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Benzylidene as Efficient Corrosion Inhibition of Mild Steel in Acidic Solution †

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Abstract: A New benzylidene derivative namely benzylidene-5-phenyl-1,3,4-thiadiazol-2-amine (BPTA), was successfully synthesized and characterized using Fourier Transform Infrared Spectroscopy, Nuclear Magnetic Resonance and elemental analysis (CHN) techniques. The inhibition efficiency of BPTA on mild steel corrosion in 1.0 N HCl was tested at various temperatures. The methodological work was achieved by gravimetric method complemented with morphological investigation. The concentrations of inhibitor were 0.1, 0.2, 0.3, 0.4 and 0.5 mM at the temperatures 303, 313, 323 and 333 K. The BPTA, molecules as become superior corrosion inhibitor with 92% inhibition efficiency of mild steel coupon in the acidic environment. The inhibition efficiency increased with increasing concentrations of BPTA and the excellent efficiency was performed with the 0.5 mM concentration and followed with 0.4 mM. In acidic environment, the 0.5 and 0.4 mM gave the optimum performance with weight loss technique and scanning electron microscopy analysis. On the other hand, the inhibition efficiency decreased with the increase of temperature. Results of BPTA indicated mixed type inhibitor and the adsorption on the mild steels surface obeys the Langmuir adsorption isotherm. It was found that the BPTA performance depend on the concentration and the solution temperature. Quantum chemical calculations have been done to correlate the electronic characteristics of BPTA with the corrosive inhibitive impact. Experimental and theoretical results are in good agreement.

Keywords: BPTA; corrosion inhibitor; sem; nmr; benzylidene

1. Introduction

Acidic solutions have many applications in industries such as cleaning, pickling, descaling, and acidizing. The uses of HCl in industries was due to its performance action in cleaning in addition to low cost and comparting with other acids. The superior procedure technique to impedance the corrosion of the acidic environment is to use of organic inhibitors. Organic inhibitors having heteroatoms such as P "phosphorus", S "sulfur" O "oxygen" and N "nitrogen" were mentioned as corrosion inhibitors. The molecules of organic inhibitors were adsorbing on the surface of the mild steel and retard the active site through blocking the reactions occur on the surface of mild steel [1,2]. Organic compounds proved their abilities for corrosion inhibition through adsorption on the metal surface, on the other hand inorganic inhibitors behavior as anodic inhibitor compounds and the iron atoms enclosed in the layer improve their corrosion impedance. The inhibition action depends on

parameters such as active groups, structure of the molecule and steric effect [3–5]. Following up of the investigations for efficient corrosion inhibitor [6–28], this investigation reports the inhibitive effects of new corrosion inhibitor. The synthesized new corrosion inhibitor namely benzylidene-5-phenyl-1,3,4-thiadiazol-2-amine (BPTA), were characterized with FTIR and NMR spectroscopies. The corrosion inhibition behavior on the surface of mild steel in corrosive environment was studied using weight loss techniques and surface investigations using scanning electron microscope. Density functional theory (DFT) were used to corroborate mythological findings.

2. Experimental

2.1. Synthesis of Benzylidene-5-phenyl-1,3,4-thiadiazol-2-amine (BPTA)

Reflux of similar quantities of benzaldehyde and 2-amino-5-phenyl-1,3,4-thiadiazol (0.02 mol) in ethanol for 5 h. Cooling and precipitated filtered and recrystallized from ethanol to obtain benzylidene-5-phenyl-1,3,4-thiadiazol-2-amine (BPTA); M.P. 169.5 °C, Yield 88 %,FT-IR spectrum (υ, cm^{-1}): 1570 (C=N); ^{1}H-NMR ($CDCl_3$-DMSO-d_6, δ, ppm): 8.82 (s, ^{1}H, azomethine CH=N), 7.36, 7.55, 7.59, 7.93 and 8.01, (10H aromatic H); ^{13}C-NMR (CDCl3-DMSO-d6, δ, ppm): 173.10 and 174.11 (carbon for thiadiazole ring); 164.31 (CH=N); 128.8, 129.76, 131.26, 134.75, 128.39, 127.38, 132.41, 137.16 (carbon for aromatic rings). Anal. Calculated/found for $C_{15}H_{11}N_3S$: C, 67.90/68.10, H, 4.18/4.14, N, 15.84/15.63.

2.2. Materials and Sample Preparation

The mild steel coupons with the composition (wt.%) of C, 0.21%; Fe, 99.21%; Si, 0.38%; Mn, 0.05%; P, 0.09%; S, 0.05% and Al, 0.01% have been used for corrosion tests and the dimensions of coupons were 3 × 3 cm^2. Silicon carbide paper were used to cleaned the coupons and 15 min of sonicated with ethanol. Finally washed with distilled water, acetone and dried. HCl with the concentration if 1.0 M was used as strong acidic environment and was prepared by diluting analytical grade 37% hydrochloric acid with distilled water. The concentration of inhibitors was 0.1, 0.2, 0.3, 0.4 and 0.5 mM.

2.3. Weight Loss Techniques

The gravimetric measurements have been achieved depending on ASTM procedure. The coupons have been immersed in the acidic solution without and with addition of inhibitor at the concentrations of 0.1–0.5 mM. The influence of immersion time 1, 5, 10 and 24 h and the tests were carried out at 303 K and stabilized using water bath. For temperature studies, similar tests were repeated for 5 h, as immersion time. Corrosion rate, Inhibition efficiency, and surface coverage were calculated through Equations (1)–(3).

$$\text{Corrosion rate } (C_R) = \frac{87.6W}{tAd} \quad (1)$$

where C_R is the corrosion rate; t is the immersion time, W is the coupon weight loss (in gram), A is the area and d is the density.

$$\text{Inhibition efficiency } (IE) = \frac{C_{Ro} - C_{Rin}}{C_{Ro}} \times 100 \quad (2)$$

$$\text{surface coverage } (\theta) = C_{Ro} - C_{Rin}/C_{Ro} \quad (3)$$

where C_{RO} corrosion rate in absence of inhibitor where as C_{Rin} corrosion rate in presence of inhibitor.

2.4. Density Functional Theory Calculation

DFT calculations have been used with GAUSSIAN 03W software [29], B3LYP function [30,31] and a 6-31G as basis set [32]. The energies including EHOMO and ELUMO were obtained to calculate further significant parameters such ΔE, η, σ, χ, and ΔN using [33,34] the Equations (4)–(8)

$$\Delta E = E_{HOMO} - E_{LUMO} \quad (4)$$

$$\eta = -\frac{1}{2}(E_{HOMO} - E_{LUMO)} \quad (5)$$

$$\sigma = \frac{1}{\eta} \quad (6)$$

$$\chi = -\frac{1}{2}(E_{HOMO} + E_{LUMO}) \quad (7)$$

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

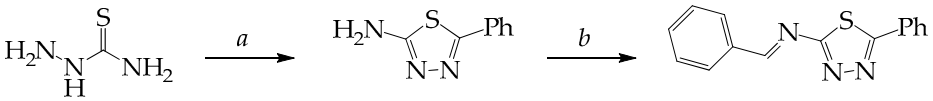
where χ_{Fe} is the electronegativity of iron and η_{inh} is the hardness of iron.

It was reported that χ_{Fe}, was of 7 eV/mol, whereas η_{Fe} was 0 eV/mol [35].

3. Results and Discussion

3.1. Chemistry

The sequences for the reaction synthesis of BPTA starting from 2-amino-5-phenyl-1,3,4-thiadiazol are outlined in Scheme 1. The reaction of benzaldehyde with 2-amino-5-phenyl-1,3,4-thiadiazol afforded benzylidene namely benzylidene-5-phenyl-1,3,4-thiadiazol-2-amine (BPTA) in good yield. The FT-IR spectrum of compound BPTA showed absorption band at 1570 cm^{-1} (azomethine C=N). The ^{1}H-NMR spectrum exhibited a singlet at δ 8.82 ppm due to the one azomethine proton and this band confirm the formation of target inhibitor. A multiplate due to the aromatic proton at δ 7.36, 7.55, 7.59, 7.93 and 8.01 ppm. The NH_2 vibrational band was disappeared for the synthesized inhibitor BPTA that was appeared in the parent compound. The disappearance nitrogen-hydrogen bond BPTA was due to condensation reaction between benzaldehyde with 2-amino-5-phenyl-1,3,4-thiadiazol and reducing H_2O to produce the target compound.



a=benzoic and sulfuric acids with refluxed for 7 h. b= benzaldehyde and refluxed for 5 hours.

Scheme 1. The BPTA chemical structure.

3.2. Gravimetric Techniques

3.2.1. Effects of Concentrations

Weight loss of coupons at different time periods, without and with addition of various concentrations of (BPTA) in 1 M hydrochloric acid at 303 K was investigated. The corrosion rate and the inhibition efficiency are displayed in Figures 1 and 2. It is obvious that the reducing C_R is related to increase in BPTA concentration which elucidates that furthe BPTA molecules adsorbed on the coupon surface, herewith supplying broader surface coverage.

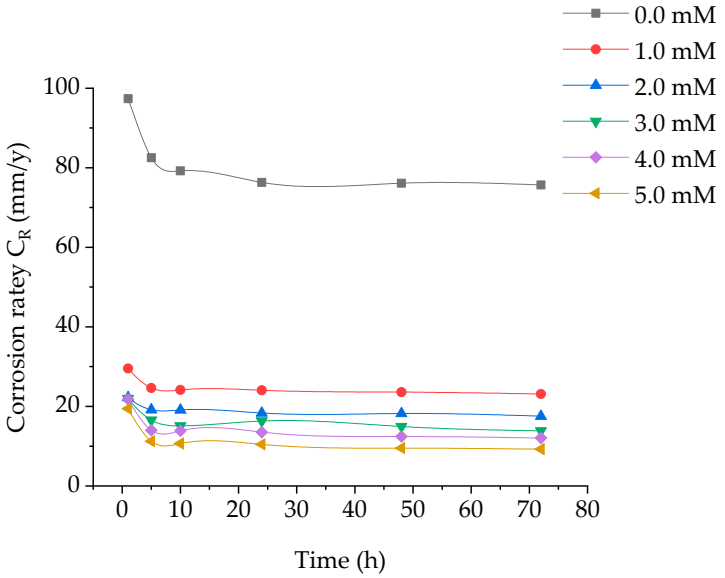


Figure 1. Effect of concentration of BPTA and time on corrosion rate of tested coupon.

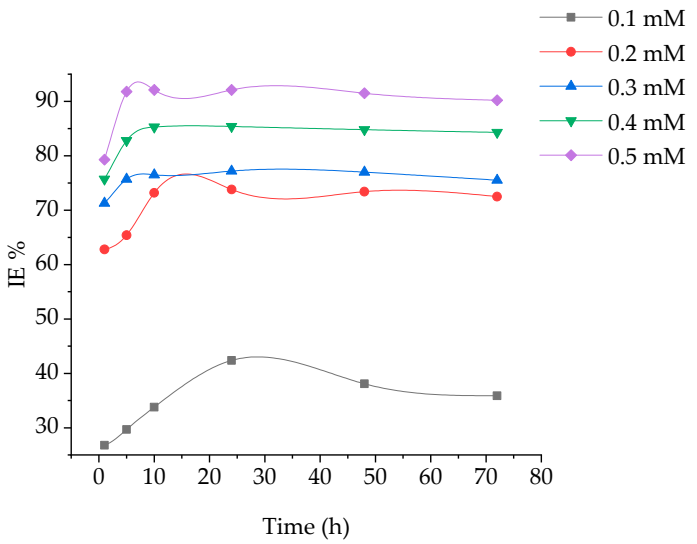


Figure 2. of immersion time of MS in corrosive environment at various concentrations of BPTA.

The maximum IE of BPTA was 92 that obtained at the optimum concentration of 5 mM at 10 h. The excellent inhibition efficiency was attributed to the heteroatoms (N and S) and aromatic rings (phenyl and 1,3,4-thiadiazole) in the molecular structure of BPTA. These hetero atoms react with the coupon surface through adsorption. The inhibition efficiency increases gradually with increasing time until it reaches its highest level at 5 h and stabilizes up to 10 h due to the rapid absorption of the BPTA molecules on the coupon surface and then start to decrease gradually due to desorption. The corrosion rate increases after 10 h of immersion due to the low inhibitor concentration in the HCl solution leading to the decay of the metal; it is clear that after dissolution BPTA molecules from the metal surface become inactive and therefore not involved in the inhibition.

3.2.2. Effect of Temperature

The effect of temperature in the range of 303–333, on Inhibition efficiency in absence and presence of BPTA is shown in Figure 3. From the plot of Inhibition efficiency vs temperature, it is obvious that the Inhibition efficiency decreases with increasing temperature. The inhibition efficiencies obtained at optimum inhibitor (BPTA) concentration were 92, 91 at 303 K and 41 at 333K respectively, in which the minimum and maximum examined temperatures were used in this study.

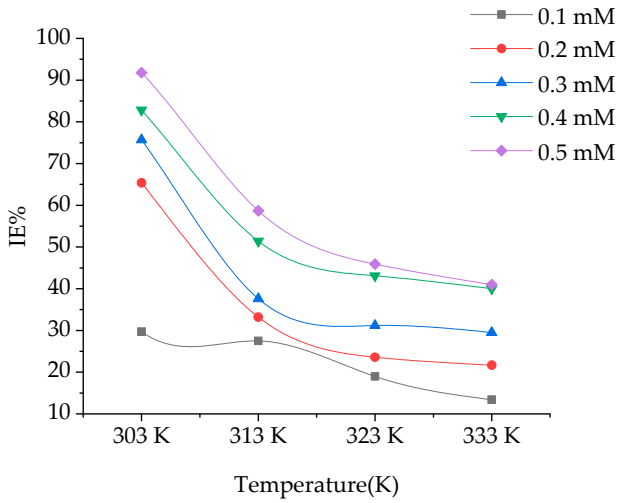


Figure 3. Effect of temperature on IE of BPTA in 1 M HCl.

3.3. Adsorption Isotherms

Adsorption isotherm demonstrate information about the mechanism of adsorption of inhibitor on coupon surface [36,37]. The adsorption of BPTA, molecules on to the coupon surface is the initial stage of adsorption mechanism. Thermodynamic parameters were calculate using the Langmuir adsorption model. The surface coverage increases with the BPTA concentration acidic media, due to formation a protected layer of BPTA molecules on to the coupon surface to impedance the corrosion. Equilibrium constant (Kads) can be calculated via equation 9.

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + B \quad (9)$$

where C is the concentration and B is an intercept.

Figure 4 illustrates the plot of the Langmuir isotherm model, which is demonstrated by the presence of correlation coefficients that were close to the unit, implying that the adsorption of the inhibitor molecules on the coupon surface assumes the adsorption of a single layer. Adsorption of inhibitor molecules tested on the coupon surface obeys Langmuir's isotherm.

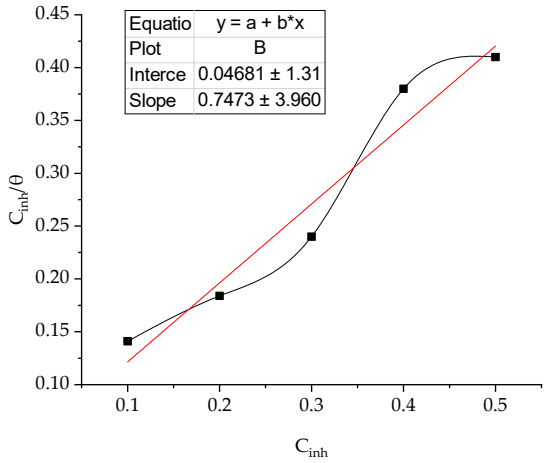


Figure 4. Langmuir plot for adsorption of corrosion inhibitor molecules on to coupon surface.

3.4. Quantum Chemistry

From Figure 5 it can be shown that the HOMO orbital BPTA is localize on the thiadiazole ring and azomethine group. These are the regions donating electrons to unoccupied d-orbital of iron atoms, whereas, LUMO orbitals for BPTA are found on all over the molecule. This region accepts electrons from iron atoms. Thus, the HOMO and LUMO orbital analyses indicate that the thiadiazole ring, benzene ring and azomethine groups play an important role as active sites for the interaction of the corrosion inhibitor with coupon surface.

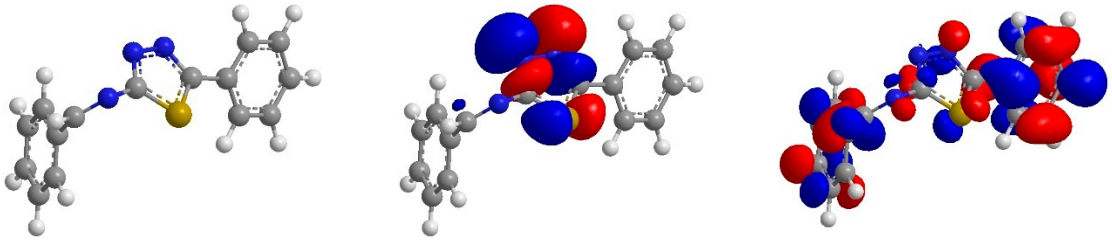


Figure 5. Optimized structure, HOMO and LUMO orbitals distribution of BPTA.

Table 1 demonstrates the parameters for the BPTA. Values of χ, σ and η as in Equations (5)–(7), were calculated by using the values of I (-LUMO) and A (-HOMO) obtained from DFT calculation. Using a theoretical χ value of 7 eV/mol and η value of 0 eV/mol for iron atom [38]. ΔN, the fraction of electrons transferred from inhibitor to the iron molecule, was calculated. According to Lukovits's study [39], if $\Delta N < 3.6$ the inhibition efficiency increased with increasing electron-donating ability at the coupon surface. In this study, BPTA molecules were donate the electrons, and the atoms of iron surface were accepting them. BPTA molecules were bonding to the coupon surface and formed inhibition adsorption layer against corrosive environment.

Table 1. Characteristics of BPTA.

Inhibitor	Energies in eV			Dipole Moment (Deby)	IE%
	EHOMO	ELUMO	EHOMO-ELUMO		
BPTA	−8.003	−1.63	6.643	2.7	92

4. Conclusions

New corrosion inhibitor namely benzylidene-5-phenyl-1,3,4-thiadiazol-2-amine (BPTA), was successfully synthesized and characterized using fourier transform infrared spectroscopy, Nuclear magnetic resonance and elemental analysis (CHN) techniques. The inhibition efficiency for mild steel in hydrochloric acid was evaluated using weight loss and scanning electron microscope techniques. Theoretical investigations were performed to evaluate the adsorption of the inhibitor molecules onto the mild steel surface. Results from the experimental and theoretical considerations are in good agreement confirming that BPTA is an excellent corrosion inhibitor for mild steel in 1 m hydrochloric acid.

Author Contributions: conceptualization, A.A.A.-A.; methodology, S.B.A.-B. and M.M.H.; software, L.M.S.; validation, J.F.O. and S.B.A.-B.; A.A.A.-A. and L.M.S.; writing—original draft preparation. Authorship must be limited to those who have contributed substantially to the work reported.

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Conflicts of Interest: There are no conflicts to declare.

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