

USE OF BROMO - INDOLE DERIVATIVE AS BIOCORROSION INHIBITOR OF MILD STEEL IN PRESENCE OF SRB

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ABSTRACT

3-(4-bromo-phenylimino)-1,3-dihydro-indol-2-one, was used to evaluate the biocorrosion inhibition efficiency on mild steel coupons in the presence of sulphate-reducing bacteria (SRB). The results were compiled from gravimetric analysis using the weight-loss method, electrochemical analysis through open-circuit potential (OCP) and potentiodynamic polarisation (PdP), and topological screening with scanning electron microscopy (SEM) and energy-dispersive X-ray analysis (EDX). The results revealed that the highest corrosion rate was observed for the untreated MS coupon, at 0.01611 mg/cm²h¹, and the highest inhibition efficiency was observed at 500 ppm, with 77.00% inhibition and a corrosion rate of 0.00322 mg/cm²/h¹ after 216 hours at 30⁰; the I.E% declined as the temperature increased. The best-fitting graph for the adsorption study was the Langmuir isotherm, which forms a monolayer of inhibitor molecules. A $-\Delta G^0_{ads}$ indicates spontaneous behaviour of the system, with correlating gravimetric and electrochemical analyses with each other. SEM and EDX show that bacterial attachment on the surface was reduced in the treated MS coupons compared to the untreated MS coupons. From the above experiments, the test synthesised compound was efficient at slowing the biocorrosion process in a simulated system.

Keywords: Sulphate Reducing Bacteria, Indole, Biocorrosion Inhibition

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INTRODUCTION

Metals are affected by biotic components in the natural environment, which can initiate electrochemical reactions and precipitate metallic salts. This electrochemical process, driven by bacteria, is called biocorrosion or microbiologically influenced corrosion (MIC). Sulphate-reducing bacteria (SRB) are primarily observed to affect metallic designs under anoxic conditions, causing biotic corrosion and producing metal sulphides and biogenic hydrogen sulphide as by-products. Mild steel or low-carbon steel is widely used due to its mechanical and chemical properties in various engineering structures, such as cooling water systems, cooling towers, and pipelines carrying fluids under low-velocity or stagnant conditions.¹⁻³ *Desulfovibrio desulfuricans* is a natural habitat SRB, identified as the main culprit in damaging MS structures under stagnant conditions. These bacterial cells do not require oxygen as their terminal electron acceptor to complete their cycles. The aerobic species in the system consume oxygen, forming an oxygen-depleted zone that creates an efficient condition for SRB to grow and form colonies. The planktonic cells adhere to the metal surface and secrete enzymatically active extracellular polymeric substances (EPS) into the environment. The role of this EPS is to attract bacterial cells, form bacterial clusters, and increase the number of cells on the surface of the metal. As conditions become unfavourable, biotic cells face difficulties in survival. To cope with these conditions, the colonies form a biofilm on the metallic surface. As conditions become favourable, the mature biofilm releases younger cells into the environment, thereby maintaining and increasing the SRB count in the system and restarting the biocorrosion process.⁴⁻⁶ The use of organic heterocyclic compounds as corrosion inhibitors and as biocides is in need. Indole, being a fused structure of benzene with pyrrole, has the heteroatom nitrogen in the ring. Due to their reactivity, indole and its derivatives are used as precursors in numerous reactions, exhibiting anti-inflammatory, antimicrobial, and pharmaceutical properties, as well as in agro-based industries and so on.^{7,8} The current study explored the efficiency of a bromo-indole derivative against microbially induced corrosion, with the influence of exposure time, concentration, and temperature. The study was conducted using gravimetric analysis to measure mass change, and electrochemical analysis with open circuit potential (OCP) and potentiodynamic polarisation (PdP) to assess corrosion parameters. The surface was analysed with scanning electron microscopy (SEM) and energy dispersive X-ray analysis

(EDX).

EXPERIMENTAL

Mild Steel Sample Preparation for Biocorrosion Exposure and Corrosive Media

Mild steel coupons with elemental composition P-0.013%, S-0.02%, Si-0.10%, C-0.16%, Mn-0.40% with remaining composition of iron. The coupons were cut to dimensions of 0.02 cm × 1.0 cm × 3.5 cm and polished with emery paper of different grades, ranging from 60, 100, 150, and 180. After polishing, these coupons were degreased with purified acetone, disinfected with purified ethanol, dried, and stored in a desiccator. Test culture *Desulfovibrio desulfuricans* utilized for stimulation of biocorrosion in Barr's media with CaSO₄ 0.29g, NH₄Cl 0.19g, Fe(NH₄)₂(SO₄)₂ 0.05g, K₂HPO₄ 0.05g, MgSO₄·7H₂O 0.29g and sodium lactate 0.79g in 100 cm³ of distilled water, and autoclaved for 15 - 20 min at 121°C for three consecutive days, with pH set in a range of 7.0 - 7.5.

Synthesis of Inhibitor 3-(4-Bromophenylimino)-1,3-dihydro-indol-2-one

4-bromoaniline, an aromatic primary amine, and isatin were taken in equal moles and dissolved in warm ethanol. Ethanol was warmed using a steam bath. The dissolved compounds were heated in a steam bath for 40 minutes. The reaction mixture was allowed to stand for 24 hours at room temperature (R.T.). The completion of the reaction was monitored using TCL. A crystalline raw compound was obtained after 24 hours. Ethanol was used for the recrystallisation of the raw product.⁹

Gravimetric Analysis Through the Mass Change Method

Gravimetric analysis was conducted in Barr's media inoculated with *Desulfovibrio desulfuricans* as the bio-corrosive agent. The MS coupons were weighed before and after the immersion in bio-corrosive media. The inhibitor solutions were prepared at concentrations of 100, 200, 300, 400, and 500 ppm and introduced into the corrosive media. The change in mass of MS coupons was observed at 303K, 313K, 323K, and 333K, and the effect of the immersion period of the coupons was noted after 96 hours, 216 hours, and 312 hours. Corrosion inhibition efficiency, corrosion rate, and surface coverage of MS coupons were calculated using equations (1), (2), and (3), respectively.

$$I.E. (\%) = \frac{CR - CR''}{CR} \times 100 \quad (1)$$

$$CR = \frac{w}{a \cdot t} \quad (2)$$

$$\theta = \frac{CR - CR''}{CR} \quad (3)$$

CR is the corrosion rate of MS before inhibitor exposure, and CR'' is the corrosion rate of MS post exposure to inhibitor in corrosive Barr's media; a is the surface area of the MS coupons, and t is the exposure time; θ is surface coverage.

Corrosion Parameters by Electrochemical Analysis and Topography

Electrochemical analysis was conducted using open-circuit potential (OCP) and potentiodynamic polarisation (PdP) techniques with a Squidstat Solo 2181 instrument. MS coupons were masked with enamel lacquer, leaving 1 cm² unmasked. For the experimental procedure, a three-electrode system was used with a Pyrex glass flask, with the reference electrode as a saturated calomel electrode, the working electrode as MS coupons, and the auxiliary electrode as a graphite electrode, with an inhibitor solution of 150 cm³. The potential was swept from +0.25V to -0.25V at a scan rate of 1 mV/s. The duration to reach steady-state current was 60 min, after which PdP curves were continued. Topographic images were obtained through scanning electron microscopy and energy dispersive X-ray analysis for MS without treatment.

RESULTS AND DISCUSSION

FTIR Characterisation of 3-(4-Bromo-phenylimino)-1,3-dihydro-indol-2-one (BDI)

FTIR characterisation was performed using a Perkin-Elmer 1710 spectrometer, with a Fourier Transform Infrared Spectrum in the range of 500 - 4000 cm⁻¹ with 2 cm⁻¹ resolution. IR (KBr, cm⁻¹): 3200 (OH, enolic), 1630 (CO), 1590 (CN), 1440 (CC), 815, 780, 740 (ArH), 660 (C-Br).⁹

Gravimetric Analysis using Mass Change Technique

As per the data, the corrosion rate was affected by a gradual increase in BDI concentration from 0 to 500 ppm, and a maximum I.E. of 77% was observed with 500 ppm BDI in the system for 303K and a minimum I.E. at 333 K at 500 ppm, 61.29%, with a decline in CR, as listed in Table-1.



Fig.-1: Synthesis of 3-(4-bromo-phenylimino)-1,3-dihydro-indol-2-one

Table-1: Mass Change Data for MS Coupons Pre- and post-BDI Treatment in Bio-Corrosive Media at 303K, 313K, 323K, and 333K

Concentration(ppm)	Inhibition Efficiency (%)			Corrosion Rate (mg/cm ² h ¹)		
	313K	323K	333K	313K	323K	333K
Temperature						
0	--	--	--	3.0640	3.8997	4.3175
100	31.81	28.57	22.58	2.0891	2.7855	3.3426
200	50.00	42.85	41.98	1.5320	2.2284	2.5069
300	59.09	50.00	48.38	1.2534	1.9498	2.2284
400	68.18	64.28	58.06	0.9749	1.3927	1.8105
500	72.72	67.85	61.29	0.8356	1.2534	1.6713

Table-2 shows the effect of exposure of mild steel specimens over time, illustrating the increase in I.E. till 216 hours of exposure, and thereby a decline in I.E. after 216 hours. The CR dropped with an increased dose of BDI and increased after 216 hours of exposure compared with 96 and 312 hours. This decline in CR and increase in I.E. are influenced by the heteroatoms and functional groups present on the inhibitor, which get adsorbed, forming a shield on MS, protecting it from the corrosive environment. The vacant d-orbitals on the iron surface and the lone pair of electrons contribute to adsorption on the metal surface, thereby affecting the corrosion rate and interfering with the system.¹⁰⁻¹³

Table-2: Mass Change Data for MS Coupons Pre- and Post-BDI Treatment in Bio-Corrosive Media at 96, 216, and 312 hours

Concentration (ppm)	Inhibition Efficiency (%)			Corrosion Rate (mg/cm ² h ¹)		
	96	216	312	96	216	312
Time (hrs)						
0	--	--	--	0.0203	0.01160	0.0102
100	28.57	38.88	21.73	0.0145	0.00709	0.0080
200	35.71	55.55	30.43	0.0130	0.00515	0.0071
300	50.00	66.66	43.47	0.0101	0.00386	0.0058
400	57.14	72.22	47.82	0.0087	0.00322	0.0053
500	64.28	77.77	56.52	0.0072	0.00257	0.0044

Activation Parameters Relating to the MS coupon

Using the Arrhenius equation (4), activation parameters were calculated for the MS coupons in a corrosive medium.

$$\ln CR = \ln A - \frac{E_a}{R.T} \quad (4)$$

Where corrosion rate is CR, activation energy is E_a , universal gas constant is R, frequency factor is A, and absolute temperature is T. Table-3 lists the activation parameters and line regression value (R^2), which are near unity, proving linearity among the acquired data. The activation energy increased with BDI concentration from 0 to 500 ppm, with the lowest activation energy for untreated specimens and the highest for the 500-ppm treated coupon. The increased value indicates the formation of a physical shield between the corrosive environment and the metal surface, which enhances mass and energy transfer, resulting in reduced damage to the metal surface.

Table-3: Activation Parameters of MS in BDI in Bio-Corrosive Medium

Concentration(ppm)	R^2	E_a (kJ/mol)	ΔH (kJ/mol)	$-\Delta S$ (J/mol)	ΔG_{ads} (kJ/mol)		
					313 K	323 K	333 K
Temperature							
00	0.960	15.097	12.417	196.23	73.765	75.725	77.685

100	0.990	20.627	17.917	181.92	74.570	76.380	78.190
200	0.932	21.727	19.011	180.82	75.351	77.151	78.951
300	0.929	25.391	22.131	172.47	75.967	77.687	79.407
400	0.996	27.133	24.339	167.83	76.610	78.280	79.950
500	0.995	30.395	28.667	155.48	77.182	78.732	80.282

An increase in E_a indicates a stronger physisorption process; E_a values increased from 15.097 kJ/mol to 30.395 kJ/mol. Another form of the Arrhenius equation (5) gives the activation enthalpy and entropy of activation; variations in the activation free energy were given by equation (6). Planck's constant is denoted by h , Avogadro's constant is N , the entropy of activation is S , enthalpy is H , the universal gas constant is R , and absolute temperature is denoted by T .^{14,15} The values for entropy were calculated using the intercept $\ln(R/Nh) + \Delta S/R$, and the values for enthalpy were given by the slope ($-\Delta H/R$), which are listed in Table-3. The enthalpy value was found to be positive, indicating that the metal dissolution process is endothermic; a negative entropy also indicates enhanced disorganisation of the reactants and the formation of complexes.

$$CR. = \frac{RT}{Nh} \exp \frac{\Delta S}{R} \exp \left(\frac{-\Delta H}{RT} \right) \quad (5)$$

$$\Delta G = \Delta H - T\Delta S \quad (6)$$

The value of Gibbs' free energy was found to be negative, indicating the spontaneous nature of the system, and it increases with temperature; the adsorption rate decreases, while the desorption rate increases. This leads to reduced effectiveness of BDI on MS coupons.

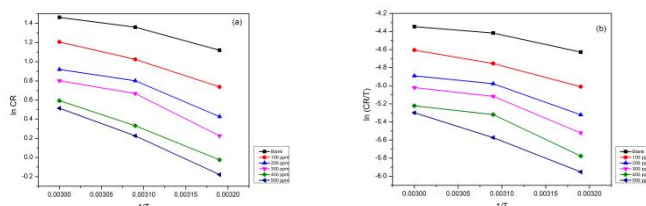


Fig.-2: MS Biocorrosion Using (a) Arrhenius Plot and (b) the Transition State of MS Coupons, respectively, without and with BDI Treatment

Adsorption Isotherm for Non-Inhibited and Inhibited Systems

A varying form of adsorption isotherm was used to predict the interaction between the synthesized inhibitor molecule and the MS surface, which gives the basic information relating to adsorption in corrosion studies. The adsorption isotherm that appropriately suited the data obtained was the Langmuir Isotherm, which relates the adsorbate of inhibitor (C) to surface coverage (θ) according to the following equation (7) and depicted by Fig.-3(A) and 7(B):

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (7)$$

With C as Concentration, θ as Surface coverage, K_{ads} as the adsorption isotherm equilibrium constant. The value of the linear regression coefficient (R^2) and slope were near unity, indicating that BDI follows the Langmuir adsorption isotherm, forming a monolayer of inhibitor, with the rate of adsorption equal to the rate of desorption.¹⁵ A thermodynamic parameter, such as standard Gibbs free energy (ΔG^0_{ads}), was given by equation (8):

$$\Delta G^0_{ads} = -RT \ln (K_{ads} 55.5) \quad (8)$$

C as Concentration, θ as Surface coverage, K_{ads} as adsorption isotherm equilibrium constant, ΔG^0_{ads} as the standard free energy, R as the universal constant, T as time, and 55.5 as the molar concentration of water in the solution. Table-4 shows the values for K_{ads} and ΔG^0_{ads} at varying time intervals, and Table-5 shows the values of K_{ads} and ΔG^0_{ads} , ΔH , and ΔS calculated for BDI w.r.t. higher temperature. The value of ΔG^0_{ads} gives an idea of the physisorption or chemisorption process occurring on the MS surface. The chemisorption process is associated with values near -40 kJ/mol, and the physisorption process is observed with values near -20 kJ/mol. The values obtained range from -40 kJ/mol to -20 kJ/mol for the

derivatives, indicating both physisorption and chemisorption, and the reaction is complex. In the present study, the ΔG^0_{ads} value is negative, indicating a spontaneous reaction on a metal surface.¹⁵

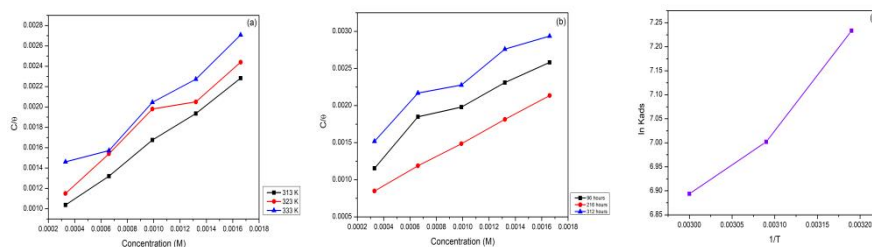


Fig.-3: Depicts the Langmuir Adsorption Isotherm Plot w.r.t (a) Temperature and (b) Time, (c) Shows the Van't Hoff Graph

Table-4: Thermodynamic Parameters of MS Specimen with Post-BDI Treatment w.r.t. Time (hours)

Incubation time (hours)	R ²	Slope	K _{ads}	−ΔG ⁰ _{ads} (kJ/mol)
96	0.939	0.998	1015.53	27.558
216	0.999	0.967	1860.56	29.083
312	0.945	1.031	763.99	26.841

Table-5: Thermodynamic-Kinetic Parameters of MS Specimen with Post-BDI Treatment w.r.t. Temperature (K)

Temperature (K)	R ²	Slope	K _{ads}	−ΔG ⁰ _{ads} (kJ/mol)	−ΔH (kJ/mol)	ΔS (J/mol)
313	0.998	0.935	1385.04	29.275	14.940	12.337
323	0.966	0.933	1098.90	29.589		
333	0.912	1.099	986.19	30.205		

Using the Van't Hoff Equation, the enthalpy and entropy for adsorption were calculated using equation (9). The adsorption enthalpy and entropy are given in Table-5. The negative H values indicate that the inhibitor adsorption on the metal surface is exothermic; also, the positive S values indicate that the adsorption process increases entropy.¹⁵

$$\ln K_{ads} = -\frac{\Delta H_{ads}^0}{R} + \frac{\Delta S_{ads}^0}{R} + \ln \frac{1}{55.5} \quad (9)$$

Electrochemical Analysis for Non-Inhibited and Inhibited MS Coupons with OCP and PdP

Steady-state potential was obtained using open-circuit potential for MS coupons without BDI treatment and at varying BDI concentrations from 100 to 500 ppm. The potential obtained from OCP illustrates that the potential shift is on the anodic part of the electrochemical reaction with respect to the MS coupon without BDI treatment. This anodic potential shift is due to the adsorption of BDI on metal coupons, which forms an interfering layer between the corrosive environment and the metal surface. Figure-4 (a) and (b), show the OCP and PdP plots without BDI and with BDI treatment.^{16,17}

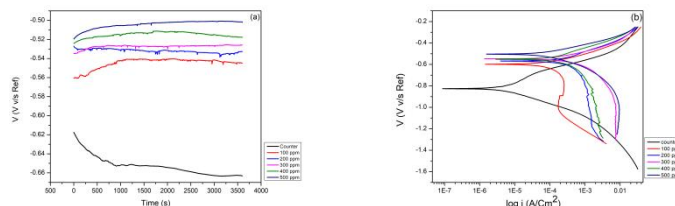


Fig.-4: Plot (a) OCP and (b) PdP without and with BDI Treatment

Table-6: PdP Parameters for MS Specimens Pre- and post-BDI Exposure

Concentration (ppm)	β _a 10 ⁻³ (v/dec)	B _c 10 ⁻³ (v/dec)	i _{corr} μA	−E _{corr} mv	CR (mpy)	I.E. (%)
0	252	360	326	809	149.326	--
100	232	211	201	808	91.802	38.34
200	84.1	162	151	524	69.34	53.68

300	93.4	155	121	537	55.554	62.88
400	72.5	84.3	85	501	38.927	73.92
500	107	38	66.4	523	30.416	79.63

Table-6 depicts the corrosion kinetic parameters like E_{corr} , I_{corr} , and Tafel slope values for anodic and cathodic reactions, and inhibition efficiencies of the synthesized BDI. The kinetics of cathodic and anodic processes were obtained from polarisation measurement studies. The decrease in β_a and β_c values with a gradual increase in the concentration of the BDI molecule was due to the BDI molecule getting adsorbed on the metal surface and protecting the MS from corrosion. An inhibitor of mixed type can affect both the dissolution of iron at an anodic reaction and the evolution of hydrogen at a cathodic site; that is, BDI is a mixed type of inhibitor affecting both the anodic and cathodic processes, which also predicts that the BDI molecules occupy the vacant d-orbitals of iron, thereby making it unavailable for corrosion attack and protecting it from biocorrosion. The rule of difference between the E_{corr} should be 85mV is not applicable, as the process of biocorrosion is a complex system.^{18,19}

Topographic Analysis using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Analysis (EDX)

The polished MS coupons were exposed to a bio-corrosive environment in the absence of BDI in Fig-5 (a), showing the growth of biofilm as well as the disturbance in mild steel coupons supporting biocorrosion. Figure-5 (c) shows the reduction in biofilm and reduced roughness on the MS coupon with BDI treatment.²⁰⁻²² Elemental changes on the corroded mild steel surface were analysed using EDX. Figure-5 (b) and (d) illustrate the effect of BDI on the MS coupon. Figure-5 (b) shows the EDX report for the corroded MS coupon with a sulphur peak indicating the biocorrosion product; on the other hand, Figure-5 (d) shows the reduced sulphur peak, indicating a reduction in biocorrosion product and protecting the metal surface from corrosion.²⁰⁻²²

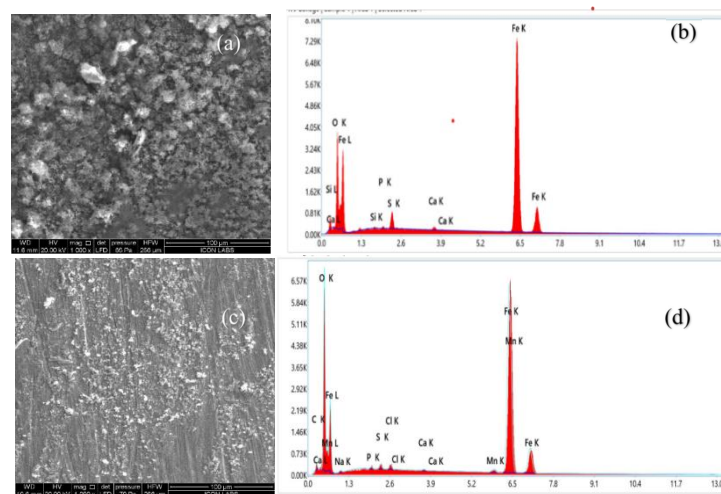


Fig.-5: SEM (a), (c) and EDX (b), (d) Images of Mild Steel Coupon with *Desulfovibrio desulfuricans* without Inhibitor Immersed in Barr's Media

CONCLUSION

3-(4-bromo-phenylimino)-1,3-dihydro-indol-2-one, was effective against the mild steel surface getting bio-corroded. Its efficiency was studied using gravimetric analysis by the weight loss method, with the highest efficiency at 303K after 216 hours of exposure, correlating the electrochemical analysis and the gravimetric analysis. The formation of the monolayer was interpreted using the Langmuir adsorption isotherm for the obtained data, and the negative $-\Delta G^0_{\text{ads}}$ values indicate spontaneous reaction. The topology of the mild steel surface was analysed using SEM and EDS, supporting the above results. The efficiency was contributed by the lone pair of electrons, the substituent -Br, which is an electron-withdrawing moiety, giving lower inhibition efficiency when compared to an electron-donating group.

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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:

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