

Effect of the structure, immersion time and temperature on the corrosion inhibition of 4-pyrrol-1-yl-*N*-(2,5-dimethyl-pyrrol-1-yl)benzoylamine in 1.0 M HCl solution

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Abstract

A benzoylamine derivative of new class, namely 4-pyrrol-1-yl-*N*-(2,5-dimethyl-pyrrol-1-yl)benzoylamine (PDPB), has been successfully synthesized and characterized by spectroscopic techniques (Fourier-transform infrared spectroscopy (FTIR) and Nuclear Magnetic Resonance (NMR)) and CHN analysis. PDPB acts as a new corrosion inhibitor for mild steel in HCl environment. The inhibition efficiency was determined by mass loss measurements and by scanning electron microscopy (SEM). The inhibitive performance of PDPB on mild steel in 1 M hydrochloric acid environment was studied as a function of PDPB concentration, immersion time, and temperature. The inhibition efficiency increases with an increase in inhibitor concentration and decreases with an increase in immersion time and temperature. The PDPB molecules are adsorbed on the surface of mild steel is a mixed mode involving physical adsorption and chemical adsorption. SEM analysis was conducted to investigate the persistency of the layer of PDPB molecules. Moreover, the relationship between the inhibitive performance and the chemical structure of the tested inhibitor molecules was investigated by density functional theory (DFT) calculations. All the experimental findings and theoretical calculations are in good agreement.

Keywords: benzoylamine, PDPB, DFT, corrosion inhibitor.

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Introduction

Mild steel has been used in petroleum, food, chemical, and engineering industries as a common structural material [1–3]. However, mild steel is adversely affected by acid solutions in industrial cleaning, acid de-scaling, and acidization of oil wells [4–6], which can cause major economic losses and safety risks. One of the most inexpensive and effective approaches was the use of inhibitors to avoid or mitigate the significant harm to mild steel

in acid environments [7–10]. It is widely accepted that organic molecules containing heteroatoms (sulfur, oxygen, and nitrogen) are efficient organic inhibitors in corrosive environments, and their molecular structure contains heterocyclic rings or polar sites. The explanation is that iron on the solid-liquid surface forms coordination bonds with the inhibitor molecules [11–13] and this blocks the activation sites on the mild steel surface, preventing corrosion of mild steel in corrosive environments [14–17]. The weight loss method [18, 19] is a traditional technique to determine the inhibitive behavior. The purpose of this investigation is to study the inhibitive performance of a newly synthesized benzoylamine, namely 4-pyrrol-1-yl-*N*-(2,5-dimethyl-pyrrol-1-yl)benzoylamine (PDPB) (Figure 1), on mild steel coupon in 1 M hydrochloric acid environment. The reason why this compound is chosen is that, first, it has a number of adsorption centers, including heteroatoms such as nitrogen and oxygen, in addition to heterocyclic and aromatic rings. Secondly, pyrrole demonstrated a significant inhibitive performance for mild steel in 1 M HCl environment at 303 K. Third, this inhibitor (PDPB) can indeed be synthesized easily in an excellent yield. The investigation was conducted utilizing gravimetric techniques and scanning electron microscopy (SEM). The correlation between the inhibitive performance of various inhibitor concentrations, immersion time and solution temperature is discussed. All experimental data are compared with the density functional theory (DFT) results to explain the process of successful coordination of the inhibitor molecules with the d-orbital of iron atoms on mild steel surface.

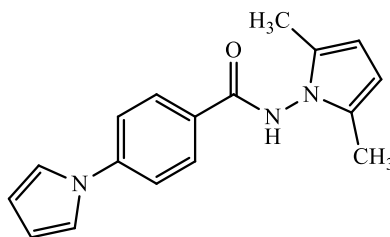


Figure 1. The chemical structure of the newly synthesized corrosion inhibitor.

2. Experimental Section

2.1. Synthesis of PDPB

The investigated benzoylamine derivative was synthesized and the chemical scheme of the reaction of 4-pyrrol-1-yl benzohydrazide is shown in Figure 2. Two solutions of 4-(1H-pyrrol-1-yl)benzohydrazide (0.005 mol) in ethyl alcohol (20 mL) and hexane-2,5-dione (0.010 mol) in glacial acetic acid (2 mL) were mixed and heated for 5 h. The concentrated residue was poured into ice, filtered, washed with distilled water, and then dried. The dry filtrate was recrystallized from ethyl alcohol. Yield 70%, m.p. 230–231°C. FT-IR (cm^{-1}): 3291 (amide group), 3071 (C–H aromatic), 2954 (C–H aliphatic group), 1658 (carbonyl group), 1563 (C=N group). ^1H NMR (DMSO- d_6 , ppm): 8.01 (d, 1H, C–H aromatic), 7.94 (d, 1H, C–H aromatic), 7.73 (s, 1H, C–H pyrrole), 6.40 (s, 2H, C–H pyrrole), 5.69 (s, 2H,

C–H pyrrole), 2.13 (s, 6H, C–H aliphatic). ^{13}C NMR (DMSO- d_6 , ppm) 166.6 (C=O), 143.7 (C aromatic), 129.4 (C aromatic), 128.1 (C aromatic), 125.9 (C pyrrole), 120.1 (C pyrrole), 111.3 (C pyrrole), 106.1 (C pyrrole), 10.8 (C methyl). CHN elemental analysis (calc/found): C, 73.10/73.81; H, 6.13/5.99; N, 15.04/14.79.

2.2. Weight loss measurements

Gravimetric measurements were conducted in 1 M hydrochloric acid environment in the presence of various concentrations of PDPB. The immersion periods were 1, 5, 10, 24, and 48 h and the solution temperature was 303 K. Coupons of mild steel having dimensions $4.0 \times 2.5 \times 0.5$ cm were used for the gravimetric measurements. The chemical composition of mild steel utilized is as follows (weight percentage): 0.210 carbon; 0.050 manganese; 0.380 silicon; 0.010 aluminum; 0.030 sulfur; 0.090 phosphorus; iron balance. After degreasing with acetone and benzene, mild steel specimens were precisely weighed. After the immersion periods, the mild steel coupons were removed, cleaned, and accurately weighed again. Distilled water was utilized to dilute HCl to the 1 M concentration. In order to estimate the inhibition efficiency at various temperatures, the measurements were performed at various inhibitor concentrations (100, 200, 300, 400, and 500 ppm) at various temperatures (303, 313, 323, and 333 K) for 5 h corroding time [20–22]. For each experiment, triplicate tests were performed and the average values are reported. The corrosion rate of mild steel was calculated using equation (1):

$$C_R = \frac{87.6W}{at\rho} \quad (1)$$

where W is the weight loss in the absence and in the presence of the synthesized inhibitor (mg), a is the coupon area (cm^2), t is the immersion time and ρ is the specific density of the tested mild steel coupons.

The inhibition efficiency ($IE\%$) was estimated from equation (2) whereas the surface coverage (θ) was determined from equation (3):

$$IE\% = \frac{C_R - C_{Ri}}{C_R} \times 100 \quad (2)$$

$$\theta = \frac{IE\%}{100} \quad (3)$$

where C_R is the corrosion rate of mild steel in the absence of PDPB and C_{Ri} is the corrosion rate of mild steel in the presence of PDPB.

2.3. SEM analysis

The images of mild steel surface after immersion for 5 h corrosion time in 1 M environment at 303 K in the absence and in the presence of PDPB was obtained with a scanning electron microscope, model TM1000 Hitachi Tabletop Microscope.

2.4. Density Functional Theory (DFT)

The geometrical structure of PDPB molecules and quantum chemical calculations were conducted utilizing the Gaussian-03 software package for DFT calculations and B3LYP function with the basis set 6–31G (d, p) [23–25]. The quantum chemical parameters, such as highest occupied molecular orbital (HOMO), lowest unoccupied molecular orbital (LUMO), energy gap (ΔE), electron affinity (A), ionization potential (I), dipole moment, softness (σ), and hardness (η) were calculated [26–29].

3. Results and Discussion

3.1. Synthesis

The molecular structure of the synthesized inhibitor was confirmed by FT–IR and NMR techniques. The reaction synthesis steps are shown in Figure 2.

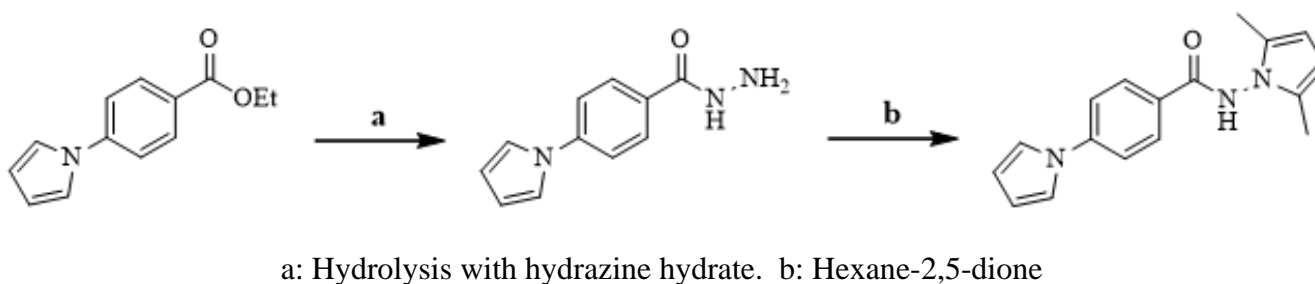


Figure 2. Scheme of inhibitor synthesis.

3.2. Gravimetric measurements – concentration effects

The inhibition effect of PDPB as a corrosion inhibitor for mild steel in 1 M HCl environment at 303 K was primarily studied by the mass loss technique. The corrosion rate (C_R) and inhibition efficiency ($IE\%$) values were determined and demonstrated in Figure 3. It is obvious from Figure 3 that the corrosion rate decreased significantly with an increase in the PDPB concentration compared to zero PDPB concentration, which was attributed to the strong adsorption of PDPB molecules on the surface of mild steel and formation of a protective film that covers the mild steel surface and prevents and/or controls the impact of the corrosive solution [30]. It is obvious that inhibitive activity increased with increasing PDPB concentration, and the highest value of the inhibition efficiency was found to be 95.8% at the optimum PDPB concentration of 500 ppm, suggesting that PDPB successfully inhibited the mild steel corrosion in the HCl solution. These findings may be attributed to the chemical structure of PDPB molecules which have pyrrole and benzene rings in addition to a carbonyl group and hetero atoms which have free electrons that they can share with the d-orbitals of iron atoms on the surface of mild steel and form coordination bonds.

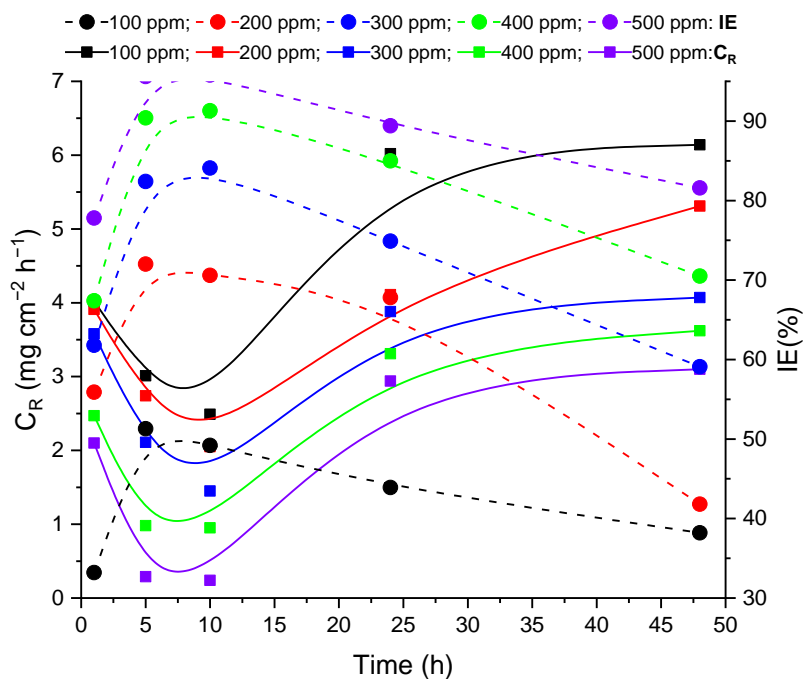


Figure 3. The effect of immersion time on the corrosion rate and inhibition efficiency of mild steel in 1 M HCl containing various concentrations of PDPB in 1 M HCl at 303 K.

3.3. Gravimetric measurements – immersion time effect

The effect of exposure time on the inhibitive efficacy of 100–500 ppm PDPB on mild steel in 1 M HCl environment at 303 K is demonstrated in Figure 3. It is obvious that during the first 1 h, the inhibition efficiency increased with the exposure period for PDPB inhibitor, whereas in the second periods of 5 and 10 h, the inhibition efficiency increased sharply. These findings may be attributed to the formation and growth of the adsorbed layer of PDPB molecules on the mild steel surface. After 10 h, the inhibitive efficacy reduced with prolongation of the exposure period, which is attributed to the dissolution of adsorbed protective PDPB molecules [31]. In addition, the inhibitive efficacy of PDPB molecules was still above 80% after the 48 h exposure period, suggesting that PDPB was a long-range efficient inhibitor for mild steel in 1 M HCl environment.

3.4. Gravimetric measurements – temperature effect

The effect of temperature on the corrosion rate and inhibition efficiency was investigated in 1 M hydrochloric acid solution at different temperatures (303, 313, 323 and 333 K) without and with addition of various concentrations of PDPB (Figure 4). The inhibition efficiency was reduced with increasing temperature from 303 to 333 K. These findings may correlate with the dissolution of adsorbed PDPB molecules on the mild steel surface. These results confirm that the inhibitive process occurs by adsorption of PDPB molecules on the mild steel surface, and on the other hand, increasing the temperature increases the corrosion rate and decreases the inhibition efficiency due to desorption of inhibitor molecules from the mild steel surface as shown in Figure 4.

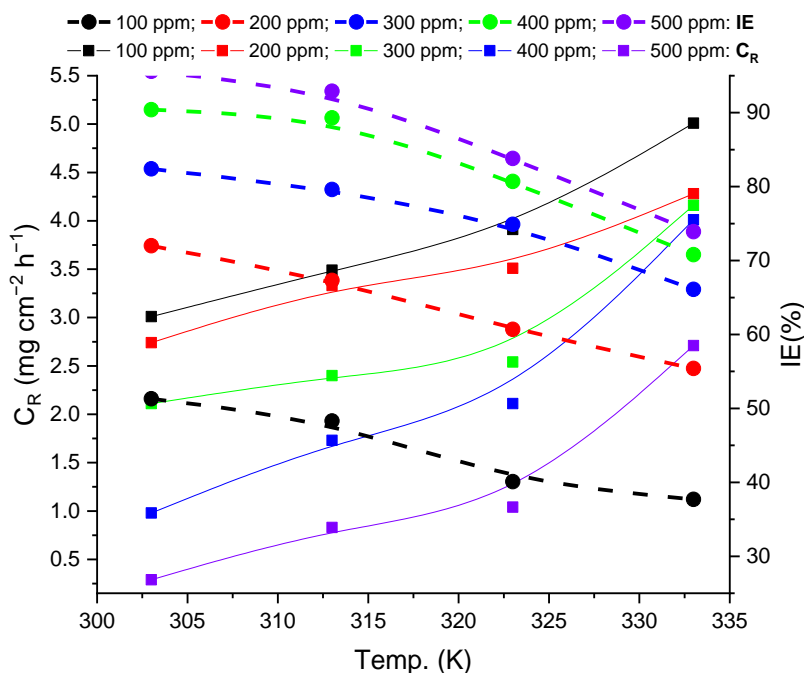


Figure 4. The effect of temperature on the corrosion rate and inhibition efficiency of mild steel in 1 M HCl containing various concentrations of PDPB on mild steel in 1 M HCl.

Weight loss tests were conducted at various temperatures (303–333 K) to evaluate the thermodynamical parameters. The rate of corrosion and inhibitive efficacy were determined at various temperatures as demonstrated in Figure 4. It is obvious that the corrosion rate increases with increasing temperature and the inhibitive efficacy decreases with increasing temperature, which is attributed to the chemical adsorption mechanism of PDPB molecules on the surface of mild steel. This effect demonstrated the interactions of PDPE molecules and the metal surface [32]. Arrhenius equation (4) was used to evaluate the activation energy (E_a) [33],

$$\log C_R = \frac{-E_a}{2.303RT} + \log(A) \quad (4)$$

where A is the Arrhenius parameter.

The value of (E_a) was determined from the slope of the Arrhenius plot as demonstrated in Figure 5. The value of (E_a) is $8.51 \text{ kJ} \cdot \text{mol}^{-1}$. The adsorption of PDPE molecules was correlated to the chemical adsorption mechanism. Based on the current results and the value of activation energy (E_a), the proposed mechanism is chemical adsorption. The value of the activation energy determines and establishes that the type of interference between the corrosion inhibitor molecules and the metal surface and suggests a chemisorption mechanism [34].

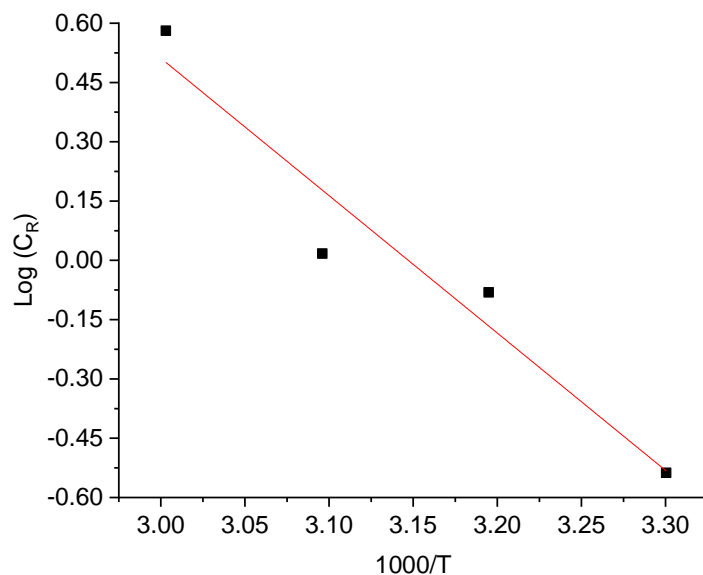


Figure 5. Log C_R versus $1/T$ for mild steel in 1 M HCl environment.

3.5. Morphology study

Scanning electron micrographs of mild steel specimens in the corrosive environment in the absence and presence of 500 ppm PDPB corrosion inhibitor are shown in Figure 6. It is clear that the surface of the tested specimens was corroded in the absence of PDPB (Figure 6a), whereas in the presence of 500 ppm PDPB (Figure 6b), the surface of mild steel was found to be smooth. These results suggest that PDPB molecules form a film on the specimen surface and impede and/or control the corrosion effect of the HCl solution.

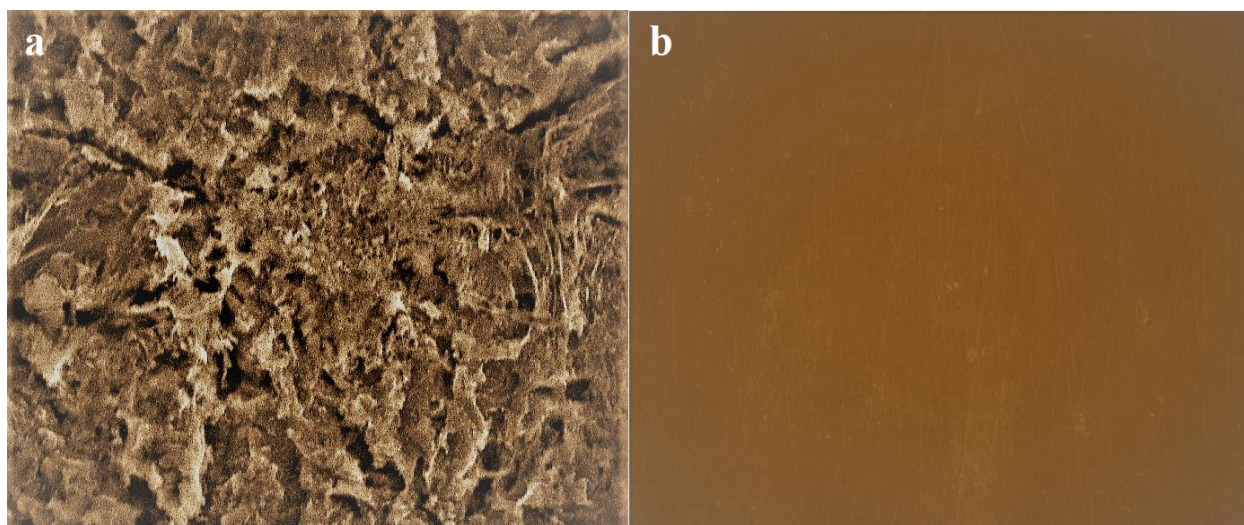


Figure 6. Surface morphology of mild steel after being corroded in 1 M HCl solution at 303 K in the absence (a) and in the presence of 500 ppm PDPB (b).

3.6. Mechanism of inhibition

The adsorption of PDPB molecules on the mild steel-solution interface is responsible for the effect of this compound on the impedance of the corrosion of mild steel in 1 M HCl. Physical adsorption or chemical adsorption or both are the primary forms of interaction between the PDPB molecules (as organic inhibitor) and the surface of mild steel. The alloy nature, the chemical composition of inhibitor molecules, the electrolyte form, the environment temperature, and the specimen morphology are the main parameters that affect the inhibitor's adsorption [35]. Generally, the inhibitive performance depends on the electronic density (which is the measure of the electron being present probability) at a functional site of the inhibitor molecule. The thermodynamic parameters show that the adsorption of the investigated inhibitor molecules on the specimen surface in 1 M HCl environment includes chemical and physical adsorptions together [36]. Chemisorption (chemical adsorption) of PDPB molecules on mild steel surface was attributed to the interactions of unshared electron pairs of heteroatoms (nitrogen and oxygen) and π -electrons of azomethine groups in addition to pyrrole and benzene rings with the unoccupied d-orbital of iron atoms on the surface of mild steel [37]. These interactions produce an insoluble and stable layer through covalent coordination bonds [38]. The enhanced inhibitive behavior of PDPB molecules is associated with the electron donor effect of a benzene ring-connected amide group which increases the electron density in the aromatic (pyrrole and benzene) rings as illustrated in Figure 7. The increased electron delocalization density in the molecule may cause corrosion inhibition. The adsorption of PDPB molecules will give more stability due to the involvement of π -electrons of benzene and pyrrole rings [39].

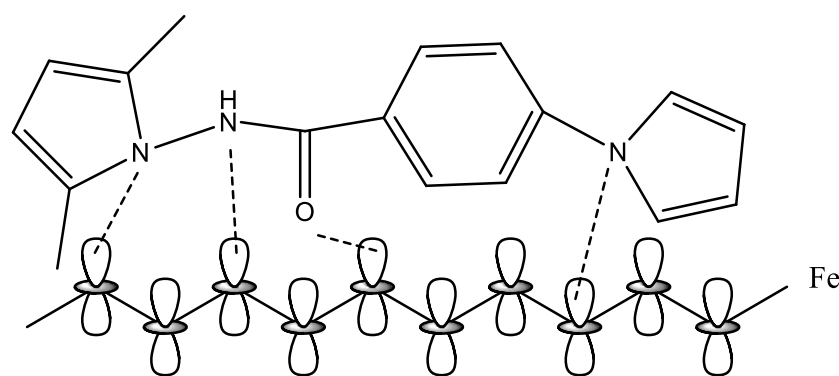


Figure 7. The suggested mechanism of inhibition of PDPB molecules.

3.7. DFT calculations

Figure 8 demonstrates the correlation between the inhibitory activity and the optimized molecular structure. The Highest Occupied Molecular Orbital (HOMO), lowest unoccupied molecular orbital (LUMO), energy gap ΔE , and dipole moment are the most significant parameters for determination of the linkage between the inhibitor and metal surface. Based on the principle of molecular orbital frontiers, E_{HOMO} refers to the ability of a molecule to

send electrons to the appropriate electron receptors, and thus, when the E_{HOMO} value is high, it appears that the molecule contributes more electrons in a moderate-vacancy d-orbital of iron atoms on the surface of mild steel. LUMO, therefore, refers to the electron acceptability of the molecule, and when the evaluated E_{LUMO} value is lower, a molecule has a higher capacity for accepting electrons, so the inhibitor has a higher inhibitory activity when its ΔE value is lower, as lower energy is required to eliminate the electron from the previously occupied molecule [40,41].

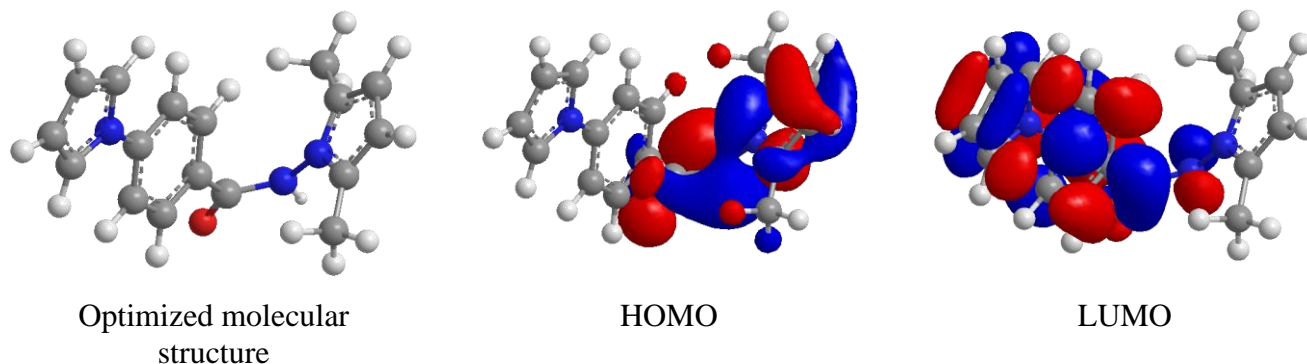


Figure 8. Optimized molecular structure and frontier molecule orbital of PDPB molecule.

The ionization potential (I) and the electron affinity (A) values may be determined [42] based on equations (5,6):

$$I = -E_{\text{HOMO}} \quad (5)$$

$$A = -E_{\text{LUMO}} \quad (6)$$

Furthermore, the softness (σ), hardness (ρ) and electronegativity (χ) of the PDPB molecule are calculated according to equations (7–9):

$$\sigma = \frac{1}{\rho} \quad (7)$$

$$\rho = -\frac{E_{\text{HOMO}} - E_{\text{LUMO}}}{2} \quad (8)$$

$$\chi = -\frac{E_{\text{HOMO}} + E_{\text{LUMO}}}{2} \quad (9)$$

The DFT calculations are presented in Table 1. It is apparent that the PDPB molecule has a high value of E_{HOMO} and low value of E_{LUMO} which is in good agreement with above hypothesis and means that the tested inhibitor has significant inhibitive efficacy. Furthermore, the PDPB molecule exhibits a considerable μ value. Even so, the association between μ and inhibition effectiveness remains controversial, *e.g.*, several researchers proposed improved inhibition efficacy with increasing μ [43–45], although others indicated that inhibitor molecules with a lower μ showed better inhibitory activity [46]. The

electronegativity value, in general, reflects the chemical reactivity, and a higher value shows improved inhibitive efficiency. Moreover, where the value of hardness is lower, as per the hard–soft acid basis (HSAB) principle, an inhibitor still has higher inhibitive effectiveness [47]. Inspection of Table 1 also reveals that the PDPB molecule has high electronegativity and low hardness, values suggesting high inhibitive efficacy, which is compatible with weight loss and SEM techniques.

Table 1. DFT theoretical parameters of PDPB molecules.

Parameter	PDPB
E_{HOMO} (eV)	–11.132
E_{LUMO} (eV)	–2.573
ΔE (eV)	8.559
μ (D)	3.7824
I (eV)	11.132
A (eV)	2.573
χ (eV)	6.852
ρ (eV)	4.279
σ [(eV) ^{–1}]	0.2336

Conclusion

Weight loss techniques and scanning electron microscopy were applied to study the inhibitive behavior of a novel synthesized corrosion inhibitor derived from benzoylamine, namely 4-pyrrol-1-yl-*N*-(2,5-dimethyl-pyrrol-1-yl)benzoylamine (PDPB) for mild steel in 1 M HCl environment. Experimental findings demonstrated that PDPB is an excellent corrosion inhibitor for mild steel surface in corrosive solution with a highest inhibitive efficacy of 95.8% at 500 ppm. The inhibitive efficacy increases with increasing PDPB concentration and decreases with rising temperature. The observations from SEM and DFT were well-conformed with those from gravimetric measurements.

Acknowledgments

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