International Journal of Trend in Scientific Research and Development (IJTSRD)
Volume 5 Issue 5, July-August 2021 Available Online: www.ijtsrd.com e-ISSN: 2456 – 6470

A Study of Newly Schiff Base as Corrosion Inhibitor for Metal Corrosion in Acidic Medium

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ABSTRACT

The Schiff base 2-[(3,4-dihydroxy-5-nitrophenyl)methylidene] hydrazine-1-carbothioamide (DHNPMHC) synthesized by condensation of 3,4-dihydroxcy-5-nitro-benzaldehyde and thiosemicarbazide. The structure of Schiff base was characterized by elemental analysis, FT-IR and UV–Vis methods. The inhibition efficiency of DHNPMHC towards the corrosion of iron in 0.5M HCl was investigated using weight loss measurement technique. Results showed that DHNPMHC is an effective inhibitor for iron corrosion in 0.5M HCl solution. The inhibition efficiency also increased with concentration of inhibitor increased. The maximum corrosion inhibition efficiency of inhibitor reported at 5% (5X10-5M) inhibitor concentration is 89.52 %. Adsorption of the inhibitor on the iron surface followed Langmuir adsorption isotherm.

KEYWORDS: *DHNPMHC, Corrosion, Inhibition efficiency, Iron, Weight loss measurement*

How to cite this paper: Sanjay Kumar | Dr. Yogesh Chahar | Dr. V. K. Swami "A Study of Newly Schiff Base as Corrosion Inhibitor for Metal Corrosion in Acidic Medium" Published in International Journal of Trend in Scientific Research and Development (ijtsrd), ISSN: 2456-6470, Volume-5 | Issue-5, August 2021, pp.1945-1950, URL: www.ijtsrd.com/papers/ijtsrd45226.pdf

IJTSRD45226

1. INTRODUCTION

Iron is the most widely used of all the metals, accounting for over 90% of worldwide metal production. Iron and its alloys are widely used in many industrial and structural applications due to high strength, ease of fabrication and low cost. Stainless steel, which is highly resistant to corrosion, it's commonly used in kitchen cutlery, cookware and hospital equipment [1-2]. Corrosion is a serious problem in all fields of application of metals. Corrosion is the destructive attack of a metal by chemical or electrochemical reaction with its environment. It is a process, where in a metal forms a compound that is more stable, thereby returning to its natural ore form. Corrosion is a reverse process of metallurgy. The loss of metal resources whose abundance is limited is cumulative and poses a danger to conservation and serious economic problem [3-5].

In the steel and ferrous alloys industries HCl is most widely used for removing the undesirable scale and rust formed on the metal surfaces during several industrial processes and exposure to corrosion environment. Corrosive attack on the virgin metal surfaces is caused by some industrial activities also. It is a constant and continuous problem, often difficult to eliminate completely. Generally now a day's paints and polymers widely used to protect the metal surface. Recently the use of synthetic inhibitors has created environmental problems due to its toxicity properties. Thus it is important and necessary to develop low cost and environmentally safe corrosion inhibitors. The use of inhibitor is one of the most practical methods for protection against corrosion to protect metal dissolution and acid consumption [6-10].

Organic compounds containing a heteroatom (N, O and S) in their structure act as good corrosion inhibitors. The corrosion inhibitor efficiency of organic inhibitor are depends on the chemical

structure and physiochemical properties of the compound like functional groups, electron density at the donor atom, *p*-orbital character, and the electronic structure of the molecule [11-12].

Schiff bases form an important class of the most widely used organic compounds and has a wide variety of applications in many fields including analytical, biological and inorganic chemistry [13]. In recent years, the Schiff bases widely tested as effective corrosion inhibitors for iron and its alloys in acidic medium [14-34]. The Schiff base 2-[(3,4-dihydroxy-5-nitrophenyl)methylidene]hydrazine-1-carbothioamide (DHNPMHC) is nontoxic, soluble in aqueous media in presence of small amount of DMSO, relatively cheap and easy to produce with high purity. These properties would justify the use of Schiff base 2-[(3,4-dihydroxy-5-nitrophenyl)methylidene]hydrazine-1-carbothioamide (DHNPMHC) as corrosion inhibitor.

The aim of the present paper is to study the anticorrosive properties of Schiff base 2-[(3,4-dihydroxy-5-nitrophenyl)methylidene]hydrazine-1-carbothioamide (DHNPMHC) on iron corrosion in a strong acidic media.

2. Synthesis and characterization of DHNPMHC:

2.1. Synthesis of DHNPMHC:

All the reagents used in this study were of analytical grade. 3,4-dihydroxcy-5-nitro-benzaldehyde and glacial acetic acid were of obtained from (SLR, India). Thiosemicarbazide, ethyl alcohol and hydrochloric acid were directly procured from MolyChem India Limited. The DHNPMHC was synthesized and characterized on the basis of past various research studies done so far [14-34]

The Schiff base 2-[(3,4-dihydroxy-5-nitrophenyl)methylidene]hydrazine-1-carbothioamide (DHNPMHC) was synthesized from 3,4-dihydroxcy-5-nitrobenzaldehyde (2gm) and thiosemicarbazide (2gm) in ethanol (20ml) in presence of glacial acetic acid(2ml). The content was refluxed in round bottom flask fitted with water condenser for about 6-7 hours at 70^{o}C. On cooling the contents the shining yellow colored solid (M.P.175-180 ^{o}C) separated out. The crystals obtained were washed several times with ethanol, air dried and recrystallized from ethanol. The yield of (DHNPMHC) is 3.35gm (81.25%). The method of synthesis is summarized in Figure-1.

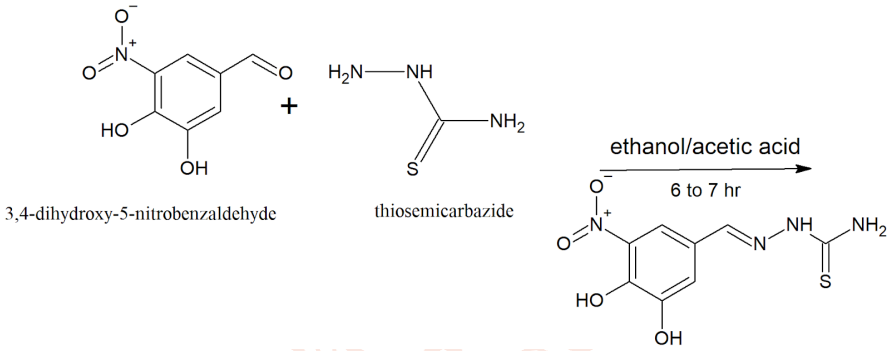


Figure 1: Reaction Scheme

2.2. Characterization of DHNPMHC:

The structure of compound was characterized by elemental analysis, IR and electronic studies. The elemental study shows the presence of C (37.42%), H (3.08%), N (21.79%), O (24.88%) and S (12.42%) in the compound. Absence of a υ(C=O) band of aldehyde and presence of υ(C=N) band occurred at 1695-1650 cm^{-1} in the IR spectra of DHNPMHC indicating the condensation between aldehyde group of 3,4-dihydroxcy-5-nitro-benzaldehyde and amino group of thiosemicarbazide. In IR spectra of DHNPMHC different variable bands like –O-H str. between 3700-3500 cm^{-1}, C-O str. of phenolic group at 1200 cm^{-1}, N-H str. of –NH_2 group between 3500-3400 cm^{-1}, -N-O str. of –NO_2 group at 1575-1500 cm^{-1}, sharp C=C str. of aromatic ring between 1650-1450 cm^{-1} and C-S str. of C=S group at 1120-950 cm^{-1} were observed. In the electronic spectrum of DHNPMHC n-Π* absorption peak of azomethine group were observed at 352,366.5,390,391,416 nm and Π*- Π* peak of benzene ring observed at 240.5nm.

3. Experimental:

For the mass loss study rectangular iron specimens of size 2.5cm x 3.0cm x 0.1 cm was employed with a small hole of about 0.5 cm. diameter near the upper edge were employed. The specimens were mechanically polished with different grades of emery paper; they were degreased in absolute alcohol, dried in acetone and stored in moisture free desiccators before their use in adsorption studies.

All the chemicals employed were of analytical grade and the corresponding solutions were prepared in double distilled water. Test solutions with given concentration of the inhibitor in the acid was prepared by properly

diluting the bulk solution of the inhibitor (.01N). To prepare stock solution of DHNPMHC small amount of DMSO was added for solubility reasons. Concentrations of inhibitor (DHNPMHC) in various test solution ranged from 1.0% to 5.0%.

Each specimen was suspended by a V- shaped glass hook made by capillary glass tube and immersed in a glass beaker containing 50 ml of test solution at room temperature. After the exposure of sufficient time the test specimen was taken out, cleaned under running water and dried in oven, after drying specimens weighted. The variation in mass loss was followed at an interval for 4 hours to 72 hours in 1M HCl as shown in the tables 1.

The percentage corrosion inhibition efficiency was calculated as:
$\eta\% = 100\ (\Delta M_U - \Delta M_i)/\ \Delta M_U$

Where, ΔM_U = Mass loss of metal in uninhibited solution.
ΔM_i= Mass loss of metal in inhibited solution.

The degree of Surface coverage (Θ) of metal specimen by inhibitor was calculated as:
$\Theta = (\Delta M_U - \Delta M_i)/\ \Delta M_U$

The corrosion rates can be calculated by the following equation:
Corrosion rate (mm / yr) = (Mass loss x 87.6)/ DAT

Where, D = density of copper
A = surface area of metal specimen
T = time exposure

4. Result and Discussion:

The inhibition of corrosion is a complex phenomenon. In this process inhibitor adsorbed and forms a protecting layer on the metal surface and decreased the corrosion rate. In this paper the inhibition efficiency of Schiff base DHNPMHC has been studied for iron metal in 0.5 M HCl by using weight loss measurement. Mainly in this paper, we discuss effect of two factors namely immersion time and inhibitor concentration. Immersion time can play a decisive role in the prevention of corrosion ability. DHNPMHC have shown a decreasing in its inhibition performance by rising exposure time or immersion time.

Corrosion rate values decrease as the concentration of inhibitor increases. Surface coverage of iron increases with increasing concentration of inhibitor. Consequently, Inhibition efficiency values increases with increasing the concentration of inhibitor which is shown in Figure-2 (concentration versus inhibition efficiency).The maximum inhibition efficiency of inhibitor reported at 5% ($5X10^{-5}$M) inhibitor concentration is 89.52 % (at 4 hour immersion time). This is due to the adsorption of inhibitor on the iron surface. It is clear from the comparison of images of metal sheet in presence and absence of inhibitor the corrosion of iron inhibit. The plot log $\theta/(1-\theta)$ v/s log C gives a linear line. So the adsorption of inhibitor on iron surface obey Langmuir isotherm that indicate formation of monolayer on iron surface. It is clearly shown in Figure 3.

Table 1 Concentration of inhibitor (COI), mass loss, inhibition efficiency, surface coverage and corrosion rate for iron metal in presence of DHNPMHC at different time interval

COI (%)	4 hours				24 hours				48 hours				72 hours			
	ΔM (mg)	η (%)	Θ	CR (mm/yr)	ΔM (mg)	η (%)	Θ	CR (mm/yr)	ΔM (mg)	η (%)	Θ	CR (mm/yr)	ΔM (mg)	η (%)	Θ	CR (mm/yr)
blank	1.7	…	…	6.3	4.1	…	…	2.53	4.8	…	…	1.48	6.5	…	…	1.35
1	.98	42.9	.429	3.6	2.0	51.2	.51	1.23	2.3	52.1	.52	.710	3.0	54.5	.545	.618
2	.58	66.2	.662	2.1	1.6	60.9	.60	.988	1.28	73.3	.73	.395	2.2	66.6	.666	.453
3	.23	86.6	.866	.85	.63	84.6	.84	.389	.72	85.0	.85	.222	1.3	80.3	.803	.267
5	.18	89.5	.895	.66	.46	88.7	.88	.284	.58	87.9	.87	.179	.92	86.0	.860	.189

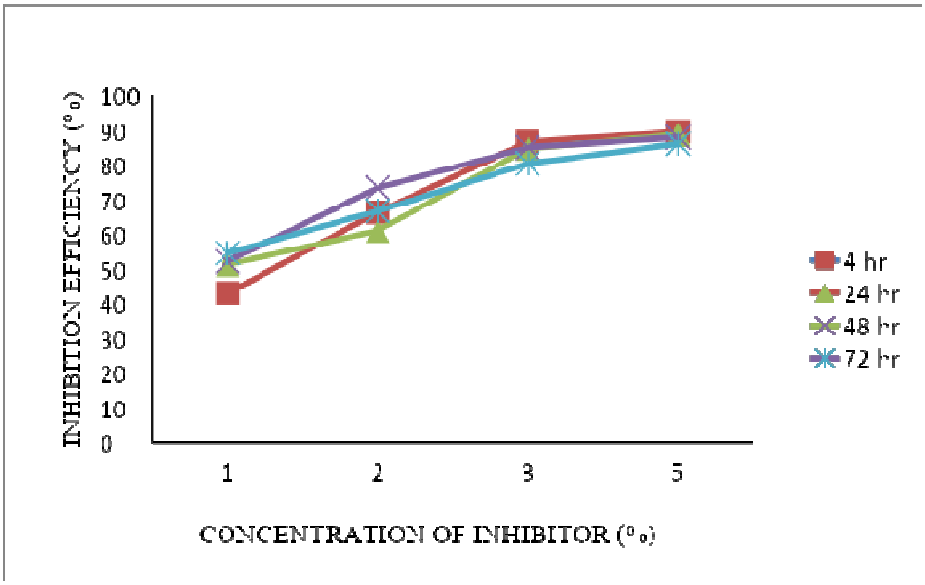


Figure 2: The graph inhibition efficiency v/s concentration of inhibitor (%) at different time interval for iron in 0.5 M HCl

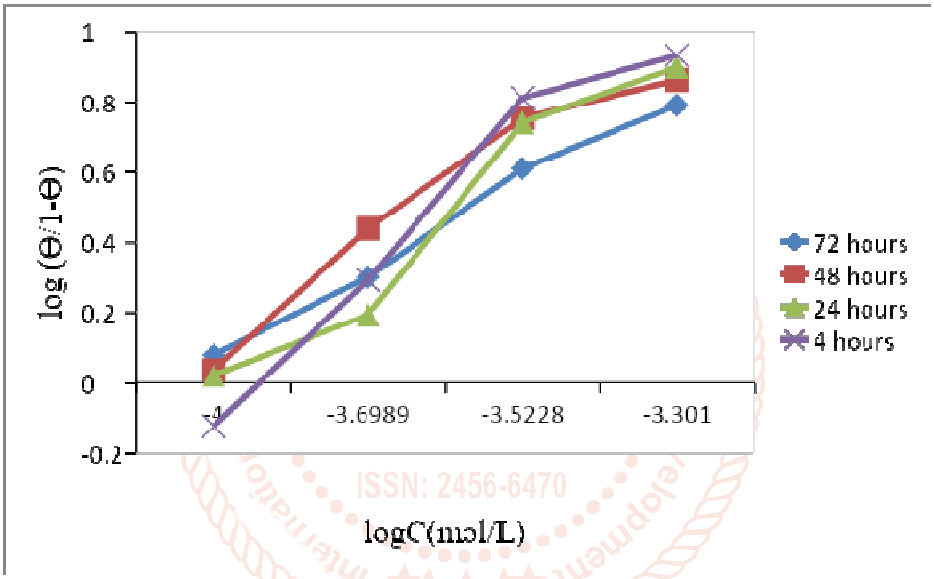


Figure 3: Langmuir adsorption isotherm plot for iron in 0.5 N HCl with Schiff base DHNPMHC

5. Conclusion:

The inhibition efficiency of synthesized Schiff base DHNPMHC for iron in 0.5 M HCl media have been studies. Results obtained from weight loss measurements indicate that Schiff base act as potential inhibitor for iron corrosion in acidic media. Corrosion rate values decrease as the concentration of inhibitor increases. Surface coverage of iron increases with increasing concentration of inhibitor. The graph between inhibition efficiency and concentration shows that the inhibition efficiency increases with concentration of inhibitor. The plot log θ/(1 – θ) v/s log C gives a linear line. So the adsorption of inhibitor on iron surface obey Langmuir isotherm. The maximum inhibition efficiency of inhibitor reported at 5% ($5X10^{-5}$M) inhibitor concentration is 89.52 %.

6. Acknowledgement:

Special thanks to department of chemistry dungar college bikaner and the Principal, Govt. Lohia (PG) College, Churu provided facility of Research Laboratory.

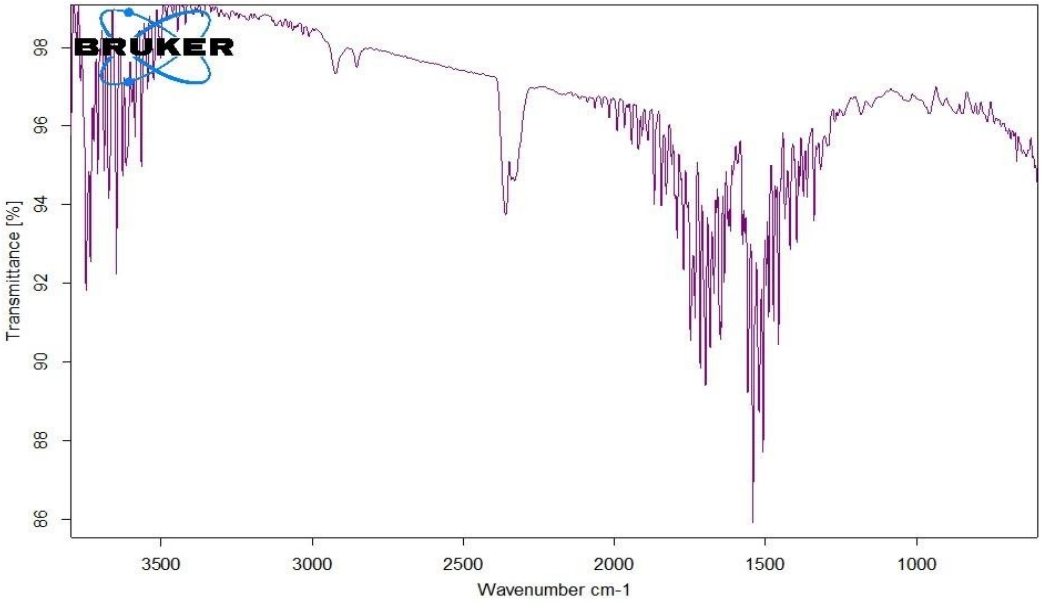


Figure 4: IR spectrum of DHNPMHC

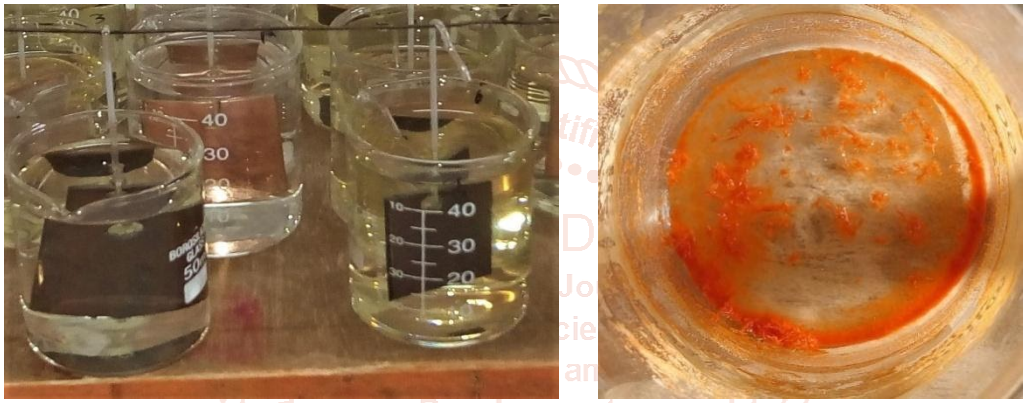
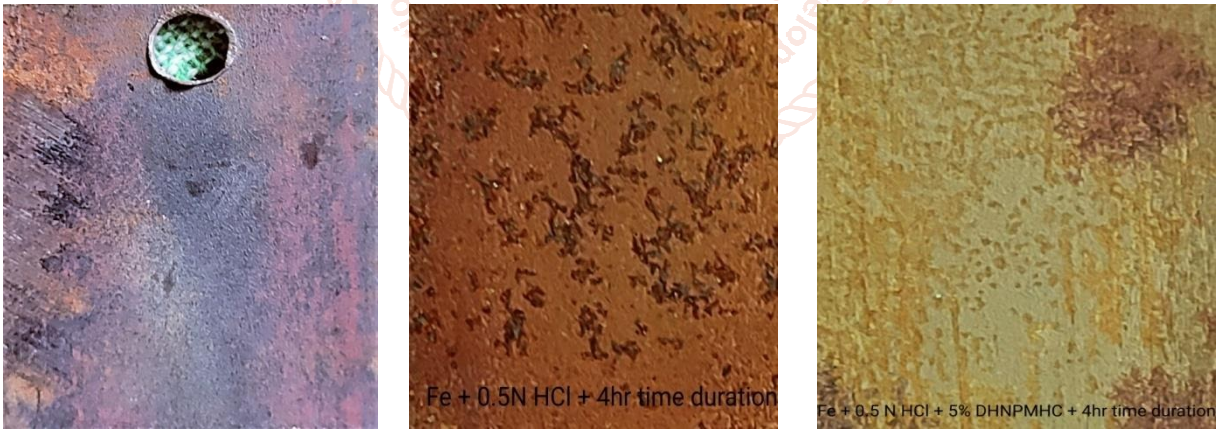


Figure 5: Weight loss measurement process and fresh crystals of DHNPMHC



(a) **(b)** **(c)**

Figure 6: (a) Fresh Iron surface (b) In presence of 0.5N HCl (c) In presence of 5% DHNPMHC

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