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Weight Loss and Surface Analysis Study of Mild Steel Corrosion Inhibition Using 4-((4-Nitrophenyl)-Diazenyl)-3,5-Dimethyl-1H-Pyrazole Compound

Chaitalee Kadam[1] and R. S. Dubey[2]
Ph.D. Scholar, Chemistry, R. J. College, Ghatkopar, India[1]
Associate Professor, Chemistry, R. J. College, Ghatkopar, India[2]

Abstract: *The organic heterocyclic 4-((4-nitrophenyl)-diazenyl)-3,5-dimethyl-1H-pyrazole was synthesized and used as a corrosion inhibitor in a saline solution containing mild steel. The corrosion inhibitor efficiency was evaluated using the weight-loss method. The inhibition efficiency of the inhibitor increases with its concentration. At a higher concentration of 600 ppm, it showed good inhibition efficiency. Characterization of pyrazole is performed using FTIR. The surface morphology of the inhibitor was investigated using SEM and EDAX techniques. This paper represented the inhibition of the inhibitor on the surface of the mild steel. The protective monolayer of the inhibitor formed on the metal surface*

Keywords: Corrosion, cooling water system, mild steel, saline solution

I. INTRODUCTION

Corrosion is a naturally occurring, irreversible process in which mild steel degrades due to electrochemical and chemical interactions between the metal surface and the surrounding environment [1-2]. The corrosion of mild steel is a crucial issue in industrial cooling water systems. Mild steel is used in pipelines, heat exchangers, and valves. Due to the mechanical properties of mild steel, its use in industries is increasing [3-5]. Saline water in the cooling water system can penetrate the metal surface, causing pitting corrosion [6].

The organic inhibitor containing heteroatoms N, O, and S formed a protective passive layer on the surface of corroded mild steel. Organic heteroatoms can be adsorbed physically and chemically on the surface of metal and reduce corrosion [7-9]. The 4-((4-nitrophenyl)-diazenyl)-3,5-dimethyl-1H-pyrazole acts as an effective inhibitor in saline solution against corroded mild steel. The inhibitor demonstrates strong potential for application in a cooling water system. Nitrogen-containing pyrazole inhibitor showed effective results and formed a barrier on the metal surface.

II. EXPERIMENTAL SECTION

Synthesis of inhibitor:

Preparation of 3-(2-(4-nitrophenyl)-hydrazono)-pentane-2,4-dione

Dissolved 4-nitroaniline (0.01mole) in a mixture of 8 ml concentrated HCl with 6 ml water. Cooled the above mixture to 00C in an ice bath. A cold aqueous solution of sodium nitrite (0.02mole) was added. The cooled solution of diazonium salt was filtered into the solution containing acetylacetone in ethanol (20ml), (0.01 mole) sodium nitrite, and sodium acetate (0.05mole). The whole solution was stirred for 2 hours. The solid was filtered and dried. Recrystallization of a solid is performed using an organic solvent, such as ethanol, to obtain 3-(2-(4-nitrophenyl)-hydrazono)-pentane-2,4-dione.

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Preparation of 4-((4-nitrophenyl)-diazenyl)-3,5-dimethyl-1H-pyrazole inhibitor

A mixture of 3-(2-(4-nitrophenyl)-hydrazono)-pentane-2,4-dione (0.01 mole) and hydrazine hydrate (0.1mole) in glacial acetic acid (15 ml) is refluxed for 4 to 5 hours. The resulting mixture was concentrated and allowed to cool. The solid was filtered, washed, dried, and recrystallized from ethanol to afford the compound [10].

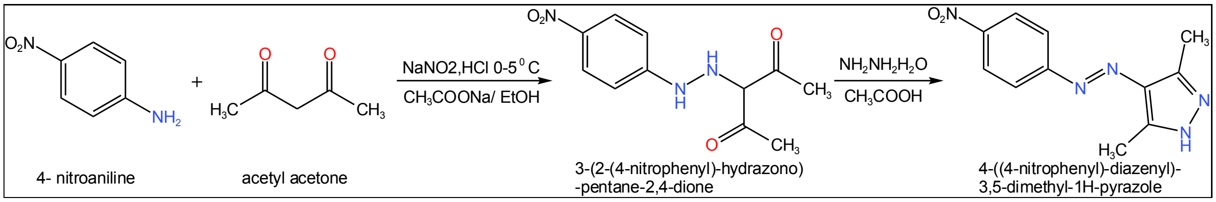

Fig.1 Synthesis of 4-((4-nitrophenyl)-diazenyl)-3,5-dimethyl-1H-pyrazole inhibitor

Material

The mild steel coupons, with dimensions 1 cm × 3.5 cm × 0.02 cm, contained 0.16% carbon, 0.40% manganese, 0.10% silicon, 0.013% phosphorus, and the remainder was iron. The iron part is used in further experimental analysis. The mild steel coupons were used, polished with emery paper, degreased with acetone, washed with double-distilled water, and dried in a desiccator.

Preparation of different concentrations of inhibitor solution:

A 3.5% NaCl solution was used to prepare inhibitor solutions ranging from 100 ppm to 600 ppm. Saline solution (3.5% NaCl) is used as a corrosion solution. It's prepared with AR-grade (35 gm) sodium chloride in one litre of double-distilled water.

Weight loss method:

Different inhibitor concentrations were used to study the weight loss of the pyrazole derivative. It is a method in which mild steel is immersed for 48 hours in solutions with and without an inhibitor. The weight loss of mild steel was studied after 48 hours. The coupons were wiped with acetone, allowed to dry, and then the weight of the mild steel was recorded. The corrosion rate is calculated by using the following equation:

$$CR = W/(S.t) \qquad (1)$$

Where C. R. is the corrosion rate of the inhibitor, w is the difference in the weight of the mild steel, S is the surface area of the used specimen, and t is the immersion time in hours.

Percentage of Inhibition efficiency of the inhibitor calculated by using equation no. 2

$$IE (\%) = (CR-CR')/CR \times 100 \qquad (2)$$

C.R. represents the corrosion rate of the mild steel sample in the absence and presence of the inhibitor.

Surface analysis:

The study compared the solution with and without an inhibitor at a higher concentration of 600 ppm. The formation of a protective layer on the surface of the mild steel was observed in the presence and absence of an inhibitor.

III RESULT AND DISCUSSION

Characterisation of inhibitor:

To evaluate the characteristics of the synthesis inhibitor by the FTIR technique. The Fourier Transform Infrared Spectrum (FTIR) was recorded from 500 to 4000 cm-1 at 2 cm-1 resolution using a PerkinElmer 1710 spectrophotometer.

Molecular weight: 245.24 g/mol, Melting point: 180-1810C

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FTIR (KBr cm-1): 3030.35 (=C-H), 1550.50 (C=N), 1450.24 (-N=N-), 3240 (N-H), 1530.65 (C-NO2), 1595.71 (C=C).

Weight loss analysis:

Effect of concentration on mild steel

The weight-loss method showed that the inhibitor inhibits the surface of mild steel. As the solution concentration increased, the inhibition efficiency also increased. Fig.2 indicates that as the inhibition efficiency increases, the corrosion rate decreases. The inhibition efficiency and the corrosion rate are inversely proportional. The mild steel was immersed in a saline solution for 48 hours, and the corrosion rate was calculated as shown in Table 1. The maximum inhibition efficiency in 600 ppm is 75%, observed at room temperature [11-13].

4-((4-nitrophenyl)-diazenyl)-3,5-dimethyl-1H-pyrazole				
Sr. No.	Concentration (ppm)	Inhibition Efficiency (I.E.%)	Surface Coverage (θ)	Corrosion Rate mgcm-2h-1
1	Blank	----	------	0.081
2	100	42.85	0.428	0.046
3	200	50	0.5	0.041
4	300	53.57	0.536	0.038
5	400	64.28	0.643	0.029
6	500	67.85	0.678	0.026
7	600	75	0.75	0.020

Table 1. Gravimetric analysis data of mild steel in saline solution in the presence and absence of the synthesized derivative of pyrazole at room temperature.

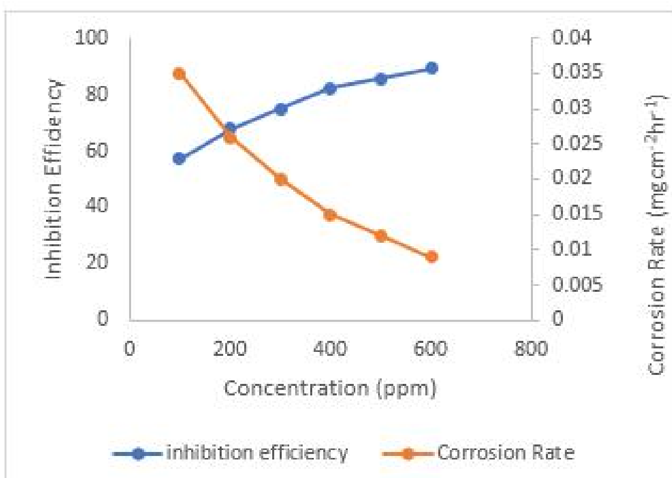

Fig. 2. Corrosion rate and Inhibition efficiencies at different concentrations

Surface analysis:

Scanning electron microscopy (SEM):

Scanning electron microscopy (SEM) analysis was performed on the surface of mild steel in the presence and absence of an inhibitor. The protective energy barrier formed on the surface of the mild steel. Fig. 3(a) shows the metal surface before immersion in the saline solution. Fig. 3(b) shows the metal surface after the mild steel coupon was immersed in a saline solution for 48 hours. The presence of Cl- ions at the surface of mild steel accelerates corrosion, causing

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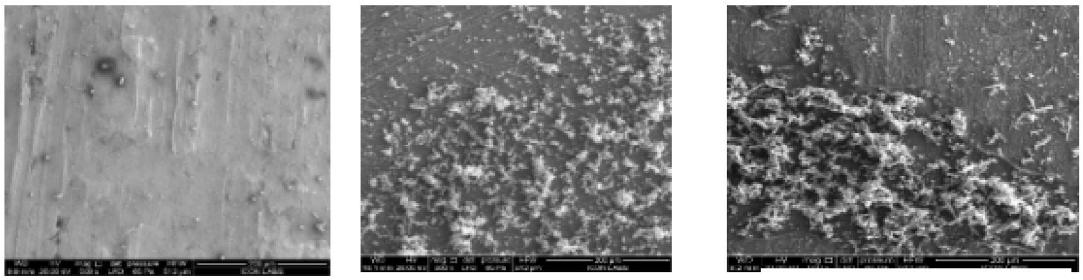
pitting. Fig. 3(c) shows that the mild steel surface has no pits or cracks, indicating that the 600 ppm solution formed a protective layer on the metal surface, thereby reducing corrosion [14-16].



Energy-dispersive X-ray analysis (EDAX)

The Energy-dispersive X-ray technique is used to detect the elements present on the surface of the mild steel. Fig. 4(a) shows that the surface of the mild steel was covered with the maximum amounts of oxygen and iron after 48 hours of immersion in the saline solution. Fig. 4(b) shows the surface of the mild steel, in which the carbon peaks, which are visible in the EDX spectrum, are from the carbon conductive tape used for fixing the sample and carrying out its analysis. The peaks of nitrogen and oxygen indicate the presence of functional groups on the metal surface [17-18]. A Decrease in Oxygen and Chlorine: Proving the barrier from the inhibitor. An Increase in Nitrogen and carbon directly originates from the inhibitor molecule itself. Higher Iron Intensity: If the protective film is thin enough to prevent bulky rust from the surface of the mild steel [19-20].

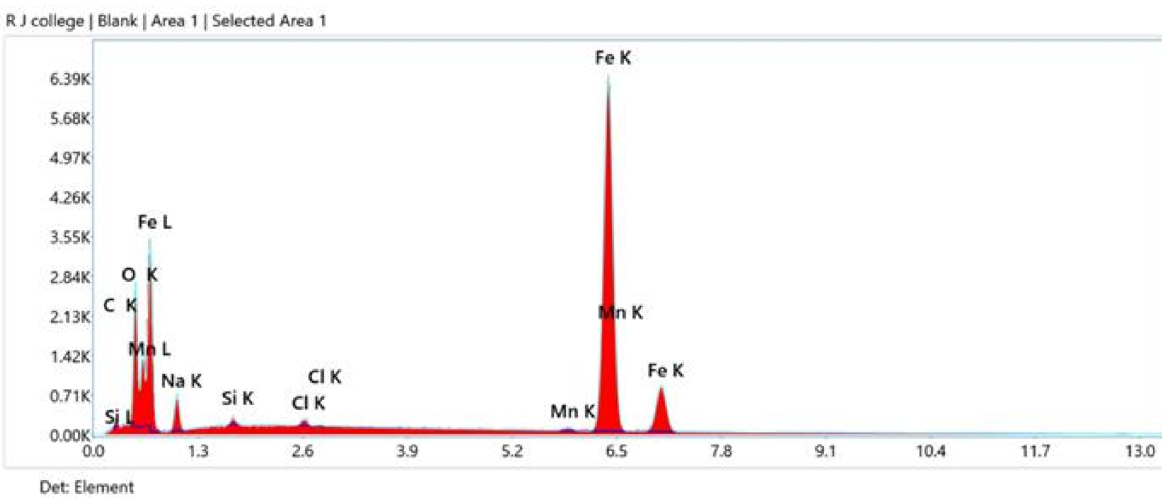


Fig.4(A) EDAX Spectra for Mild Steel After Immersion in 3.5% NaCl Solution for 48 hours

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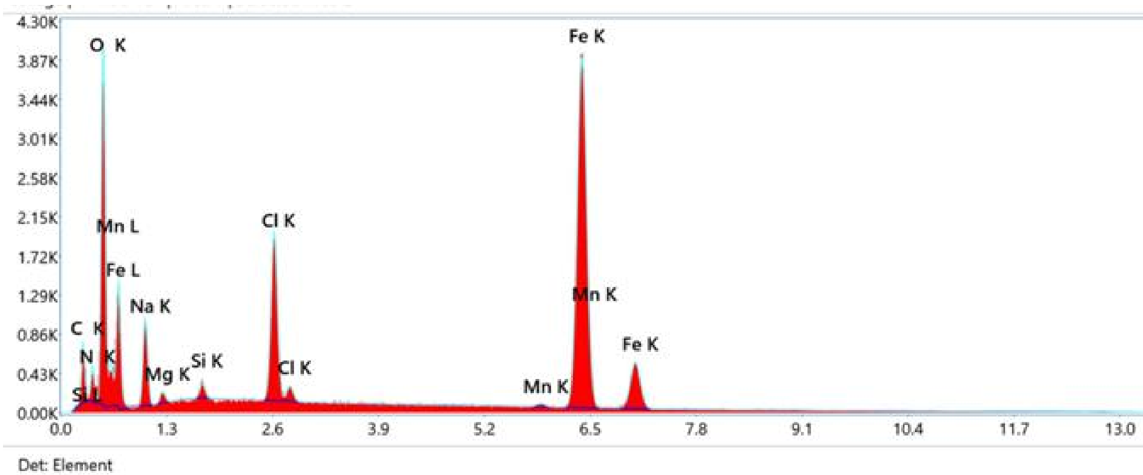

Fig.4 (B) EDAX Spectra for Mild Steel in the Presence of 600 Ppm of Studied Inhibitor

Mechanism of adsorption:

The organic heterocyclic compounds containing heteroatoms that can donate electrons to the surface of the mild steel. Corrosion occurs due to electrochemical reactions in which both oxidation and reduction take place.

Oxidation reaction:

Fe → Fe2+ + 2e- (metal loses electrons, iron gets oxidized)

Reduction reaction:

O2 + 2H2O + 4e- → 4OH- (Oxygen gains electrons, oxygen is reduced)

Mild steel consists of an iron component, and when it is oxidized by losing electrons, it becomes positively charged [21-23]. The inhibitor has electron-rich sites. Two types of adsorptions are observed on the surface of metal. Physisorption, i.e., physical adsorption, and chemisorption, i.e., chemical adsorption, are hence mixed types of inhibition. The observed physical adsorption is due to electrostatic attraction between the polar inhibitor molecule and the charged metal surface. Chemical adsorption is due to the presence of the azo group, which contains nitrogen atoms. The inhibitor has π-electron conjugation with the compound's aromatic ring. NO2 is an ambident ligand that can attach to the surface of the metal and form a strong and stable coordinate bond with the surface of the metal. The inhibitor molecule blocks the metal's active site and forms a compact barrier layer, reducing the corrosion rate of the metal surface [24-27].

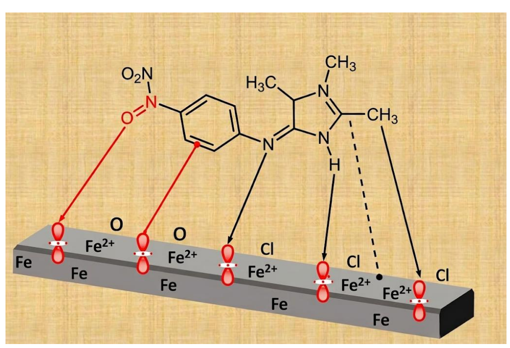

Fig. 5: Corrosion Inhibition Mechanism

II. CONCLUSION

The synthesized pyrazole derivative effectively inhibits corrosion in saline environments. It shows high inhibition efficiency and strong adsorption behaviour, making it suitable for a cooling water system.

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CONFLICT OF INTERESTS

The authors declare there is no conflict of interest.

Author Contributions

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

Chaitalee Kadam https://orcid.org/0009-0007-1818-5735
R.S. Dubey https://orcid.org/0009-0002-3803-1075

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