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Corrosion Inhibition Study of Thio-Urea Based Schiff Base (BDHNPMTU) on Mild Steel in Acidic Medium

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Abstract
A new thiourea-based Schiff base, N,N′-bis[(3,4-dihydroxy-5- nitrophenyl) methylidene] thiourea (BDHNPMTU), was synthesized by condensation of 3,4- dihydroxy-5-nitro-benzaldehyde with thiourea under acidic conditions. The synthesized compound was characterized using elemental analysis, FT-IR, and UV–Visible spectroscopy. The anti-corrosive effect of BDHNPMTU was evaluated on steel corrosion in 0.5 M HCl using the mass-loss method. The inhibition efficiency was found to increase with inhibitor concentration, showing a maximum efficiency of approximately 96.5 % at 5×10^{-5} M concentration. The adsorption of the inhibitor molecules on the mild steel surface obeyed the Langmuir adsorption isotherm, indicating monolayer adsorption. The results suggest that BDHNPMTU acts as an efficient, stable, and environmentally benign inhibitor for steel in acidic medium.

Keywords – Mild Steel, Corrosion, Thiourea, Schiff base, Langmuir adsorption isotherm, Mass loss measurement, BDHNPMTU

1. Introduction

Corrosion is a natural electrochemical process that deteriorates metals through interaction with the environment, converting pure metals into oxides, hydroxides, or sulphides. It causes severe economic and structural losses, estimated at 3–4 % of global GDP annually. Steel, an alloy of iron with manganese, chromium, zinc, and carbon, is extensively used in industrial applications including construction, pipelines, and manufacturing. However, it is highly susceptible to corrosion in acidic media such as HCl used for pickling, cleaning, and descaling operations.[1-7]
Among corrosion control methods—such as coatings, cathodic protection, and alloying—the use of organic inhibitors is practical and cost-effective. Organic compounds containing heteroatoms like N, O and S are especially effective due to their ability to adsorb on metal surfaces via lone-pair electrons and π bonds. Schiff bases derived from aldehydes and amines have attracted much attention because of their structural versatility, planarity, and high electron density on donor atoms.
Thiourea derivatives represent an important class of Schiff bases that show high inhibition efficiency due to the presence of nitrogen and sulphur donor sites and an aromatic framework, which enhances adsorption stability. In this study, we report the synthesis, characterization, and

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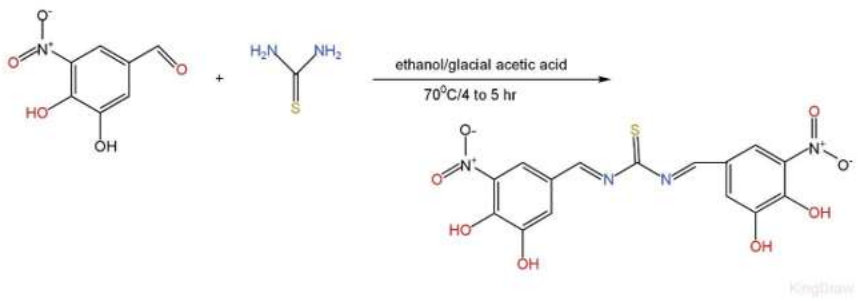
evaluation of a thiourea-based Schiff base (BDHNPMTU) as a potential corrosion inhibitor for steel in 0.5 M HCl.[7-15]

2. Synthesis of Thio-Urea Compound (BDHNPMTU)

All reagents used were of analytical grade. The compound was synthesized according to a modified literature procedure. For the synthesis of N,N'-bis[(3,4-dihydroxy-5-nitrophenyl)methylidene]thiourea (BDHNPMTU) Equimolar quantities of 3,4-dihydroxy-5-nitro-benzaldehyde and thiourea were dissolved in 20 mL of ethanol containing 2 mL glacial acetic acid. The reaction mixture was refluxed at 70 °C for 4–5 hours using a water condenser. Upon cooling, a dark-brown solid precipitated, which was filtered, washed with 50 % ethanol, and recrystallized from ethanol.[16-26]

Reaction Scheme:



Melting Point ≈ 110 °C.

3. Characterization of BDHNPMTU

The structure of the synthesized Schiff base BDHNPMTU was confirmed by elemental analysis, FT-IR spectroscopy, and UV–visible spectral studies. The elemental analysis results (C = 44.34 %, H = 2.48 %, N = 13.79 %, O = 31.50 %, and S = 7.89 %) were found to be in
good agreement with the calculated values, supporting the proposed molecular formula of the compound. The FT-IR spectrum exhibited a broad band in the region 3500–3700 cm^{-1}, which can be attributed to the ν(O–H) stretching vibration, while another broad band observed at 3100–3400 cm^{-1} corresponds to the ν(N–H) stretching vibration. The absence of the characteristic ν(C=O) band of the aldehyde group clearly indicates the completion of the condensation reaction and the formation of the Schiff base. A strong band appearing in the region 1690–1640 cm^{-1} is assigned to the ν(C=N) azomethine linkage, confirming the formation of the imine group. The presence of the nitro group is evidenced by the asymmetric and symmetric ν(N–O) stretching vibrations observed in the regions 1515–1560 cm^{-1} and 1345–1385 cm^{-1}, respectively.

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Additionally, the bands appearing around 725 cm^{-1} and 1100 cm^{-1} are attributed to the ν(C–S) vibrations of the thiocarbonyl (C=S) group. [Fig. -2] The UV–visible spectrum of BDHNPMTU shows an absorption band in the range 310–370 nm, which is assigned to the n→π* transition of the azomethine (C=N) group, while the band observed at 249 nm is due to the π→π* transition of the aromatic ring system. Collectively, the elemental and spectral data confirm the successful synthesis and structural integrity of the Schiff base BDHNPMTU.

4.Experimental Theory

Rectangular steel specimens with a zinc coating measuring 3.0 cm × 2.5 cm × 0.045 cm were used for the mass loss measurements. Prior to being used in mass loss experiments, every specimen was dried in acetone and kept in moisture-free desiccators. Double-distilled water was utilized to generate the acid and inhibitor solutions. The mass loss investigation employed in a .01M inhibitor solution. A V-shaped glass hook formed of capillary glass tube was used to suspend each specimen, which was then submerged in a glass beaker filled with 50 milliliters of room-temperature test solution. The test specimen was removed after being exposed for an adequate amount of time, cleaned under running water, and dried in an oven before being weighed. For weight loss measurement time interval taking followed 4 to 48 hours as shown in table 1.

Equations:

1. For the calculation of percentage corrosion inhibition efficiency (η %)

$$\eta\% = 100\frac{(\Delta M_a - \Delta M_i)}{\Delta M_a}$$

Where ΔMa = Mass loss of metal in acid solution.

ΔMi = Mass loss of metal in presence of inhibitor.

2. For the calculation of Surface coverage (Θ) of metal surface by inhibitor

$$\theta = \frac{(\Delta M_a - \Delta M_i)}{\Delta M_a}$$

3. For the calculation of corrosion rates (mm/yr)

$$CR = \frac{(Massloss \times 87.6)}{DAT}$$

Where D = 7.84 g/cm^3, A = area (cm^2), T = exposure time (h).

5. Results and Discussion

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The corrosion rate of mild steel in 0.5 M HCl increases with immersion time, but the addition of BDHNPMTU significantly reduces metal dissolution. The inhibition efficiency increases with concentration due to higher surface coverage by inhibitor molecules. At 5 × 10^{-5} M concentration, maximum efficiency of ≈ 96.5 % was observed at 48 hours.

Adsorption of BDHNPMTU molecules on the mild steel surface followed Langmuir adsorption isotherm, indicating monolayer coverage. The presence of azomethine (–C=N–) and thiocarbonyl (C=S) groups facilitates coordination of lone pairs with Fe atoms on the steel surface, forming a protective film that reduces metal ion release and hydrogen evolution. SEM micrographs of steel surfaces show smooth and protected films in the presence of inhibitor as compared to rough and damaged surfaces without inhibitor.

The adsorption process may be attributed to physisorption at low concentrations and chemisorption at higher concentrations due to the formation of coordination bonds between nitrogen/sulphur atoms and Fe surface atoms.

Table 1: Concentration of inhibitor (COI %), mass loss ΔM (in mg), inhibition efficiency (η), surface coverage and corrosion rate CR (in mm/year) for steel in presence of BDHNPMTU at different time interval

COI	4 HOUR				12 HOURS				24 HOURS				48 HOURS			
	ΔM	η	Θ	CR	ΔM	η	Θ	CR	ΔM	η	Θ	CR	ΔM	η	Θ	CR
BLANK	.06	…	….	.508	.13	…	…	.367	.18	….	…..	.254	.24	…	….	.169
1	.031	48.3	.483	.262	.055	57.7	.577	.155	.08	55.5	.555	.113	.093	61.2	.612	.065
2	.020	66.6	.666	.169	.039	70.0	.700	.110	.050	72.2	.722	.070	.056	76.6	.766	.039
3	.016	73.3	.733	.135	.026	80.0	.800	.073	.035	80.5	.805	.049	.037	84.6	.846	.026
5	.008	86.7	.867	.0677	.010	92.3	.923	.028	.012	93.3	.933	.017	.008	96.7	.967	.005

6. Conclusion

1. The thiourea based Schiff base BDHNPMTU acts as an excellent corrosion inhibitor for mild steel in 0.5 M HCl.
2. Inhibition efficiency increases with inhibitor concentration and exposure time, showing maximum ≈ 96.5 % at 48 hours.
3. Adsorption of inhibitor follows Langmuir adsorption isotherm, confirming monolayer formation.
4. The compound is non-toxic, water soluble and environmentally safe, making it a potential green corrosion inhibitor for industrial applications

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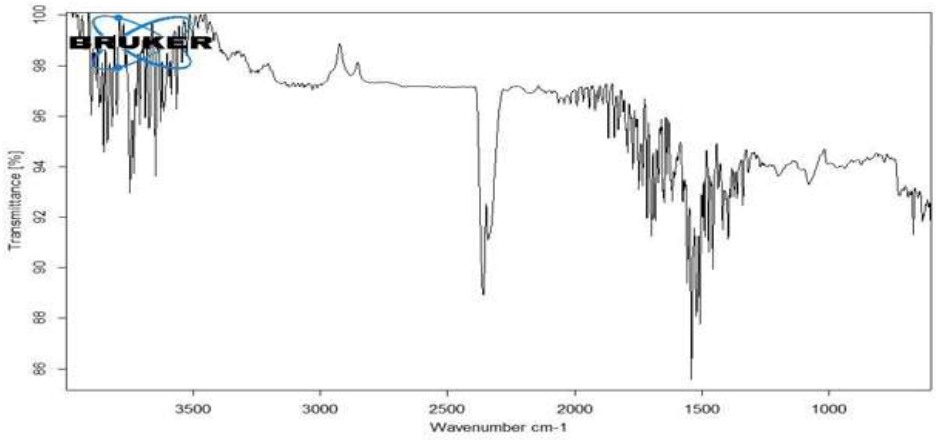
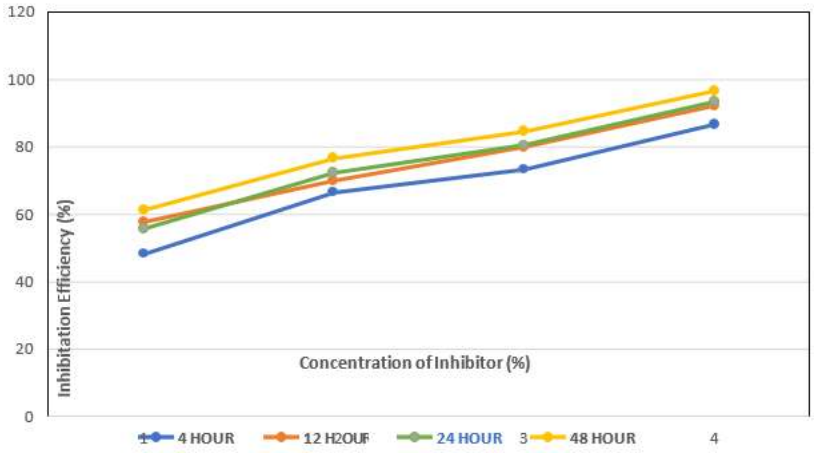
7. Research Gap and Future Scope

Although BDHNPMTU shows excellent performance against steel corrosion in acidic medium, further studies are required to explore:

1. Electrochemical (EIS, Tafel) measurements to understand kinetic and mechanistic aspects.
2. Thermodynamic studies to evaluate adsorption energy and activation parameters.
3. Effect of temperature and different acidic media (H_2SO_4, HNO_3).
4. Development of metal complexes of BDHNPMTU to enhance inhibition efficiency and film stability.



The graph inhibition efficiency v/s concentration of inhibitor (%) at different time interval for mild steel in 0.5 M HCl



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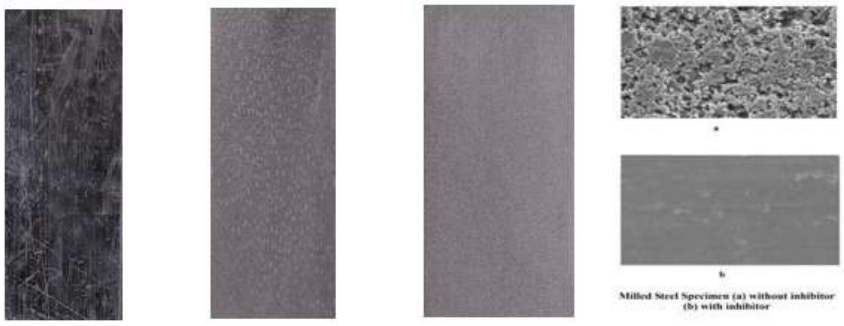

(A) (B) (C)

Mild Steel surface (A) Fresh (B) In 0.5 M HCl (C) In presence of BDHNPMTU

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