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Inhibition of Acidic Corrosion of Low Carbon Steel by Novel Synthesized Benzothiazole Derivatives

Evrim HÜR[1]♠, Görkem Ali VAROL[1], Kamuran GÖRGÜN[1], Handan Can SAKARYA[1]

[1]*Eskişehir Osmangazi University, Faculty of Arts and Science, Department of Chemistry, 26480, Eskişehir, TURKEY*

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ABSTRACT

Two novel benzothiazole derivatives (2-[(1,3-benzothiazol-2-ylamino)methyl]-1-naphthol (1) and 2-{[(6-methyl-1,3-benzothiazol-2-yl)amino]methyl}-1-naphthol) (2) have been synthesized. The protecting effect of these compounds on low carbon steel (LCS) in 0.1M hydrochloric acid (HCl) solutions as corrosion media was investigated using potentiodynamic polarization technique, and electrochemical impedance spectroscopy (EIS). The potentiodynamic polarization curves indicate that investigated benzothiazole derivatives were acting as anodic inhibitor. The inhibitive action of inhibitor molecules may be interpreted in view of their adsorption on the LCS surface. The adsorptions of benzothiazole derivatives on metal surface in 0.1M HCl solution were found to obey Temkin adsorption isotherm. Thermodynamic parameters of adsorption (Kads, ΔGads) of investigated inhibitors were calculated using Temkin adsorption isotherm.

Key Words: *Benzothiazole, corrosion, steel, inhibitor.*

1. INTRODUCTION

The corrosion of metal and alloys in acidic medium and its inhibition is a mainly important problem in chemistry and industrial application [1-4]. Especially, steel finds wide application in broad spectrum of industry and machinery. However its tendency to corrode makes it unsuitable for exposure to acids [5]. The most of the efficient inhibitors used in industry are organic compounds which mainly contain oxygen, sulphur, nitrogen atoms and multiple bonds in the molecule through which they are adsorbed on the metal surface. The selection of a suitable inhibitor for a particular system is a difficult task because of the selectivity of the inhibitors and wide variety of corrosive environment [6]. Thus the synthesis of new organic compounds as corrosion inhibitor is important.

Benzothiazole and its derivatives is fused heterocyclic molecules and have ability of bonding with metal ions via donor atoms (N and S) to form heterocyclic ring-metal complexes. Moreover benzothiazole derivatives are already used as antioxidant, rust inhibitors and metal deactivators [7-10]. The inhibition efficiency of the organic compounds is related to the structure and the properties of the layer formed on the metal surface [11]. In the conception of ‘‘molecular structure’’, two components can be distinguished. The electronic structure is determined by the electronic density at the reaction centre in the molecule while the chemical structure is determined by the volume (area) and the steric distribution of eventual substituent.

The goal of this work is to synthesize and characterize novel bezothiazole derivatives (2-[(1,3-benzothiazol-2-ylamino)methyl]-1-naphthol, 2-{[(6-methyl-1,3-benzothiazol-2-yl)amino]methyl}-1-naphthol) and to investigate their inhibition efficiency against to corrosion of low carbon steel in HCl solution using electrochemical technique.

♠Corresponding author, e-mail: evrimhur@ogu.edu.tr

2. MATERIALS AND METHODS

2.1. Synthesis of Benzothiazole Derivatives

Melting points (mp) were determined on Gallenkamp apparatus, Infrared (FT-IR) spectra were recorded as pressed KBr disc, using Perkin Elmer Precisely Spectrum 100 FT-IR Spectrophotometer, ^{1}H NMR (500 MHz) were recorded in DMSO d_6 using Bruker DPX FT NMR (500 MHz) spectrometer instrument at 298 K. Chemical shifts are given in δ (ppm) relative to TMS. Elemental analyses were obtained on Elementar Analsensysteme GmbH varioMICRO CHNS (Turkish Technical and Scientific Research Council Laboratories, Ankara, Turkey). All the reactions were monitored by thin layer chromatography.

Typical procedure for the reactions for naphtaldehyde and heteroaryl amine to procedure imines

A solution of 2-amino-6-methylbenzothiazole (1eq.) and 2-hyroxy naphtaldehyde (1eq.) in methanol (100mL) was refluxed for 3h [12]. The mixture was left to stand at room temperature for overnight and then concentrated. The residue was washed with n-hexane (2x100 mL) and filtered off. The residue was hydrolyzed in water and extracted with EtOAc (4x50 mL). After drying in Na_2SO_4 and evaporation, the crude product was obtained. Products were recrystallized from CH_2Cl_2 to get corresponding Shiff bases in 75% yield.

Typical procedure for the synthesis of substituted 2-[(1,3-benzothiazol-2-ylamino)methyl]-1-naphthol (1)

2-[(1,3-benzothiazol-2-ylimino)methyl]-1-naphthol (1eq.) were dissolved in methanol. $NaBH_4$ was added to the stirred solution at room temperature until the yellow color of the Schiff bases were disappeared [13]. Cold water was added to the reaction mixture to precipitate the products 1-2. The precipitates were recrystallized from methanol to obtained compound (91%).

2-[(1,3-benzothiazol-2-ylamino)methyl]-1-naphthol (1) White crystals. Yield: 6.27g (91%) mp 186-187°C. IR vmax 3316, 1623, 1550 cm^{-1}; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 4.55 (s, 2H, Ar-CH_2N), 7.06 (t, 1H, J=7.51 Hz, H-5), 7.21 (d, 1H, J=8.84 Hz, H-4), 7.27 (t, 1H, J=7.38 Hz, H-6), 7.33 (t, 1H, J=7.28 Hz, H-14), 7.48 (d, J=8.02 Hz, 1H, H-7), 7.50 (t, 1H, J=7.23 Hz, H-13), 7.68 (d, 1H, J=7.77 Hz, H-16), 7.79 (d, 1H, J=8.87 Hz, H-12), 7.84 (d, 1H, J=8.07 Hz, H-15), 7.98 (d, 1H, J=8.54 Hz, H-17), 8.57 (s, 1H, <u>NH</u>CH_2Ar), 10.7 (s, 1H, Napht-OH). Anal. Calcd. for $C_{18}H_{14}N_2OS$: C, 70.56; H, 4.61; N, 9.14; S, 10.47. Found: C, 70.67; H, 4.67; N, 9.18; S, 10.53

2-{[(6-methyl-1,3-benzothiazol-2-yl)amino]methyl}-1-naphthol (2) White crystals. Yield: 6.52g (90%) mp 176-177°C. IR vmax 3323, 1623, 1599 cm^{-1}; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 2.33 (s, 3H, CH_3-Benzothiazole), 4.91 (d, 2H, $J_{CH_2N}=4.64$ Hz, Ar-CH_2N), 7.06 (d, 1H, J=8.10 Hz, H-5), 7.20 (d, 1H, J=8.85 Hz, H-4), 7.34 (d, 1H, J=7.31 Hz, H-16), 7.35 (t, 1H, J=8.18 Hz, H-14), 7.48 (s, 1H, H-7), 7.51 (t, 1H, J=8.23 Hz, H-13), 7.70 (d, 1H, J=8.87 Hz, H-12), 7.83 (d, 1H, J=7.98 Hz, H-15), 7.98 (d, 1H, J=8.53 Hz,H-17), 8.49 (t, 1H, $J_{NHCH_2}=4.68$ Hz, <u>NH</u>CH_2Ar), 10.6 (s, 1H, Napht-OH). Anal. Calcd. for $C_{19}H_{16}N_2OS$: C, 71.22; H, 5.03; N, 8.74; S, 10.01. Found: C, 71.34; H, 5.10; N, 8.76, S; 10.04.

2.2. Corrosion Tests of Benzothiazole Derivatives

Corrosion tests were carried out in a three electrode type cell using the reference electrode (Ag/AgCl) in a luggin capillary and the counter electrode (Pt wire). The working electrode was prepared from a cylindrical low carbon steel rod (%0.3 Mn, %0.1 C %0.05 S, %0.04 P and remainder iron) insulated with polyester resin such that the area exposed to solution was 0.1548 cm^2. Distilled water and analytical reagent grade HCl were used for solutions. The electrode was polished with 600, 800, 1000 and 1200-grit emery papers prior to each experiment using Metkon Grinding and Polishing Machine. Prior to measurements, working electrode was pre-treated in ethyl alcohol and acetone using an ultrasonic cleaner solution to remove the impurities of the metal. Electrochemical corrosion measurements were carried out using electrochemical impedance spectroscopy (EIS), and ***potentiodynamic*** techniques. Electrochemical impedance measurements were obtained at measured E_{ocp} values applying 8 mV of amplitude, in frequency range from 10^5 to 10^{-3} Hz. Potentiodynamic polarization studies was performed with a scan rate of 2 mV/s in the potential range from -400 to +400 mV relative to the corrosion potential. All corrosion measurements were performed in 0.5M HCl solution at room temperature and in open air with Gamry Model Reference 600 Potentiostat/Galvanostat. Each experiment was repeated at four times to check the reproducibility.

3. RESULTS AND DISCUSSION

3.1. Synthesis of Benzothiazole Derivatives

Condensation of substituted 2-aminobenzothiazole and 2-hydroxynaphtaldehyde analogues afforded benzothiazole Schiff bases. The products were checked by comparing the R_f values of TLC with the starting materials, which resulted in a single spot different from the starting materials.

The reduction of benzothiazole Schiff bases by $NaBH_4$ in methanol afforded the corresponding amine compounds 1-2 in good yields (Table 1). The structures of the compounds 1 and 2 were elucidated by elemental analyses, IR, ^{1}H NMR and ^{13}C NMR. IR studies of each amine compound confirm the formation –NH- groups, which were observed in the range of 3316-3323 cm^{-1}. The integral intensities of each signal in the ^{1}H NMR spectra of the amine compounds were found to agree with the number of different types of protons present. In compounds (1-2) -NH- proton signals were appeared in the range of δ 8.56-8.49 ppm as triplet. The signals for the hydrogens of the –CH_2-NH- group were detected in the region expected in range of δ 4.54-4.91 ppm for -CH_2-. Additionally, ^{1}H NMR spectra of benzothiazole amines give rise to multiplet signals at δ 7.06-7.98 ppm which is assigned to aromatic protons. These data confirm the proposed structures.

Table 1. Physical and analytical data of compounds.

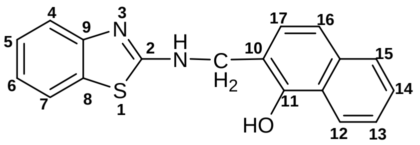
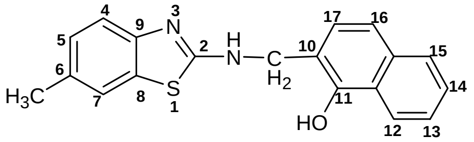
Compound	**mp°C**	**Yield (%)**	**Formula**	**M.W.**	**C%Found (Calc.)**	**H%Found (Calc.)**	**N%Found (Calc.)**	**S%Found (Calc.)**
1	186-187	91	$C_{18}H_{14}N_2OS$	306	70.67 (70.56)	4.67 (4.61)	9.18 (9.14)	10.53 (10.47)
2	176-177	90	$C_{19}H_{16}N_2OS$	320	71.34 (71.22)	5.10 (5.03)	8.76 (8.74)	10.04 (10.01)

3.2. Corrosion Tests

Potentiodynamic polarization

Figure 1 shows potentiodynamic polarization curves for LCS in 0.1M HCl solutions in the absence and presence of different concentrations of compound 1 and 2 (Table 2).

Table 2. The investigated molecules of inhibition efficiency.

Structure of molecules	Name of molecules	No
	**2-[(1,3-benzothiazol-2-ylamino)methyl]-1-naphthol**	1
	2-{[(6-methyl-1,3-benzothiazol-2-yl)amino]methyl}-1 naphthol	**2**

The corrosion current densities and corrosion potentials were calculated from the intersection of extrapolated anodic and cathodic Tafel regions. The changes observed in the polarization curves after addition of the inhibitor are usually used as criteria to classify inhibitors as cathodic, anodic, or mixed type [14]. From the Figure 1 with increasing concentrations of inhibitors, the anodic polarization curves shift towards anodic direction, and cathodic polarization curves towards cathodic direction.

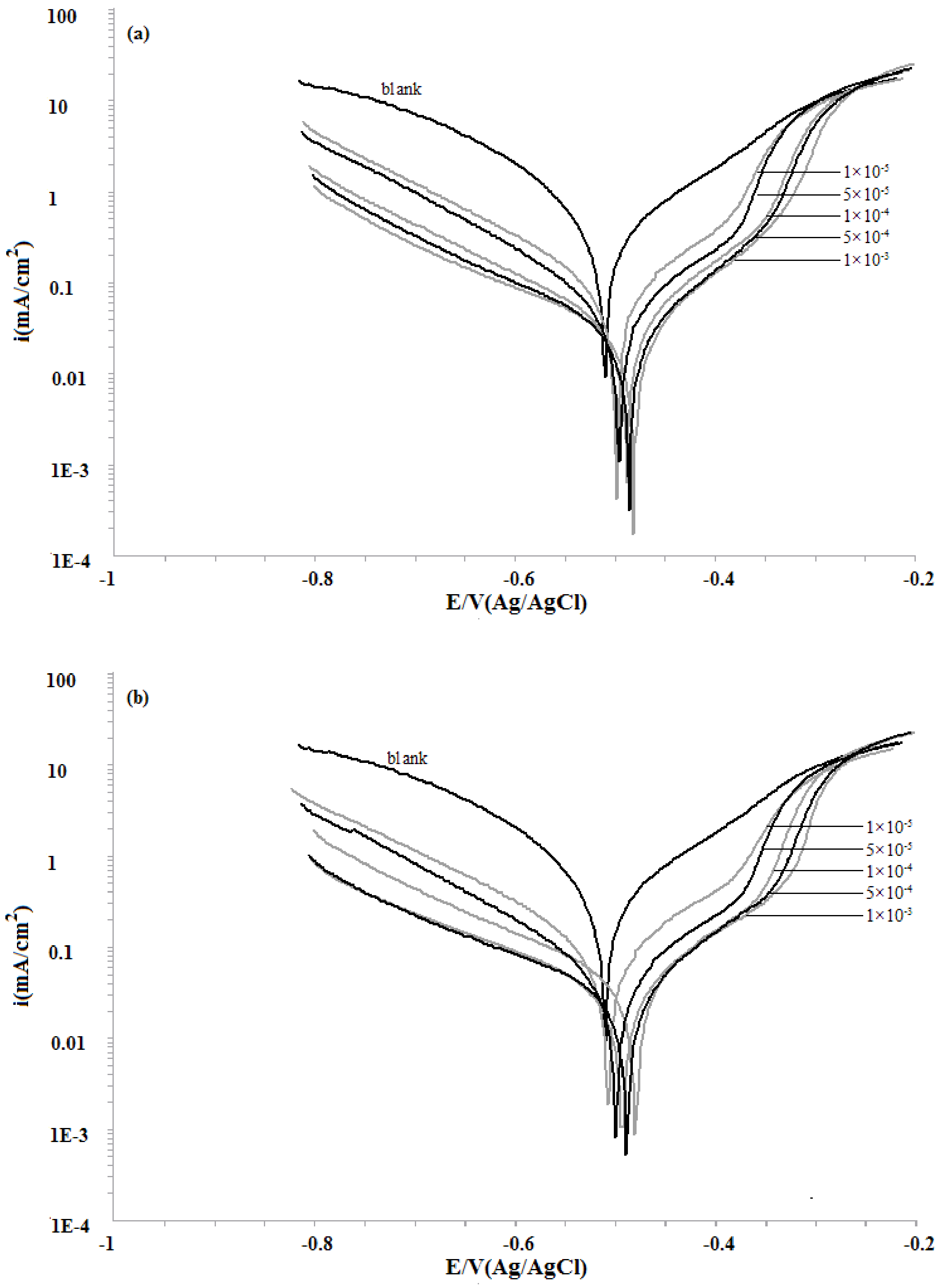


Figure 1. Polarization curves of LCS obtained in 0.1M HCl in the presence of various concentration of **(a)** inhibitor 1 **(b)** inhibitor 2.

The corrosion potential shifted towards more positive direction as regular than corrosion potential of reference curves. These results suggest that inhibitors can be classified as anodic corrosion inhibitors with predominantly control of anodic reaction [14, 15]. On the other hand, the corrosion current density decreased with increasing of inhibitors concentration. The percentage inhibition efficiency at different inhibitor concentrations was calculated using the following equation:

$$IE(\%) = \left[1 - \frac{i_{corr.}(inh.)}{i_{corr.}(blank)} \right] \times 100$$

where, $i_{corr.}$ *(inh)* and $i_{corr.}$ *(blank)* are the corrosion current densities for in the presence and absence of inhibitor, respectively. The calculated electrochemical parameters can be seen from Table 3. Inspections of the percentage inhibition efficiency show that inhibition efficiency increases with an increase in the inhibitor concentration. This result proposes that the inhibitor molecules are adsorbed on the steel surface by acting as adsorption inhibitor.

Table 3. Potentiodynamic polarization result and inhibition efficiency for the corrosion of LCS in 0.1 M HCl with and without addition of different concentration of compounds.

Compounds	**Inhibitor Concentration (M)**	**E_{corr} vs Ag/AgCl (mV)**	**i_{corr} (A/cm^2)**	**IE (%)**	**θ**
Blank	---	-511	438 x 10^{-6}	---	---
1	1×10^{-5}	-499	145 x 10^{-6}	66.9	0.669
	5×10^{-5}	-496	78.1 x 10^{-6}	82.2	0.822
	1×10^{-4}	-489	56.0 x 10^{-6}	87.2	0.872
	5×10^{-4}	-486	46.0x 10^{-6}	89.5	0.895
	1×10^{-3}	-482	40.8 x 10^{-6}	90.7	0.907
2	1×10^{-5}	-508	131.0x10^{-6}	70.1	0.701
	5×10^{-5}	-500	58.3 x 10^{-6}	86.7	0.867
	1×10^{-4}	-480	50.2 x 10^{-6}	88.5	0.885
	5×10^{-4}	-489	54.2 x 10^{-6}	87.6	0.876
	1×10^{-3}	-494	47.0 x 10^{-6}	89.3	0.893

In the HCl solution the following mechanism is proposed for the corrosion of iron and steel [16, 17]. According to this mechanism anodic dissolution of iron is as following:

$$Fe + Cl^- \rightleftharpoons (FeCl^-)_{ads}$$

$$(FeCl^-)_{ads} \rightleftharpoons (FeCl)_{ads} + e^-$$

$$(FeCl) \longrightarrow (FeCl^+) + e^-$$

$$FeCl^+ \rightleftharpoons Fe^{+2} + Cl^-$$

add cathodic hydrogen evolution is as following:

$$Fe + H^+ \rightleftharpoons (FeH^+)_{ads}$$

$$(FeH^+)_{ads} + e^- \longrightarrow (FeH)_{ads}$$

$$(FeH)_{ads} + H + e^- \longrightarrow Fe + H_2$$

Electrochemical Impedance Spectroscopy

Figure 2 shows the Nyquist plots obtained in 0.1M HCl solution for absence and the presence of different concentrations of inhibitors. Obtained Nyquist plots are depressed almost semicircles indicating a barrier layer formed on the steel surface. As can be seen from Figure 2, the Nyquist plots consist of one capacitive loop. The capacitive loop was attributed that corrosion process of metal is controlled by charge transfer [14, 18].

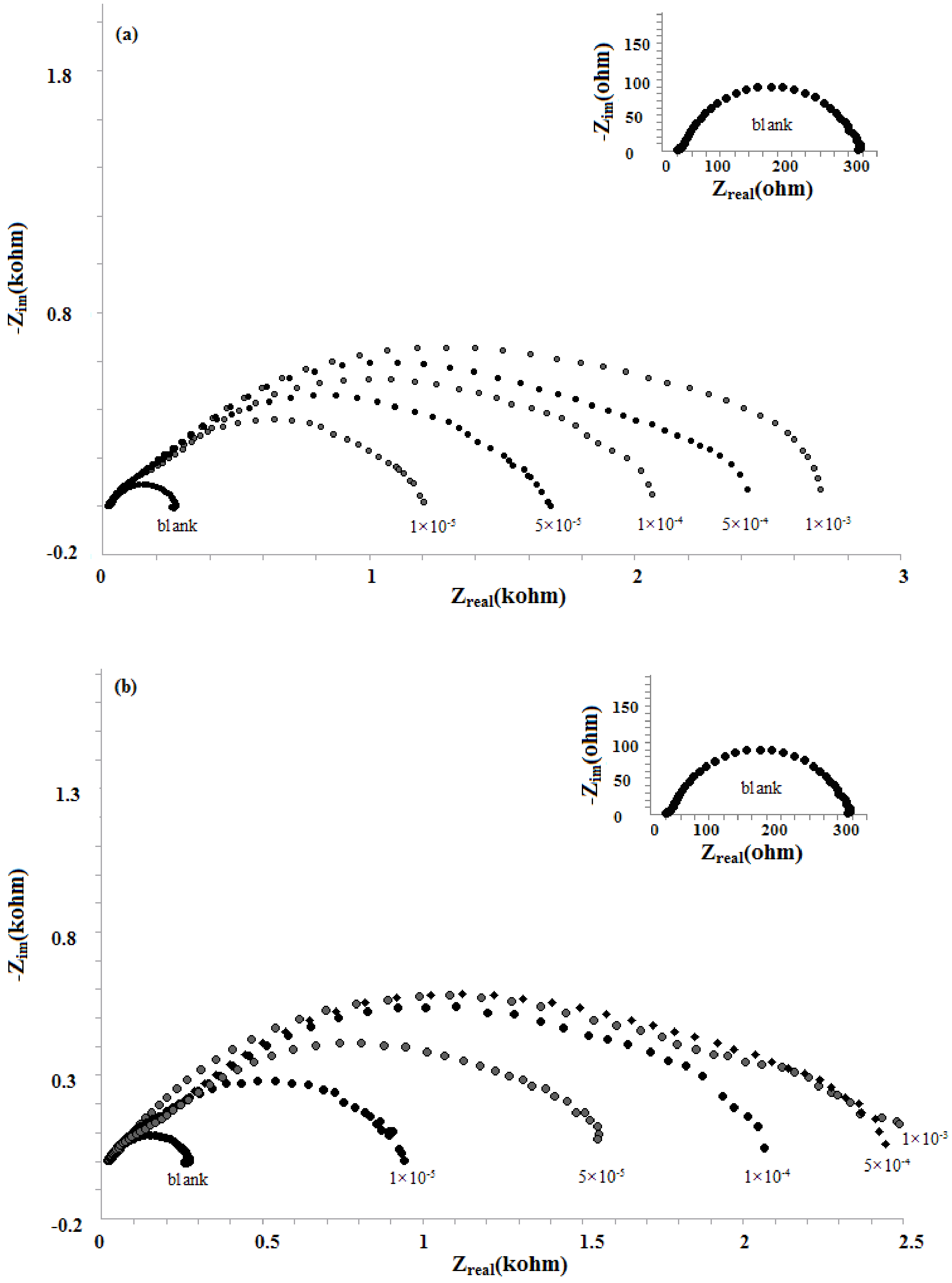


Figure 2. Impedance plots of LCS obtained in 0.1M HCl in the presence of various concentration of (a) 1 (b) 2.

The percent protection efficiency values were also calculated by using the following equation according to electrochemical impedance measurements and obtained results are given in Table 4.

$$IE(\%) = \left[1 - \frac{R_{ct.}(blank)}{R_{ct.}(inh.)}\right] \times 100$$

where, R_{ct} *(inh.)* and R_{ct} *(blank)* are the charge transfer resistance, with and without inhibitor, respectively. As can be seen from Table 4, R_{ct} values of each inhibitor increase with the addition of inhibitor when compared without inhibitor. In the same time, the values of R_{ct} were increased with the increasing inhibitor concentration. This result can be related to the formation of surface film.

Table 4. Impedance parameters and inhibition efficiency for the corrosion of LCS in 0.1 M HCl with and without addition of different concentration of compounds.

Compounds	**Inhibitor Concentration (M)**	**E_{corr} vs Ag/AgCl (mV)**	**R_{ct}(Ω)**	**IE (%)**	**θ**
Blank	---	-518	283	---	---
1	1×10^{-5}	-503	1303	78.3	0.783
	5×10^{-5}	-508	1735	83.7	0.837
	1×10^{-4}	-500	2151	86.8	0.868
	5×10^{-4}	-493	2486	88.6	0.886
	1×10^{-3}	-500	2892	90.2	0.902
2	1×10^{-5}	-507	1021	72.3	0.723
	5×10^{-5}	-480	1629	82.6	0.826
	1×10^{-4}	-493	2182	87.0	0.870
	5×10^{-4}	-495	2518	88.8	0.888
	1×10^{-3}	-495	2612	89.2	0.892

Application of Adsorption Isotherms

The surface coverage ratios (θ) for various concentrations of the inhibitors in 0.1M HCl were evaluated from EIS measurements. The obtained experimental data for the studied inhibitors have been applied to different adsorption isotherm equation. The best correlations have been obtained from EIS results and these results obey to Temkin adsorption isotherm equation [19, 20].

$$\exp(f\theta) = K_{ads}C$$

where, f is the molecule interaction parameters depending upon molecular interactions in the adsorption layer and the degree of heterogeneity of metal surface. C, K_{ads} and θ are the inhibitor concentration in bulk solution, adsorption equilibrium constant and ratio of surface coverage respectively. The surface coverage values were calculated using EIS data by following equation:

$$\theta = \left[1 - \frac{R_{ct.}(blank)}{R_{ct.}(inh.)}\right]$$

Typical plot of θ versus lnC for 1-2 were given in Figure 3. From the obtained straight lines in θ - lnC graphs, equilibrium constants of adsorption (K_{ads}) were obtained. The equilibrium constants of adsorption is releated to the free energy of adsorption (ΔG_{ads}) by;

$$K_{abs.} = \frac{1}{55.5} e^{-\Delta G_{abs.}/RT}$$

where 55.5 is the molar concentration of water in the solution, T is the temperature, and R is a gas constant. The calculated values of K_{ads} and ΔG_{ads} are given Table 5.

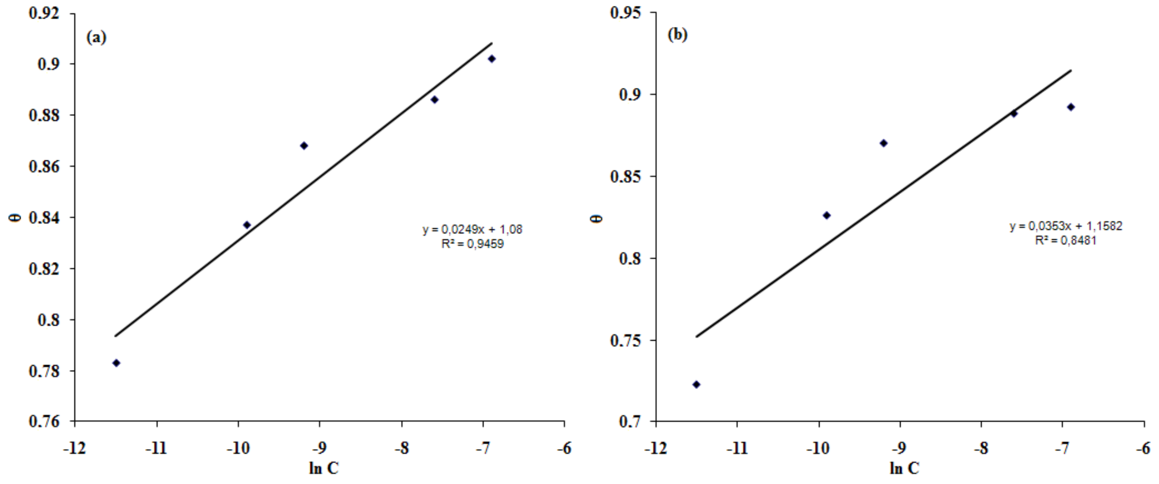


Figure 3. Plot of Temkin adsorption isotherm of (a) 1 and (b) 2 obtained by using surface coverage values calculated by the EIS.

Table 5. Thermodynamic parameters of adsorption obtained by EIS measurements for investigated benzothiazole derivatives on LCS in 0.1M HCl solutions.

Inhibitor	***f***	R^2	K_{ads}	ΔG_{ads} **(kJ/mol)**
1	40.16	0.95	43.37	-19.28
2	28.32	0.85	30.59	-18.42

The low and negative values of ΔG_{ads} indicate the spontaneous adsorption of inhibitors on the surface of steel. The negative values of ΔG_{ads} also suggest the strong interaction of the inhibitor molecules onto the steel surface [6, 21].

The studied inhibitor molecules show perfect inhibitor efficiency even in low concentrations of inhibitor in acidic solutions. The inhibition efficiencies of inhibitor 1 are slightly higher than that of inhibitor 2. The effectiveness of a compound as a corrosion inhibitor depends on the structure of the organic compound. The molecular structure is one of the major factors influencing the adsorption of the organic molecules on the metal surface, and hence the inhibitor properties, especially in the case of chemisorptions, which involve charge sharing or charge transfer from the inhibitor molecules to the metal to form coordinate type of bonds [9, 22]. Investigated benzothiazole derivatives in this study have heteroatoms N, O, and S. These heteroatoms can share their lone pair electrons with metal. Additionally, π electron clouds of aromatic rings can interact with the metal via π-d electron transfer [6,16, and 23].

4. CONCLUSION

The benzothiazole derivatives results of electrochemical investigations show that the benzothiazole derivatives decrease dissolution of steel in HCl solution. The inhibition efficiency increased with increasing of inhibitor concentration. The potentiodynamic polarization curves suggest that inhibitors 1-2 used as anodic inhibitor. The observation of capacitive loop in EIS curves indicates dielectric properties of the surface film. The R_{ct} values of inhibitor increased with increase in concentration of benzothiazole derivatives. The inhibition effect of the investigated benzothiazole derivatives is mainly due to their adsorption on the steel surface. The adsorption process obey Temkin adsorption isotherm. The K_{ads} and ΔG_{ads} values were calculated from adsorption isotherm. The negative ΔG_{ads} values were depicted to spontaneous adsorption process.

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