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EXPLORATION OF CORROSION INHIBITION POTENTIAL OF NOVEL *N, N′*-BIS(2-HYDROXY-3,5-DINITROBENZYL)-1,4-PHENYLENEDIAMINE FOR MILD STEEL CORROSION IN 3M NACL SOLUTION

P. Karuppasamy[1,✉], S. Meena[1], C. Shanmuga Sundari[2], N. Edayadulla[3], and K. Gayathri[4]

[1]Department of Chemistry, Dayananda Sagar College of Engineering, Bangalore- 560111, Karnataka, India
[2]Department of Chemistry, The Oxford College of Engineering, Bangalore-560068, Karnataka, India
[3]Department of Chemistry, Vel Tech Rangarajan Dr. Sagunthala R&D Institute of Science and Technology, Chennai-600055, Tamil Nadu, India
[4]Department of Physics, Academy of Maritime Education and Training, Kanathur-603112, India
✉Corresponding author: periyakaruppankaruppasamy@gmail.com

ABSTRACT

This study explored the corrosion inhibition potential of a novel reduced Schiff base compound, such as *N, N′* -bis(2-hydroxy-3,5-dinitrobenzyl)-1,4-phenylenediamine (BNSPDA), on mild steel (MS) in a 3 M NaCl by weight loss, adsorption isotherm, FE-SEM, EDX, AFM, PDP, and EIS studies. Inhibition efficiency increases with rising inhibitor concentrations but decreases with temperature. Maximum inhibition efficacy reached 65.89 %. The Langmuir adsorption model was found to be the best fit. Polarization data indicated mixed-type inhibition, while EIS revealed a diffusion-controlled mechanism. The $-\Delta H^*$ and $-\Delta S^*$ values supported the exothermic process and confirmed active adsorption. Additionally, $-\Delta G^o$ value reinforced spontaneous adsorption. These findings pave the way for promising mechanisms and future applications in corrosion science.

Keywords: Mild Steel; *N, N′*-bis(2-hydroxy-3,5-dinitrobenzyl)-1,4-phenylenediamine; Adsorption Isotherm; Thermodynamic Parameters; Electrochemical Study

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INTRODUCTION

Mild steel (MS) is one of a significant iron alloy known for cost-effectiveness and wide applications in engineering.[1] Sodium chloride (NaCl) is a versatile compound used in metal pretreatment, plastic production, and textile finishing. In corrosion control, high salt concentrations can reduce the corrosion rate of MS. Additionally, NaCl is utilized in electroplating to prepare conductive solutions for metal plating.[2] Schiff bases are proven, eco-friendly corrosion inhibitors that enhance the resistance of mild steel through compounds containing heteroatoms like oxygen, nitrogen, and sulfur.[3] This study focuses on synthesizing a novel reduced Schiff base compound (BNSPDA) to assess its corrosion inhibition efficacy on MS in a saline environment by various techniques.

EXPERIMENTAL

Materials and Synthesis of Reduced Schiff Base

Salicylaldehyde, *p*-phenylenediamine, and NaCl were sourced from Sigma-Aldrich Chemicals, India. A polished plate (1 cm × 1 cm) was exposed in 3 M NaCl for electrochemical tests. All the solutions were prepared using DI-H_2O and CH_3CN. The Schiff base (**a**) was synthesized using a literature method.[4] To synthesize reduced form (**a1**), 0.01 M of **a** and $NaBH_4$ were dispersed in C_2H_5OH, stirred well for 2 hrs resulting in a colour change. The stirred mixture was filtered, dried, and recrystallized from CH_3OH, yielding BNSPDA (**a1**).

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Detection Method

Morphology and elemental analysis were performed in a Sigma FE-SEM-BRUK ER Nano-X Flash model: 5030. Electrochemical parameters were analyzed using a CH-analyzer (model CHI660E) with an electrochemical cell containing three electrode model. The weight loss method was carried out in a 50 mL Borosil beaker containing 15 mL of solution at different concentrations of BNSPDA and temperatures. The equations used to calculate the various corrosion parameters are given below.

$$\Theta = \frac{W_0}{W_{i-w_0}} \quad (1)$$

$$\text{IE (\%)} = \frac{W_0}{W_{i-w_0}} \times 100 \quad (2)$$

$$\text{Corrosion rate (mpy)} = \frac{87.6 \times \Delta W}{\rho A T} \quad (3)$$

$$\text{IE \%} = \frac{R_{ct(i)} - R_{ct(b)}}{R_{ct(i)}} \times \text{ x } 100 \quad (4)$$

$$\text{\% of IE} = I_{corr(b)} - I_{corr(i)} / I_{corr(b)} \quad (5)$$

$$\log (CR) = \log(A) - \frac{E_a}{2.303RT} \quad (6)$$

$$\log \left(\frac{CR}{T}\right) = \log \left(\frac{R}{hN_A}\right) + \left(\frac{\Delta S^*}{2.303R}\right) - \left(\frac{\Delta H^*}{2.303RT}\right) \quad (7)$$

$$\Delta G_{ads} = -RT\ln (K_{ads} \times 55.5) \quad (8)$$

$$C/\Theta = 1/K_{ads} + C \quad (9)$$

$$Z_{CPE} = \left(\frac{1}{Y_0}\right) [(j\omega)_n]^{-1} \quad (10)$$

$$C_{dl} = \frac{1}{2\pi f max \times Rct} \quad (11)$$

Where, w_i and w_b denote the weight losses of MS, ρ- density, T- time; A-exposed area, Rct- charge transfer resistivity, E_{corr}- corrosion potential, I_{corr}- corrosion current, β_a and β_c- anodic and cathodic slopes, E_a- activation energy, A- frequency factor, R- gas constant, T- temperature, N_A- Avogadro's number, ΔH^*-change of enthalpy, ΔS^*- change of entropy at activated state, K_{ads}- equilibrium constant, θ- surface coverage, C- concentration, Y_0- proportional factor, CPE- constant phase element, ω- angular frequency, j- imaginary unit, n-phase shifting, C_{dl}- double-layer capacitance, and fmax- impedance frequency.

RESULTS AND DISCUSSION

Evaluation of Corrosion Parameters

The corrosion parameters, such as CR (corrosion rate, mpy), IE, % (inhibition efficiency), as well as θ (surface coverage) in the absence and presence of BNSPDA at dissimilar concentrations between 10 ppm and 50 ppm, as well as temperatures between 303 K and 323 K in 3 M NaCl were evaluated. All the weight loss data were calculated by equations (**1-5**). Increasing BNSPDA concentration leads to a substantial decrease in CR and an increase in IE. For instance, the CR decreased from 4039.9 mpy to 992.83 mpy while IE increased from 23.83 % to 65.89 % at 313 K. The highest IE reached about 65.89 % with 50 ppm of BNSPDA. Additionally, θ values were increased from 0.2383 to 0.6589 when increasing BNSPDA concentration and temperature, resulting in enhanced adsorption of BNSPDA on the MS surface (Fig.-1a). To increase the concentration of BNSPDA above 50 ppm, exhibited decreased CR due to desorption of adsorbed BNSPDA on MS.[5]

Evaluation of Thermodynamic and Activation Parameters

All the parameters were evaluated using the equations (**6-8**).[5-7] The evaluated activation data reveal that the E_a, ΔH^*, and ΔS^* increase with the BNSPDA concentration from 10 ppm to 50 ppm. The -ΔS values (148.3 – 130.5 $\times 10^3$ kJmol^{-1}) suggest the BNSPDA adsorbs onto the MS in a more ordered state in the transition phase compared to the reactant state, while the - ΔH values (-46.0 kJ mol^{-1}) indicate an exothermic reaction during corrosion. Figure-1b illustrates the graph between log CR and 1000/T at dissimilar BNSPDA concentrations, which appear as straight lines. From these plots, ΔH_{ads} and ΔS_{ads} were determined. The spontaneity of adsorption is confirmed by the $-\Delta G_{ads}$ = 1.147 – 2.499 kJmol^{-1}

value.[5] The graph between log (CR/T) and 1000/T is depicted in Fig.-1c; indicate the values of ΔH^* and ΔS^*. The $-\Delta H^*$ values (16.42 -32.12 kJ mol^{-1}) and the $-\Delta S^*$ values (190.13-191.66 $Jmol^{-1}/K$). This data vindicates the development of protecting layer on MS due to an interaction of BNSPDA. Linear relationship between C/θ and BNSPDA concentration as shown in Fig.-1d at different temperatures. Figure-1e shows a straight line with an R^2 value close to one, confirming monolayer formation (Eq.-9).

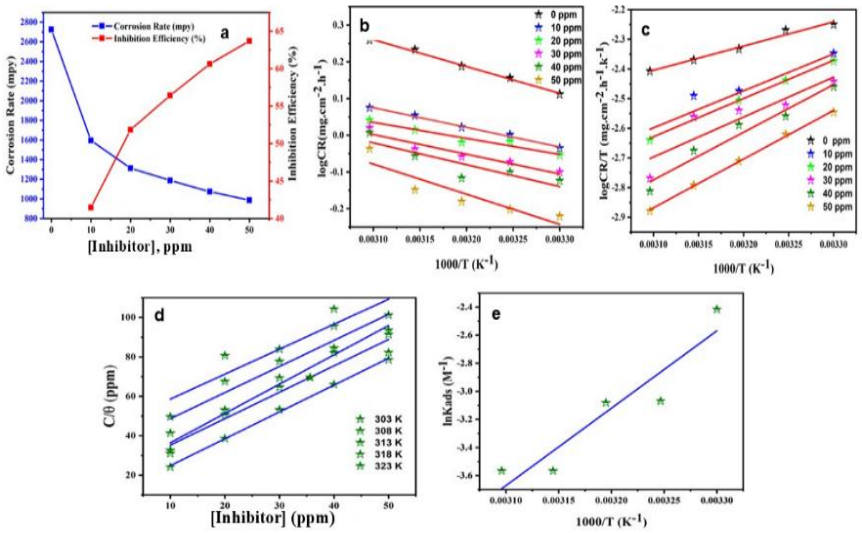

Fig.-1: (a). Effect of Concentrations of BNSPDA (10 ppm -50 ppm) on CR of MS and IE, (%) in 3 M NaCl Environment at 313 K. Thermodynamic Activation Parameters for Corrosion Inhibition of MS in 3 M NaCl, (b). Arrhenius Plot and, (c). Transition Plot in different Concentrations of BNSPDA. Adsorption Isotherm: (d). Langmuir Adsorption Isotherm Plots and (e). Plot of $\ln K_{ads}$ *vs*. 1000/T in different Concentrations of BNSPDA at different Temperatures

EIS and PDP Analysis

Figure-2a displays the EIS data noted that Rct increased (728.87-1386.56 Ω/cm^2) and Rs increased (4.13-180.94 Ω/cm^2) with BNSPDA concentrations, consistent with weight loss method data. Meanwhile, Cdl values decreased (34.35-10.81 $\mu F/cm^2$) suggesting active charge transfer between MS and BNSPDA. The maximum IE observed was 54% at a 50-ppm concentration of BNSPDA. The Bode and phase angle figures indicated an increased phase angle while varying BNSPDA concentrations (Fig.-2b, c). The values of CPE and Cdl were evaluated by equations(10 and 11).[6] Bode plots indicate a single charge transfer mechanism and show that phase angles rise with higher BNSPDA concentrations suggesting the development of a protective layer over the MS surface.[6,7] Potentiodynamic polarization (PDP) study was carried out to analyze interface kinetics at cathodic and anodic sides during the corrosion inhibition of MS using BNSPDA in 3 M NaCl at 313 K. The obtained Tafel polarization curves are shown in Fig.-2d.

Furthermore, the I_{corr} values of corrosion inhibition of MS reduced from 3600.12 to 1091.66 μA cm^2. But the IE decreased and the maximum value found to be 69.67% in 3 M NaCl using 50 ppm of BNSPDA and also the rate of corrosion of MS decreased. The E_{corr} values shifted to the anodic side while varying BNSPDA concentrations. Therefore, the evaluation rate of H_2 decreased owing to anodic corrosion inhibition using BNSPDA. The obtained Tafel data support that the BNSPDA acts as a mixed type of inhibitor.[7] The calculated IE, % values (eq.5) are in close agreement with weight loss and EIS values.

Morphological and Elemental Analysis

SEM micrographs of a polished MS surface (Fig.-3(a-a_2)) displayed a rough surface, elemental composition and mapping analysis of elements. Figure-3(b-b_2) showed the morphology of MS in 3M NaCl and provides information about Cl, Fe, Na, O and C. SEM micrographs of MS surface in 3M NaCl with 50 ppm of BNSPDA is shown in Fig.-3(c-c_2) and gives information about Fe, N, C, and O.

The obtained SEM micrographs confirmed the existence of hetero-atoms which is responsible for strong interaction between MS and BNSPDA. AFM micrographs of MS in 3M NaCl and in 3M NaCl containing are shown in Fig.-3B (a, b).

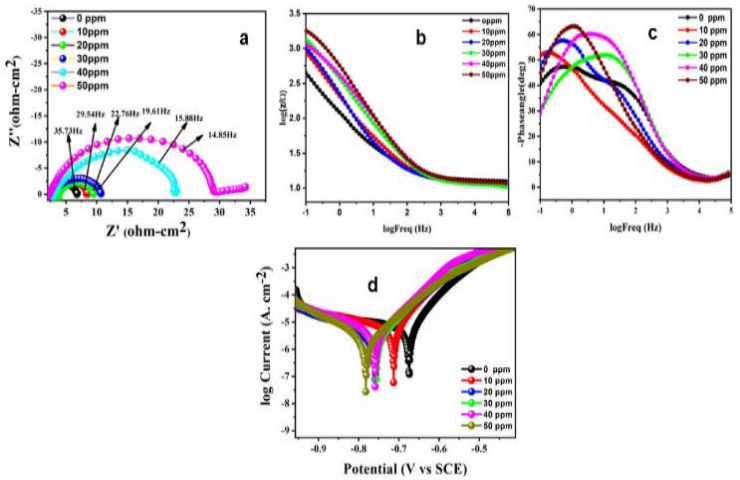

Fig.-2: (a). Nyquist, (b). Bode, (c). Phase Angle Curves and (d). Tafel Curves for MS dipped in 3 M NaCl Containing and without BNSPDA in different Concentrations (10 ppm-50 ppm) at 313 K

AFM data, such as R_a- surface roughness, R_q- root mean-square roughness, as well as peak to valley intensity (P-V) data was found to be 245.2 nm, 227.4 nm and 962.6 nm. AFM data support the formation of a protective layer via electrostatic interaction between the heteroatoms of BNSPDA on MS.[8]

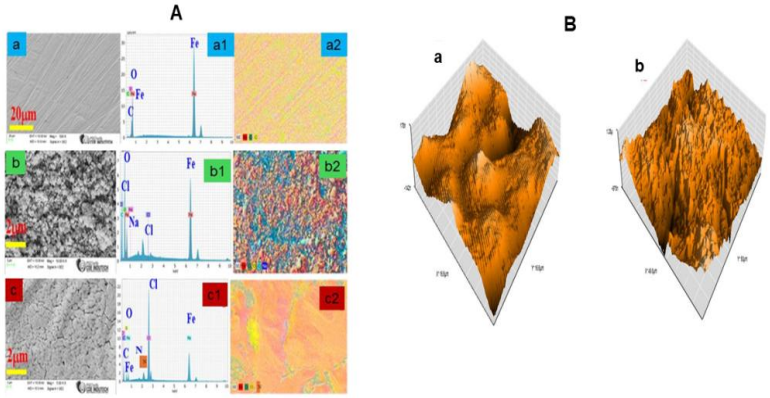

Fig.-3: (A). FE-SEM, EDX and Mapping Images of Polished MS (a - a_2), MS Immersed in 3M NaCl (b-b_2) and MS Immersed in 3M NaCl Containing BNSPDA (c-c_2). (B). AFM Images of MS Dipped in (a). 3M NaCl, and (b). 3M NaCl Containing BNSPDA

Mechanism

BNSPDA helps to inhibit the corrosion of MS in a corrosive environment in three different possible pathways, i). Interaction between lone pair electrons present in the heteroatoms of BNSPDA with MS, ii). Interaction of π-electron cloud present in aromatic moiety of BNSPDA with MS and iii) Charged BNSPDA molecules in saline medium have an electrostatic interaction with MS.[8,9]

CONCLUSION

Corrosion inhibition of MS using BNSPDA in a saline environment was studied using various analytical techniques. Experimental results confirmed the protective action of BNSPDA over MS by physisorption. The electrochemical data supported for mixed type inhibitive action. The morphological and elemental analysis data confirmed an interaction between the heteroatoms of BNSPDA and the MS surface.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9: Industry, Innovation and Infrastructure*. 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:

Periyakaruppan Karuppasamy http://orchid.org/0000-0002-9503-1728
Subramaniam Meena http://orchid.org/0000-0001-9979-8689
C. Shanmuga Sundari http://orchid.org/0000-0001-7814-2074
N. Edayadulla http://orchid.org/0000-0002-3970-5775
K. Gayathri http://orchid.org/0000-0003-0398-0094

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