



-RESEARCH ARTICLE-

The Effects of Sodium Nitrite on Corrosion Resistance of Steel Reinforcement in Concrete

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Abstract

The aim of this study is to investigate the effect of 0.1 M nitrite ions on the corrosion of the reinforcing steel and the compressive strength of the concrete. The electrochemical tests of the corrosion resistance of steel and reinforced concrete in 3.5% NaCl and 3.5% NaCl + 0.1 M NaNO₂ solutions was examined for 90 days. In the presence of 0.1 M nitrite ions polarization resistance (R_p) values of reinforced concrete were higher than those without Sodium nitrite. AC impedance spectra revealed the similar results with R_p measurements. The compressive strength of concrete specimens containing 0.1 M nitrite ions was measured and an increase of 14.7-38.9% was observed.

Keywords:

Reinforcing steel, Concrete, Corrosion, Inhibition, Free Energy (ΔG).

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Introduction

Corrosion is the deterioration of a metal, its alloys or steel rebars in concrete by reaction with the environment. The type of metal, the composition of the surrounding medium, the pH of the solution, whether the solution is aerated or deaerated, the solution temperature and the design effect are among the effective factors in corrosion.

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The loss of alkalinity in concrete provides cracks in the concrete therefore chlorides approach the reinforcing steel and prevent re-passivation reaction that leads to pitting corrosion. The accumulations of corrosion products such as iron oxides and hydroxides causes an internal stress within the concrete. Many methods have been applied to protect corrosion of reinforced steel; these methods include variation of the concrete formulation, cathodic protection, surface treatment of rebars (e.g., epoxy coating), and addition of inhibitors (Kılınççeker, 1991; Kılınççeker, 1998; Kılınççeker et al., 2002; Kılınççeker et al., 1999; Erbil et al., 1978; Pruckner et al., 1996; Kahyaoğlu et al., 2002; Dhouibi et al., 2002; Loto, 1992). A corrosion inhibitor decreases the corrosion rate of a material, typically a metal or an alloy and offer an effective, reliable solution when concrete is exposed to environment containing chloride. They have no adverse effect on the properties of the fresh and hardened concrete admixtures and prevent corrosion of the embedded steel during added in an adequate amount. Corrosion inhibitors are either added to concrete during its preparation or penetrated into the hardened concrete surface. Therefore, corrosion inhibitors obtain a protection by forming a protective film at the steel surface and reducing the ingress of aggressive ions into the concrete matrix (Monticelli et al., 2000; Goni et al., 1990; Dhir et al., 1990).

Inhibitors such as chromate and nitrite are used as passivating inhibitors. They oxidize the metal and make it passive. As nitrite ions, form a protective barrier; have been frequently studied for prevention of corrosion of various metals, under various conditions. Especially for iron based materials, 0.1 M nitrite ions act as anodic inhibitor and that this ion continues to exhibit this behaviour in environments which contains other aggressive ions (Lipkowski et al., 1992; Gaidis, 2004; Hou et al., 2000; Kılınççeker, 2008). Nitrite ions accompany the natural ferric oxide layer and prevent chloride ions from complexing with ferrous ions.

The aim of this study is to study the effectiveness of 0.1 M nitrite ions (NO_2^-), in providing corrosion protection to reinforcing steel in 3.5% NaCl solution. The effect of the inhibitor on compressive strength of concrete was also determined. The corrosion resistance was evaluated by measuring corrosion potentials, resistance to linear polarization, AC impedance spectroscopy, potentiodynamic and EIS (electrochemical impedance spectroscopy) instruments were used.

Materials and Methods

- **Materials and specimen preparations**

The characteristic properties of the concrete is given in Table 1. The density of saturated dry surface specimen of aggregate was $2.30 \times 10^3 \text{ kgm}^{-3}$ and the size varied from 0.5 to 7 mm. Cylindrical reinforced concrete samples of 70 mm long and 40 mm in diameter with embedded reinforcing steel bar were used for electrochemical studies (Fig. 1). The water compositions and solutions are indicated in Table 2. The water/cement (w/c) ratio was 0.50. Steel bars having the following composition in wt%: 0.11 C, 0.64 Mn, 0.032 S, 0.015 P, 0.015 Si and the rest Fe used for reinforcement. The electrodes 50 mm in length with 17.27 cm^2 contact area and 10 mm diameter coated with epoxy paint were used. Before each measurement, the samples were glazed using 600–1200 grades wet SiC papers. The electrodes were post-cured for 7 d and they were put in the external solution. Concrete specimens were separated into three groups. The first group was prepared using distilled water (specimen A) whereas the second was mixed with 3.5% NaCl (specimen B). The third group was prepared using 0.1 M NaNO_2 whereas the was mixed with

3.5% NaCl (specimen C). The data obtained on the external and mixing water solutions given in Table 2.

Table 1. Composition of the concrete.

	Mass (g)	Density (gcm ⁻³)	Volume (cm ³)
Water	225.00	1.00	225.00
Cement	450.00	2.95	152.54
Aggregate	1355.00	2.60	521.15
Air	-	-	101.31

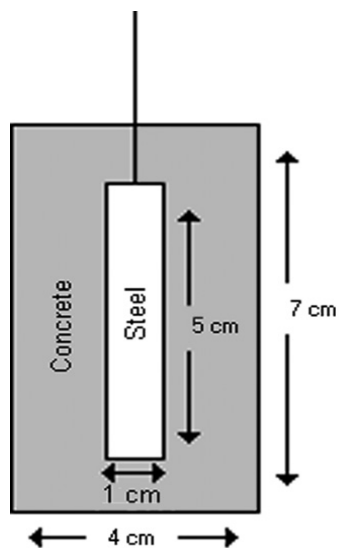


Figure 1. Working electrode (test electrode).

Table 2. External solution and mixing water environment.

Specimens	External and mixing solution
A	Distilled water
B	3.5%NaCl
C	3.5%NaCl + 0.1 M NaNO ₂

- **Test methods**
- **Measurements of corrosion potential,**

Measurements of corrosion potential, helps to explain the corrosion of reinforced concrete. Routine examination of reinforced concrete structures is one of the very important tasks. Therefore, to date 90 th corrosion potentials were measured and recorded at regular intervals (Kılınççeker et al., 2008).

- **AC impedance spectroscopy**

Impedance analysis were performed at the corrosion potential (E_{corr}), using a computer-controlled potentiostat and frequency response analyzer and the three-electrode assembly. Prior to the impedance analysis, a stabilization period of 30 min was allowed, which proved to be sufficient to reach a stable value for E_{corr} . The analysis applied with a sinusoidal potential waves of 5 mV amplitudes and frequencies ranging from 105 to 10-1 Hz. The measurements were achieved on the 7, 14, 21, 28, 60 and 90th day of the curing period (Kılınççeker et al., 2009). Concrete capacitance is given within the equivalent circuit in Fig. 2.

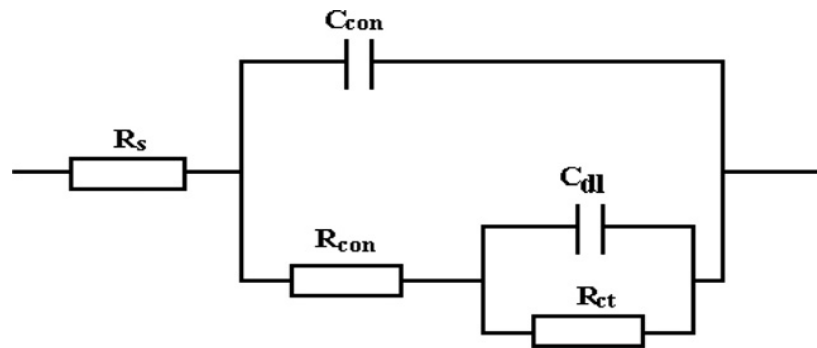


Figure. 2. Equivalent circuit for concrete (R_s , solution resistance; R_{con} , concrete resistance; R_{ct} , charge transfer resistance; C_{con} , concrete capacitance; C_{dl} , diffuse layer capacitance).

- **Potentiodynamic measurements**

The studied electrodes were polarized starting from the equilibrium state value (with respect to Ag/AgCl), first in cathodic and then in anodic direction at a rate of $0.01 \text{ V}\cdot\text{s}^{-1}$ in the range $-1.8 \text{ V} - +1.8 \text{ V}$. The current values measured were then converted to current density by taking into account the electrode surface area and semi logarithmic current-potential curves were also drawn (Kılınççeker et al., 2010).

- **Compressive strength**

In this part, cubic sized concrete samples (15 cm x 15 cm x 15 cm) were used. The compressive strength was carried out after 28 days preparing in external solution (Distilled water, 3.5% NaCl and 3.5% NaCl + 0.1 M NaNO₂) with a Humboldt testing machine. The average compressive strength was obtained from the four concrete cubes by loading at a constant rate of 5 kN/s using a hydraulically operated digital compression machine of 2000 kN capacity according to ASTM C 31 and ASTM C 39. When the evaluation was made, the pressure resistance was taken into consideration. The percentage of the compressive strength was calculated according to the equation below:

$$\% = [(A-B)/A] \times 100,$$

where A is the average compressive strength of four samples cured under distilled water, MPa; and B is the average compressive strength of four samples subjected to the test solutions, MPa.

Table 3. Compressive strength of the concrete specimens at 28 days.

Test series	Compressive strength (MPa)
Specimen A	20.5
Specimen B	15.2
Specimen C	28.6

Results and Discussion

- **Corrosion potential (E_{corr}) of embedded steel**

The effects of 0.1 M nitrite ions addition on the corrosion behavior of rebar in the concrete were monitored and the results are shown in Fig. 3 and Table 4. E_{corr} for the specimens in all media have varied from -0.154 to -0.929 V (versus Ag/AgCl) for a period of 90 days (Fig. 3). E_{corr} values in the concrete specimen without corrosion inhibitor (specimen A and B) was more negative than the corrosion potentials of the steel in the concrete specimen prepared with 0.1 M nitrite ions (specimen C).

On the average, the E_{cor} of the rebar in specimen B was ~0.100 V more negative than that of rebar in specimen A as is shown in Fig. 3. This indicated that the rebar embedded in the concrete containing 0.1 M nitrite ions (specimen C) had become more positive than the rebar in concrete without 0.1 M nitrite ions (specimen A, B). In the literature, it was informed that the corrosion potential obtained was more negative than -0.270 V / SHE and the corrosion target would be higher than 95% by decreasing -0.320 V / SHE, which meant that corrosion was occurred there. (Kılınççeker et al., 2008; 2009; 2010; 2011; ASTM C876-91, 1991). Hence, the adding of 0.1 M nitrite ions into concrete have decreased the corrosion rate and also have reduced

distilled water as external solution. However, the addition of 0.1 M nitrite ions in chloride solution could be effective on corrosion resistance of rebar in concrete. Specimen C that contained NO_2 ions more than specimen B were found to be less corrosive just as expected. It was shown that, nitrite ions could inhibit anodic dissolution of reinforcing steel in aggressive chloride media. The inhibition effect of nitrite ions was resulted from complex formations (between nitrite ions and corrosion products) which physically adsorbed on the surface. Therefore, the inhibition effects decreased with the chloride ions.

Due to nitrite presence at stable potentials lowers the flow density and also lowers the speed of diffusion oxygen molecules which causes the cathodic reaction happened on surface..

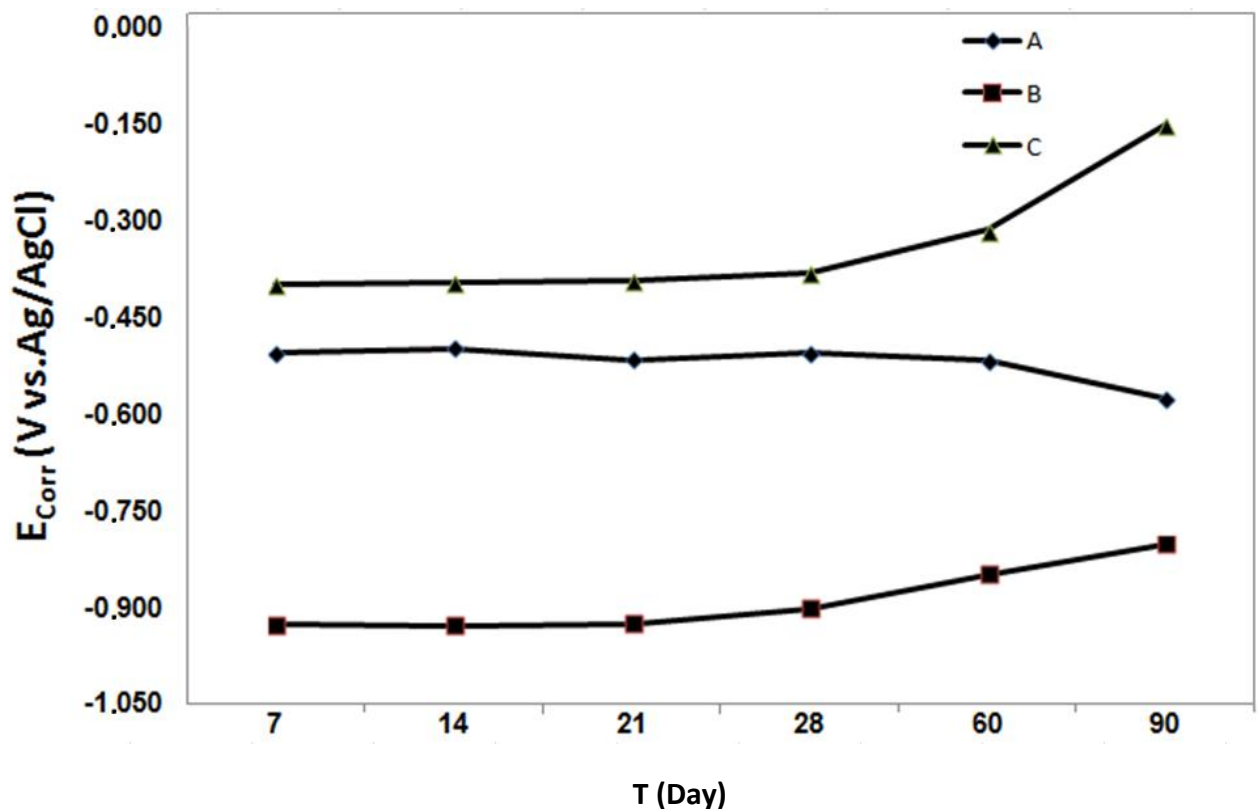


Figure 3. The corrosion potential versus reciprocal time for reinforcing steel in distilled water (A), 3.5% NaCl (B), 3.5% NaCl + 0.1 M NaNO_2 (C).

Table 4. The electrochemical data determined from polarization and impedance measurements for reinforcing steel at *pH* 8.0.

Solution	Time (Day)	E_{corr} (V vs. Ag/AgCl)	$E_{I=0}$ (V vs. Ag/AgCl)	R_p (ohm)	$I_{-1.400V}$ (mA.cm ⁻²)	$I_{-0.250V}$ (mA.cm ⁻²)	I%
Distilled Water	7	-0.506	-	3981.1	-	-	
	14	-0.500	-	4305.3	-	-	
	21	-0.517	-	4008.7	-	-	
	28	-0.507	-	4477.1	-	-	
	60	-0.518	-	4698.9	-	-	
	90	-0.578	-1.0469	4325.1	2.98×10^{-4}	2.91×10^{-4}	58.50
3.5%NaCl	7	-0.928	-	1258.9	-	-	
	14	-0.929	-	1374.0	-	-	
	21	-0.926	-	1402.8	-	-	
	28	-0.902	-	1603.3	-	-	
	60	-0.850	-	1527.6	-	-	
	90	-0.803	-0.8853	1794.7	2.15×10^{-3}	1.14×10^{-3}	-
3.5%NaCl + 0.1 M NaNO ₂	7	-0.401	-	9794.9	-	-	
	14	-0.397	-	9484.2	-	-	
	21	-0.395	-	11614.5	-	-	
	28	-0.383	-	12531.4	-	-	
	60	-0.316	-	13583.1	-	-	
	90	-0.154	-0.8955	12882.5	1.24×10^{-3}	1.81×10^{-4}	86.07

- **AC impedance method**

The impedance measurements on steel bar in the concrete samples were carried out using platinum electrode as a counter electrode, Ag/AgCl as a reference electrode and the steel bar embedded concrete as the working electrode (frequency range 0.1–10⁵ Hz). Figs. 4-6 (a, b) show impedance diagrams (as Nyquist and Bode diagrams) of steel in all concrete specimens. As shown in Fig. 4(a), during the first day, the diagram for the specimens has exhibited a semicircular change which is $R_s + R_{ct}$ until the real impedance value has reached 3981.1 Ω

(specimen A) during the first day in Table 4. This region, which is known as semicircle, can attain only one section because the surface structure of the steel is not disturbed at the applied maximum frequency. The solution resistance (R_s) can be obtained by extrapolating the semicircle curve to the real impedance axis (Z'/Ω). The region of the real impedance and the imaginary impedance, which increases continuously, is known as Warburg impedance. This resistance that is due to coating, accumulating of surface and concrete, causes shielding effect or resistance effect for the metal corrosion (Kılınççeker et al., 2008; 2009). Charge transfer resistance is the rate determining resistance at the reinforcing steel corrosion. During the seventh day, while $R_s + R_{ct}$ value for specimen A which was made of steel embedded in concrete containing only distilled water as a mixing solution and 3.5% NaCl as external solution, was 1258.9 Ω (specimen B), it increased to 9794.9 Ω for specimens C, respectively. These data indicated that specimen A from severe damage on its passive layer and that the addition of 0.1 M nitrite ions to the concrete, has composed a protective layer on the steel and concrete surface. Nevertheless, the resistance values gradually increased when the electrodes were held in the outer solution for longer periods. Due to the diffuse layer effect and the R_d resistance addition to the $R_{ct} + R_s$. After 90 days, the values of $R_s + R_{ct} + R_d$ have showed an increments from 9794.9 and 12882.5 Ω for specimen C,

respectively. The change in percent inhibitions ($I\%$) was calculated using $I\% = \frac{R_{p_{(inh)}} - R_p}{R_{p_{(inh)}}} \cdot 100$.

The calculated percent inhibitions were given in Table 4. Inhibitory activity % of the 3.5% NaCl + 0.1 M NaNO₂ solution varies from 85.51 to 88.75 (7th and 90 th day).

It is worth mentioning here that in all media, Warburg impedance region has showed similar changes. R_{ct} and C_{dl} that show resistance and capacitance and also R_{con} , concrete resistance, C_{con} , concrete capacitance is given within the equivalent circuit in Fig. 2. The rebar present in reinforced concrete has a pore structure where the capillary channels are in contact with external medium. When these capillary channels approach the metal ends, electrical double layer forms thus allowing charge transfer in this layer. Charge transfer resistance and double layer capacitance at equivalent circuit are shown as R_{ct} and C_{dl} in Fig. 2. The outside concrete resistance (R_{con}) and the proposed capacitance for the concrete (C_{con}) were fixed onto the circuit. The outermost double layer formed within the capillary channels and the solution resistance (R_s) that arose between the outermost of the concrete and the potential point measurement is also shown in Fig. 2. According to the equivalent circuit; high frequency region of Nyquist diagram shows the charge transfer resistance and diffuse layer resistance. However, increasing imaginary impedance in the lower frequency region is also considered as Warburg impedance (Fig. 4-7). These impedances are evaluated as a total resistances produced from both the concrete and corrosion products. At low frequency region (Warburg region), 0.1 M nitrite ions influence was not observed and thus this can be attributed to changes in $R_s + R_{ct}$ by chemical adsorption effect. However, a weak influence of 0.1 M nitrite ions indicates that a physical adsorption is present. Therefore, the effect of 0.1 M nitrite ions is thought to have increased the widespread layer resistance. By making the use of the corrosion potentials measured in different solutions, the change in free energy (ΔG) was calculated using $\Delta G = -nFE_{corr}$. The calculated free energy values were given in Table 5. The free energy of adsorption of 0.1 M nitrite ions (ΔG), in distilled water, 3.5% NaCl and 3.5% NaCl + 0.1 M NaNO₂ solutions was calculated from polarization and impedance data and found to be equal to 111.549, 154.973 and 29.720 kJ mol⁻¹, respectively (90th day). Reducing the free energy of adsorption decreases with the 0.1 M nitrite ions. It is well known that values of ΔG in the order of -20 kJ mol⁻¹ or lower indicate a physisorption; those

about -40 kJ mol^{-1} or higher involve charge sharing or a transfer from the inhibitor molecules to the metal surface to form a co-ordinate type of bond (Kılınççeker et al., 2008; 2010). The calculated value for the adsorption of 0.1 M nitrite ions on reinforcing steel shows that the adsorption is of a chemical nature, and there is chemical interaction between the inhibitor molecule and the metal surface. Oxygen diffusion comes from the external environment to the metal through the sipes and pores. As the added additives block these formed sipes and pores, diffusion becomes more difficult and the layer resistance increases (Kılınççeker et al., 2010; Pech-Canul, et al., 2002). The increase of the resistances in Nyquist diagrams and the decrease in shift of Warburg region to low frequencies supports this observation very well. Since the capillary channels in the concrete are not blocked, it is easier for the additive (0.1 M nitrite ions) to pass through and reach the metal surface from the external solution (solution without 0.1 M nitrite ions) in chloride medium. While 0.1 M nitrite ions in added to the mixing solution, molecules losses their mobility and thus the effect will be much less than that in the external solution. The resistance values of the specimens with 0.1 M nitrite ions in the mixing water and those with 0.1 M nitrite ions in the exposure solution are given in detail in Table 4.

The R_p values at various time were also obtained using polarization curves and Nyquist plots measured for these time (Figs. 4–6). The results are given in Table 4, but the potentiodynamic and EIS measurements were not given as separate figures.

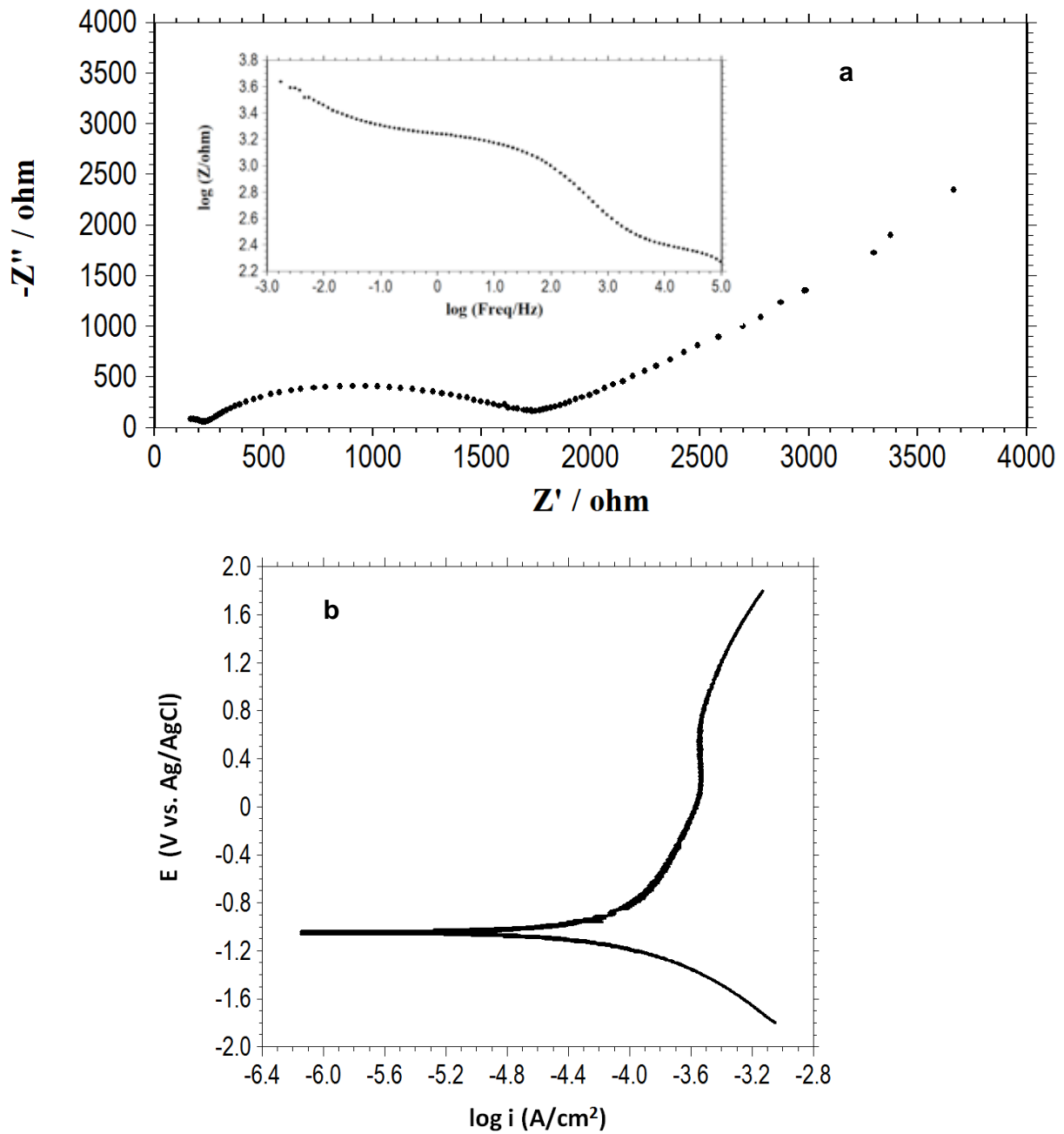


Figure 4. The Nyquist and Bode plots (a) and current-potential curves (b) of reinforcing steel in distilled water (90th day)

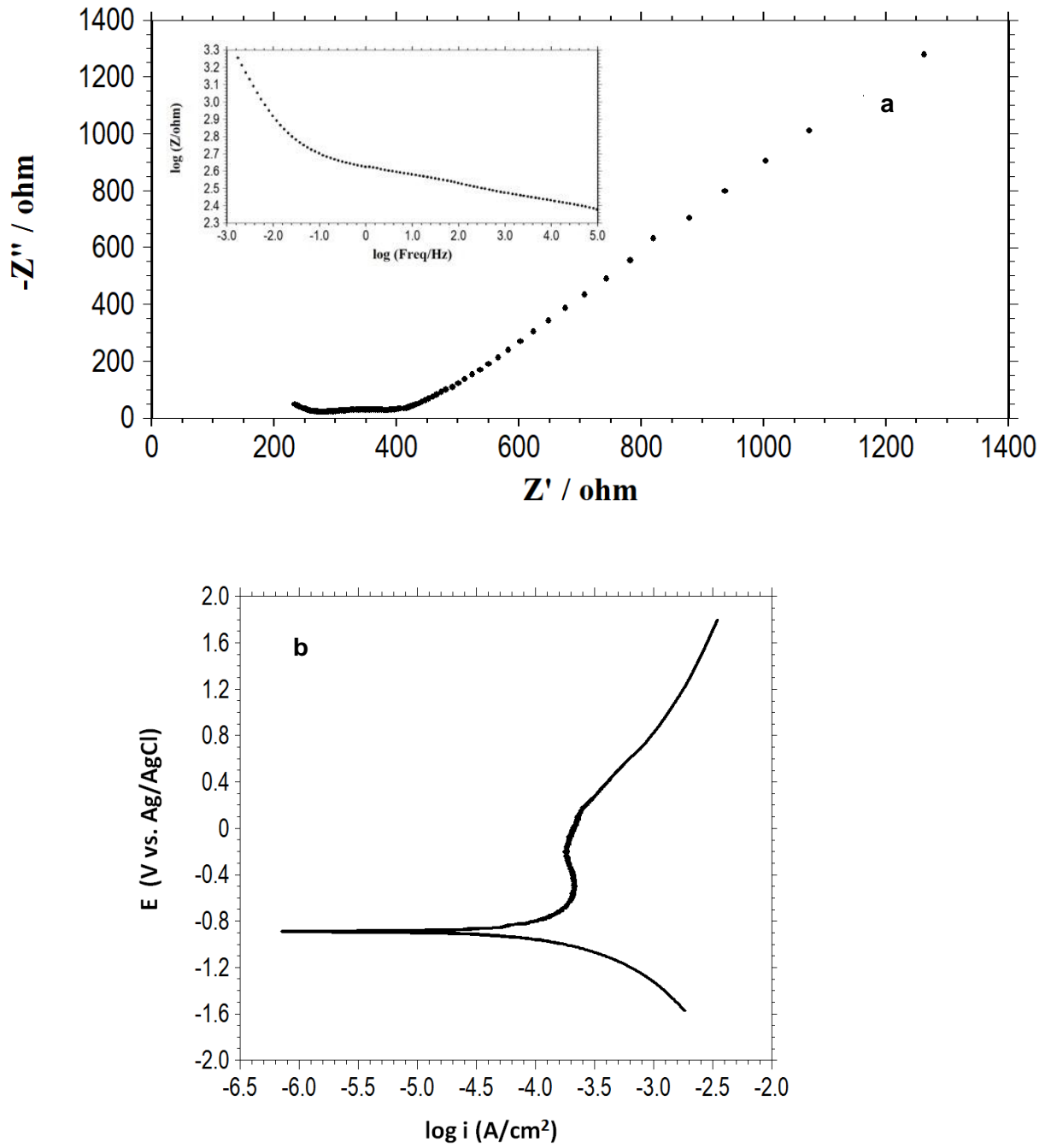


Figure 5. The Nyquist and Bode plots (a) and current-potential curves (b) of reinforcing steel in 3.5% NaCl solution (90th day).

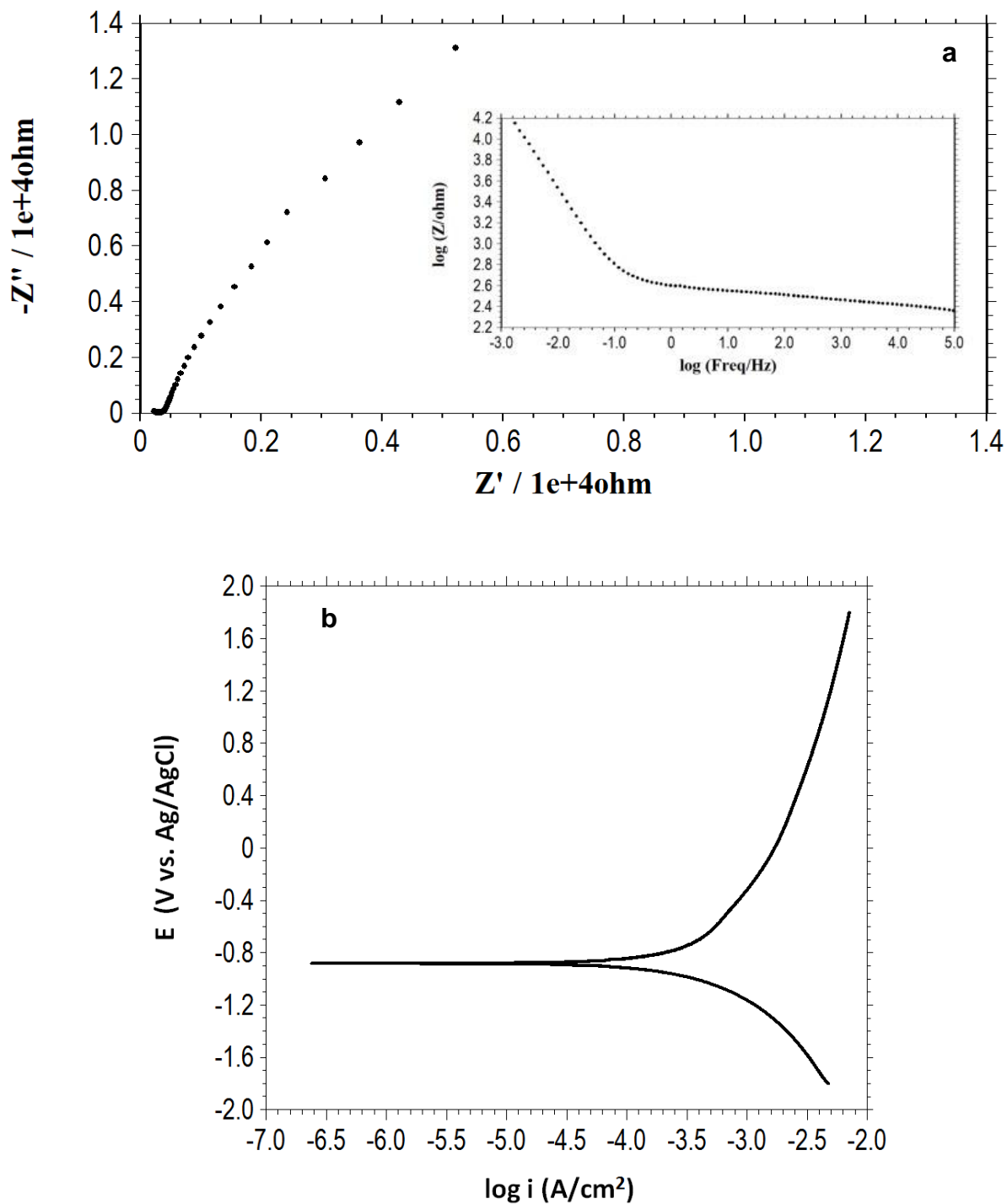


Figure 6. The Nyquist and Bode plots (a) and current-potential curves (b) of reinforcing steel in 3.5% NaCl + 0.1 M NaNO₂ solution at $pH=8.0$ (90th day).

Table 5. The corrosion potentials (E_{corr} (V vs. Ag/AgCl)), change in corrosion current and change in free energy values for reinforcing steel in different solutions having pH value of 8.0 under different days.

Solution	Time (Day)	E_{corr} (V vs. Ag/AgCl)	$\log i_{corr}$ (Acm^{-2})	DG ($kJ.mol^{-1}$)
Distilled Water	7	-0.506	-6.53	77.390
	14	-0.500	-6.04	96.496
	21	-0.517	-6.48	99.777
	28	-0.507	-5.80	97.847
	60	-0.518	-5.53	99.970
	90	-0.578	-6.01	111.549
3.5%NaCl	7	-0.928	-2.06	179.097
	14	-0.929	-1.89	179.290
	21	-0.926	-1.85	178.711
	28	-0.902	-1.62	174.079
	60	-0.850	-1.70	164.043
	90	-0.803	-1.44	154.973
3.5%NaCl + 0.1 M NaNO ₂	7	-0.401	-2.65	77.389
	14	-0.397	-2.74	76.617
	21	-0.395	-2.24	76.231
	28	-0.383	-2.07	73.915
	60	-0.316	-1.91	60.985
	90	-0.154	-2.01	29.720

• Potentiodynamic method

The potentiodynamic measurements on steel bar in the concrete samples were carried out using platinum electrode as a counter electrode, Ag/AgCl as a reference electrode and the steel bar embedded concrete as the working electrode (-1.8 V - +1.8 V). The current-potential curves of reinforcing steel in distilled water at pH 8.0 are given in Fig. 4-7 (c). The corresponding electrochemical data such as zero current potentials ($E_{I=0}$), the current density at -1.400 V ($I_{-1.400}$ v), the current density at -0.250 V ($I_{-0.250}$ v), percentage inhibition efficiency ($I\%$) values were calculated from Fig. 4-7 (c). As observed when time is increased the rate of anodic and cathodic reactions also increased the zero current potentials shift to more negative values (90th day). The obtained data are given in Table 4. In Fig. 4-7 (c) at 298 K, when the potential variation is considered in cathodic direction, oxygen reduction is seen partly and for the cathodic reaction in more negative potentials, it is seen that hydrogen reduction is predominant (Al-Amoudi, et al., 2003; El-Shafei, et al., 2001; Türkmen, et al., 2003; Pech-Canul, et al., 2002).

The corrosion current (i_{corr}) values for reinforcing steel in different solutions having pH value of 8.0 under different days. The obtained data are given in Table 5. The corrosion current distilled water, 3.5% NaCl and 3.5% NaCl + 0.1 M NaNO₂ solutions was calculated from polarization data and found to be equal to 9.77×10^{-4} , 36.3 and 9.77 mA.cm⁻², respectively (90th day). In the absence of Cl⁻ ions, which are known to have corrosive properties, according to the current-potential curves obtained at 298 K a protective film is formed on the surface of reinforcing steel (Kılınççeker, et al., 2015) and the current values decline (Table 4, 5).

- **Compressive strength**

All samples were tested for compressive strength on day 28th.(Table 3) Pressure value of concrete in distilled water were observed 20.5 MPa and 3,5% NaCl solution 15.2 MPa .Concrete strength in 3.5% NaCl solution (pH=8) with presence of nitrite measured as 28.6 MPa. The specimen C which contains chloride with nitrite ion is found the most resistible concrete. Admixed chlorides to mortar tends to react with the cement aluminates during the hydration of cement in this way, allowing a quicker entry of chlorides that comes from the environment once the concrete has hardened (Dehri, et al., 2000; Gürten, et al., 2005; Batis et al., 2001; Erbil, 1978). These results show that there was an increase of 39.5% in the strength of concrete by adding only 0.1 M nitrite ions.

Conclusion

The effect of 0.1 M nitrite ions on the corrosion of steel embedded in concrete was investigated and the adding of 0.1 M nitrite ions to the mixing or external solution was established to have increased the $R_{ct} + R_s + R_d$ resistance and have minimised the corrosion of the rebar in concrete. However, the specimen that contained distilled water only as mixing solution and 0.1 M nitrite ions as mixing or external solution, have demonstrated an analog rising in resistance. The corrosion of the rebar in concrete containing chloride was found to be inactive even when 3.5% NaCl with 0.1 M NaNO₂ were used as external solution. The tendency of corrosion is largely dependent on the corrosion potential and polarization resistance. Polarization resistance values showed a significant increase in the mixing or addition of 0.1M nitrite ions to the outer solution. Although the addition of distilled water containing 0.1M nitrite ions to concrete is significantly lower than the steel corrosion rate, the chloride ion is capable of spreading into the concrete mold and capable of depassivating a film on the reinforced steel surface. However, the addition of 0.1 M nitrite ions containing chloride have effected weakly the corrosion resistance of rebar in concrete. Corrosion potential values represented while the addition of 0.1 M nitrite ions to concrete used up by distilled water was effective in delaying the initiation of reinforcement corrosion, the addition of 0.1 M nitrite ions with chloride could not be as clearly effective on corrosion resistance of rebar. By the way, the increase in the strength varied from 20.5 MPa to 28.6 MPa in the concrete specimens used together with 0.1 M nitrite ions together with chloride ion. Finally, according to our research, the compressive strength of concrete was not adversely effected by 0.1 M nitrite ions.

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