Anticorrosion Properties of Indazole Derivative for Corrosion Inhibition of Carbon Steel in 1 M HCl

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Abstract—The inhibitory effect of a new derivative of the indazole family, namely, (*Z*)-2-(1-methyl-5-nitro-1*H*-indazol-4-yl)-3-(3-nitrophenyl) acrylonitrile (51K2) against corrosion of carbon steel in 1 M HCl medium at a temperature of 25°C has been evaluated by different techniques such as gravimetric methods, potential-intensity curves, and electrochemical impedance spectroscopy. The impact of the concentration on the inhibition efficiency was also studied. The results of weight loss measurements and electrochemical analyses obtained showed that the addition of 51K2 increases the inhibition efficiency by decreasing the corrosion rate, with a maximum inhibitory efficacy of 80%. In other words, 51K2 is a good anodic-type corrosion inhibitor of carbon steel in 1 M HCl.

Keywords: corrosion, carbon steel, 1 M HCl, indazole, weight loss measurements, electrochemical techniques

INTRODUCTION

The high mechanical strength of carbon steel and its low manufacturing cost have made it possible to use steel widely as building material in several applications, such as chemical industries, transportation, energy production, industries of electrochemicals and petrochemicals, and others [1, 2]. Due to a serious damage to metals and alloy structures and economic significance in terms of replacing damaged products, those and other industries that use hydrochloric acid have researched and developed methods to solve the problems of steel corrosion in HCl [3–5]. Among effective anti-corrosion methods is the use of corrosion inhibitors [6–8].

Many well-known corrosion inhibitors are natural extracts and also heteroatomic synthetic organic compounds that contain atoms such as phosphorus, nitrogen, sulphur, and oxygen, these atoms having the ability to adsorb on the surface of a metal, thus forming a protective layer [9–17]. The most effective metal corrosion inhibitors for hydrochloric acid are organic nitrogen inhibitors, while for sulphuric acid the best organic inhibitors are those that contain sulphur. Organic compounds with nitrogen and sulphur act as perfect corrosion inhibitors for both acidic media [18–22].

The effectiveness of inhibitors with heteroatoms is inversely proportional to their electronegivity, with performance inhibition followed in the order of O, N, S, and P [23, 24]. The addition of organic inhibitors in acid solutions changes the electrochemical behavior of the medium: it generally decreases the aggressiveness of the solution [25–31].

MATERIALS AND METHODS

Sample and Test Solution Preparation

The material used as working electrode in this study was carbon steel S235JR whose chemical composition is given in Table 1.

Carbon steel samples of size $5 \times 1 \times 0.2$ cm^3 were first successively treated with abrasive paper of grades 180, 400, 600, 800, and 1200, and then cleaned by distilled water and degreased by acetone, according to the Block-on-Ring standard test method (ASTM G77) [32].

The corrosive solution of this study was 1 M HCl, as prepared via the dilution of an analytical solution of HCl 37% by distilled water. The concentrations of the studied inhibitor (*Z*)-2-(1-methyl-5-nitro-1*H*-indazol-4-yl)-3-(3-nitrophenyl) acrylonitrile (51K2) varied from 1×10^{-5} to 1×10^{-3} mol L^{-1} in the 1 M HCl medium.

Table 1. Chemical composition of used carbon steel

EU grade	C, %	Mn, %	P, %	Cu, %	Al, %	Si, %	Cr, %	Fe, %
S235JR	0.07	0.19	0.02	0.12	0.02	0.03	0.05	Rest

Synthesis of 51K2

The studied compound 51K2 was obtained via the Knoevenagel condensation reaction (Fig. 1) of nitroindazolylacetonitrile (A), analyzed in [33] with the aromatic aldehyde *m*-bromo-benzaldeyde. The reaction was performed in an ethanol solution with piperidine (1 equivalent), and the mixture was stirred at room temperature for 48h−96h. A thin layer chromatography of the reaction mixture revealed the consumption of the starting material and the formation of a precipitate. The filtration and crystallization in ethanol of the isolated solid afforded to produce 51K2 in good yield (79%) [34].

The structure of 51K2 obtained via the Knoevenagel reaction was confirmed by spectroscopic and spectrometric techniques, namely, 1D (1H and 13C spectra) and 2D [(1H,1H) COSY, (1H, 13C) HSQC and (1H, 13C) HMBC] nuclear magnetic resonance and mass spectrometry techniques.

Weight Loss Measurements

Weight loss measurements of carbon steel were performed by immersion of test coupons in 1 M HCl without and with the addition of 1×10^{-3}, 1×10^{-4} and 1×10^{-5} mol L^{-1} of the inhibitor tested at 25°C. Steel samples were weighed before and after 24 h immersion time, and before the final weighing the coupons were rinsed with distilled water and dried. Corrosion rate values (*CR*), the surface coverage (θ), and the inhibition efficiency (*IE%*) were calculated using Equations 1, 2, and 3, respectively:

$$CR = \frac{W - W'}{S \times T};$$ (1)

$$\theta = \frac{W - W'}{W};$$ (2)

$$IE\% = \frac{W - W'}{W} \times 100,$$ (3)

where W and W' are the weight loss values of carbon steel without and with the addition of the inhibitor, respectively, S is the total steel area in cm^2, and T the immersion time in h.

Electrochemical Experiments

An OrigaStat type potentiostat with the analytical software OrigaMaster was utilized for the electrochemical analysis. The corrosion cell used had three electrodes: a platinum countertop electrode, a saturated calomel reference electrode, and a carbon steel working electrode of a 1 cm^2 surface, submerged in the analysis solution. Before launching the Tafel potentiodynamic polarization (PDP) and the electrochemical impedance spectroscopy (EIS) tests, the open circuit potential (E_{ocp}) was measured under its stable condition during 30 min of immersion of the working electrode in the solution.

The EIS measurements were performed at the open circuit potential in the 1 kHz to 100 mHz frequency range, with a voltage of 5 mV of the AC sine wave. The diagrams obtained by EIS have been converted into the Nyquist plots. Following the EIS measurements, the Tafel PDP curves were obtained using a scanning rate of 20 mV s^{-1} of −750 to −100 mV in the direction from the cathode to the anode.

RESULTS AND DISCUSSION

Weight Loss Measurements

The study of corrosion and corrosion inhibition by the weight loss technique as a powerful tool is reported in many investigations [21, 35−38]. The gravimetric measurements of carbon steel in 1 M HCl without and
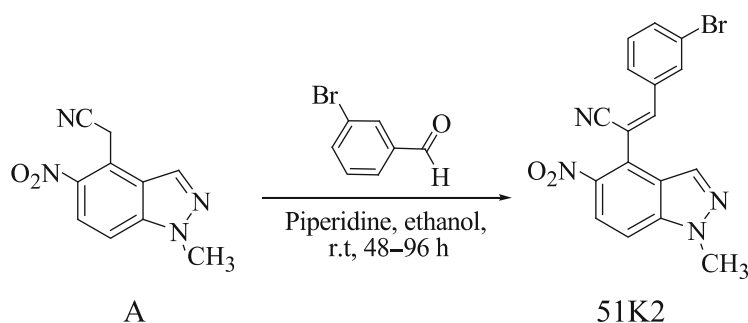

Fig. 1. Synthetic reaction of 51K2.

Table 2. Gravimetric parameters of carbon steel in 1 M HCl at 25°C in absence and in presence of different concentrations of 51K2 after 24 h immersion

Inhibitor concentration, mol L^{-1}	Weight loss, g	Corrosion rate, mpy	Surface coverage, θ	IE%
Blank	0.0715	262.69	–	–
51K2				
1×10^{-5}	0.0307	112.62	0.57	57
1×10^{-4}	0.0224	81.98	0.69	69
1×10^{-3}	0.0158	57.83	0.78	78

Table 3. PDP parameters for carbon steel in 1 M HCl without and with 51K2

Inhibitor concentration, mol L^{-1}	E_{corr}, mV	i_{corr}, mA/cm^2	b_c, mV	b_a, mV	IE%
Blank	−494.2	0.193	−84.3	80.6	–
51K2					
1×10^{-5}	−562.3	0.084	−93.3	83.2	56
1×10^{-4}	−556.2	0.062	−97.2	85.2	68
1×10^{-3}	−552.6	0.038	−92.5	90.7	80

with the addition of various concentrations of 51K2 were determined after 24 h of immersion at 25°C.

The effect of the inhibitor concentration was studied for concentrations of 1×10^{-3}, 1×10^{-4} and 1×10^{-5} mol L^{-1} of 51K2. The results were summarized in Table 2. The corrosion rate decreased at higher concentrations of the studied inhibitor while the inhibition efficiency increased and reached a maximum value of 78% for 1×10^{-3} mol L^{-1} of 51K2.

The inhibitor molecular structure is responsible for its adsorption at the metal/solution interfaces, the compound 51K2 is characterized by a high electronic density of benzene due to the presence of aromatic ring π electrons and pairs of heteroatoms electrons and also this molecule has active groups such as the nitro group, and cyanide that can be hydrolyzed in the presence of 1M HCl to the group amide, which has increased efficiency in addition to the bromide group. An increase in the inhibitor concentration promotes the amount of inhibitory molecules adsorbed at the metal/solution interface. Thus an insulating layer on the metal/solution interface is formed, which decreases the contact between the corrosive medium and the metal surface [39−43].

Potentiodynamic Polarization Measurements

Figure 2 gives the PDP curves of carbon steel in HCl 1 M solutions without and with different concentrations of 51K2 at 25°C. Electrochemical parameters

such as the corrosion current density (I_{corr}), the corrosion potential (E_{corr}), the Tafel slopes (b_c and b_a), and the inhibition efficiency (IE%) are given in Table 3. The inhibition efficiency was calculated using Eq. 4:

$$IE\% = \frac{i'_{corr} - i_{corr}}{i'_{corr}} \times 100, \qquad (4)$$

where i'_{corr} and i_{corr} are the corrosion current densities without and with the inhibitor, respec-tively.

Examination of Fig. 2 and Table 3 reveals that the addition of an indazole derivative slightly modifies the values of E_{corr}. The studied inhibitor 51K2 acts as anodic type one. Because it anodic current is controlled, so the corrosion mechanism is controlled by anodic dissolution. While according to b_c values, the inhibitor is simply blocking sites in the cathodic direction.

The IE% values in Table 3 demonstrate that the inhibition efficiency is proportional to the 51K2 concentration, at higher inhibitor concentrations and reaches 80%.

Electrochemical Impedance Spectroscopy

The Nyquist diagrams obtained in the absence and in the presence of different concentrations of the inhibitor in the 1 M hydrochloric acid solution are illustrated in Fig. 3, and Table 4 shows the correspond-
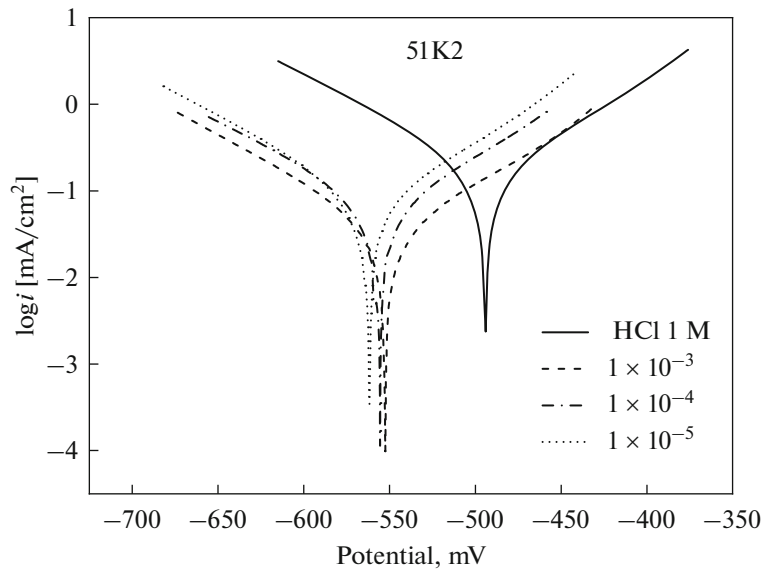
Fig. 2. PDP curves of carbon steel in 1 M HCl with different concentrations of 51K2.
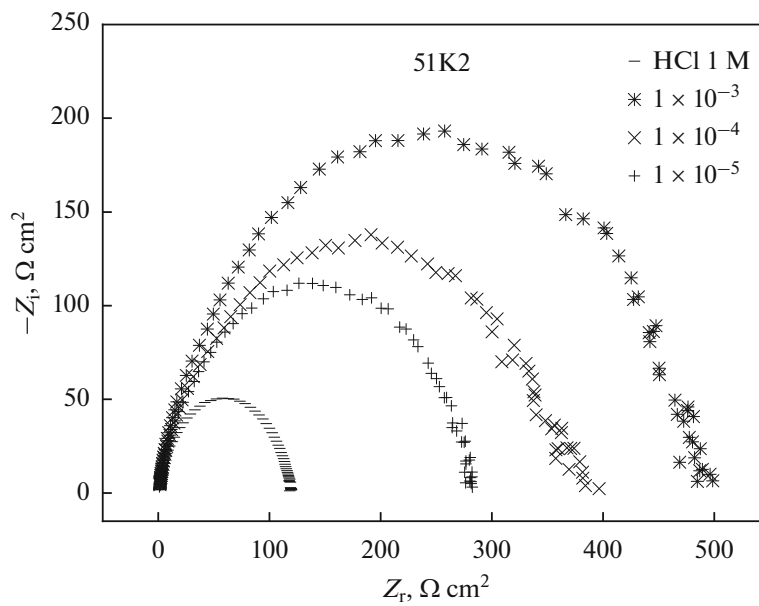

Fig. 3. Nyquist plots for carbon steel in 1 M HCl solution in absence and in presence of 51K2.

ing electrochemical parameters. The resistance to the load transfer was calculated using relationship (5):

$$E_R\% = \frac{R_t' - R_t}{R_t} \times 100, \qquad (5)$$

where $E_R(\%)$ is the inhibition efficiency, R_t' and R_t are the charge-transfer resistances in the inhibited and in the blank solutions.

Figure 3 shows that all impedance plots have single semicircles, indicating a formation of load transfer complexes between the metal surface and the inhibitor molecules at the electrode/solution interface, and that corrosion reactions are controlled by the transfer process [44, 45]. The imperfect semicircles in Fig. 3 are explained by the frequency dispersion due to the roughness of the steel surface and its inhomogeneous [46].

The impedance parameters as values of the charge transfer resistance (R_t), the double layer capacitance (C_{dl}), and the inhibition efficiency ($E_R(\%)$)) are given in Table 4. It is evident that increasing the inhibitor concentration increased the load transfer resistance

Table 4. Impedance parameters obtained for carbon steel in 1 M HCl solution in absence and in presence of different concentrations of 51K2

Inhibitor concentration, mol L^{-1}	C_{dl}, μF Cm^{-2}	Charge transfer resistance, Ω cm^2	E_R, %
Blank	133	119	–
51K2			
1×10^{-5}	63	282	58
1×10^{-4}	48	384	69
1×10^{-3}	45	499	76

and reduced the double layer capacity. The obtained impedance results, for comparison, are similar to those obtained by Naothers [37]. An increase in R_t values is directly related to a decrease in the local dielectric constant and an increase in the thickness of the double electrical layer, which leads to the formation of a protective layer on the surface of steel [47, 48]. A decrease in the double layer capacity values is most likely attributed to a gradual replacement of water molecules with organic molecules that is adsorbed on the surface of the metal [49].

A comparison of the results obtained via two electrochemical methods (EIS and PDP) shows that they are in agreement. The noticeable difference in the values of the effectiveness of inhibition via these methods may be related to the nature of each technique: PDP measurements provide real time kinetics of electrochemical processes and the polarization range of the potential with an irreversible change due to the measurement process [50], while EIS data are obtained at the open circuit potential and provide measured values of the overall resistance to the electrode/electrolyte interface.

CONCLUSIONS

The following conclusions can be drawn from the present study:

(1) The synthesized organic compound of the indazole family 51K2 acts as good carbon steel corrosion inhibitor in the 1 M HCl medium.

(2) The corrosion rate of carbon steel increases at higher concentrations of 51K2 in 1 M HCl solution.

(3) The inhibition efficacy values obtained by PDR, EIS, and weight loss measurements are in good agreement.

(4) The maximum inhibition efficiency of 82% was obtained at the concentration of 1×10^{-3} mol L^{-1} of 51K2.

(5) The PDP measurements demonstrate that 51K2 acts as anodic-type inhibitor.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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