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Predicting long-term durability of steel reinforced concrete with calcium nitrite corrosion inhibitor ☆

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Abstract

Steel reinforced concrete is one of the most durable and cost effective construction materials, but it can suffer in high chloride environments from corrosion due to chloride induced breakdown of the normal passive layer protecting the steel. One way of protecting embedded steel reinforcement from chloride induced corrosion is by the addition of corrosion inhibiting admixtures. The most widely used corrosion inhibiting admixture is calcium nitrite, due to its excellent inhibitor properties and its benign effect on concrete properties.

One advantage to calcium nitrite is that its protection mechanism is well defined. In this paper data are presented that show the levels of chloride to which given levels of calcium nitrite will protect. Furthermore, it will be shown that once corrosion initiates, the rates are lower with calcium nitrite present. Finally, it is demonstrated how these results can be used by the design engineer in an integrated durability model to produce reinforced concrete structures with durabilities in excess of 50–100 years.

Keywords: Concrete; Durability; Corrosion; Life prediction; Corrosion inhibitor; Calcium nitrite; Steel reinforcing; Modeling; Design life

1. Introduction

To predict the corrosion service life of a structure it is necessary to be able to predict the rate of chloride ingress to the steel reinforcing, the cover over the steel and the level of chloride necessary to start corrosion. Corrosion inhibitors that increase the chloride threshold level for corrosion initiation provide a major advantage in that the engineer can use extensive data on protection levels provided to design for service life. Using historical chloride exposure levels for a given concrete quality and known concrete cover over the reinforcement steel, the engineer is able to estimate the time for reaching the chloride level for corrosion initiation. Thus, an inhibitor that increases the threshold value of chloride will extend the time before the onset of corrosion and provide an increase in service life.

In this paper we present a methodology for predicting an increased threshold for chloride induced corrosion, which can then be utilized with models that address the issue of chloride ingress into concrete over time, to predict service life extension through the use of calcium nitrite.

1.1. Corrosion principles

A brief review of the corrosion behavior of steel in concrete is useful in understanding how the protection tables for calcium nitrite corrosion inhibitor have been developed. Even in the absence of chloride or carbonation, steel in concrete is always demonstrating a small trickle of background corrosion current. This trickle of current will subside to very low levels as the steel approaches the passive state caused by the high pH (>12.5) environment existing in concrete. Over the course of a few months this highly alkaline environment builds up a protective oxide layer on the steel. Even in the passive state there is always a small corrosion current present from the process of maintaining the protective passive oxide. This results in no significant rust buildup or expansive processes, therefore, the steel will remain in this condition for centuries with no loss in performance.

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When chloride enters the concrete and reaches the steel the normal passivity is disrupted, and active pitting corrosion initiates. Pitting is a very localized form of corrosion and the corrosion rates at the pits are orders of magnitude higher than the passive rate. However, since only a small portion of the steel is initially pitting, the actual corrosion rates measured over the entire exposed area of steel will initially show only a 4–10 fold increase. As corrosion occurs in a pit the local pH becomes lower and a more active corrosion process occurs in which more of the surface is corroding, and the measured corrosion rates increase. In other words, the entire corrosion process is accelerated.

Anodic inhibitors, such as calcium nitrite, strengthen the process of producing a passive layer on the steel, and thereby raise the concentration of chloride at which the passive corrosion process turns into an active or pitting process. A higher concentration of nitrite strengthens the passive layer to resist a higher level of chloride. This is the threshold chloride content for that calcium nitrite dosage. This effect is well documented by Rozenfeld in his book on inhibitors [1]. Numerous researchers outside our laboratory have also documented that nitrite increases the chloride content at which pitting and active corrosion initiate for steel in concrete and concrete-type environments [2–12]. As will be explained in detail later, even though these references clearly show an increase in threshold values for active corrosion initiation, the precise values need to be determined from longer-term chloride ingress experiments, which accurately reflect the exposure conditions of field concrete.

Admixed chloride tests are useful to show that calcium nitrite is an inhibitor, but as pointed out by Rozenfeld [1], could require more inhibitor than what would be needed if the inhibitor was present before exposure to chloride. Therefore, protection levels are based upon ingressed chloride in better quality concretes to better represent the slow ingress of chloride in the field.

1.2. Development of calcium nitrite to chloride content protection tables

Prior to the early 1980s our studies in concrete used corrosion potentials to indicate the performance of calcium nitrite [13,14]. Chloride analyses were performed and active corrosion was considered not to be occurring until corrosion potentials were more negative than −350 mV vs. copper–copper sulfate electrode (CSE). This was based upon an earlier ASTM C 876 test method version replaced in 1991, and reflected the state of the art at that time. Original nitrite-to-chloride protection levels were based upon these methods.

In the 1980s, extensive work by us and others made it clear that the corrosion potential measured in ASTM C 876 is very much affected by the moisture content in the concrete. For water saturated concretes, potentials more negative than −350 mV vs. CSE are not necessarily indicative of corrosion [15,16]. On the other hand, in test regimes where severe drying occurs, corrosion has been reported at more positive potentials in the −240 mV vs. CSE range [17]. This is either due to more oxygen availability or most likely to junction potential errors that can cause the measured voltage to be as much as 200 mV more positive [18]. As an example, via autopsy in our laboratory we found that no corrosion was occurring in one of the decks in Ref. [14] even though the corrosion potentials taken before the autopsy were at −473 mV vs. CSE. Since the decks were ponded daily and always wet, this is not unexpected in light of current knowledge. Thus there is no single corrosion potential values which uniformly defines the limit between passive and active corrosion behavior.

Accordingly, based upon internal observation of this effect, in the mid 1980s a reevaluation of the procedure for determining the chloride-to-nitrite ratio for corrosion initiation was started, and we chose to use only direct autopsy results in future laboratory work in support of corrosion protection dosage recommendations, Table 1.

It should be strongly emphasized that confirmation of the correctness of Table 1 was carried out on this basis. As will be shown later the time for autopsy was determined by using electrochemical measurements of the corrosion rate. The autopsies involve a physical examination of the reinforcing steel and measurement of the average chloride content at the steel level. If corrosion is observed, the chloride content at the exact corrosion location is determined provided the sample permits such analysis. Determining the chloride content at the exact corrosion site is critical since the chloride content is not constant due to the inhomogeneity of the concrete. Comparisons between chloride and nitrite contents are based on the calcium nitrite dosage added to the concrete.

A paper by Rosenberg and Gaidis [14] used corrosion potentials to determine the chloride-to-nitrite protection ratio. The high scatter in their Fig. 3 reflects the poor correlation of corrosion potential to actual chloride content. Since this work was done well prior to 1985, it does not take into account the effects of concrete

Table 1
Calcium nitrite protection table for ingressed chloride

Calcium nitrite (30% solution) l/m^3 (gal/yd^3)	Maximum chloride ion kg/m^3 (lb/yd^3)	Chloride/nitrite
10 (2)	3.6 (6.0)	1.3
15 (3)	5.9 (9.9)	1.5
20 (4)	7.7 (13)	1.4
25 (5)	8.9 (15)	1.3
30 (6)	9.5 (16)	1.2

moisture on corrosion potential readings, and does not reflect the methods we use today to validate our dosage table. However, further studies do confirm the original dosage conclusions of the Rosenberg and Gaidis paper. Recently both Feliu et al. [19] and Weyers et al. [20] have shown that the corrosion potential is a poor indication of absolute corrosion activity.

2. Results of field and laboratory chloride/nitrite measurements

Fig. 1 shows the ingressed chloride contents at the reinforcing level for many concretes with different addition rates of calcium nitrite, different concrete qualities, and various covers, as reported in numerous references [5,7,19–29]. The line represents the dosage recommendations given in Table 1. Open symbols represent those cases where the steel was not corroding, as noted by visual analysis of the steel, and the solid symbols are where actual corrosion damage was found on the steel. Specimens are from both laboratory studies and field sites, and represent work conducted at our laboratories and by others. For the seven data points from field sites corrosion rate or potential measurements and physical appearance of the concrete were used to assess performance, since the structures could not be sacrificed for complete autopsy. These data are presented in Fig. 2.

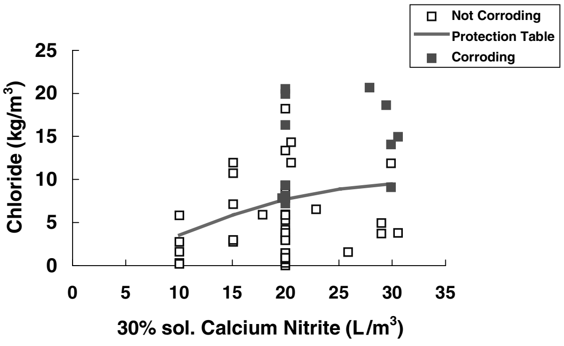

Fig. 1. Chloride vs. nitrite—all data.

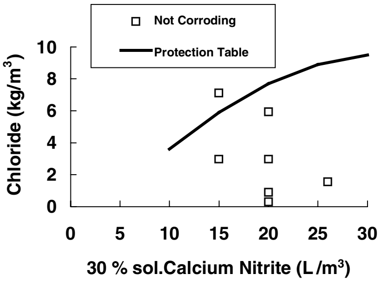

Fig. 2. Chloride vs. nitrite content—field data.

It can be seen that by far the greatest number of points follow the prediction of the Table 1, and that in many cases concretes expected to be in corrosion due to chloride content in excess of the predicted protection level are nevertheless not corroding.

In Fig. 1 there are only two cases where corroding points are very slightly below the protection line from Table 1. They are both from laboratory studies using a low cover, short curing, and harsh drying cycle exposure regime. These specimens had 25 mm of cover and the chloride values were obtained at that depth. Since these data were obtained in an outside lab, we were not able to sample and perform chloride analysis of the concrete at the actual points of corrosion. However autopsy of a similar concrete specimen from that laboratory showed as much as a factor of three increase in chloride contents at individual corroding points vs. the average value at 25 mm. This was due to a high chloride content right next to a 19 mm coarse aggregate particle. Had actual chloride analyses been performed next to the corrosion sites in these samples, the ratios would most likely have been well above the recommended protection line in Fig. 1. These data are included for completeness despite the less than precise knowledge of the chloride content at the corroding sites. It should also be noted that these concretes were of relatively low quality (0.5 water-to-cement ratio [w/c] and low cover), and thus would not meet ACI recommendations for cover and quality in corrosive environments [30]. Finally, very rapid ingress of chloride as occurred in these concretes can lead to misleading conclusions, since the chloride can reach the steel before the full passive barrier is established. Overall the data collected from laboratory and field structure studies for the last 20 years clearly support the protection levels for ingressed chloride given in Table 1.

As shown above, corrosion potentials are not directly related to the corrosion rate. The lack of oxygen in water saturated concrete results in more negative corrosion potentials. Drier concrete can result in junction potentials which cause the corrosion potential to be more positive [18], and which accounts for some observations of corrosion occurring at potentials more positive than −350 mV vs. CSE. Thus, statements about corrosion activity made solely on the basis of corrosion potentials are unwarranted in light of current knowledge. The current version of ASTM C 876 has moved the interpretation of corrosion potentials into the nonmandatory appendix; this reflects the lack of consensus regarding their use in predicting corrosion activity.

On the other hand, when properly interpreted, corrosion rate measurements using polarization resistance or electrochemical impedance can be a good indication of corrosion activity in the laboratory [31–33], where the area of steel affected in the test is known and results can be verified by autopsies. Macrocell measurements can be misleading because corrosion could be occurring due to local cathodic sites. In any event we use these tests only in developmental research and as an indication that an autopsy should be performed. A few examples of how these were used in determining autopsy times are given below.

Figs. 3 and 4 provide laboratory corrosion rate data for several of the specimens used to develop the chloride threshold levels. They are given to illustrate how corrosion rate measurements can be used to determine autopsy times, and to show that the corrosion rate techniques give a good representation of the corrosion activity.

Fig. 3 gives corrosion rate measurements on concrete lollipops before removal for autopsy, from data in Ref. [21]. The corrosion rate is expressed in terms of the inverse of the polarization resistance, R_p. Values above 25 μS/cm^2 are indicative of severe corrosion [21,31]. The corresponding chloride-to-nitrite ratio at the time of autopsy and autopsy results are also given for each curve. These data clearly show that corrosion rate measurements are a good method to zero in on when to autopsy. The highly corroding specimens were produced with high w/c ratios to accelerate chloride ingress and in some cases the rapid ingress of chloride might be providing an over accelerated test as noted in the previous section.

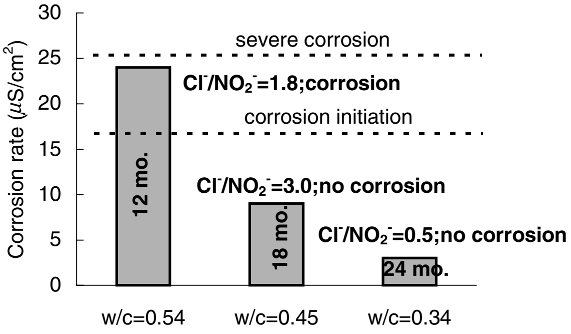

Fig. 3. Corrosion rate of concrete lollipops.

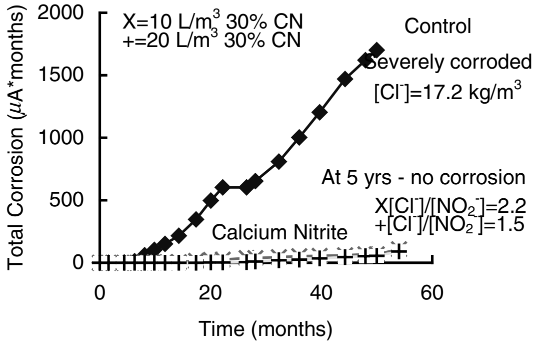

Fig. 4. Minideck corrosion vs. time Ref. [26] w/c = 0.46, CF = 348 kg/m^3, cover = 19 mm.

Table 2
South Florida cooling tower beams

Calcium nitrite (l/m^3)	0	29.7
Air (%)	4.8	7.0
28 days compressive strength (MPa)	48.7	50.4
E_{corr} (mV vs. SCE)	−47	25
$1/R_p$[a] (μs/cm^2)	5.2	1.9
Chloride content at the rebar level (kg/m^3)	0.95	0.36
Chloride/nitrite	–	0.045
Autopsy	Light corrosion	No corrosion

[a] Determined at 22 °C. Temperatures in application could have resulted in values over four times those at 22 °C.

Fig. 4 shows modified ASTM G109 type minibeam specimens tested for 5 years from data in Ref. [24]. The chloride-to-nitrite levels were determined after 5 years and autopsy data showed no corrosion on the bars, in good agreement with the macrocell currents. Since calcium nitrite does not significantly change resistivity between the top and bottom mats the macrocell technique is useful in determining corrosion activity. The corrosion rate is measured in μA, and integration over time is the total macrocell corrosion and is expressed in units of current × time (μA × months).

In field structures the area of steel polarized in a corrosion test is not exactly known, so corrosion rate tests are best used as a guide as to where cores or autopsies are to be performed. Corrosion potential contours can be used to identify sites for autopsies as noted in ASTM C 876. However in marine structures potential values in the tidal zone and below can be misleading, as noted in the literature [15,16]. Table 2 provides some corrosion rate data on beams that were exposed in a power plant cooling tower using brackish water and returned to the laboratory for polarization resistance measurements of corrosion rate [28]. The data show that corrosion rate measurements clearly identified corrosion in control specimens and that these relatively early age specimens with low chloride-to-nitrite ratios were not corroding.

2.1. Corrosion rates after corrosion initiation

In addition to increasing the threshold value for corrosion initiation, several laboratory tests show that calcium nitrite reduces corrosion rates after corrosion initiation as shown in Figs. 5 and 6. After corrosion has occurred corrosion rates are less for concretes containing calcium nitrite as seen by the lower slopes in the integrated total corrosion curves. Fig. 7 shows that a reduction in chloride levels, made possible by the addition of two dosages of silica fume, can increase time to corrosion initiation, but not reduce the rate once cor-

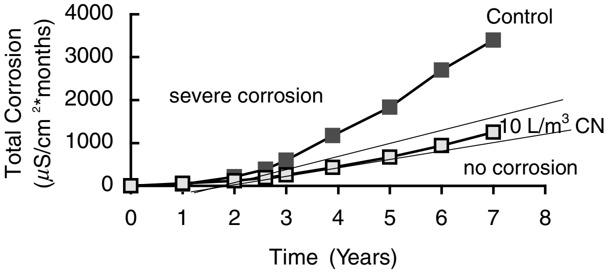

Fig. 5. Total corrosion of lollipops, w/c = 0.38, CF = 356 kg/m^3.

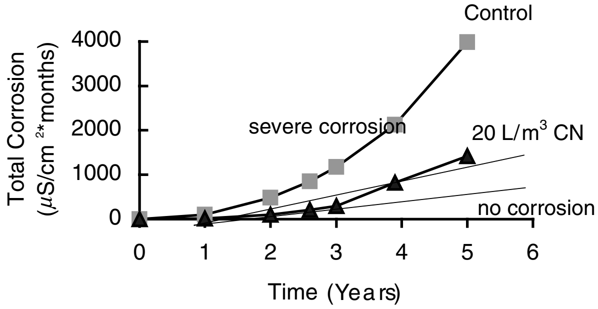

Fig. 6. Total corrosion of lollipops, w/c = 0.48, CF = 356 kg/m^3.

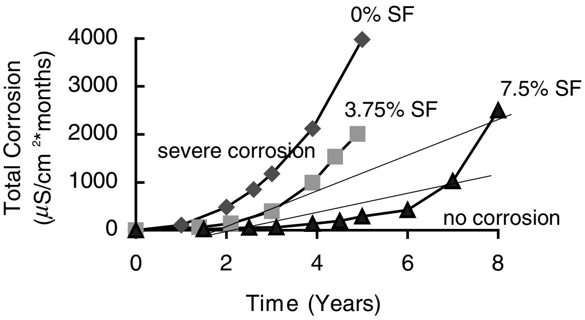

Fig. 7. Total corrosion of lollipops with various amounts of silica fume, w/c = 0.48, CF = 356 kg/m^3.

rosion starts. A combined increase in chloride threshold levels and lower corrosion rates after initiation should lead to significantly improved times to repair in reinforced concrete structures subjected to chlorides.

A recent study in the Strategic Highway Research Program indicated that there was about 2.5–5 years from severe corrosion initiation to repair in US highway bridges [34] Reduced corrosion rates after initiation could extend these times, and more laboratory and field work is in progress to quantify the potential improvements. Thus, a conservative approach for now would be to base future time to repairs on the time to corrosion initiation.

2.2. Life-cycle modeling

Considerable work has been conducted in the area of life-cycle modeling using Ficks' second law of diffusion to obtain an effective diffusion coefficient for chloride in concrete, which is based upon total chloride content [24,35–40]. Corrosion is typically considered to start at approximately 0.9 kg/m^3 of chloride at the reinforcement level and as noted earlier repairs might occur within 2.5–5 years of initiation. In this section chloride ingress is modeled for typical structures that would be found in a marine environment. The benefits of calcium nitrite in increasing chloride threshold values are highlighted. Note that with good cover and low w/c ratios cracks under approximately 0.2–0.3 mm play a minor role in the overall corrosion [41,42].

Effective diffusion coefficients (D_{eff}) as a function of temperature are given for two good quality concretes in Table 3.

These represent a typical 0.4 w/c concrete with ordinary portland cement and a similar concrete produced with of silica fume, fly ash, or granulated ground blast furnace slag (mineral admixtures). In general, silica fume is the most efficient of the three in reducing diffusion coefficients, and its addition rates would be lower for an equivalent diffusion coefficient. Corrections for temperature in this case were made by converting data at 22 °C (295 K) by using the following equation [43]:

$$D_2 = D_1\left(\frac{T_2}{T_1}\right)e^{\left[k\left(\frac{1}{T_1}-\frac{1}{T_2}\right)\right]}$$

where $k = 5450$ and D_2 is the effective diffusion coefficient at temperature T_2 and D_1 is the effective diffusion coefficient at T_1 which is 295 K in this case. Chloride ingress tests on the particular concrete designs of interest will provide more accurate estimates of D_{eff}.

Fig. 8 illustrates the estimated chloride concentrations 75 mm in from each face at the corner of a square pile in the splash–tidal zone at an average yearly temperature of 19 °C, which is similar to that of the water in Melbourne, Australia. A two-dimensional solution to Fick's second law was used, since chloride is entering from two faces. Even though the concrete cover is high, and the both concretes are of good quality, chloride contents are in excess of 1 kg/m^3 in only 8 years for the ordinary Portland cement at 0.4 w/c and 18 years when

Table 3

D_{eff} ($\times 10^{-12}$ m^2/s) as a function of temperature and concrete type

Concrete	T (°C)			
	10	15	19	22
w/c = 0.4	1.3	1.9	2.5	3.0
w/c = 0.4 + SF, FA, or GGBFS	0.6	0.8	1.1	1.3

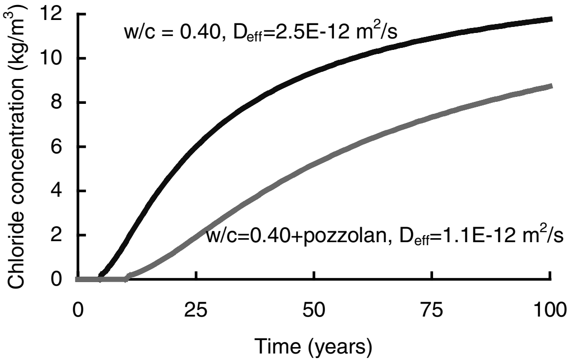

Fig. 8. Estimated chloride concentrations of square piles (360 mm side) in a marine environment in the splash/tidal zone $C_o = 15$ kg/m^3; Avg $T = 19$ °C, depth = 75 mm.

mineral admixtures are added. Thus, reducing permeability, while clearly beneficial is insufficient alone to reach design lives in excess of 50 years.

A conservative design life of 50 years can be obtained by adding calcium nitrite corrosion inhibitor. The 0.4 w/c concrete will have an estimated chloride content of 9.4 kg/m^3 at 50 years and the addition of mineral admixtures will reduce the chloride levels to 5.2 kg/m^3. Thus, from Table 1, 30 or 15 l/m^3 of 30% calcium nitrite would be needed depending upon the concrete chosen to extend severe corrosion initiation times to 50 years. Furthermore, there is a clear advantage in the combination of calcium nitrite with lower permeability concrete as can be seen by the reduced dosage needed. To extend corrosion initiation to 100 years, 25 l/m^3 of 30% calcium nitrite can be used with the low permeability concrete with mineral admixtures.

The above example involved relatively high average temperatures. Fig. 9 shows that in environments that are colder significantly less chloride reaches the steel. However, chloride will still surpass 1 kg/m^3 levels for the concrete with mineral admixtures within 33 years and supplemental protection such as using calcium nitrite would be needed to reach longer design lives.

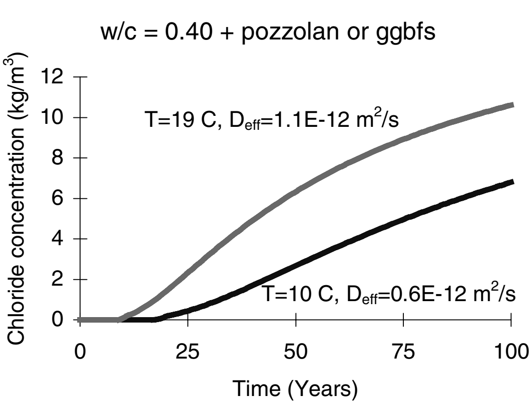

Fig. 9. Estimated chloride concentrations of square piles in a marine environment at two different temperatures in the splash/tidal zone, $C_o = 18$ kg/m^3, depth = 75 mm.

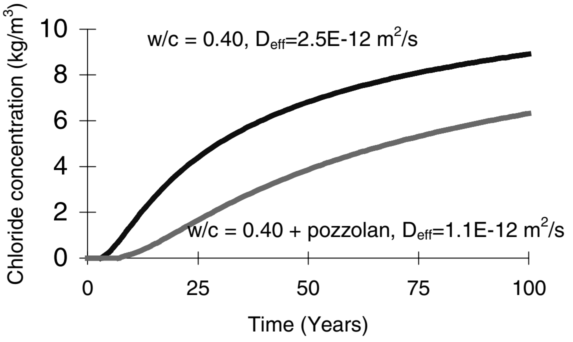

Fig. 10. Estimated chloride concentrations of a marine wall in the splash/tidal zone at 65 mm depth $C_o = 15$ kg/m^3, $T = 19$ °C.

Marine walls in the splash–tidal zone have a reduced ingress of chloride as they are subjected to ingress in only one-dimension. Fig. 10, shows the estimated profiles for the same quality of concretes as in Fig. 8 in the same marine environment. At a cover of 65 mm the times to chloride reaching 1 kg/m^3 are the same as in the square pile case with 75 mm of cover, however, there is considerably less chloride at 50 and 100 years. For protection to corrosion initiation at 100 years the 0.4 w/c needs approximately 25 l/m^3 and the 0.4 w/c with mineral admixtures needs approximately 18 l/m^3 of 30% calcium nitrite.

Reinforced concrete that is not directly in the splash–tidal zone still needs to be protected from airborne chlorides. This is illustrated in Fig. 11 which shows the estimated chloride content at 40 mm for a wall subjected to a mild chloride buildup from airborne chlorides of 0.2 kg/m^3 per year to a maximum of 18 kg/m^3 occurring after 90 years. Chloride contents of 1 kg/m^3 are exceeded at 22 and 33 years for concretes at 0.4 w/c and 0.4 w/c with mineral admixtures respectfully. The addition of approximately 12 l/m^3 of 30% calcium nitrite will extend

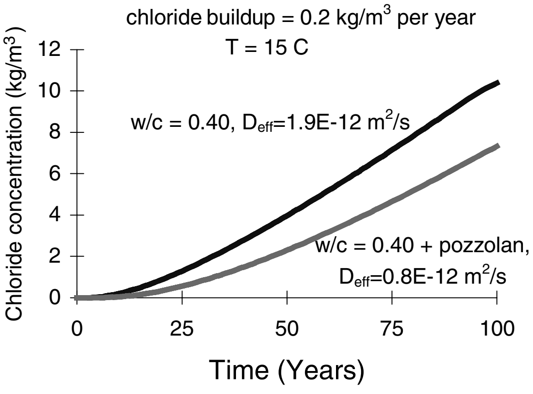

Fig. 11. Estimated chloride concentrations of a marine wall subjected to airborne chloride in a marine environment at 40 mm depth.

the time to corrosion initiation to approximately 50 years for the 0.4 w/c case. The minimum recommended dosage of 10 l/m^3 will extend the time to corrosion initiation to approximately 65 years for the lower permeability concrete.

The above examples show how one can utilize corrosion and chloride ingress data to estimate service lives of reinforced concrete structures. By varying the dosage of calcium nitrite, concrete quality and concrete cover, the design engineer has significant control in meeting a particular service life requirement.

3. Conclusions

Based upon extensive work performed on the corrosion of steel in concrete with and without calcium nitrite corrosion inhibitor the following conclusions can be made:

- The 30% calcium nitrite solution protection levels given in Table 1 are appropriately but not excessively conservative. The method used today to support the table values is straightforward examination and analysis of concrete samples.
- In no test where actual chloride content at the corroding sites has been measured have dosages, predicted by the protection table, failed to give the required protection.
- Arguments questioning the table based on interpretations from corrosion potential measurements are not justified, based on the known lack of direct correlation between potential and corrosion activity, and based on the fact that the table is supported by direct measurement.
- Review of test data should be carefully performed with special consideration to the conditions used in the actual testing. Rapid ingress of chloride as occurs in some testing protocols can lead to misleading conclusions, since the chloride can reach the steel before the full passive barrier is established.
- Corrosion rate data on laboratory specimens are in good agreement with autopsy results, but are not a substitute for verification by autopsy.
- The data collected from laboratory and field structure studies for the last 20 years clearly support the protection levels for ingressed chloride given in Table 1.
- Calcium nitrite does not increase corrosion rates after chloride protection values are surpassed, and in contrast often lowers them.
- A major advantage to the use of calcium nitrite corrosion inhibitor is that the engineer can use rational procedures based on chloride exposure, concrete quality, and quantity of calcium nitrite to design for service life on the basis of expected chloride-to-nitrite ratios.

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