrete slab to serve as a counter electrode. The diagram of a typical concrete slab is given in Fig. 1a.

In order to induce a transverse crack of a certain width penetrating to the rebar level, the mold was divided into two halves. The first half of the specimen was cast and de-molded on the next day, and a rubber sheet with a dimension of 150 × 300 mm was used to form a crack and the remaining part of the mold was then filled. The rubber sheet was slightly greater than 0.2 (0.4) mm thick and it was stretched so that the rubber provides a space of 0.2 (0.4) mm between the two separate castings of concrete. Also holes in the rubber, accommodating rebars and a counter electrode, were cut slightly smaller

Table 2
Concrete mixture proportions

Mixture number	1	2	3
w/c	0.40	0.40	0.40
Cement (kg/m^3)	505	505	505
Coarse aggregate (kg/m^3)	890	890	890
Fine aggregate (kg/m^3)	610	610	610
Water (kg/m^3)	202	202	202
A/E adm. ml/10 kg of cement	6	5	5
HRWA (l/m^3)	–	–	3
Set retarder ml/100 kg of cement	–	–	125
Corrosion inhibitor (l/m^3)	–	5 l (OCI)	25 l (CNI)

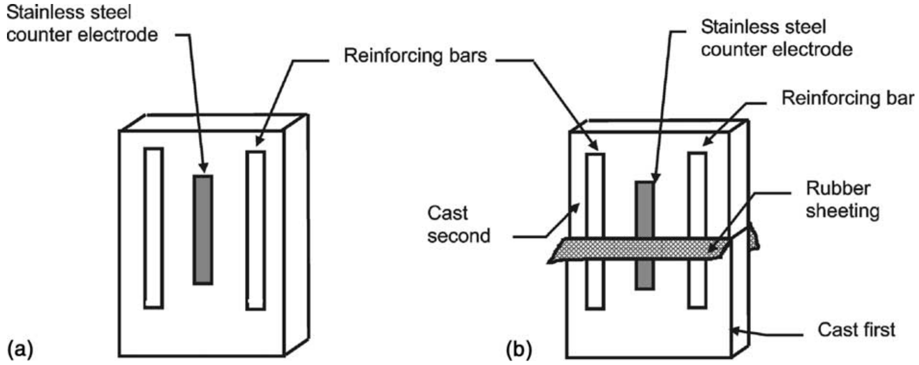

Fig. 1. Diagram of uncracked and precracked specimens with two embedded bars: (a) uncracked and (b) precracked.

than the diameter of the bar and, when the rubber was stretched to a thickness of 0.2 or 0.4 mm, holes had the appropriate diameter to fit tightly around the rebar. The diagram of the casting procedure for precracked specimens is given in Fig. 1b. After casting, concrete specimens were moist cured for 28 days at 23 ± 2 °C at a relative humidity of 95% prior to being exposed to chlorides. For each mixture duplicate concrete specimens were made.

2.2. Corrosion inhibitors

The details on the corrosion inhibitors used in this testing program are given in Table 3. Mixture #1 was a control mixture and did not contain any corrosion inhibitor. Commercial organic corrosion inhibitor (OCI) was added to concrete mixture #2 during the mixing process. The addition amount was 5 l/m^3 of concrete that was based on the manufacturer's recommendations. According to manufacturer's information, this OCI should provide dual protection in terms of corrosion of reinforcement in concrete. Firstly, it coats the pores of the concrete matrix thus slowing the ingress of chlorides and moisture into the concrete, and secondly, it provides additional protection by adsorbing onto the reinforcing steel to form a thin protective film that suppresses both the cathodic and anodic reactions on the metal surface.

The commercial anodic calcium nitrite-based corrosion inhibitor (CNI) that was also used in this study contains a minimum 30% of calcium nitrite. The inhibitor was added to the concrete during the mixing process for concrete mixture #3. According to the manufacturer, this anodic inhibitor stabilises the protective oxide film layer on the steel surface and covers defects in the protective oxide film. When added to the concrete in sufficient quantity, it should maintain an active corrosion controlling system within the concrete matrix. In this study, 25 l/m^3 of the CNI corrosion inhibitor was used as recommended by the manufacturer. The addition of corrosion inhibitors did not have any noticeable effect on the workability of fresh concrete and on the concrete's compressive strength.

Table 3
Details on types of corrosion inhibitors used in the testing program

Type of inhibitor	Active ingredient
OCI—mixed anodic/cathodic organic corrosion inhibitor	Combination of amines and esters in water
CNI—anodic corrosion inhibitor	30% calcium nitrite

2.3. Experimental procedures

Reinforced concrete slabs were installed on the beach at a natural marine environment site at Treat Island in the Bay of Fundy, Maine. Weather conditions at Treat Island are typical for North Atlantic with an average of 120 freezing and thawing cycles per year. Chemical analysis of seawater is typical for North Atlantic.

The slabs were placed about 1 m below the mean high tide level which provides the most severe exposure in terms of corrosion of embedded reinforcement as was confirmed by the results of previous testing [17].

The corrosion activity of the reinforcing steel bars in concrete slabs was monitored on an annual basis using the linear polarization resistance (LPR) technique. Electrochemical measurements were taken using the corrosion measurement system—CMS 105 (by Gamry Instruments Inc.) with concrete resistance (IR) compensation capability.

The corrosion system was installed in a portable computer enabling on-site measurements. The diagram of the LPR testing set-up is given in Fig. 2. The embedded reinforcing steel bars served as working electrodes, 316 SS stainless steel rod served as a counter electrode, and saturated calomel electrode was used as a reference electrode. The proportionality constant, B, in the calculation of the corrosion rate of the reinforcing steel was assumed to be 26 mV for active corrosion [18].

Visual examination of specimens was performed annually during the electrochemical testing procedure.

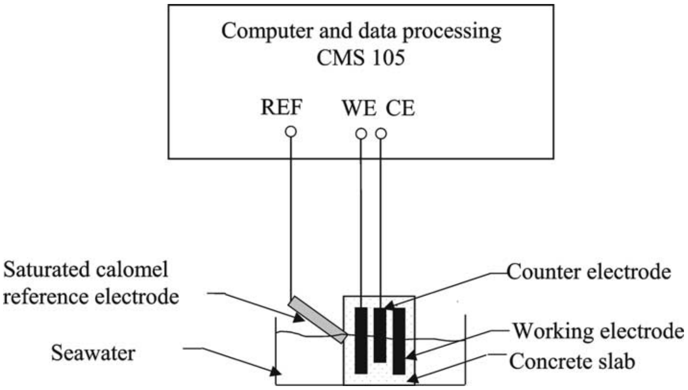

Fig. 2. LPR testing set-up.

After 12 months of marine exposure, one uncracked and one precracked slab for each concrete mixture were cut open along the reinforcing bars using a diamond concrete saw. The surface of the reinforcing steel was exposed and the extent of corrosion was evaluated. Water-soluble chloride-ion content profiles for the examined slabs were determined using the conventional titration procedure, and the results are given in Figs 3 and 4. For the cracked specimens, the sampling with a 20 mm drill was done at a distance of 10 mm from the crack.

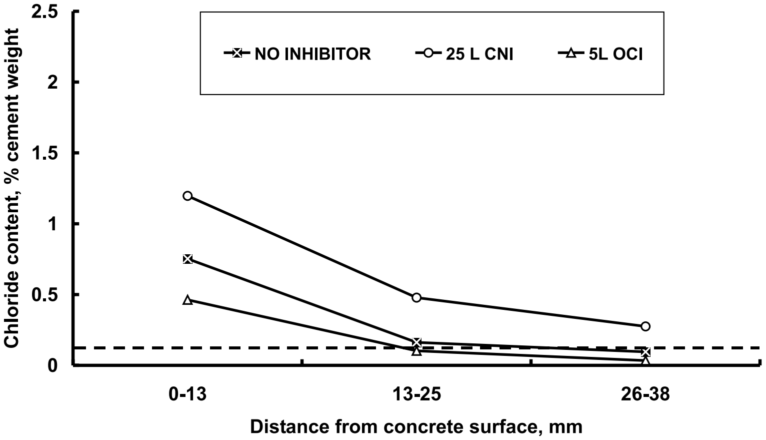

Fig. 3. Chloride profiles for uncracked specimens with corrosion inhibitors after 12 months of exposure.

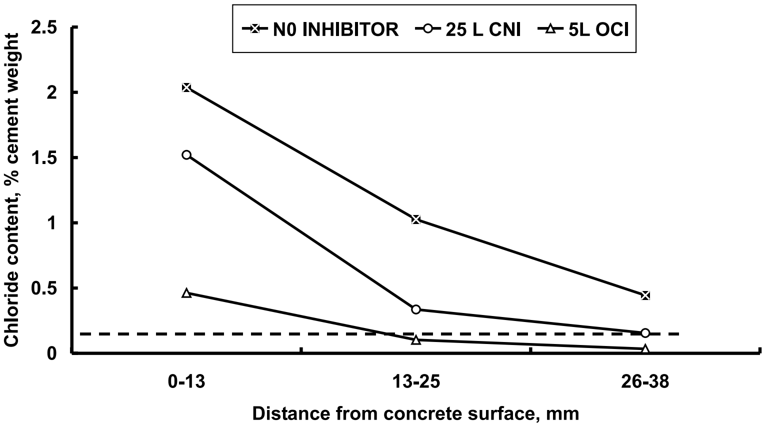

Fig. 4. Chloride profiles for precracked specimens with corrosion inhibitors after 12 months of exposure.

3. Results

3.1. Uncracked specimens

Results of the linear polarization testing for uncracked specimens are presented in Fig. 5. After two years of exposure, the values of calculated current densities were below 0.5 μA/cm^2, which is considered to be the transition limit from low to moderate corrosion rate [19]. When examined after 12 months of exposure, the surface of the reinforcing bars embedded in uncracked concrete slabs with and without corrosion inhibitors was free of corrosion products. After the third year of exposure, the corrosion current densities for both, control specimens and specimens with CNI corrosion inhibitor, became higher than 0.5 μA/cm^2 and, according to the adopted classification, shifted to the "moderate–high" range. However, the specimens with inhibitors showed lower corrosion rates than control specimens with the OCI corrosion inhibitor specimens being the lowest. In general, for all uncracked $w/c = 0.40$ concrete specimens, there were no visible signs of corrosion on the surface of the slabs after three years of natural marine testing.

3.2. Precracked specimens

Cracking had a significant effect on the corrosion rate of embedded steel for the $w/c = 0.40$ concrete as is evident in Figs 6 and 7. After only 12 months of exposure, the corrosion current densities for all precracked concrete slabs with 0.2 and 0.4 mm crack exceeded the moderate corrosion limit of 0.5 μA/cm^2. More than this, the corrosion current densities of specimens with corrosion inhibitors at 12 months of exposure exceeded those for control specimens and were significantly higher than the "high" limit of 1 μA/cm^2. This could be explained by a more pronounced effect of chloride ions on the initiation of corrosion process in the crack area for specimens with corrosion inhibitors. Steel embedded in

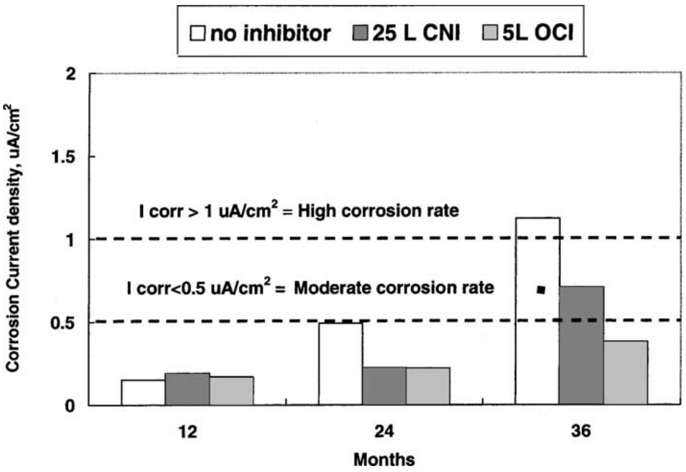

Fig. 5. Corrosion current densities for uncracked concrete slabs with corrosion inhibitors.

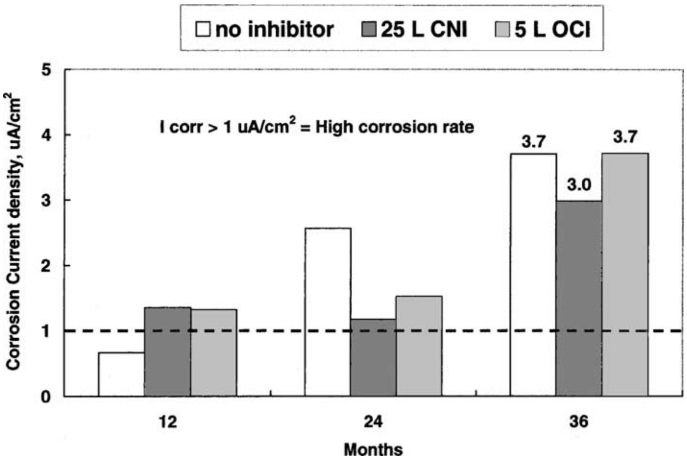

Fig. 6. Corrosion current densities for precracked concrete slabs with 0.2 mm crack.

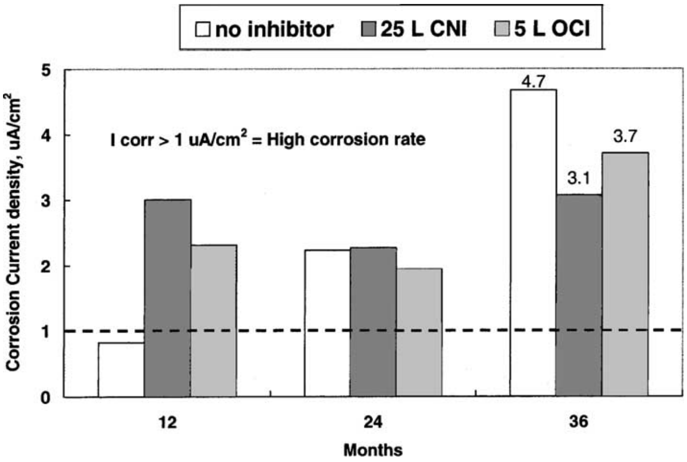

Fig. 7. Corrosion current densities for precracked concrete slabs with 0.4 mm crack.

the uncracked portion of concrete slabs with corrosion inhibitor apparently remained relatively more passive, than the steel in slabs without corrosion inhibitors, thus creating a more potent macrocell. A visual examination of the surface of steel reinforcement for specimens broken open after 12 months of exposure revealed localised pitting corrosion in the crack area for all precracked specimens examined, with pitting damage being more severe for specimens containing corrosion inhibitors.

After the second year of exposure, the corrosion rates for precracked slabs with corrosion inhibitors became closer to the corrosion rates for "no inhibitor" slabs. The plausible explanation could be the diminishing power of the corrosion macrocell, created between the steel sections in cracked and uncracked concrete, due to the general increase of the chloride content at the rebar level after the second year of exposure [20] and the consequent reduction of the area of passive steel. In general, after the second year of exposure, the corrosion current densities for all of the precracked concrete slabs became higher than 1 $\mu A/cm^2$, indicating an active corrosion process. At three years of exposure for precracked slabs with 0.2 and 0.4 mm crack the corrosion current density was correspondingly 3.7 and 4.7 $\mu A/cm^2$ for the control specimen, 3.0 and 3.1 $\mu A/cm^2$ for 25 l of CNI, and 3.7 and 3.7 $\mu A/cm^2$ for 5 l of OCI. As a result, after three years of exposure all of the initially transversely precracked reinforced concrete slabs exhibited corrosion-caused longitudinal hairline cracks along the embedded reinforcing steel.

3.3. Effect of crack width

Corrosion rate values recorded for uncracked specimens were always lower than for precracked specimens, as described above, which corroborates that cracks play an important role in the development of corrosion in 0.4 w/c ratio concrete as reported by Ohno et al. [6]. The results also suggest the easier degree of anodic dissolution in cracked sections of concrete. In this way, the crack section may reduce the accumulation of corrosion products around the steel surface resulting in less confinement of the anodic dissolution products. This should verify the hypothesis that anodic dissolution is an important factor in macrocell corrosion of concrete.

After 12 and 24 months of exposure, corrosion current density values for most of the 0.2 mm crack specimens were smaller than for 0.4 mm crack specimens, regardless if a corrosion inhibitor was used or not. Similar results were reported by Ohno et al. [27]. However, after 36 months, the corrosion values registered were essentially the same for both crack sizes. This may be due to the gradual depassivating of the steel bar when chloride ions penetrate the concrete cover.

4. Discussion

4.1. Chloride penetration and corrosion

The chloride-blocking effect of OCI corrosion inhibitor is evident from the chloride profiles in Figs 3 and 4. After 12 months of exposure in marine environment, only the specimens incorporating OCI corrosion inhibitor had a soluble chloride-ion content at the level of reinforcement (13–25 mm from the surface of concrete) that was below the threshold limit for corrosion initiation (0.15% of chloride by weight of cement, ACI 222R-9 [21]). These results are in good agreement with the reported beneficial effect of OCI in terms of reducing chloride permeability of concrete [22].

Chloride profiles for uncracked slabs with CNI corrosion inhibitor after 12 months of exposure were higher than for specimens without corrosion inhibitor. This result confirms reported higher chloride permeability of concrete with CNI corrosion inhibitor. In a recent study on the effect of calcium nitrite on compressive strength and chloride diffusion in high performance concrete [23] it was found that the addition of calcium nitrite influences the hydration process of cement paste. Apparently, calcium nitrite has the function of accelerating and stabilising the formation of the crystal phase of calcium hydroxide which leads to an increase in the micropore diameter in the hardened cement paste and thus to an increase in chloride permeability compared to concrete without inhibitor. It was also reported [24] that calcium nitrite can decrease the resistivity of concrete and also tends to increase concrete chloride permeability values.

For uncracked reinforced concrete slabs the addition of corrosion inhibitors was effective in reducing corrosion activity compared to the control "no inhibitor" specimens as can be seen in Fig. 5. The lowest corrosion rate observed for concrete slabs of mixture #2 at 36 months could be explained by the beneficial role of OCI corrosion inhibitor in reducing chloride-ion content in the vicinity of the reinforcing steel.

4.2. Effect of precracking on corrosion rate

From the results of corrosion monitoring and visual observations, it is evident, that precracking has a significant effect on corrosion of embedded steel for $w/c = 0.40$ concrete. Corrosion current densities, depending on the crack width, observed for precracked concrete slabs after three years of exposure were higher than for uncracked slabs by a factor of three to four. For precracked specimens with both, 0.2 and 0.4 mm crack width without corrosion inhibitors added, corrosion current density increased linearly with time.

For precracked specimens with corrosion inhibitors added, rates of corrosion in general, were slightly lower or equal to rates for specimens without inhibitors, with the exception of 12 months test results, where they were significantly higher than the ones for the "no inhibitor" specimens.

At the early stages of exposure, corrosion inhibitors should be effective in maintaining the passivity of steel in the uncracked portion of the concrete slab. As a result of this passivation, a large cathode-small anode effect is created, that, in turn, results in the accelerated localised corrosion in the crack (anodic site). This could explain the higher 12 months' values of corrosion current densities for precracked concrete slabs with corrosion inhibitors and also more severe localised corrosion in the crack area for specimens with inhibitors. Similar results were reported in another study also involving cracked specimens with $w/c = 0.40$, where it was observed that corrosion rates for cracked specimens with corrosion inhibitor were higher, than for specimens without inhibitor added [25]. In yet another study on the performance of a sodium nitrite inhibitor in cracked concrete [26] it was found that in cracked concrete specimens immersed in seawater corrosion became more severe in the presence of sodium nitrite, and the severity of corrosion increased with increasing crack width.

After more prolonged exposure time, the effect of large cathode-small anode couple diminishes due to the effect of chloride diffusion through the concrete cover and initiation of chloride-induced corrosion over the entire surface of reinforcing steel. Under these circumstances, the steel in the uncracked region of the slab can become less effective as a cathode, resulting in relatively lower corrosion rates compared to 12 months' values. After three years of exposure, for all precracked slabs, with and without corrosion inhibitors, an additional corrosion-caused cracking of concrete due to accumulation of corrosion products is taking place, leading to even higher corrosion rates.

5. Conclusions

(i) In general, the use of corrosion inhibitors in uncracked $w/c = 0.40$ concrete is beneficial, but usually concrete cracks in service;
(ii) For precracked reinforced concrete slabs with 20 mm concrete cover, significant localised corrosion in the crack area was noted after only one year of marine exposure, and corrosion-induced concrete cracking occurred after only three years of exposure;
(iii) OCI was more efficient than CNI in terms of corrosion rate reduction in uncracked concrete; and
(iv) In cracked concrete both corrosion inhibitors were relatively inefficient.

6. Continuing research

Testing is continuing of the specimens with a water-to-cement ratio of 0.40 with crack widths of 0.1, 0.2, and 0.3 mm and the results on the influence of crack width on corrosion rates for concrete exposed in natural marine environment will be reported later.

Acknowledgements

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