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Efficiency of Cathodic Prevention to Control Corrosion in Seawater Mixed Concrete

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ABSTRACT

Large amount of potable water is used in construction industry. Due to shortage of potable water, use of seawater for both mixing and curing concrete seems to be an ideal alternative. However, major drawback of using seawater in concrete is large amount of chloride present in it, which leads to corrosion of embedded reinforcement in concrete. The main objective of the present study is to explore use of Cathodic Prevention (CPre) technique to control corrosion and successfully use seawater for making concrete. In the present study, three types of specimens were tested i.e. (i) mixed with potable water, (ii) mixed with seawater, and (iii) mixed with seawater and with CPre to compare corrosion rate in seawater mixed concrete and to check efficiency of CPre to control it. Corrosion in specimens was monitored using both direct current (HCP and LPR) and alternating current (ac) based (EIS) technique. Effectiveness of CPre is monitored both in terms of 100 mV decay criterion and using EIS. The results show that application of the CPre technique just after casting of concrete can allow successful use of seawater for making steel reinforced concrete. Moreover, compared to dc technique, EIS technique provides more detailed and accurate data during monitoring of steel corrosion, CPre and it is more helpful in identifying prevalent corrosion control mechanisms in seawater mixed concrete.

Keywords: Electrochemical Impedance Spectroscopy; Cathodic Prevention; Non-Destructive Technique; Corrosion; Seawater; Reinforced Concrete

1. INTRODUCTION

Shortage of water is major issue around the globe and large amount of potable water is used in construction industry [1]. Seawater on the other hand is available in large quantity. Thus, use of seawater for making concrete could be an alternative solution to conserve freshwater. However, corrosion of steel bars embedded in the

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seawater concrete is a major issue due to presence of large amount of salts in it [2]. One of the possible remedial measure to control corrosion in seawater concrete is application of cathodic prevention technique. Cathodic protection is an electrochemical technique used for preventing ongoing corrosion in reinforced concrete structures subjected to chloride penetration [3–5]. U.S. Federal Highway Administration memorandum stated that ‘the only rehabilitation technique that has been proven to stop corrosion in salt-contaminated bridge decks regardless of the chloride content of the concrete is cathodic protection’ [6]. CP can be applied to control corrosion in chloride contaminated structures or to prevent corrosion in new structures [7, 8]. The latter technique is referred as cathodic prevention [9]. Application of cathodic prevention technique increases the critical chloride threshold required for corrosion initiation and, therefore, maintain steel reinforcement in passive conditions even in the presence of very high chloride content [10–12]. Thus, cathodic prevention could be beneficial to control corrosion in seawater mixed concrete.

Corrosion of steel in concrete and effectiveness of different corrosion control techniques are monitored using various electrochemical and non-destructive techniques [13]. These techniques analyze corrosion as an electrochemical phenomenon [14]. These techniques are relatively less time consuming, less destructive and show greater reliability compared to other used destructive techniques [15]. The techniques can be grouped into direct current (dc) and alternating current (ac) techniques. Most common “dc” techniques used for corrosion monitoring on site and in laboratories are Linear Polarization Resistance (LPR), Half-Cell Potential (HCP) and Tafel extrapolation, while Electrochemical Impedance Spectroscopy (EIS) is the only “ac” technique [16–18].

One of the limitations of HCP method is that it does not provide quantitative information about corrosion rate (CR) of reinforcement [19, 20]. Determination of CR of steel in concrete has great significance for the evaluation of structural assessment, service life prediction and evaluation of repair methods [20]. LPR method provides a quantitative information on corrosion rate, however, the value obtained is instantaneous value and is largely influenced by climatic changes such as temperature, humidity [20, 21]. Moreover, it cannot separate the contribution of various processes involved in the corrosion process [22].

To monitor CP in RC structures, the satisfactory performance criteria typically used is 100 mV depolarization [23]. However, there is a problem of ohmic drop (IR) with the potential measurement, which adds a negative voltage to the potential measurement. Therefore, a technique is required which can directly detect corrosion and determine the efficacy of CP [23].

Among the current testing methods, EIS is relatively new and powerful method [24, 25]. It can be used to obtain various information for a steel-concrete system such as the presence of surface films, concrete characteristics, presence or absence of diffusion processes, interfacial corrosion and so on [14, 24]. The main advantage of this technique is that it is non-destructive and allows the modifications that can take place in the same material to be monitored [26]. Previous studies on EIS were focused on suggesting different equivalent circuits [14, 15, 27, 28], investigating cement hydration [29, 30] and carbonation behavior of OPC and other blended cement [24, 25]. However, its applications are limited due to the difficulties involved in interpreting the data, amount of work involved and high data dispersion, which requires specialist operators [31]. There is still a lack of information about the EIS method for analyzing corrosion in seawater mixed concrete, detailed investigation of identifying processes responsible for the change and to monitor cathodic protection in concrete. Due to limited knowledge about this technique, its use is restricted despite being more detailed and accurate than any other “dc” technique.

This paper investigates use of CPre to control corrosion in seawater concrete by application of EIS as a monitoring technique to determine corrosion rate of steel and check the CPre efficiency.

2. THE ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY (EIS)

2.1 Principle

The principle of this technique is to apply a small amplitude voltage (5-20 mV) to the working electrode at a wide range of frequencies and measure response in terms of current, where both current and voltage are sinusoidal in nature [16]. EIS technique generally works in the frequency range of MHz to Hz [32]. The response of the corroding system to “ac” excitation can be examined from EIS. In EIS, the measurements are generally made using three electrode system. They are a reference electrode, an auxiliary electrode and a working electrode. In the case of reinforced concrete, the working electrode is steel.

Since, in “ac” excitation, the frequency is not zero, Ohm’s law is represented by Eq. (1):

$$V = IZ \quad (1)$$

Where V and I are waveform amplitudes for potential and current, respectively, Z is impedance and shows the relationship between ΔV and ΔI .

2.2 Interpretation of Results

EIS data is often represented in the form of Nyquist plot or Bode diagram or both. In Nyquist diagram, the imaginary component (Z'') is plotted vs. the real component (Z') for each excitation frequency (Fig. 1). The abscissa is the real part and shows resistive terms, whereas the ordinate is imaginary part and shows capacitive or inductive terms [15]. Each point in the Nyquist plot represents magnitude and direction of impedance vector at a particular frequency. Another representation is in the form of Bode diagram, which shows logarithm of impedance modulus ($\log |Z|$) and the phase displacement (θ) as a function of the logarithm of frequency ($\log \omega$). These parameters are used for determination of absolute impedance [14].

For more precise information about the electrochemical process involved, data from EIS are fitted into an electrical circuit using the basic circuit elements e.g. resistors, capacitors and inductors. A typical impedance spectrum and its equivalent circuit for cement based materials are shown in Fig. 1 which consists of a solution resistance (R_s) connected in series with a combination of the resistor-capacitor circuit. Several electrochemical processes are involved in a corrosion process, thus equivalent circuit is composed of various circuit elements. The equivalent circuit may vary as per the processes involved [14].

3. EXPERIMENTAL PROCEDURE

3.1 Materials

All concrete specimens were made using OPC-43 grade cement, compliant with IS 8112:2013 [33] and locally available river sand (S). The specific gravity of sand was 2.6 and fineness modulus was 2.77. Both 12.5 mm and 20 mm down size coarse aggregate (CA) mixed in the proportion of 7:3 were used to achieve better packing. For reinforced concrete specimens, 500 grade ribbed steel rebar of 10 mm diameter was used. Artificial seawater was prepared as per the guidelines given in IS 8770:1978 [34] and used for mixing and curing, while potable tap water was used for mixing and curing of controlled specimens. The composition of artificial seawater is given in Table 1.

Table 1. Chemical composition of artificial seawater [34]

Compounds (Anhydrous)	NaCl	MgCl ₂	Na ₂ SO ₄	CaCl ₂	KCl	NaHCO ₃	KBr	NaF
Concentration (g/l)	23.5	5.0	3.9	1.1	0.66	0.20	0.10	0.003

3.2 Composition and preparation of the specimens

All the concrete specimens of M30 grade were cast during the study. Three sets of specimens were cast i.e. (i) mixed with potable water, (ii) mixed with seawater and (iii) mixed with seawater and protected with CPre.

Corrosion monitoring was carried out on slab specimens of size 300 x 300 x 50 mm having mix proportioning as given in Table 2. A ribbed steel bar of 10 mm diameter was placed concentrically in the specimens. Before using the steel bar, it was cleaned with a sand paper. The exposed length of the steel bar inside the specimen was 260 mm (Fig. 2). Specimens were demolded after 24 hours. Potable water and seawater mixed specimens are cured in potable and seawater, respectively for 28 days.

Table 2. Mix proportioning of concrete specimens

Mix Designation	Mixing and Curing Water	w/c Ratio	Binder (kg/m ³)				Admixture SP (by % of binder)
			W	C	S	G	
PW	Potable Water	0.45	180	400	700	1170	0.2
SW	Seawater	0.45	180	400	700	1170	0.2

For CPre, a 20 mm thick secondary anode consisting of a conductive mortar layer was provided over the top surface of the cured seawater concrete specimens. The conductive mortar was prepared with a sand to cement ratio of 1, water-cement ratio of 0.3 and carbon fiber (0.6% by volume of the mix). A 2 mm diameter titanium wire was placed into the mortar layer as a primary anode for providing the electrical connection to the secondary anode mortar layer (Fig. 3).

3.3 Cathodic Prevention and Monitoring

Three specimens of each potable water concrete and seawater concrete were left to corrode, while on the remaining 3 specimens of seawater concrete, CPre technique was applied. For initiating cathodic prevention current in the specimen, negative terminal of the rectifier was connected to the steel bar and positive terminal to titanium wire. The system was energized using a rectifier and a current density of 2 mA/m² of the steel bar. After 28 days of curing, the specimens were uniformly polarized for 10, 20 and 30 days, respectively and specimens were allowed to depolarize for 4 hours before evaluating the performance of cathodic prevention. EIS technique was used to monitor corrosion rate and performance of CPre in the concrete specimens and the potential shift was recorded using a copper/copper sulfate electrode (CSE).

The EIS measurements were carried out using a corrosion analyzer (ACM Field Machine) from ACM Instruments (UK), in the frequency range between 100 kHz and 100 Hz, and the amplitude of the AC signal was 10 mV/RMS. To avoid spatial distribution of impedance versus frequency, an electrical guard with specific auxiliary electrode was used. The test setup is shown in Fig. 4. The test was performed after 28, 56 and 90 days of casting and the obtained EIS data was analyzed using Zman 2.2 software by choosing an appropriate

equivalent circuit. For the present study, an equivalent circuit shown in Fig. 5 was selected for analyzing the experimental data received from the equipment [14, 15, 28]. Here R_0 is the offset resistance and R_2 represents the concrete resistance. Both are related to characteristics of concrete surrounding and protecting the rebar; Q_2 represents solid-liquid interface capacitance; Q_e represents electrode-cement system interface or double layer capacitance, which is formed due to redistribution of mobile charges at the boundary between electrode and cement; R_e represents the electrode resistance or charge transfer resistance and therefore related to corrosion phenomenon and is a measure of electric charge transfer through the electrode surface.

HCP and LPR tests were also performed to compare the results with EIS data. HCP was measured with the help of a copper-copper sulfate (CSE) reference electrode and a high impedance voltmeter. Electrical connection was established between the working electrode (WE) steel bar and the reference electrode. The potential difference between them was measured through a voltmeter. The polarization curves were recorded between -700 mV and +700 mV vs. OCP at a sweep rate of 121 mV/min using IVIUMSTAT equipment of IVIUM Technologies (UK) to obtain Stern–Geary constants. For the linear polarization measurements, a sweep range of -20 mV to +20 mV vs. OCP at a sweep rate of 6 mV/min was used, and the polarization resistance (R_p) was determined. Both HCP and LPR readings were recorded at an interval of a week for 90 days after curing.

4. RESULTS & DISCUSSIONS

4.1 Corrosion of Steel in Concrete

4.1.1 Corrosion monitoring using “dc” techniques

Potential values of seawater concrete specimens are found to be more negative than -450 mV when measured against Copper/Copper Sulphate (CSE) reference electrode (Fig 6). While in the case of potable water concrete specimens the potential values were more positive than -200 mV. These values indicate severe corrosion in seawater specimens and no corrosion in the controlled specimens as per ASTM C-876-2015 [35]. Thus, it can be observed that HCP data only gives an indication of the state of steel in concrete, no direct indication of corrosion rate can be obtained from it.

Corrosion current (I_{corr}) measured using the LPR technique is plotted in the Fig 7. From the obtained results, it was observed that seawater concrete shows high corrosion rate. The initial peak at 28 days was observed followed by a decrease in I_{corr} at 56 days. Again at 90 days increase in I_{corr} up to $4.8 \mu\text{A}/\text{cm}^2$ is observed indicating severe corrosion of steel bar. However, I_{corr} is almost constant in the controlled specimens; indicating negligible corrosion. Thus, it can be noted that though LPR provides corrosion rate values, it cannot separate the

contribution of various processes involved in the corrosion process. Moreover, polarization resistance value obtained from LPR varied in a wide range due to changing conditions on site.

4.1.2 Corrosion monitoring using “ac” technique

The slab specimens were subjected to AC impedance analysis after 28, 56 and 90 days. All the specimens showed semi- circular impedance spectra.

The impedance data depicted in Fig. 8 allow comparing the characteristics of concrete after 28 days of curing. The decrease in the diameter of the semicircle of seawater specimens is attributed to decreased polarization resistance and thus increased corrosion rate. As seen from the plot, seawater concrete presents the smaller impedance value as compared to potable water concrete. The possible reason could be the increased chloride ion concentration in seawater concrete, leading to increased electron movement due to corrosion reaction and thus reducing the polarization resistance.

Also, from Fig. 9 (Bode diagram), it can be observed that use of seawater reduces the impedance value at each frequency. The degree of phase angle describes the degree of surface corrosion of steel [22]. As seen from the plot, reduced absolute phase angle in seawater concrete attributes to decreased corrosion resistance of rebar. This shift in frequency shows the properties of passive film that undergoes different paths, becoming more stable in potable water concrete [36].

Fig. 10 corresponds to the impedance data after 56 days and 90 days. Comparing with Fig. 8, it is clearly seen that at all the ages seawater concrete has smaller impedance value. Moreover, the bulk arc is found to be expanding in its diameter when concrete specimens at 56 and 90 days, when compared with concrete specimens at 28 days. The reason may be the consumption of pore fluid with the progress of hydration process and thus resulting in increased bulk resistance. However, at 90 days seawater concrete shows reduced impedance value compared to 56 days, depicting considerably reduced resistivity of concrete.

All the impedance spectra have been set into the equivalent circuit shown in Fig. 5 using impedance analysis software Zman 2.2. Detailed analysis of model parameters is further carried out to identify the mechanism involved for the change and to detect the contributions of the various electrochemical processes involved. Table 3 summarizes the evaluation of the main model parameters with the age.

From the Table 3, it can be observed that in seawater concrete both- concrete bulk resistance (R_2) and charge transfer resistance (R_c) are much lower as compared to potable water concrete. With the progress of cement hydration, C-S-H gel is formed, which has a larger volume than cement. Hence the expanding cement gel

particles during hydration process blocks the continuity of conductive paths and thicken the discontinuous paths [27]. All these changes increase R_e and R_2 values with hydration time. The variation in the data was observed to be less than 5%, which is and within experimental error.

Table 3. Results obtained by equivalent circuit fitting of the impedance spectra

Model Parameters	Potable Water Concrete			Seawater Concrete		
	28 Days	56 Days	90 Days	28 Days	56 Days	90 Days
R_0 (Ω -cm ²)	1380	3130	857	954	498	1200
R_2 (Ω -cm ²)	5090	9280	8870	274	371	2.53×10^{-03}
Q_2 (F)	7.65×10^{-10}	2.46×10^{-10}	1.32×10^{-08}	6.90×10^{-04}	3.09×10^{-09}	1.78×10^{-08}
Q_e (F)	2.23×10^{-08}	2.46×10^{-10}	3.45×10^{-05}	1.42×10^{-08}	2.18×10^{-08}	1.76×10^{-01}
R_e (Ω -cm ²)	5510	9280	8990	2770	7330	6930
I_{corr} (μ A/cm ²)	4.71	2.80	2.89	9.39	3.54	3.75

Due to volume expansion of corrosion product and micro-crack initiation, hardened concrete deteriorates as observed. This leads to lower resistance (R_2) in the case of corroding seawater concrete specimens. Moreover, due to increased chloride ion concentration in seawater concrete specimens, critical chloride content is exceeded the limiting value required for corrosion initiation, chloride ion reaches reinforcement and destroys passive layer around the reinforcement locally. This may be the possible reason for the decrease in R_e value. The corresponding interfacial capacitance (Q_e) for seawater concrete specimens is higher than that of control specimens, which shows higher adsorption of chloride ions on the steel-concrete interface, decreased passivity of rebar and corrosion damage on the large surface area.

Using R_e values, corrosion rate was calculated using Eq. 2. The corrosion rate is evaluated using Stern-Geary relationship [37], where I_{corr} is the corrosion rate (μ A/cm²), B is a constant (mV) and R_p is polarization or charge transfer resistance ($k\Omega$ -cm²).

$$I_{\text{corr}} = \frac{B}{R_p} \quad (2)$$

For calculation of I_{corr} , B value of 26 mV was considered. Table 3 shows almost 34% higher corrosion rate in corroded specimens as compared to controlled specimens at 90 days.

From these results, it can be concluded that EIS technique provides reliable and more detailed results on corrosion rate, concrete bulk resistance, and polarization resistance in a reasonable time and can be successfully used as a non-destructive field technique. The overall scatter of the data is not bigger than in any other instrumentation used for corrosion rate determination in the field [20].

4.1.3 Comparison of CR from ac and dc technique

Fig. 11 shows a linear relationship between corrosion rate obtained from LPR and EIS. The graph shows a good co-relation exists between the two methods. However, LPR results show too high R_p values, as a consequence, the corrosion rates obtained from LPR are strongly underestimated. However, wide application of EIS is limited by the difficulties involved in interpreting the data and greater amount of work involved compared to dc techniques.

4.2 Application of Cathodic Prevention Technique to Control Corrosion in Seawater Concrete

The principle of CPre or CP is to deliver an appropriate cathodic polarization current to the protected structure so that the potential of the protected structure is negatively shifted and the corrosion is arrested or steel moves into passive zone [38]. The suitability of CP can be assessed on two bases: (1) first involves thermodynamic considerations which include moving steel to the immune zone or passive zone in Pourbaix diagram (2) second involves examining the kinetics of involved reactions based on experimental measurements of current to potential relationships of both cathodic reactions and metal dissolutions [39]. Some of the methods used for monitoring are Absolute Potential, Polarization curves, Depolarization method and AC impedance response.

4.2.1 CPre monitoring using 100 mV decay criteria

For monitoring CPre or CP in concrete structures, the most frequently used monitoring criteria is 100 mV depolarization, which relies on the fact that 100 mV cathodic polarization is sufficient to mitigate corrosion. Fig. 12 shows the initial potential and potential decays determined on steel in concrete specimens containing seawater.

The open circuit potential just prior to applying CPre was -450 mV (CSE). After 20 days of application of 20 mA/m² of current density, the 100 mV decay criteria was achieved. The data also shows that it is difficult to achieve large potential shifts on actively corroding steel in short term. However, the potential measurement does not provide a direct measurement of corrosion rate and the results may show some error due to the voltage drop. However, CR can be indirectly calculated by putting the applied current density and steel potential shift values into the Butler Volmer equation (Eq. 3) [40–42].

$$i = i_{corr} \left(\exp\left(\frac{2.3\Delta E}{\beta_c}\right) - \exp\left(\frac{2.3\Delta E}{\beta_a}\right) \right) \quad (3)$$

Where β_a and β_c are the anodic and cathodic Tafel Slopes (Both β_a and β_c are assumed as 26 mV in this study), i is the applied current density, i_{corr} is corrosion rate and ΔE is the electrode potential shift. Results show that application of CPre decreases corrosion rate in seawater concrete.

4.2.2 CPre monitoring using EIS

The Nyquist plot obtained from EIS technique is depicted in Fig. 13 and model parameters obtained after fitting into an equivalent circuit is listed in Table 4. The variation in the data was seen to be less than 5% and within instrumental error. The R_e value is predominant in the case of cathodic prevention and is considerably affected by the presence of a passive film or other conductive layers covering the steel reinforcement.

Table 4. Model parameters obtained by equivalent circuit fitting of the impedance spectra before and subsequent to cathodic polarization

Condition	R_s ($\Omega\text{-cm}^2$)	R_2 ($\Omega\text{-cm}^2$)	Q_2 (F)	R_e ($\Omega\text{-cm}^2$)	Q_e (F)	I_{corr} ($\mu\text{A}/\text{cm}^2$)
Before CPre	673.73	1912.18	1.21×10^{-05}	4908.95	2.84×10^{-08}	5.29
After 10 Days of CPre	2094.11	3670.99	9.38×10^{-09}	8959.79	7.33×10^{-05}	2.90
After 20 Days of CPre	634.13	18684.70	2.41×10^{-08}	18972.26	3.02×10^{-14}	1.37
After 30 Days of CPre	889.50	20717.97	1.66×10^{-08}	22398.98	1.91	1.16

Interestingly, it can be observed from Table 4 that charge transfer resistance values increase with the polarization time. The increased R_e value shows that progressive repassivation process was induced after application of CPre. This is primarily because of cathodic reaction at steel (Eq. 4), which increases pH near the steel surface.



Table 4 also shows that application of CPre technique is decreasing corrosion rate with polarization time in corroded concrete specimens. I_{corr} value drops to almost 78% after 30 days of polarization, indicating the efficiency of CPre to control corrosion in seawater concrete.

For a correct determination of potential measurement, the compensation of IR drop is considered essential [21] and this adds negative voltage to potential measurement [23]. Compared to 100 mV criteria, EIS technique has the advantage that R_e is determined as an integral part of the measurement, thus R_e values obtained are IR free. Comparing the corrosion rate from two techniques, it can be observed that corrosion rate obtained from 100 mV

criteria are overestimated (Fig. 14). Hence, it can be stated that EIS seems to be a more feasible technique to directly measure corrosion rate or to monitor a structure being cathodically prevented.

5. CONCLUSIONS

The present study reveals the efficiency of cathodic protection to control corrosion in seawater concrete and applicability of electrochemical impedance spectroscopy for monitoring corrosion and cathodic prevention of steel in concrete. Conclusions drawn from the study are:

1. Corrosion in seawater mixed concrete can be controlled by adopting suitable cathodic prevention technique. However, the risk of damage due to the presence of chlorides should be considered. Moreover, the extrapolation of the results still needs to be studied in more detail.
2. In comparison to dc techniques, both for corrosion and cathodic prevention monitoring, EIS is more accurate. However, its applications are limited due to the difficulties involved in interpretation of the data, amount of work involved and high data dispersion, which require specialist knowledge.
3. In the case of CPre, the problem with conventional measurement technique is a voltage drop (IR) during potential measurement, which adds a negative voltage to the potential measurement. For this case, EIS has shown to be a better measurement technique to directly detect corrosion and determine the efficiency of CPre.
4. Future work involves comparing the cost of the suggested method of using seawater for making concrete with the cost of concrete made using potable water where it is not scarcely available.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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Figure Captions

Fig. 1 A Nyquist plot (a) and its equivalent circuit (b)

Fig. 2 A slab specimen for EIS study

Fig. 3 Specimen for cathodic prevention and its schematic diagram

Fig. 4 Setup for EIS measurement

Fig.5 Equivalent circuit according to Ribeiro et al. 2015 (Ribeiro et al. 2015) used for analysis

Fig.6 HCP vs No. of days

Fig.7 Change in corrosion current density with age

Fig.8 Comparison of the Nyquist plots of the impedance spectra of controlled and corroded specimens after 28 days

Fig.9 Comparison of the Bode diagram of the impedance spectra of controlled and corroded specimens after 28 days

Fig.10 Comparison of the Nyquist plots of the impedance spectra of controlled and corroded specimens after (a) 56 days (b) 90 days

Fig.11 Co-relation between CR from LPR and EIS

Fig.12 Depolarization behavior of steel in seawater concrete under CPre

Fig.13 Comparison of Nyquist plot of the impedance spectra before and subsequent to cathodic polarization

Fig.14 Co-relation between CR from 100 mV decay and EIS