

Research Article

Corrosion Inhibition of Carbon Steel in 0.5 M H₂SO₄ by Phtalocyanine Blue

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Phtalocyanine blue dye has been investigated as a carbon steel corrosion inhibitor in 0.5 M sulfuric acid by using polarization curves, electrochemical impedance spectroscopy, and gravimetric tests. Dye concentrations included 0, 100, 200, 400, 600, 800, and 1000 ppm, whereas testing temperatures were 25, 40, and 60°C. Results indicated that phtalocyanine blue is a good corrosion inhibitor with its efficiency increasing with the concentration up to 40°C, but it increases at 60°C. Inhibitor improves the passive film properties and it forms an adherent, compact, protective film, acting, therefore, as an anodic-type inhibitor. At 25 and 40°C the corrosion process was under charge transfer, whereas at 60°C the adsorption/desorption of some species from the metal surface controlled the corrosion process.

1. Introduction

Organic compounds containing atoms such as nitrogen, sulphur, and oxygen are commonly used to reduce the corrosion attack on steel in acidic media. These compounds adsorb on the metal surface, block the active sites on the surface, and thereby reduce the corrosion process [1–6]. The inhibiting action of such compounds is based on the adsorption ability of their molecules, where the resulting adsorption film isolates the metal from the aggressive media. The corrosion inhibition of organic compounds is related to their adsorption properties. Adsorption depends on the nature and the state of the metal surface, on the type of corrosive environment, and on the chemical structure of the inhibitor [7]. Studies report that the adsorption of organic inhibitors mainly depends on some physicochemical properties of the molecule, related to its functional groups, to the possible steric effects and electronic density of donor atoms; adsorption is supposed, also, to depend on the possible interaction of the π -orbitals of the inhibitor with the d-orbitals of the surface atoms, which induce greater adsorption

of the inhibitor molecules onto the surface of metal, leading to the formation of a corrosion protection film [8].

Recently, there has been a lot of attention paid to the use of organic dyes as potential corrosion inhibitors for different metals environments [9–24]. Thus, Oguzie evaluated the use of Congo red [9], methylene blue [10], indigo blue [11], safranin-o, thymol blue, and fluorescein [12] as corrosion inhibitors for mild steel in acidic media, although in some cases they evaluated these dyes for aluminum in hydrochloric acid [13]. Alizarin yellow and Bromophenol blue have been used also but now studying the synergistic effect of iodide ions for the corrosion inhibition of carbon steel in acidic media [14, 15]. Additionally, alizarin violet [16], 3H-phenothiazin-3-one, 7-dimethylamin [17], phenylazopyrazolones [18], acid violet 6B [19], and methyl violet [20] have also been studied as corrosion inhibitors for cold rolled steel in acidic environments. Finally, some traditional analytical indicators, for example, methyl red [21], cresol red [22], methyl orange [23], and fast green [24], behave as good corrosion inhibitors for steel in acid media. Thus, the aim of this work is to evaluate the corrosion inhibitory properties

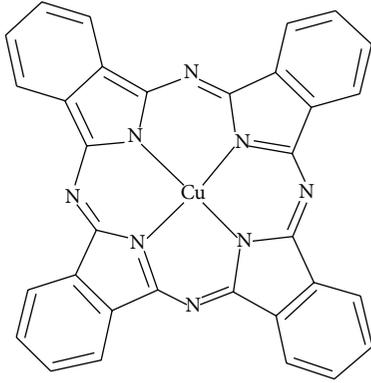


FIGURE 1: Chemical structure of phthalocyanine blue.

of phthalocyanine blue for carbon steel in acidic environment, an organic dye with good solubility in water, whose chemical structure is given in Figure 1 and possesses N and Cu atoms.

2. Experimental Procedure

Corrosion tests were performed on coupons prepared from 1018 carbon steel rods containing 0.14% C, 0.90% Mn, 0.30% S, 0.030% P, and as balance Fe, encapsulated in commercial epoxic resin with an exposed area of 1.0 cm². The organic dye phthalocyanine blue was obtained from a local manufacturer. The aggressive solution, 0.5 M H₂SO₄, was prepared by dilution of analytical grade H₂SO₄ with double distilled water. Weight loss experiments were carried out with steel rods 2.5 cm length and 0.6 cm diameter abraded with fine emery paper until 1200 grade, rinsed with acetone, and exposed to the aggressive solution during 72 h. After being corroded, specimens were taken out, washed with distilled water, degreased with acetone, dried, and weighed accurately. Tests were performed by triplicate at room temperature (25°C), 40 and 60°C by using a hot plate. Corrosion rates, in terms of weight loss measurements, ΔW , was calculated as follows:

$$\Delta W = \frac{m_1 - m_2}{A}, \quad (1)$$

where m_1 is the mass of the specimen before corrosion, m_2 the mass of the specimen after corrosion, and A the exposed area of the specimen. For the weight loss tests, inhibitor efficiency, IE, were calculated as follows:

$$IE (\%) = \frac{100 (\Delta W_1 - \Delta W_2)}{\Delta W_1}, \quad (2)$$

where ΔW_1 is the weight loss without inhibitor and ΔW_2 the weight loss with inhibitor. Specimens were removed, rinsed in water and in acetone, dried in warm air, and stored in a dessicator. Specimens were weighed in an analytical balance with a precision of 0.1 mg. Electrochemical techniques employed included potentiodynamic polarization curves and electrochemical impedance spectroscopy measurements, EIS. In all experiments, the carbon steel electrode was allowed to

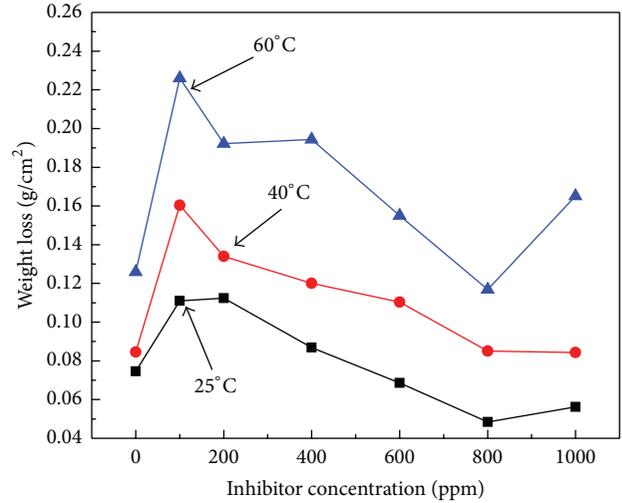


FIGURE 2: Effect of inhibitor concentration in the weight loss for 1018 carbon steel in 0.5 M H₂SO₄ at different temperatures.

reach a stable open circuit potential value, E_{corr} . Polarization curves were recorded at a constant sweep rate of 1 mV/s at the interval from -1000 to +1500 mV with respect to the E_{corr} value. Measurements were obtained by using a conventional three electrodes glass cell with two graphite electrodes symmetrically distributed and a saturated calomel electrode (SCE) as reference with a Lugging capillary bridge. Corrosion current density values, i_{corr} , were obtained by using Tafel extrapolation. Inhibitor efficiency, IE, was calculated as follows:

$$IE (\%) = \frac{100 (i_{\text{corr}1} - i_{\text{corr}2})}{i_{\text{corr}1}}, \quad (3)$$

where $i_{\text{corr}1}$ is the corrosion current density value without inhibitor and $i_{\text{corr}2}$ the corrosion current density value with inhibitor. EIS tests were carried out at E_{corr} by using a signal with amplitude of 10 mV in a frequency interval of 100 mHz–100 KHz. An ACM potentiostat controlled by a desk top computer was used for the polarization curves, whereas for the EIS measurements, a model PC4 300 Gamry potentiostat was used.

3. Results and Discussion

Figure 2 shows the corrosion rate results, in terms of the weight loss, for carbon steel in 0.5 M H₂SO₄ at different inhibitor concentrations at 25–60°C. It can be seen that when 100 ppm of inhibitor is added the corrosion rate decreases compared to that for uninhibited solution, at the three different temperatures, but with a further increase in the inhibitor concentration the corrosion rate decreases, reaching its lowest value more or less around 800 ppm, increasing again at 1000 ppm. The fact that the corrosion rate decreases with the inhibitor concentration is due to the fact that the inhibitor adsorption coverage on the steel surface starts to increase [25]. It can also be seen that the corrosion rate increases with an increase in the temperature in both

TABLE 1: Inhibitor efficiency values obtained from the weight loss tests at different concentrations and temperatures.

C_{inh} (ppm)	I.E. (%)		
	25°C	40°C	60°C
0	—	—	—
100	-48.806	-79.781	-89.513
200	-50.759	-52.564	-58.637
400	-16.549	-2.8457	-54.426
600	7.942	-23.521	-30.119
800	35.052	-0.7194	7.1672
1000	24.6	0.2282	-31.134

TABLE 2: Potentiodynamical polarization parameters for the corrosion of 1018 carbon steel in 0.5 M H_2SO_4 at 25°C.

C_{inh} (ppm)	E_{corr} (mV)	i_{corr} (mA/cm ²)	E_{pas} (mV)	i_{pas} (mA/cm ²)	I.E. (%)
0	-503	0.3	725	6.2	—
100	-456	0.2	535	3.7	40
200	-457	0.15	645	5.3	15
400	-460	0.13	530	2.8	55
600	-462	0.12	535	1.7	67
800	-459	0.11	286	1.1	82
1000	-461	0.1	207	4.9	26

uninhibited and inhibited solutions. Inhibitor efficiency at the different concentrations and temperatures is given in Table 1, indicating that the highest efficiency values are found between 800 and 1000 ppm. It should be noted that the inhibitor efficiency decreases with increasing the temperature up to 40°C but it increases with a further increase in the temperature up to 60°C. This is because inhibitor molecules are physically adsorbed on the metal surface, whereas a chemical adsorption implies an increase in the inhibitor efficiency with an increase in the temperature [8–13]. It has been reported that inhibitor can form coordination bonds between the unshared electron pair of N atom and the empty d iron electron [8, 22]. It means that coordinate bond could be formed by donating the lone electron pairs of N atoms to the unfilled orbits of Fe. Blue phtalocyanine can be protonated in acidic solution [24]; SO_4^{2-} ions could accumulate gradually close to the metal/solution interface, being specifically adsorbed; they create an excess negative charge towards the solution and favor more adsorption of the cations; then the protonated inhibitor may adsorb through electrostatic interactions between the positively charged molecules and the negatively charged metal surface. When protonated blue phtalocyanine adsorbed on metal surface, coordinate bond may be formed by partial transference of electrons from the N polar atom to the metal surface.

The effect of inhibitor concentration in the polarization curves for carbon steel in 0.5 M H_2SO_4 at 25°C is shown in Figure 3, whereas the electrochemical parameters are given in Table 2. It can be seen that the steel exhibits an active-passive behavior. The addition of the inhibitor did not affect very

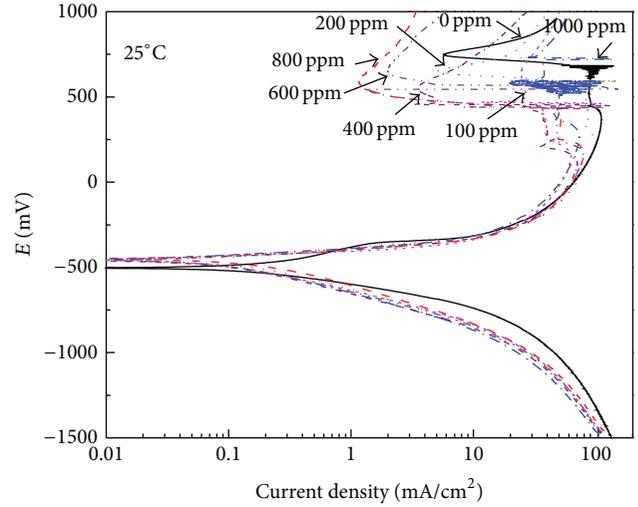


FIGURE 3: Effect of inhibitor concentration in the polarization curves for 1018 carbon steel in 0.5 M H_2SO_4 at 25°C.

much neither the E_{corr} nor the i_{corr} values, but it improves the passive current density and passivation potential values, i_{pas} and E_{pas} , respectively. Thus, the E_{corr} value for the uninhibited solution was -503 mV, and with the addition of inhibitor this value was around -460 mV approximately regardless of the inhibitor concentration. The i_{corr} slightly decreased from 0.3 down to 0.1 mA/cm², and the lowest value obtained was with the addition of 1000 ppm. On the other side, as can be seen in Table 2, the passivation potential value, E_{pas} , for the uninhibited solution was 725 mV, but it decreased with the addition of inhibitor, obtaining the lowest value, 286 mV, with the addition of 800 ppm. Similarly, the passivation current density value, i_{pas} , was 6.2 mA/cm² in absence of inhibitor, but it decreased as the inhibitor concentration increased, obtaining the lowest value, 1.1 mA/cm² by adding 800 ppm of inhibitor; a further increase in the inhibitor concentration to 1000 ppm increased the i_{pas} value, similar to the behavior of the weight loss results shown in Figure 2. Thus, it could be concluded that the addition of phtalocyanine blue decreases the corrosion rate of carbon steel in acidic media by improving the passive film properties. In Table 2, inhibitor efficiency values were calculated by using the i_{pas} values instead of the i_{corr} ones, since the inhibitor effect is much clearer in the former than in the later.

Polarization curves at 40°C, Figure 4, were not very different to those obtained at room temperature: the steel exhibited an active-passive behavior, neither the E_{corr} nor the i_{corr} values were drastically affected by the addition of the inhibitor, Table 3. Unlike this, the E_{pas} and i_{pas} values were improved by the addition of the inhibitor, reaching the best effect with 1000 ppm. Once again, in Table 3, inhibitor efficiency values were calculated by using the i_{pas} values instead of the i_{corr} ones, since the inhibitor effect is much clearer in the former than in the later. This time, the highest efficiency value was obtained at 1000 ppm, and the efficiency values were lower than those obtained at 40°C. Finally, at 60°C, the steel did not show an active-passive behavior, only

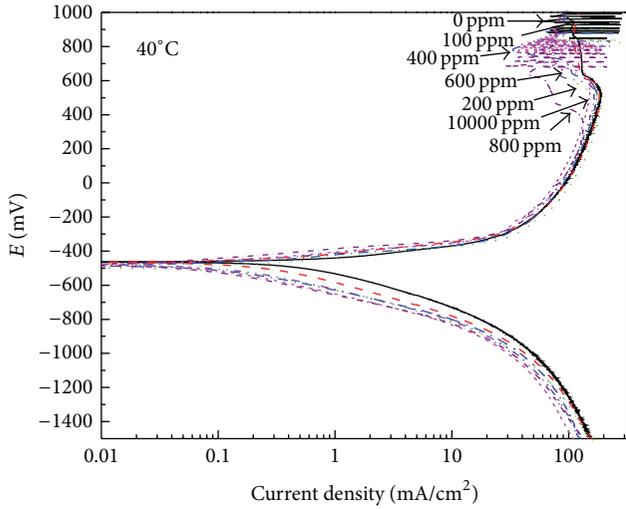


FIGURE 4: Effect of inhibitor concentration in the polarization curves for 1018 carbon steel in 0.5 M H_2SO_4 at 40°C.

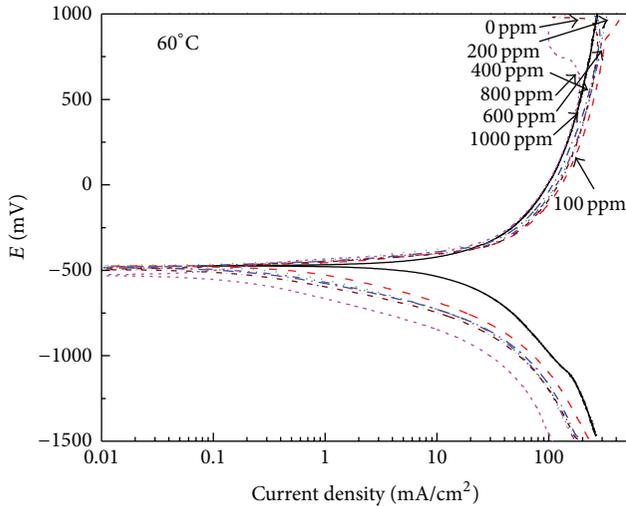


FIGURE 5: Effect of inhibitor concentration in the polarization curves for 1018 carbon steel in 0.5 M H_2SO_4 at 60°C.

anodic dissolution, Figure 5. The effect of adding inhibitor in the i_{corr} value at 60°C was somehow different, Table 4, since it increased with increasing the inhibitor concentration, reaching its lowest value with the addition of 1000 ppm. The anodic branch of the polarization curve was not affected by the inhibitor; instead the cathodic branch was affected, indicating that in this case the inhibitor acts as a cathodic-type of inhibitor. Additionally, the corrosion current density values increased with increasing the temperature. This time, efficiency values were higher than those obtained at both 25 and 40°C. The fact that the inhibitor does not affect the E_{corr} has been referred as it acts by a geometric blocking effect [22]; that is, the inhibition effect comes from the reduction of the reaction area on the surface of the corroding metal. However, it has been shown in this work that the main effect of the inhibitor is to improve the passive film properties,

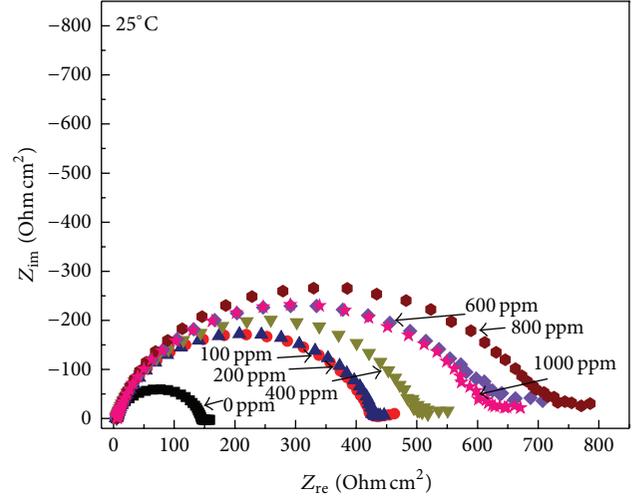


FIGURE 6: Nyquist plots for 1018 carbon steel in 0.5 M H_2SO_4 at 25°C.

TABLE 3: Potentiodynamical polarization parameters for the corrosion of 1018 carbon steel in 0.5 M H_2SO_4 at 40°C.

C_{inh} (ppm)	E_{corr} (mV)	i_{corr} (mA/cm ²)	E_{pas} (mV)	i_{pas} (mA/cm ²)	I.E. (%)
0	-480	0.8	650	133	—
100	-470	0.46	660	122	8
200	-500	0.2	640	110	17
400	-490	0.17	610	85	36
600	-483	0.12	630	77	42
800	-483	0.1	650	72	46
1000	-480	0.08	450	45	66

which is an anodic process, except at 60°C, where the cathodic slopes were affected. At 25 and 40°C the cathodic slopes were slightly affected; thus, the addition of the inhibitor implies the inhibitor molecules adsorption on both anodic and cathodic sites, resulting in an inhibition of both anodic and cathodic reduction reactions.

Impedance data in the Nyquist format for carbon steel corroded in 0.5 M H_2SO_4 at 25°C is given in Figure 6. Data describe a single, capacitive-like depressed semicircle with their centers in the real axis and with their diameters increasing with the inhibitor concentration up to 800 ppm, after which the diameter decreases with a further increase in the inhibitor concentration up to 1000 ppm. The single semicircle indicates that the corrosion process is under charge transfer control from the metal surface to the electrolyte through the double electrochemical layer and that the presence of the inhibitor does not change the mechanism of metal dissolution. Additionally, these impedance diagrams are not perfect semicircles which are related to the frequency dispersion as a result of the roughness and inhomogeneous of electrode surface [18]. Even more, the real axis intercepts at high and low frequencies in presence of the inhibitor is bigger in presence of the inhibitor than that in absence of the inhibitor, meaning that the impedance of the steel

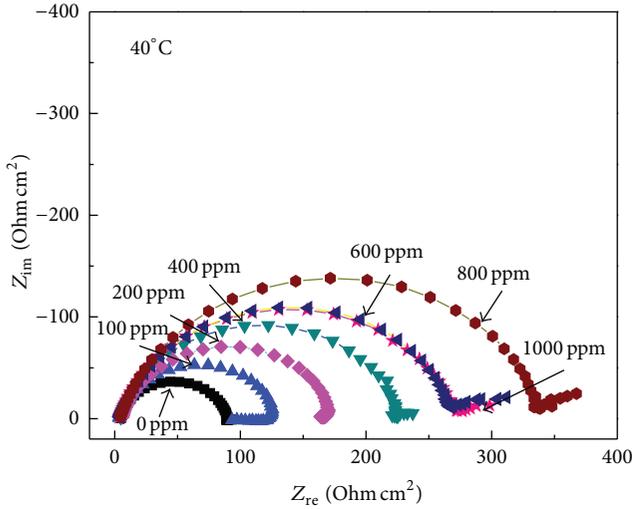


FIGURE 7: Nyquist plots for 1018 carbon steel in 0.5 M H₂SO₄ at 40°C.

TABLE 4: Potentiodynamical polarization parameters for the corrosion of 1018 carbon steel in 0.5 M H₂SO₄ at 60°C.

C_{inh} (ppm)	E_{corr} (mV)	i_{corr} (mA/cm ²)	I.E. (%)
0	-508	22	—
100	-503	11	50
200	-487	7.5	65
400	-490	5.6	75
600	-501	4.1	81
800	-503	3.5	84
1000	-500	2.9	87

in the inhibited environment increases with the inhibitor concentration, decreasing, thus, the metal corrosion rate. Thus, according to data in Figure 6, the highest inhibitor efficiency (81%) is reached with the addition of 800 ppm.

When the temperature is increased up to 40°C, Figure 7, Nyquist data also describe a single, depressed, capacitive-like semicircle with its centre increasing with increasing the inhibitor concentration, obtaining its biggest diameter value at 800 ppm of inhibitor. Thus, just like the tests at 25°C, the corrosion process is under charge transfer from the metal to the electrolyte and does not change with the presence of the inhibitor. The impedance values obtained at 40°C, however, are smaller than those obtained at 25°C, which implies that the corrosion rate increases with the temperature. The highest inhibitor efficiency, obtained with the addition of 800 ppm, was 75%, smaller than that obtained at 25°C. However, with a further increase in the testing temperature up to 60°C, Figure 8, Nyquist data described a capacitive-like, depressed semicircle at high and intermediate frequency values followed by an inductive semicircle at low frequencies. The capacitive semicircle diameter increased with increasing the inhibitor concentration reaching its maximum value when 800 ppm of inhibitor is added, with an efficiency value of 65%. The presence of an inductive loop at low frequencies means that the corrosion process is not any

TABLE 5: EIS Parameters for the corrosion of 1018 carbon steel in 0.5 M H₂SO₄ at 25°C.

C_{inh} (ppm)	R_{ct} (Ohm cm ²)	C_{dl} (μF/cm ²)	IE (%)		
			25°C	40°C	60°C
0	148	156	—	—	—
100	421	59	62	28	90
200	430	56	65	42	96
400	530	50	72	60	97
600	702	30	78	66	97
800	790	12	81	73	98
1000	671	22	78	67	96

longer under charge transfer control but under the control of the adsorption/desorption process of aggressive species or the inhibitor from metal surface.

EIS data can be calculated by using equivalent electric circuits as that shown in Figure 9. In this figure, R_s represents the solution resistance, R_{ct} the charge transfer resistance, C_{dl} the capacitance of the double electrochemical layer, and R_f represents the resistance of the corrosion products film and C_f its capacitance. However, the double-layer capacitance, C_{dl} , can also be calculated from the equation bellow:

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}}, \quad (4)$$

where f_{max} is the frequency value at which the imaginary component of the impedance is maximal. For the uninhibited solution, a C_{dl} value of 156 μF/cm² was found. Table 5 gives the results for the R_{ct} and C_{dl} for the solution with and without inhibitor together with the inhibitor efficiency values at the three testing temperatures. It is important to note that by increasing the inhibitor concentration brings an increase in the charge transfer resistance value and a decrease in the double-layer capacitance. The decrease in the C_{dl} value can be interpreted as due to the adsorption of the inhibitor onto the electrode surface [20]. The double-layer formed at the metal-solution interface is considered as an electric capacitor, whose capacitance decreases due to the displacement of water molecules and other ions originally adsorbed on the electrode by the inhibitor molecules, forming a protective film. The thickness of the film formed increases with increasing concentration of the inhibitor, since more inhibitor adsorbs on the surface, resulting in lower C_{dl} values. It has to be noted that, according to Table 5, inhibitor efficiency values decrease when the temperature increases to 40°C; then they increase with a further increase in the temperature up to 60°C, similar to the reported values by using polarization curves, Tables 2–4.

It has been reported that the natural logarithm of the corrosion rate is a linear function with $1/T$, where T is the absolute temperature, for the corrosion of carbon steel in acidic conditions [2, 6, 9]. The apparent activation energy, E_a , associated with 1018 carbon steel in uninhibited and inhibited

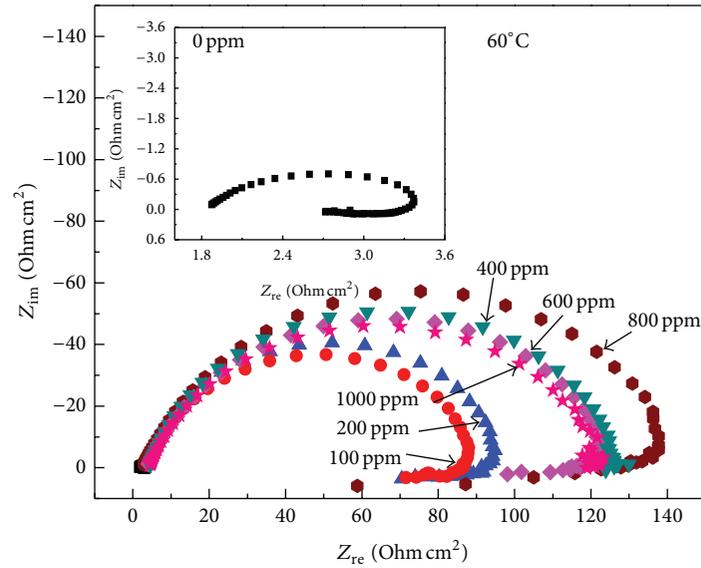


FIGURE 8: Nyquist plots for 1018 carbon steel in 0.5 M H₂SO₄ at 60°C.

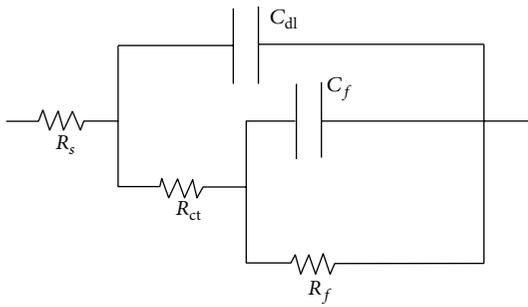


FIGURE 9: Electric circuit used to simulate EIS data.

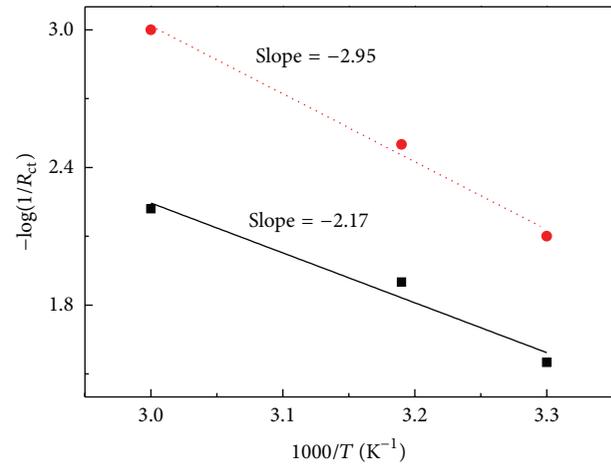


FIGURE 10: Arrhenius plot for the $1/R_{ct}$ values without and with 800 ppm of phtalocyanine blue.

acid solution was determined by using an Arrhenius-type plot according to the following equation:

$$\ln CR = \frac{-E_a}{RT} + \ln F, \quad (5)$$

where CR is the corrosion rate, R is the molar gas constant, T is the absolute temperature, and F is the frequency factor. The corrosion rate can be given in terms of the weight loss, ΔW , corrosion current density, i_{corr} , or $1/R_{ct}$, where R_{ct} is the charge transfer resistance, which is the semicircles diameter of the Nyquist data. Arrhenius plots of $\ln 1/R_{ct}$ against T^{-1} for 1018 carbon steel in 0.5 M H₂SO₄ in absence and presence of inhibitor are shown in Figure 10. The apparent activation energy obtained for the corrosion process in the inhibitor-free, uninhibited acid solution was found to be 7.7 and 3.2 kJ mol⁻¹ in presence of the inhibitor. The energy barrier of the corrosion reaction decreased in the presence of the inhibitor, which can be due to the adsorption of the inhibitor on the steel surface. Many studies [20, 21] showed that, in the presence of inhibitor, the decrease of E_a with respect to uninhibited solution is interpreted as physical adsorption.

Some micrographs of corroded specimens without and with the addition of inhibitor are shown in Figure 11. For the uninhibited solution, Figure 11(a), the steel surface shows very porous corrosion products, indicating the aggressive attack of the corroding environment on the steel surface. However, when the steel is corroded in the aggressive solution containing 800 ppm of phtalocyanine blue at 25°C Figure 11(b), very compact, rough, porous, or cracks-free, protective corrosion products are formed on top of the steel, which reduced the steel corrosion rate. When the steel is immersed in the inhibited solution but at higher temperatures, Figures 11(c) and 11(d), the corrosion products become more porous and some microcracks can be seen now, indicating that the aggressive environment can penetrate through them and corrode the underlying metal,

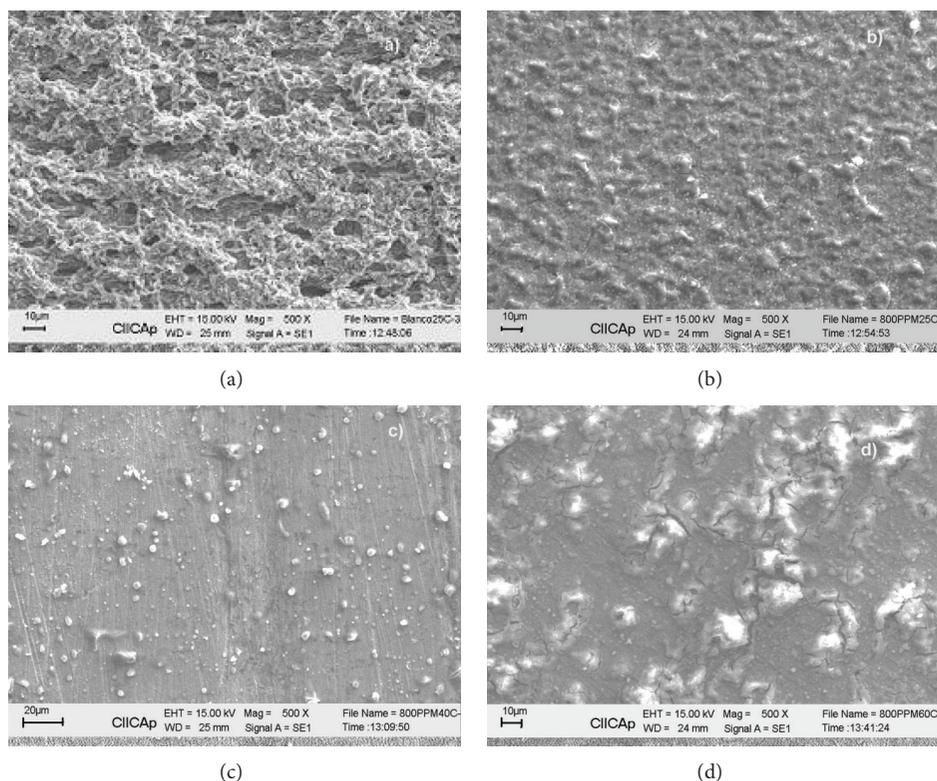


FIGURE 11: Micrograph of corroded 1018 carbon steel at (a) 25°C with 0 ppm, and 800 ppm of phtalocyanine blue at (b) 25, (c) 40 and (d) 60°C.

increasing the corrosion rate and decreasing the inhibitor protectiveness.

4. Conclusions

A study of the use of phtalocyanine blue dye as corrosion inhibitor for 1018 carbon steel in 0.5 M H_2SO_4 has been carried out. It has been found that phtalocyanine blue acts as a good corrosion inhibitor with its efficiency increasing with the inhibitor concentration, reaching its highest value by adding 800 or 1000 ppm due to the formation of an adherent, compact, protective film. Additionally, inhibitor efficiency decreases with an increase in the temperature up to 40°C, but it increases at 60°C. Phtalocyanine blue dye improved the properties of the passive film formed on the steel surface, that is, decreases both the passivation current density and passivation potential values; therefore, it acts as an anodic-type inhibitor. At 25 and 40°C, Nyquist data displayed a single capacitive, depressed loop, indicating that the corrosion process is under charge transfer control, but at 60°C there appears an inductive loop, indicating that the corrosion process is now under adsorption/desorption control.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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