



Study of Corrosion Behavior of N'-acetyl-4-pyrrol-1-ylbenzohydrazide for Low-Carbon Steel in the Acid Environment: Experimental, Adsorption Mechanism, Surface Investigation, and DFT Studies

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ABSTRACT

ew corrosion inhibitors, namely N'-acetyl-4-pyrrol-1-ylbenzohydrazide were synthesized by the reaction of 4-pyrrol-1-(NAPB). ylbenzohydrazide with acetic anhydride. NAPB was characterized by FTIR and NMR spectroscopy. Weight loss measurement was used to evaluate the corrosion inhibition of Low-carbon steel in a 1 M hydrochloric acid medium. The inhibition efficiency (IE%) increased as NAPB concentration increases and decreases as the solution temperature increases. The inhibition efficiency reached 94.6 % at the optimum concentration (500 ppm) of NAPB. The scanning electron microscopy technique proved the formation of a protective layer from NAPB molecules as corrosion inhibitors on the Low-carbon steel surface. The adsorption isotherm of NAPB molecules on the surface of Low-carbon steel was confirmed to follow the Langmuir adsorption isotherm. Further, through quantum chemical calculations using density functional theory (DFT), the importance of inhibition performance and molecular structure of an inhibitor has been theoretically investigated. Both experimental and theoretical findings are consistent with one another. Prog. Color Colorants Coat. 15 (2022), 133-141[©] Institute for Color Science and Technology.

1. Introduction

The corrosion phenomena of alloys, especially lowcarbon steel, are the most significant chemical reactions concerning the low-carbon steel and its surroundings [1-3]. Low-carbon steel has variously been used, and particularly in the areas of manufacturers. An example of the numerous well-known techniques used to eliminate oxide from the low-carbon steel surface is to clean the alloy in a corrosive solution named "stripping, a physical separation process where oxides are removed" [5-7]. Due to the highly corrosive pickling media, natural or synthesized organic inhibitors remain an initial, efficient, and non-expensive technique to guarantee sufficient protection [8-11]. The natural or synthesized organic inhibitors are added with a small quantity to reduce the interactions of corrosive solutions. The inhibition performance and the adsorption on the inhibitor molecules' metal surface are related to the

chemical structure of the inhibitor molecules, spatial planarity, the types of the functional sites, and attraction tendencies [12]. Different organic molecules, natural and/or synthetic, are appropriated as a chemical structure with heteroatoms such as nitrogen, oxygen, sulfur, phosphorous, and pi-electron conjugated systems [13, 14]. These organic molecules are responsible for the inhibitor molecule's adsorption process on the lowcarbon steel surface. These inhibitor molecules retard or control the corrosion by block the active site on the surface of low-carbon steel [15, 16]. It is known and scientifically famous that pyrrole molecules are used in various chemical, medicinal, and pharmaceutical applications. In addition to their use in industrial fields, they are utilized as inhibitors to controlling or retarding steel corrosion.

Continuing to search for the progress of heterocyclic compounds as efficient inhibitors [17-20] for the corrosion low carbon steel of in HCl solution, the novelty in the current investigation is assessing the inhibition efficiency of organic compound namely "N'acetyl-4-pyrrol-1-ylbenzohydrazide" toward corrosion of low-carbon steel. An easy and efficient technique has synthesized N'-acetyl-4-pyrrol-1-ylbenzohydrazide (NAPB) with a large yield. Furthermore, the chemical structure of NAPB has been illustrated by proton-NMR, carbon-NMR, and CHN analysis. Weight loss techniques have achieved the corrosion inhibition evaluation of NAPB. The low-carbon steel surface was examined by scanning electron microscopy (SEM), and the results support its inhibitory performance.

Moreover, the adsorption of inhibitor molecules

was studied by interaction with the surface of low carbon steel to understand the mechanism of inhibition. The impact of different temperatures in the HCl of NAPB was studied, and various thermodynamic coefficients were evaluated. Finally, a theoretical calculation by the DFT method has been conducted to validate the methodological findings.

2. Experimental

Table 1 shows the chemical composition of low-carbon steel used for corrosion measurements. In preparing, 1 M hydrochloric acid environment from double-distilled water, HCl (37 %), Sigma-Aldrich Malaysia has been used. Inhibitor (NAPB) stock solution was conducted in 1 M HCl and then diluted to make different NAPB concentrations. In 1 M HCl solution with distinctive concentrations of 100, 200, 300, 400, and 500 ppm, the procedures were performed for weight loss measurements. The low-carbon steel surface was polished with emery paper before each experiment and was washed with purified water and acetone.

2.1. Synthesis of inhibitor

N'-acetyl-4-pyrrol-1-ylbenzohydrazide was synthesized by gentle refluxing 4-pyrrol-1-ylbenzohydrazide (0.01 mol) with acetic anhydride (17 mL) for one hour, demonstrated in Figure 1. First, the yellow solid precipitate thus obtained was filtered and dried. Then, the new corrosion inhibitor was recrystallized using ethyl alcohol to get pure crystals with Yield 69 % and M.P. 233 °C.

	Elements	Carbon	Manganese	Silicon	Aluminum	Sulfur	Phosphorus	Iron
ĺ	wt %	0.210	0.050	0.380	0.010	0.050	0.090	balance
	O II	0		NHNH	[₂		HN-N	H
		↓ + [)N-()	\neg	→	N-	\rightarrow	CH ₃
	H ₃ C ^r O ^r	CH ₃		Ň			ٽ ٽ 🖌 🛏	

Table 1: The composition of low-carbon steel.

Figure 1: Scheme of the synthesized inhibitor.

2.2. Gravimetric techniques

The measurements of gravimetric techniques were conducted using Low-Carbon Steel coupons of size $4.5 \times 2 \times 0.025$ cm³. Gravimetric techniques have been carried out in 1 M HCl according to ASTM G1 [21]. In this technique, various inhibitor concentrations given above at 303 K were used. The measurements of gravimetric methods were conducted at 303 K selected in parallel with room temperature. The coupons were exposed to various environments in the immersing position and suspended with corks. The weight of the tested coupons was reliably measured after the experiments had been completed. For precise weighing measuring of low carbon steel coupons, a digital electronic weighing balance was used. All experiments were conducted using a wireless thermostat to keep steady temperatures. Weight loss tests were also conducted at different temperatures, 303, 313, 323, and 333 K, to investigate temperature effects on the corrosion phenomenon. Weight losses for low carbon steel coupons were calculated, which is essentially the weight differential of the coupons before and after they were exposed to the corrosive environment. All tests were conducted in three sets to obtain accurate data. Corrosion rate (C_R units mmy^{-1}) and inhibition efficiency (IE%) can be evaluated from gravimetric techniques as according to Equations 1 and 2 [22].

$$C_R = \frac{87.6w}{atd} \tag{1}$$

$$IE\% = \frac{W_o - W_i}{W_o} \times 100$$
 (2)

where *W* represents the weight loss (mg) of low-carbon steel, *a* is an area (cm^2) of tested coupon, *t* is time (h), and *d* is the density ($g.cm^{-3}$) of tested coupon, W_o is the weight loss of low carbon steel coupons in 1 M HCl in the absence of the tested inhibitor, and W_i is the weight loss of low carbon steel in presence of inhibitor.

2.3. Surface morphology

The surface Characterization of low-carbon steel coupons was evaluated by scanning electron microscopy (SEM; Hitachi TM1000 tabletop microscope). The surface characterization was conducted by utilizing low-carbon steel coupons which were exposed in 1 M hydrochloric acid solution without addition and with a 500 ppm concentration of NAPB as a corrosion

inhibitor. After five h, the low-carbon steel coupons were washed with acetone, dried, and utilized for characterization.

2.4. Theoretical chemical calculations

Quantum chemical calculations were performed to a theoretical realization of the efficacy of the inhibitor molecules and the identification of the most important factors that could regulate the inhibitor's action. DFT was used to define quantum chemical parameters. Neutral mode using DFT with B3LYP function was used to carry out the quantum calculations for tested inhibitor molecules. The energies of ionizing and electronic affinity were evaluated employing HOMO (highest occupied molecular orbital) and LUMO (lowest unoccupied molecular orbital) orbitals, which measured the electron negative (χ) effect of the inhibitor molecule and its global hardness (η) . Moreover, the parameters such as energy gap (ΔE), softness (σ), and the number of transferred electrons (ΔN) were also calculated (E.q. 3-8) [23].

$$I = -E_{HOMO} \tag{3}$$

$$A = -E_{LUMO} \tag{4}$$

$$\chi = \frac{I+A}{2} \tag{5}$$

$$\eta = \frac{I-A}{2} \tag{6}$$

$$\sigma = \frac{1}{\eta} \tag{7}$$

$$\Delta N = \frac{\phi \cdot \chi_{inh}}{2(\eta_{Fe} + \eta_{inh})}$$
(8)

The ironwork function (ϕ) and hardness were evaluated as 4.82 eV and zero, respectively [24].

3. Result and Discussion

3.1. Synthesis

The chemical structure of the synthesized corrosion inhibitor was confirmed by spectroscopically techniques (Fourier transform infrared (FT-IR) and Nuclear magnetic resonance (NMR). FT-IR (cm⁻¹): 3341 and 3317 cm⁻¹ (N-H), 3079 cm⁻¹ (C-H aromatic), 1689 cm⁻¹ (CH₃C=O), and 1649 cm⁻¹ (C=O lactam). ¹H NMR (400 MHz, DMSO- d_6) chemical shift (ppm): δ 10.13 (s, 1H), 8.99 (s, 1H), 7.88 (d, 1H), 7.71 (d, 1H),

7.26 (s, 1H), 6.69 (s, 2H). ¹³C NMR (400 MHz, DMSO- d_6) chemical shift (ppm): δ 20.7, 110.3, 111, 121.8, 127.3, 128.6, 130.1,131.4, 144.5, 166.8, and 168.9. CHN-elemental analysis (calculated/found): C, 64.19/63.91; H, 5.39/5.97; N, 17.27/17.88

3.2. Weight loss measurements

3.2.1. Effect of inhibitor's concentration

The synthesized inhibitor (NAPB) is highly effective in preventing low-carbon steel corrosion. Changes in the corrosion rate and inhibition efficacy have been seen in Figure 2, where the inhibitor concentration was varied. Measuring weight loss at 303 K was achieved. The corrosion rate decreased as the inhibitor concentration increased, and the inhibition was improved, as shown in Figure 2. This reduction rate indicates that the inhibitor molecules are mainly adsorbed more widely on the steel surface as concentrations are increased. The interaction of steel and corrosive environment is therefore prohibited. Thus, NAPB showed the highest inhibition efficiency at 500 ppm and 96.8 % of inhibition efficiency. The significant inhibition efficiency of NAPB may be attributed to the electron donate sites, such as benzoyl and acetyl, which enhance the efficiency of inhibitor molecules to donate electrons to the unoccupied d-orbitals of iron atoms on the steel surface, which retards the process of corrosion [25-29]. Figure 2, however, shows no identifiable changes in inhibition performance due to an additional increase in the concentration of the inhibitor, which may be because of the surface saturation. Therefore, 500 ppm is the best-selected concentration.

3.2.2. The effect of temperature

The temperature effect was being investigated on corrosion rate and inhibition efficiency. The experimental findings were presented in Figure 3. In general, the corrosion rate increases with temperature, increasing linearly. More significant temperature influences have the advantage of accelerating a chemical reaction and reducing oxygen solubility, allowing a cathodic reaction. Moreover, the solution viscosity decreases with a rise in temperature, which improves the spread of the solution (iron ions). In this case, this would allow for improved transport by the metal surface of reactants (dissolved oxygen or other electric receivers). The temperature effect on corrosion rate without investigated inhibitor is four times that on corrosion rate in the presence of studied inhibitor. These findings attributed to the inhibitor molecules forming a protected layer that isolates the low-carbon steel surface from the acidic environment. Furthermore, the temperature has an unfavorable impact on the inhibition efficiency, around 20 %. This raise in temperature contributes to an increase in inhibitor molecules' dynamic energy. This increases the rate of the impact. This prevents and reduces the deposition of the inhibitor protecting layer on the surface of the steel. The temperature increase induces adsorption molecules to be strong on the surface of the steel.



Figure 2: Variation of corrosion rate and inhibition efficiency with the various inhibitor concentrations.



Figure 3: Effect of temperature on inhibition efficiency at various concentrations of tested inhibitor for five h as exposure period.

3.3. Adsorption isotherm

Regarding the inhibitor molecules' adsorption on the surface of low-carbon steel, a significant amount of knowledge is revealed by means of various adsorption models. Of all adsorption models, Langmuir adsorption isotherm was reported as being the most suitable to explain the adsorption of synthesized inhibitors on the low-carbon steel surface for reasons that the linear regression parameter is obtained quite nearest to unity [30]. Langmuir adsorption isotherm is quite well illustrated according to Equation 9.

$$\frac{C_{\text{inh}}}{\theta} = \frac{1}{K_{\text{ads}}} + C_{\text{inh}}$$
(9)

This relationship essentially briefly refers to the dependence of the inhibitor concentration on the coverage surface (θ) of the inhibitor. The portion of the surface area protected by an inhibitor molecule shall be specified as the fracture and measured according to Equation 10.

$$\theta = \frac{W_{o} - W_{i}}{W_{o}} \tag{10}$$

At 303 K, Figure 4 demonstrates the plot of C_{inh}/θ versus C_{inh} which has a straight line, and thus the inhibitor molecules' adsorption is much more efficient than other isotherms and obeys the Langmuir adsorption isotherm. K_{ads} means the affinity between adsorbents

and adsorbents.

Higher K_{ads} Value resulted in increased adsorption and ultimately improved performance in inhibition. Higher values of K_{ads} inferred greater adsorption and subsequently way better inhibition efficiency. K_{ads} can be ascertained using the intercepts of the straight line (Equation 11).

$$\Delta G_{ads}^{o} = -RT \ln(55.5K_{ads}) \tag{11}$$

where R and K_{ads} are the gas and equilibrium constant, respectively, whereas T signifies the absolute temperature.

The absorption of negative Gibbs energy (ΔG_{ads}^o) indicates a spontaneous process; that is, the NAPB molecules are efficiently adsorbed on the low-carbon steel surface [31]. It is evident that the interactions and hence physical adsorption of the NAPB molecule to the low-carbon steel surface is described by $\Delta G_{ads}^o \leq$ $-20 \ KJ \ mol^{-1}$. On the other hand, a ΔG_{ads}^o with high negative value $\Delta G_{ads}^o > -20 \ KJ \ mol^{-1}$ refer to a chemical adsorption which represents the chemical reaction of NAPB molecules with d-orbitals of iron atoms and the formation of the coordination bonds [32]. Hence, the ΔG_{ads}^o value for synthesized inhibitor is evaluated as $-38.9 \ KJ \ mol^{-1}$. This proposed that the inhibition mechanism involve physical and chemical adsorption.



Figure 4: Langmuir adsorption isotherm plot for the synthesized inhibitor.



Figure 5: SEM photographs of low-carbon steel for (a) in the absence of inhibitor, (b) in the presence of 500 ppm of NAPB.

3.4. Morphology characterization

Figures 5a and b represent the SEM photographs of low-carbon steel's surface in a 1 M hydrochloric acid environment in the absence and presence of optimum NAPB concentration. The surface of low-carbon steel becomes coarse and damaged when immersed in the corrosive solution, as demonstrated by Figure 5a. On the opposite, due to the existence of NAPB assynthesized corrosion inhibitor (Figure 5b), the lowcarbon steel surface remained significantly unchanged, suggesting suppression of the corrosion phenomenon.

3.5. DFT Calculations

Quantum chemical simulations provide unmatched and detailed data into novel synthesized corrosion inhibitor molecules in geometrical and electronic configuration [33]. In the current study, DFT was applied to determine the most optimized geometrical isomer and Frontier molecular orbital's density (HOMO and LUMO) shown in Figure 6. The calculated DFT parameters are demonstrated in Table 2.

Table 2: DFT Parameters for the Four NAPB Molecules.

$E_{\rm HOMO}~({\rm eV})$	$E_{\rm LUMO}~({\rm eV})$	$\Delta E_{\rm gap}~({ m eV})$	Ι	A	η	X	σ	ΔN
-8.590	-2.438	6.152	8.590	2.438	3.76	5.514	0.265	7.8



Figure 6: Optimized structure and Frontier molecule orbital density distributions (HOMO and LUMO) of NAPB molecules.

The hypothesis is that a relatively planar inhibitor molecule can coat a considerable area of steel surface when interacting with a low-carbon steel surface [34]. Furthermore, active sites are quite known for their capability to increase the reacting power of an inhibitor molecule. From these findings, an important conclusion for the geometry of the NAPB molecule and the NAPB structure appears to be almost planar.

The introduction of pyrrole, carbonyls, amines, methyl, and benzoyl alters the planarity of NAPB, while an unobvious effect was observed in the case of the aromatic and pyrrole rings. The energies of HOMO and LUMO, it can be shown that the distribution density in HOMO and LUMO of NAPB molecule as in Figure 6, is throughout the entire NAPB molecule for LUMO and particularly, in the case of HOMO orbital, which is concentrated on the amines and carbonyls. The Frontier molecular orbital's density in inhibitor molecules might further enhance inhibitor molecules' adsorption on the steel surface by dual interaction. In this term, interrelationships between the Unpaired pairs of electrons pair of heteroatoms unoccupied d-orbitals of Fe on the surface of low-carbon steel. Unshared electrons are drawn to the surface of low-carbon steel. That makes them further active.

The evaluated quantum parameters, such as E_{HOMO} , E_{LUMO} , the energy gap $\Delta \text{E}=\text{E}_{\text{HOMO}}-\text{E}_{\text{LUMO}}$, and the number of transferred electrons (ΔN) is presented in

Table 2. The E_{HOMO} this correlate to the tendency for ionization and therefore the tendency to exchange an electron in a NAPB molecule. E_{HOMO} with high value indicates to a high tendency of electron donating, whereas the E_{LUMO} value leads to the affinity of the electron. E_{LUMO} with low value corresponds to high tendency to accepting electron of the NAPB molecule. The number of transmitted electrons from the corrosion inhibitors to the metal surface can also be determined by the donor-acceptor interaction. An inhibiting molecule's potential to exchange its electrons and vice versa with a negative value suggests a positive ΔN value and it normally follows the E_{HOMO} order. The main factor in predicting chemical reactivity and stability of inhibitor molecules is the energy difference in the energy gap, according to general studies [35]. The inhibitor molecule becomes more reactive as the importance of ΔE is lowered. The values of ΔE , E_{HOMO} and E_{LUMO} as in Table 2 suppose that the E_{HOMO} and E_{LUMO} effect predict the inhibitive performance was accurate and agree to the experimental findings. The DFT findings demonstrated that the inhibitor molecule's ability to donate electrons was improved by introducing electrons of active sites such as amines and aromatic rings, which give a glance at the behavior of NAPB molecules as superior corrosion inhibitors with high inhibition efficiency. Global hardness and softness are other significant parameters that provide essential

data about the stability and reactivity of the molecule. The ΔE is large and low for a complicated molecule and a soft one, respectively, and these data suggest a high inhibition efficiency.

4. Conclusions

The synthesized corrosion inhibitor NAPB exhibited a superior inhibitive efficacy of 94.6 at 500 ppm. NAPB having a pyrrole and benzoyl rings in addition to carbonyl and amino groups substituent showed a

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significant inhibitive performance. Weight loss techniques exhibited that an increase in the concentration of NAPB increases the inhibition efficiency on the low-carbon steel surface and The decreases corrosion rate. morphological investigations of the low-carbon surface using the SEM technique confirm the formation of a protected layer covering the low-carbon steel surface. The DFT and experimental findings were observed to be in relatively good agreement.

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