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# AN ASSESSMENT OF ELECTROCHEMICAL CHLORIDE EXTRACTION AS A REMEDIATION TECHNIQUE FOR STEEL REINFORCED CONCRETE

Dr G Sergi & Dr C I Goodier  
Building Research Establishment Ltd  
Garston  
Watford WD25 9XX  
UK  
sergig@bre.co.uk

Dr AMG Seneviratne  
School of Engineering & Applied Science  
Aston University  
Birmingham B4 7ET  
UK  
amg.seneviratne@aston.ac.uk

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## ABSTRACT

Steel reinforced concrete blocks were subjected to chloride extraction after they were dosed with either NaCl or CaCl<sub>2</sub>. All the blocks were then exposed to the elements at the BRE exposure site for a total period of about 6 years. The steel reinforcement was regularly monitored electrochemically to determine its level of corrosion. Core samples were also analysed before and after treatment to determine the chloride concentration profiles. A selection of blocks were cut into smaller steel-containing prisms after 4 ½ years of exposure and were exposed to controlled laboratory conditions for 6 months while determining the level of corrosion electrochemically. At termination, each steel bar was removed and examined visually. The total weight loss was assessed gravimetrically. The final chloride concentration profile of each prism was also determined.

Results suggested that corrosion was reduced significantly following chloride extraction but that the primary long-term controlling factor, both for the desalinated and control specimens, appeared to be the level of chloride present at the depth of the steel reinforcement. As, under normal procedures, a proportion of chloride remains after chloride extraction, a significant level of corrosion of the reinforcement, although greatly reduced, is still likely.

## INTRODUCTION

In the search for methods to inhibit corrosion in reinforced concrete or repair damage caused by reinforcement corrosion, electrochemical chloride extraction (desalination) has been claimed to be a permanent solution. Independent justification was sought to ascertain these claims and place the technique in the context of other remedial methods. This paper reports on the procedure employed and on the main findings of the resulting six year research project undertaken by BRE.

The primary objective of the project was to monitor the long term performance of desalinated concrete to quantify and give definitive advice on the efficacy of the treatment.

### The concept of the electrochemical remediation techniques

Electrochemical chloride extraction, commonly known as desalination, is a technique currently available for the rehabilitation of steel reinforced concrete suffering from reinforcement corrosion (Polder et. al.,1992, Bennett & Schue,1993).

Commonly, chloride extraction, used in cases where reinforcement corrosion was caused by chloride contamination of the concrete, involves the positioning and fixing of a tank onto the surface of the concrete element to be treated, into which water or a saturated solution of calcium hydroxide is circulated by pump. A conductive anode material, often a steel mesh, is contained within the tank stretching over the whole area of concrete and is connected to the positive terminal of a transducer / rectifier. The negative terminal is connected to the steel reinforcement after it is checked for electrical

continuity. The power source maintains a constant direct current of the order of 1-5 A/m<sup>2</sup> of steel reinforcement between the external anode and the steel cathode for a period of a few weeks. The resultant electrical field enables the anionic species (calcium, sodium and potassium ions present in the concrete pore-solution) to migrate along the current lines towards the steel reinforcement (Fig. 1). At the steel cathode, cathodic reactions produce hydroxyl ions which both balance the positive charge of the arriving anions and, along with other cations such as chlorides, migrate towards the external anode. Hydroxyl ions are consumed at the anode by anodic reactions while chlorides are washed away by the circulating electrolyte.

The efficiency of chloride removal is dependant on the relative concentration of the chlorides to the total ionic concentration of the concrete pore-solution so that as chlorides diminish, less proportion of the current is used to transport the chloride ions to the external electrolyte and the process becomes uneconomical (Elsener & Bohni, 1994, Ismail et. al., 2003). As a consequence, a proportion of chloride always remains in the concrete. Furthermore, chlorides that may have been present beyond the influence of the current lines (see Fig. 1), i.e. a little behind the steel reinforcement and possibly between adjacent bars, are likely to remain there after the treatment. These chlorides then become available to re-migrate towards the steel. Although this re-migration was seen experimentally (Ismail et. al., 2003), its effect on the subsequent level of corrosion of the steel reinforcement has not yet been adequately documented.

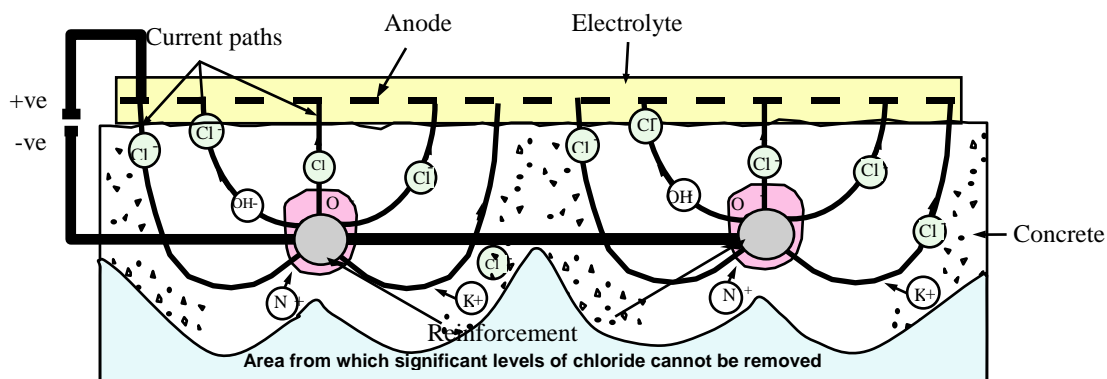


Figure 1. Diagrammatic representation of a typical electrochemical chloride extraction set-up showing direction of current lines and area from which little chloride can be extracted

## EXPERIMENTAL PROCEDURE

### Exposure site blocks

Fifty concrete blocks (360 x 360 x 150 mm) were cast between December 1995 and February 1996. Two layers of 8 mm diameter steel reinforcement made into 80 mm square grids were cast in the blocks with a minimum cover of 30 mm. The concrete mix used, its fresh properties (slump, fresh density) and the compressive strengths obtained are shown in Tables 1 and 2. A number of the specimens were cast with a silver chloride reference electrode in the centre of the specimen (identified in Table 2).

Table 1. Concrete mix details

Material	Kg/m <sup>3</sup> of concrete
OPC	238
20-10mm agg	755
15-10mm agg	343
5-0mm agg	794
water	200
TOTAL	2330
Agg to cement ratio	0.13
Water to cement ratio	0.84
Free water to cement ratio	0.70

Table 2. Cast details and compressive strength

Specimen ID (date cast)	Slump (mm)	Density (kg/m <sup>3</sup> )	Comp strength (Nmm <sup>2</sup> )		Added chloride % by weight of cement	Specimens with embedded silver chlorides
				Mean		
DF 1-5 (8/12/95)	80	2340	30, 28.5, 30	30	0	DF1
DC 1-10 (15/12/95)	50	2330	34.5, 35, 35.5	35	1	DC1(control) DC2, DC7
DC 11-15 (19/12/95)	60	2330	31, 34, 34.5	33	1	DC11 (cntrl)
DF 6-15 (18/1/96)	40	2330	30.5, 31, 32	31	0	DF14 DF15 (cntrl)
DF 16-18 (23/1/96)	40	2330	31.5, 31, 31.5	31.5	0	DF8 (cntrl)

The blocks produced for the desalination process were either exposed to cyclic chloride ponding (5% NaCl solution) or had chlorides cast into them (1% chloride as calcium chloride by weight of cement). All the chloride-contaminated specimens were desalinated by one of the industrial partners, according to their normal procedures, in February 1997 (current density = 4.5 A/m<sup>2</sup> of steel, time = 10 days). The desalination process was repeated in April 1997 at a reduced current density and increased time (current density = 1.6 A/m<sup>2</sup> of steel, time = 24 days) to determine if further chloride removal could be achieved with prolonged polarisation. In order to determine the level of chloride that was removed from the concrete following the desalination treatment, dust samples were drilled down to the level of the steel mesh. These were analysed by acid extraction. From the mean initial concentration at the level of the steel of around 2.35% by weight of cement (0.24% by wt of sample) for the blocks exposed to NaCl before treatment the concentration directly over intersecting steel bars (location A in Fig. 3) was reduced to a mean of 1.05% (a reduction of around 55%) following the second treatment (Fig. 2). The reduction was less away from the steel (i.e. 46% to 1.27% at locations 25-50mm from the steel, denoted as C in Fig. 3). In the case of the cast-in chlorides, the mean reduction from an initial concentration of 0.89% by weight of cement was 52% above the intersecting steel bars compared to only 18% at location C. The proportion of reduction from the second treatment was only of the order of 3-5%. This, as well as the lower overall reduction in the case of the cast-in chlorides, shows clearly that as the total chloride concentration diminishes, the process of removing chlorides becomes less efficient. The proportion of

chloride removed is consistent with that predicted by modelling (Hassanein et. al., 1998). The reductions obtained were deemed to be acceptable according to normally accepted criteria.

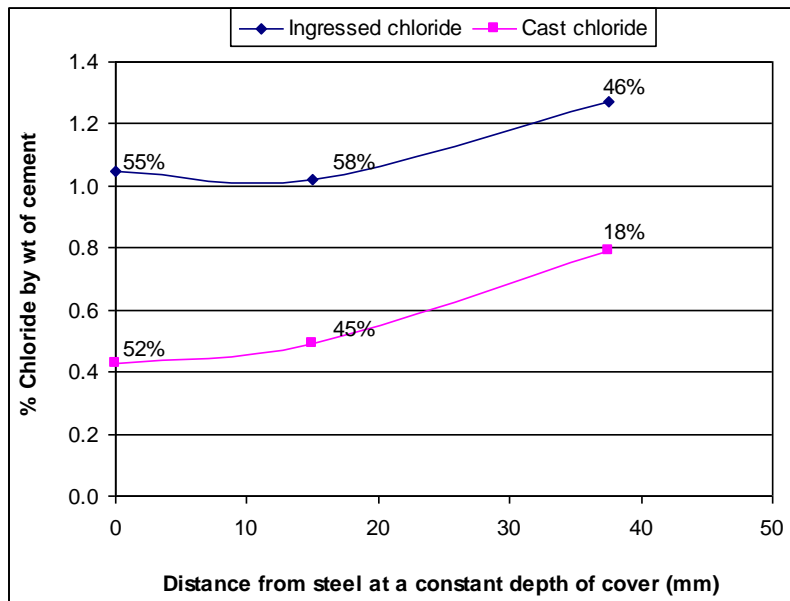


Figure 2. Remaining chloride and change in the level of reduction with distance from the steel at a constant depth from the surface following desalination treatment

All the blocks were then placed with the long faces vertical at the BRE outdoor exposure site. Each block had one of the larger faces exposed in a northerly and the other in a southerly direction.

The corrosion potential of the steel reinforcement was monitored against either the embedded reference electrode or a separate electrode positioned centrally on both the front and rear faces of each block. The corrosion intensity of a length of steel near the centre of each uncoated block was also determined in-situ with the use of GECOR-6, a commercial device based on the principle of linear polarisation (see also section on Corrosion Monitoring).

### Laboratory experiments

After about 4½ years of exposure, seven reinforced concrete specimens were selected for more detailed analysis. Details of the blocks are shown in Table 3.

Table 3. Details of the reinforced concrete blocks selected for detailed analysis

Code	Description	Condition
C 1	Control	Rust stains, rough surface
C 3	Control	Smooth surface
DC 13	Cast-in chlorides, desalinated	Rough surface
DF 8 C	Control, ingressed chlorides	Rust stains, cracks
DF 15 C	Control, ingressed chlorides	Rust stains, cracks
DF 5	Ingressed chlorides, desalinated	Some rust stains, some cracks
DF 12	Ingressed chlorides, desalinated	Few small cracks

Each of the chosen blocks was cut in such a way as to obtain three ~100 x 60 x 150mm smaller concrete prisms (Figure 3) each containing two separate sections of embedded steel bar for corrosion monitoring as shown in Figure 4. The full details of the specimens are given in Table 4. Electrical connections were made to the steel bars by drilling, tapping and fixing a length of electric wire with stainless steel screws.

The cut surfaces of the steel were then masked with a duplex layer of styrene butadiene rubber (SBR) modified white cement slurry and a layer of epoxy resin in order to minimise crevice corrosion. The specimens were then weighed and immersed up to a depth of 5mm for a total of 48 hours, on each of the two opposite originally exposed faces, turning the prisms through 180° after the first 24 hours.

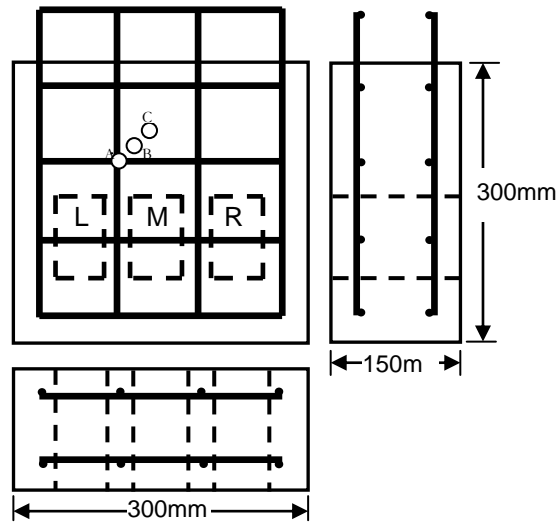


Figure 3. Details of large concrete block showing cutting to obtain three prisms (L, M & R) for corrosion monitoring

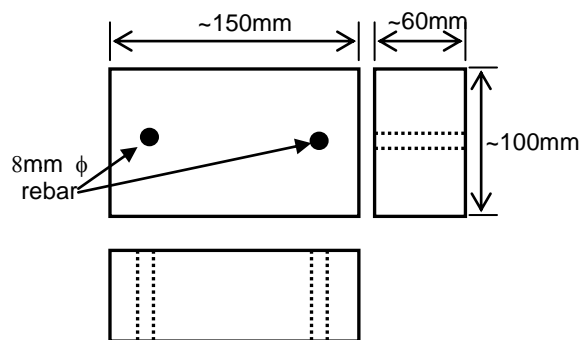


Figure 4. Details of prism used for corrosion monitoring

Table 4. Details of prisms for corrosion monitoring

CODE	Size (mm)	Diameter of rebar (mm)	Length of Bar-1, (mm)	Length of Bar-2, (mm)	Cover to Bar-1, (mm)	Cover to Bar-2, (mm)
C1/L	150x60x100	8	61	62	33	35
C1/M	150x62x102	8	62	62	34	35
C1/R	150x62x101	8	62	62	34	35
C3/L	150x80x98	8	79	78	39	34
C3/M	150x59x97	8	59	60	39	35
C3/R	150x65x98	8	65	65	38	36
DC13/L	150x58x98	8	58	60	35	35
DC13/M	150x61x98	8	62	62	35	35
DC13/R	150x61x100	8	61	62	35	35
DF8C/L	150x63x105	8	63	63	33	33 (cracks)
DF8C/M	150x57x105	8	57	56	33	34 (cracks)
DF8C/R	150x67x103	8	67	67	33 (cracks)	33
DF5/L	150x62x103	8	63	65	36	34
DF5/M	150x60x100	8	60	60	36	33
DF5/R	150x60x101	8	61	61	35	33
DF12/L	150x60x107	8	61	62	34	34
DF12/M	150x56x106	8	56	57	35	36
DF12/R	150x62x104	8	62	62	36	37
DF15C/L	150x56x85	8	58	57	31 (cracks)	32 (cracks)
DF15C/M	150x55x85	8	57	57	32 (cracks)	32 (cracks)
DF15C/R	150x62x85	8	63	64	30 (cracks)	30 (cracks)

### Corrosion Monitoring

The corrosion current ( $i_{corr}$ ) and potential ( $E_{corr}$ ) of each steel bar and weight of each individual prism were determined, both after the initial exposure to water and subsequently at regular time intervals until steady state conditions were established. Following each set of measurements the prisms were stored in a high humidity constant temperature ( $>98\% \text{ RH}$ ,  $20 \pm 2^\circ\text{C}$ ) environmental cabinet. Corrosion potentials of the two steel bars were measured with a high impedance voltmeter, versus a saturated calomel reference electrode (SCE) positioned on a pre-determined marked spot in the centre of the concrete surface parallel to the bars, half-way between the bars. The corrosion current of the bars was determined by a linear polarisation technique (Gowers & Millard, 1999). This involved shifting the potential of the steel by  $\Delta E$  ( $\sim 20 \text{ mV}$ ) from its rest potential using a potentiostat (Amel 550) fitted with a positive feedback  $iR$  compensation facility and measuring the resultant current density ( $\Delta i$ ) passing between the first steel bar, acting as the working electrode, and the second steel bar, acting as a counter-electrode, after 60 seconds. Sufficient time was allowed for depolarisation to occur before the two bars were reversed and the procedure repeated to measure the current through the second bar. The polarisation resistance ( $R_p$ ) was taken to be the measured ratio  $\Delta E/\Delta i$ . The corrosion current,  $i_{corr}$ , was then calculated assuming  $B=26\text{mV}$  in the Stern and Geary equation (Stern & Geary, 1957, Gonzalez et. al., 1985).

$$i_{corr} = B/R_p$$

where

$$B = \frac{\beta_a \times \beta_c}{2.3 (\beta_a + \beta_c)}$$

and,  $\beta_a$  and  $\beta_c$  are the anodic and cathodic Tafel constants respectively.

At the end of the corrosion monitoring, concrete powder samples were obtained by drilling at 15mm increments from the exposed surface down to the depth of the steel reinforcement and beyond. The collected powder samples were dried in an oven at 105°C and allowed to cool in a desiccator before chemical analysis to determine the chloride concentrations using a standard analytical technique.

#### *Gravimetric weight loss measurements*

The prisms were split along the length of the steel bars perpendicular to the longest dimension. The bars were carefully removed revealing the concrete substrate and the surface of each bar. The corrosion products were removed by pickling in a solution of 25% HCl containing a proprietary corrosion inhibitor. The weight of each bar was recorded at 10 minute intervals until a constant decrease with time was achieved. Extrapolation back to the origin gave the weight of the uncorroded metal allowing the determination of the weight loss due to corrosion.

## RESULTS AND DISCUSSION

### Exposure site blocks

The corrosion potential (v's silver/silver chloride standard reference electrode) determined from measurements on the blocks over a three year period are summarised in Figure 5.

If a simple criterion is used whereby a corrosion potential more negative than -200 mV is considered to signify at least some risk of corrosion to the reinforcement, it appears that the control chloride-free blocks and the desalinated blocks containing cast-in chlorides are in the main showing no evidence of corrosion. The cast-in chloride-containing control blocks appear to suffer from significant corrosion during some periods, particularly towards the end. Overall, the potential of the chloride-containing controls is more negative throughout.

Using the same criterion, the steel bars of the blocks containing ingressed chlorides, judging by their corrosion potentials, are expected to have suffered from significant corrosion over the whole period of exposure. Those subjected to desalination, however, tended to approach the -200 mV level signifying perhaps a degree of success in lowering the level of corrosion. It is known that the potential of the steel, following polarisation to very negative potentials during the desalination process, requires a period of several months, possibly a year, to achieve de-polarisation owing to large enforced chemical, and possibly, physical changes. Some of the physical changes such as reduced porosity by the possible precipitation of certain phases, are likely to be permanent. The simple assumed absolute potential criterion, therefore, is unlikely to always apply, particularly during the early months following treatment. Nonetheless, an indication of trends is possible so at least a reduction in the corrosion activity of the steel could be confidently assumed after desalination.

Although some sense could be made from the equivalent mean corrosion current results of the steel grids in the desalinated and control ingressed chloride blocks when looking at the mean values (Fig. 6), as a whole individual results were very inconclusive. Apart from considerable scatter, the chloride-free controls, ingressed chloride controls and desalinated samples, all show the same range of corrosion intensity of the steel in the region 2-80  $\mu\text{A}$ , much higher than the assumed 2-3  $\mu\text{A}$  ( $0.1\text{-}0.2 \mu\text{A}/\text{cm}^2$ ) normally considered as a limit below which the corrosion current is insignificant (Grantham. & Broomfield, 1995). Several reasons may have contributed to the apparent unreliability of these results. The most important is that the current applied during the linear polarisation test is not likely to have been adequately confined to a single length of bar, as the narrowness of the grid would have allowed current to be distributed to both horizontal and vertical bars as well as to bars in the lower grid. Furthermore, the current level and distribution varies with the resistivity of the concrete, which in turn is related to the level of moisture content in the concrete pore-system, and with ambient temperature. The net effect would have been both the overestimation and the large variation of the corrosion current.



Of some importance, at least in the desalinated samples, could also have been both the prolonged polarisation of the steel owing to the treatment itself and the possible acidification and etching of the surface of the concrete owing to the proximity of the anode during the passage of current.

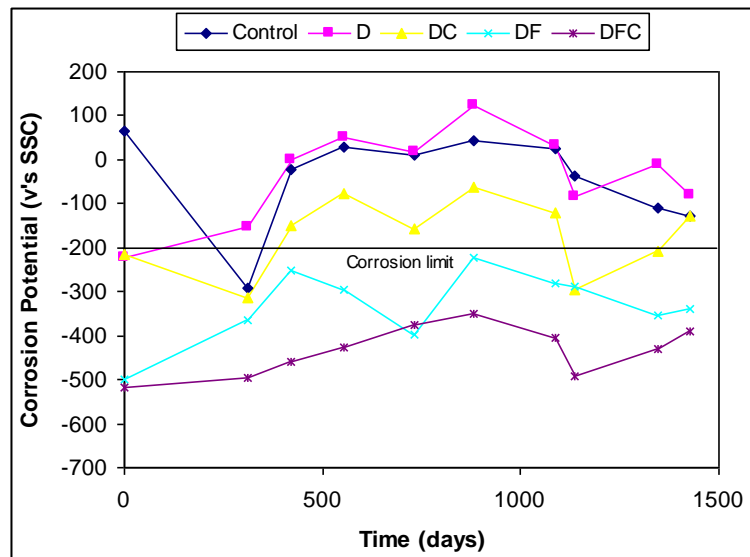


Figure 5. Mean corrosion potential of steel grids in blocks

Control = chloride-free controls, DC = desalinated, cast-in chlorides, DCC = Cast-in chlorides, control, DF = desalinated, ingressed chlorides and DFC = ingressed chlorides. controls

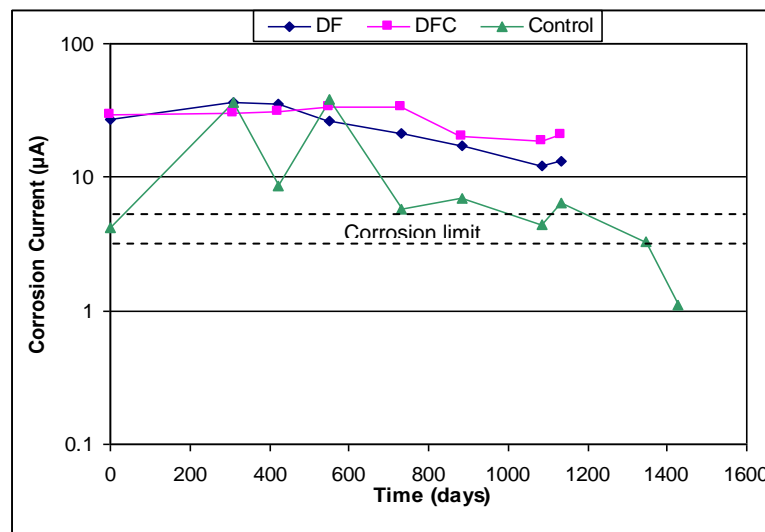


Figure 6. Corrosion intensity of steel grids in blocks

Control = chloride-free controls, DF = desalinated, ingressed chlorides and DFC = ingressed chlorides, controls

### Laboratory

A much more accurate assessment was achieved from the laboratory investigations as temperature, humidity and size of steel bars were all controlled parameters. The corrosion potentials confirmed the in-situ findings but the corrosion intensity of the single bars appeared to be much more realistic and reliable. Figures 7 and 8 summarise the results with the mean values from each condition. The corrosion current (Fig. 8) is lowest for the bars in the control chloride-free prisms and highest for the bars in the control 'ingressed-chloride' prisms, as would be expected, the latter being an order of magnitude higher. The order of conditions in an increasing trend of corrosion is then desalinated cast-in chlorides and desalinated ingressed chlorides, these being in-between the two extreme conditions.

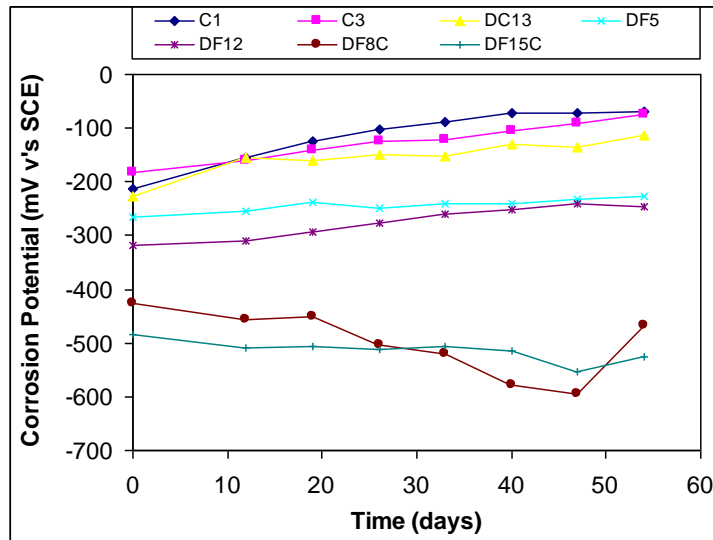


Figure 7. Variation of mean corrosion potential with time of steel bars in the cut 'corrosion-monitoring' prisms  
 C = chloride-free controls, DC = desalinated, cast-in chlorides, DF = desalinated, ingressed chlorides and  
 DFC = ingressed chlorides, controls

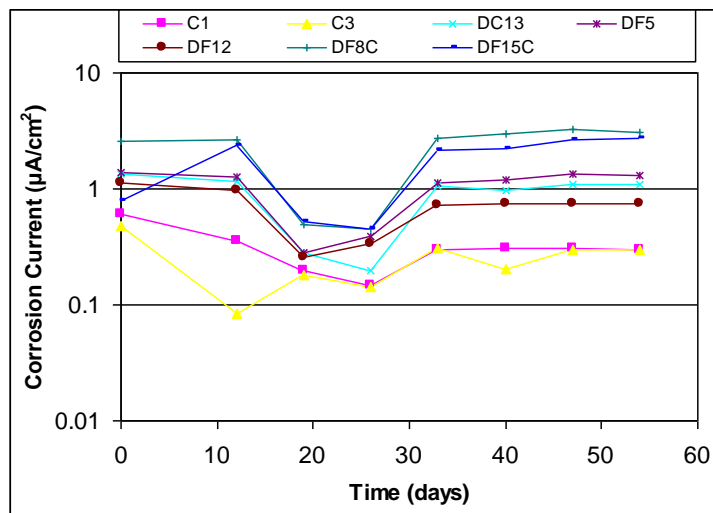


Figure 8. Variation of mean corrosion current with time of steel bars in the cut 'corrosion-monitoring' prisms  
 C = chloride-free controls, DC = desalinated, cast-in chlorides, DF = desalinated, ingressed chlorides and  
 DFC = ingressed chlorides, controls

All the individual results are summarised as plots of corrosion potential versus corrosion current (Figs 9 & 10). Such plots can both indicate differences between conditions and reveal the possible corrosion mechanisms involved. Points lying in the top left hand corner of the plots show a low incidence of corrosion. Those lying in the bottom right corner suggest a high level of corrosion. If all the points lie in a straight line between the two extremes the likelihood is that corrosion is controlled by polarisation/depolarisation of the anodic half of the corrosion process, that of the dissolution of iron (Glass et.al, 1991). The two plots (Figs 9 & 10) as well as suggesting that anodic control is the predominant mechanism, they also clearly show that the different conditions have resulted in separate populations of points. In Figure 9 the differences are small and relatively low but significant corrosion was seen in the desalinated cast-in chloride condition. A somewhat higher corrosion level had resulted in the desalinated ingressed chloride condition but was significantly lower than the equivalent controls (Fig. 10).

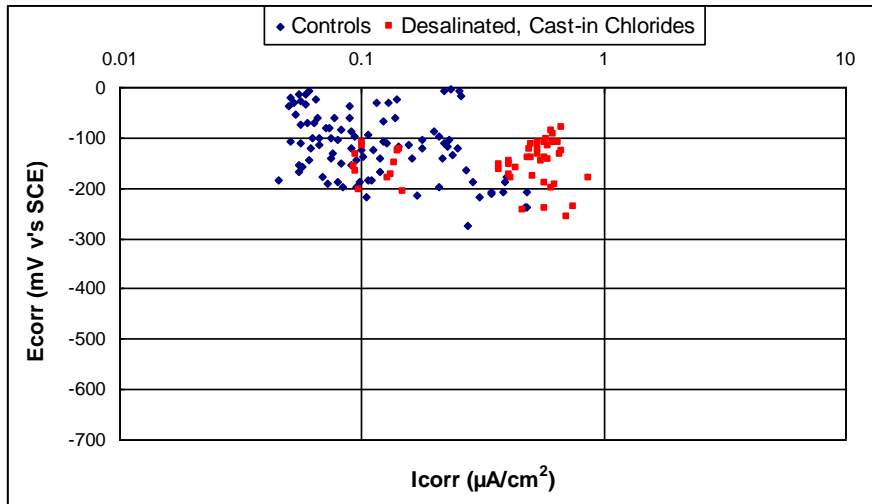


Figure 9. Corrosion current versus potential of all measurements taken for the chloride-free control and desalinated cast-in chloride conditions

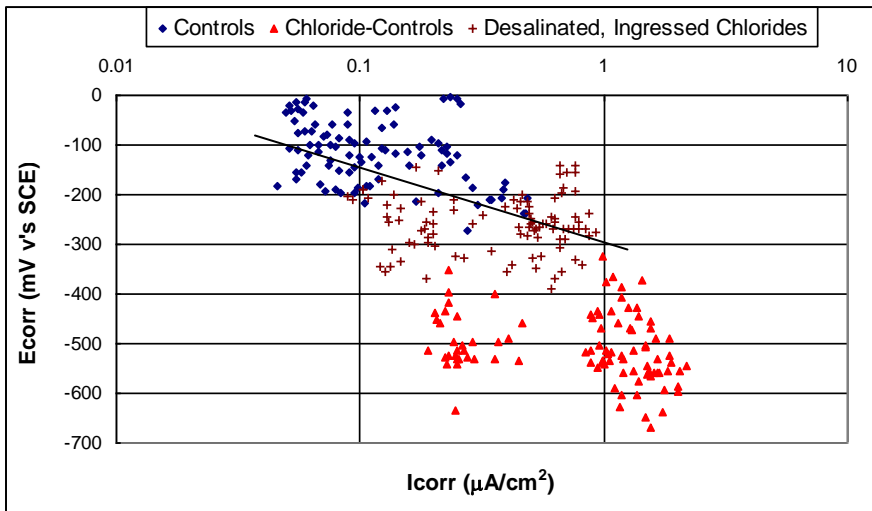


Figure 10. Corrosion current versus potential of all measurements taken for the chloride-free control, ingressed chloride control and desalinated ingressed chloride conditions

The chloride controls appeared also to exhibit cathodic control behaviour. This is signified by a potential that is reducing in value but a corrosion current that is essentially unchanging. In view of the low level of potentials ( $\sim 600\text{mV}$ ) the controlling parameter seems to be the low availability of oxygen. This is supported by the formation of magnetite (black low-oxygen corrosion product) seen on steel in the chloride control prisms.

The results suggest, therefore, that the process of desalination in concretes containing relatively high levels of chloride reduces the level of corrosion but not to a level that may be considered insignificant. Cracking and some rust staining appearing on a number of blocks along the steel bars (Tables 3 & 4), particularly on those containing ingressed chlorides, confirm that the desalination process as currently applied can only be partially successful particularly if corrosion of the steel had been occurring prior to the treatment. This is likely to be related to the fact that not all chlorides can be successfully removed. As explained earlier, the process becomes less efficient as the chloride concentration diminishes in relation to the total ionic concentration.

Figures 11 and 12 indicate this. A significant level of chloride had been removed by desalination but the concentration remained significantly higher than the chloride-free control. Even though there was no convincing evidence in the results to suggest that re-migration of chlorides towards the steel had occurred even after more than 4 years following the desalination process, the relatively high remaining chloride concentration, in the region of 0.5-0.9% by weight of cement, had allowed significant corrosion of the steel bars to continue. The possibility of chlorides re-migrating to the steel with time must be a possibility as chlorides are unlikely to be removed efficiently if they exist behind the steel reinforcement and in regions between the steel bars. That removal of chlorides is less efficient between steel bars can be seen in Figure 2 which shows the variation of chloride concentration with distance from a bar towards the centre of the steel grid at a constant depth from the exposed surface.

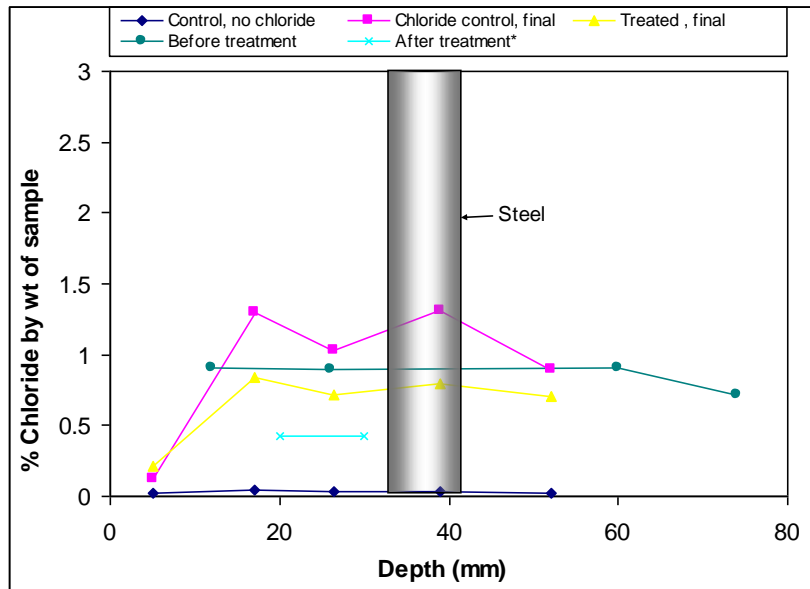


Figure 11. Cast-in chloride concentration profiles before and after desalination treatment  
 \* Concentration determined by industrial partner after carrying out treatment

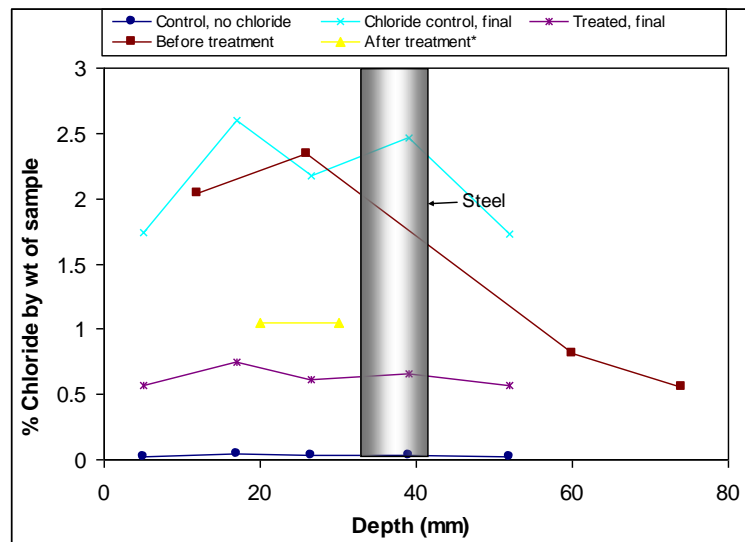


Figure 12. Ingressed chloride concentration profiles before and after desalination treatment  
 \* Concentration determined by industrial partner after carrying out treatment

Visual inspection and weight loss determinations of the bars confirmed the results, enabling a ranking of degree of corrosion, starting with the least, as follows:

1. Chloride-free Controls
2. Desalinated Cast-in Chloride
3. Desalinated Ingressed Chloride
4. Ingressed Chloride Controls

This is clearly shown in Figure 13 where the corrosion rate as cross sectional loss/year determined electrochemically is compared to the corrosion rate as the total cross sectional loss determined gravimetrically.

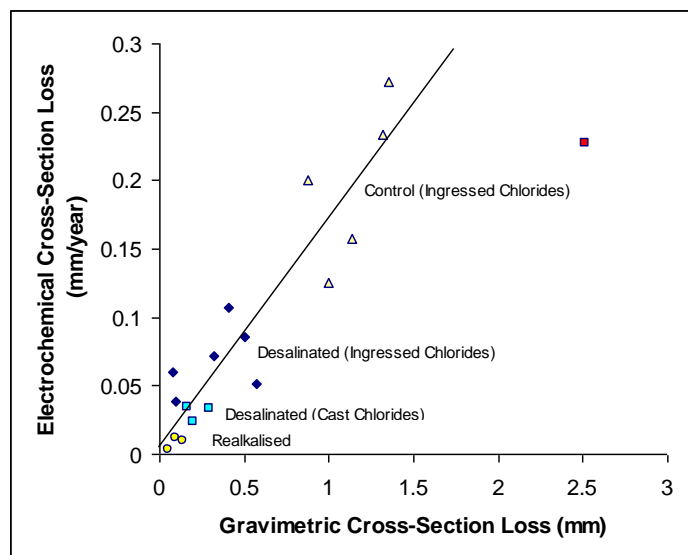


Figure 13. Comparison of cross sectional loss of steel bars in concrete prisms determined either electrochemically or gravimetrically

## CONCLUSIONS

The efficiency of chloride removal by the process of desalination appears to be reduced sharply with time of treatment. Following the initial treatment a total reduction of chloride in the region of 50% was achieved but a second identical treatment a few weeks later achieved no more than a further 3-5% reduction. This was not unexpected since, as the concentration of chloride diminishes, less proportion of the current is used to transport the chloride ions to the external electrolyte as opposed to that carried by the other more numerous ions.

Under the specific conditions of these trials, corrosion of the steel reinforcement was reduced after desalination of chloride-contaminated concrete slabs but remained at a significant level, particularly in the case where chlorides were ingressed from an external source as opposed to a lower concentration of chlorides cast-in.

The level of corrosion appeared to be related primarily to the chloride concentration remaining adjacent to the steel. As a significant level of chloride remains or may re-migrate to the area of the steel from the bulk, corrosion is unlikely to be totally eliminated in the long term.

## ACKNOWLEDGMENTS

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