International Journal of Engineering, Science and Humanities
An international peer reviewed, refereed, open-access journal
Impact Factor 8.3 www.ijesh.com ISSN: 2250-3552

Exploration of DHNPMHC Complex as an Efficient Corrosion Inhibitor for Copper in Acidic Medium

Suresh Kandoy
Research Scholar, Department of Chemistry, Shri Khushal Das University, Hanumangarh, Rajasthan
Prof. (Dr.) V. K. Swami
Research Guide ,Shri Khushal Das University, Hanumangarh, Rajasthan.

Abstract
The present investigation evaluates the corrosion inhibition performance of a thiourea-based Schiff base, 2-[(3,4-dihydroxy-5-nitrophenyl)methylidene]hydrazine-1-carbothioamide (DHNPMHC), and its metal complex for copper dissolution in hydrochloric acid medium. DHNPMHC was synthesized via condensation of 3,4-dihydroxy-5-nitrobenzaldehyde with thiosemicarbazide and subsequently complexed with metal ions to enhance its surface activity. Structural characterization was carried out using UV–Visible spectroscopy, FT-IR spectroscopy, and elemental analysis. Corrosion inhibition efficiency was evaluated using the weight loss method, adsorption isotherm analysis, and surface morphology studies.
The results demonstrate that DHNPMHC exhibits strong adsorption on the copper surface, leading to a significant reduction in corrosion rate. Inhibition efficiency increases with increasing inhibitor concentration and decreases with prolonged immersion time. Adsorption of the inhibitor follows the Langmuir adsorption isotherm, indicating monolayer formation on the metal surface. SEM analysis confirms the formation of a smooth and protective film in the presence of the inhibitor. The findings suggest that the DHNPMHC complex can serve as an efficient and environmentally benign corrosion inhibitor for copper in acidic industrial environments.
Keywords: Corrosion, DHNPMHC, Schiff base, Copper, Inhibitor, Weight loss method, SEM

1. Introduction

Copper is extensively used in electrical, thermal, and industrial applications owing to its high electrical conductivity, good mechanical properties, and resistance to atmospheric corrosion. However, copper is highly susceptible to corrosion in strongly acidic environments such as acid pickling, descaling, heat exchanger cleaning, and petrochemical processes. Corrosion of copper under such conditions leads to material degradation, economic loss, and reduced operational reliability.
The use of organic corrosion inhibitors is one of the most effective approaches to mitigate metal dissolution in acidic media. Organic inhibitors containing heteroatoms such as nitrogen, oxygen,

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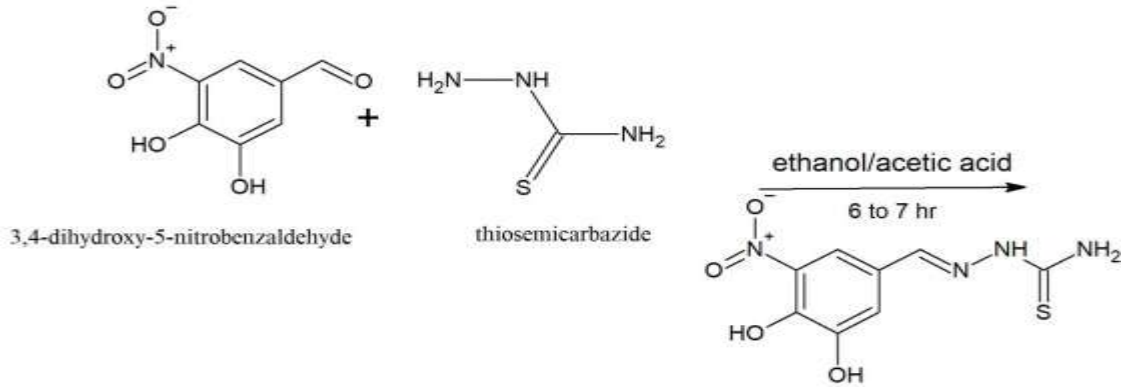
and sulfur exhibit strong adsorption on metal surfaces through donor–acceptor interactions. Schiff bases have attracted considerable attention as corrosion inhibitors due to their ease of synthesis, structural versatility, high inhibition efficiency, and relatively low toxicity.(1-4)

The Schiff base DHNPMHC is particularly attractive because it contains multiple adsorption centers, including azomethine (–C=N–), phenolic (–OH), nitro (–NO_2), and thiocarbonyl (C=S) functional groups. Furthermore, the formation of metal complexes enhances the stability and surface coverage of the inhibitor film. Motivated by earlier reports on related thiourea and Schiff base derivatives, the present study focuses on the corrosion inhibition behaviour of the DHNPMHC metal complex for copper in hydrochloric acid medium.(5-9)

2. Synthesis of DHNPMHC

DHNPMHC was synthesized by the condensation reaction between equimolar amounts of 3,4-dihydroxy-5-nitrobenzaldehyde and thiosemicarbazide in ethanolic medium under mildly acidic conditions. The reaction mixture was refluxed at approximately 70 °C until the formation of a yellow crystalline precipitate was observed. The product was filtered, washed with cold ethanol, and dried under vacuum. The obtained yield of DHNPMHC was 3.35 g, corresponding to 81.25 %. (9-14)The schematic representation of the synthesis is shown in Figure 1.



3. Characterization of DHNPMHC

The synthesized compound was characterized using elemental analysis, FT-IR spectroscopy, and UV–Visible spectroscopy. [fig. -2]

Elemental Analysis: Calculated values were found to be in good agreement with the theoretical composition: C = 37.42 %, H = 3.08 %, N = 21.79 %, O = 24.88 %, S = 12.42 %.

FT-IR Spectral Analysis:

- Broad ν(O–H) stretching band observed at 3470–3650 cm^{-1}
- Broad ν(N–H) stretching vibration in the range 3100–3400 cm^{-1}
- Disappearance of the aldehydic ν(C=O) band confirms Schiff base formation
- Strong azomethine ν(C=N) band observed at 1650–1695 cm^{-1}

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- ν(N–O) vibrations of the nitro group at 1515–1560 cm^{-1} and 1345–1385 cm^{-1}
- ν(C=S) vibrations observed near 725 and 1100 cm^{-1}

UV–Visible Spectral Analysis:

- π→π* transition of the aromatic ring at 249 nm
- n→π* transition associated with the azomethine group at 352–416 nm

These spectral features confirm the successful synthesis of the Schiff base DHNPMHC.

4. Experimental Procedure

Rectangular copper specimens of dimensions 3.0 cm × 2.0 cm × 0.01 cm with a small suspension hole of 0.02 cm diameter were used for weight loss measurements. Prior to experimentation, the specimens were degreased with acetone, washed with distilled water, dried, and stored in a desiccator. Test solutions were prepared using double-distilled water. Corrosion experiments were carried out in 0.5 M HCl solution in the absence and presence of varying concentrations of DHNPMHC.

Each specimen was suspended in 50 mL of test solution using a V-shaped glass hook and immersed for exposure periods ranging from 4 to 72 hours at room temperature. After immersion, specimens were removed, washed thoroughly, dried, and weighed to determine mass loss.

The inhibition efficiency (η %), surface coverage (θ), and corrosion rate (CR) were calculated using standard equations. (15-16)

Equations:

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

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

Where ΔM_a = Mass loss of metal in acid solution.

ΔM_i = 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 = Density of Copper(8.96 g/cm^3), A = area (cm^2), T = exposure time (h).

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For weight loss measurement time interval taking followed 4 to 72 hours in 0.5N HCl as shown in table 1**.**

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

COI	4 HOUR				24 HOURS				48 HOURS				72 HOURS			
	ΔM	η	Θ	CR	ΔM	η	Θ	CR	ΔM	η	Θ	CR	ΔM	η	Θ	CR
BLANK	.09	…	….	.366	.16	…	…	.108	.24	….	…..	.081	1.45	…	….	.328
1	.018	80.0	.800	.073	.055	65.6	.656	.037	.09	62.5	.625	.030	.55	62.0	.620	.124
2	.008	91.1	.911	.032	.039	75.6	.756	.026	.05	79.1	.791	.016	.38	73.8	.738	.086
3	.005	94.4	.944	.020	.024	85.0	.850	.016	.035	85.4	.854	.011	.21	85.5	.855	.047
5	.003	96.6	.966	.012	.010	93.7	.937	.006	.015	93.7	.937	.005	.13	91.0	.910	.029

Scanning electron microscope (SEM) were carried out at 25.0kv and 10.0μm resolution for the surface morphology of Copper surface after its immersion in acidic medium in the absence and presence of DHNPMHC.

5. Results and Discussion

The corrosion behaviour of copper in 0.5 M HCl shows a clear dependence on immersion time and inhibitor concentration. In the uninhibited acidic solution, the corrosion rate of copper increases continuously with immersion time due to enhanced anodic dissolution of copper and cathodic hydrogen evolution. However, the presence of DHNPMHC significantly suppresses copper dissolution by forming a protective layer on the metal surface.

The inhibition efficiency of DHNPMHC increases with increasing inhibitor concentration, which can be attributed to greater surface coverage by inhibitor molecules. At a concentration of **5** × 10^{-4} M, the maximum inhibition efficiency of approximately 96.6 % is observed at 4 hours of immersion. A gradual decrease in inhibition efficiency with prolonged immersion time is noticed, which may be due to partial desorption of inhibitor molecules or weakening of the adsorbed protective film over time.

The adsorption behaviour of DHNPMHC on the copper surface was analysed using adsorption isotherms. A straight-line relationship obtained from the plot of log C versus log θ/(1−θ) confirms that the adsorption process follows the Langmuir adsorption isotherm, indicating monolayer adsorption of inhibitor molecules on the copper surface without significant lateral interaction between adsorbed species.

The high inhibition efficiency of DHNPMHC is primarily attributed to the presence of active functional groups such as the azomethine (–C=N–) linkage and thiocarbonyl (C=S) group. These groups contain lone-pair electrons on nitrogen and sulphur atoms, which can coordinate with

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vacant d-orbitals of copper atoms, resulting in the formation of a stable protective film. This adsorbed film effectively reduces metal ion dissolution and retards the hydrogen evolution reaction.

Surface morphology analysis using SEM further supports the inhibition mechanism. SEM micrographs of copper specimens immersed in acid without inhibitor show a rough and severely damaged surface due to aggressive corrosion. In contrast, copper surfaces exposed to acidic solution containing DHNPMHC appear relatively smooth and uniform, confirming the formation of a protective inhibitor layer on the metal surface.

The overall adsorption mechanism of DHNPMHC on copper can be explained by a mixed adsorption process. At lower concentrations, electrostatic interactions between protonated inhibitor molecules and the metal surface favour physisorption, whereas at higher concentrations, the formation of coordinate bonds between nitrogen and sulphur atoms of DHNPMHC and copper surface atoms leads to chemisorption, resulting in enhanced inhibition efficiency.

6. Conclusion

The present study clearly demonstrates that the thiourea-based Schiff base DHNPMHC acts as an effective corrosion inhibitor for copper in hydrochloric acid medium. The important conclusions drawn from this investigation are as follows:

- DHNPMHC exhibits excellent corrosion inhibition performance for copper in 0.5 M HCl, significantly reducing metal dissolution.
- The inhibition efficiency increases with increasing inhibitor concentration and decreases with prolonged exposure time, attaining a maximum value of approximately 96.6 % at 4 hours at an inhibitor concentration of 5×10^{-4} M.
- Adsorption of DHNPMHC molecules on the copper surface follows the Langmuir adsorption isotherm, confirming the formation of a protective monolayer film.
- The compound is non-toxic, water-soluble and environmentally benign, making it a promising green corrosion inhibitor for industrial applications involving copper in acidic environments.

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Fig. 2 : **IR spectrum of BDHNPMTU**

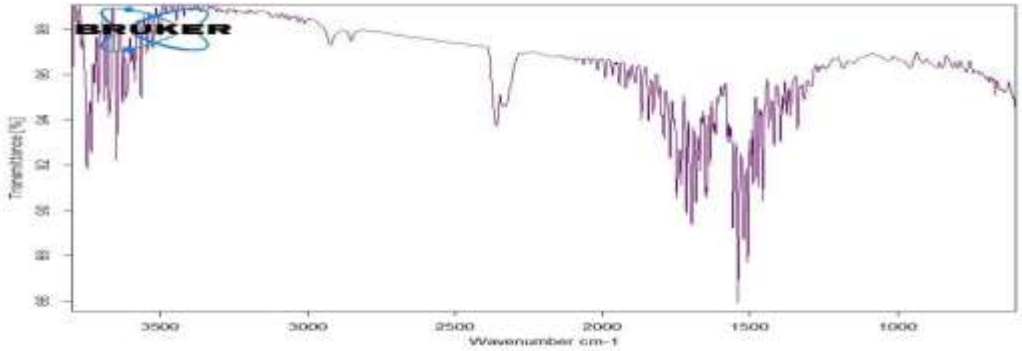

Figure 3: The graph inhibition efficiency v/s concentration of inhibitor (%) at different time interval for Copper in 0.5 M HCl

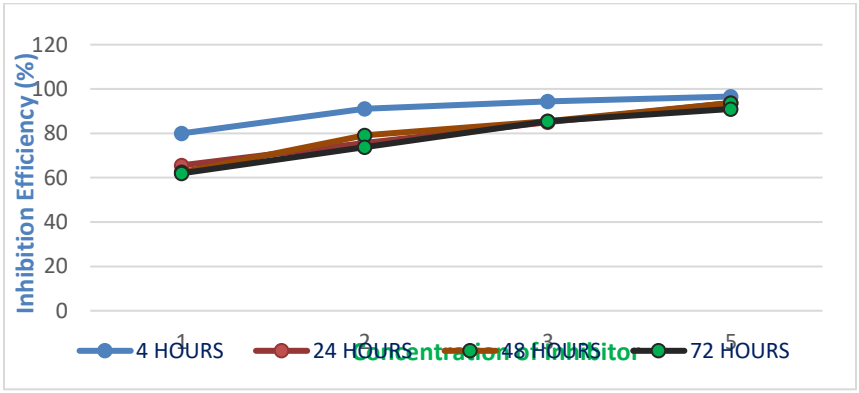

Figure 4: Langmuir adsorption isotherm plot for copper in 0.5 N HCl with Schiff base DHNPMHC

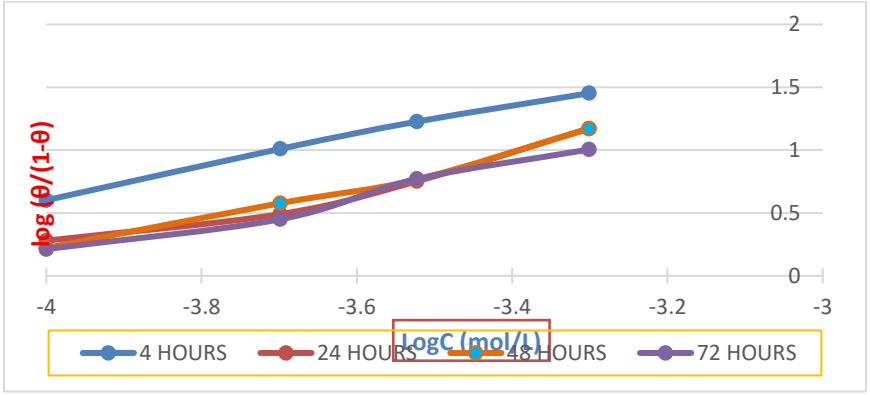

Figure 5: (a) Fresh Copper. SEM Image for 4 hour time duration (b) Cu + 0.5 N HCl (c-d) Cu + 0.5 N HCl + 5% DHNPMHC

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-4	0.602	0.28	0.222	0.213
-3.699	1.01	0.491	0.578	0.45
-3.523	1.227	0.753	0.767	0.771
-3.301	1.453	1.172	1.172	1.005

7. Acknowledgement

Sincere thanks are expressed to Prof. (Dr.) V. K. Swami, Research Supervisor, for his valuable guidance, constant support, and constructive supervision throughout this research work. Gratitude is also extended to the Department of Chemistry, Shri Khushal Das University, where the Ph.D. programme is registered, for providing the necessary facilities for the characterization of the synthesized Schiff base compounds. The support and cooperation of the Principal, Govt. Lohia College, Churu, are gratefully acknowledged for permitting the use of the research laboratory to carry out the experimental work.

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