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Colocid Dye - A Potential Corrosion Inhibitor for the Corrosion of Mild Steel in Acid Media

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Corrosion inhibition nature of Colocid dye on the corrosion of mild steel in acid media was analyzed using weightloss and electrochemical methods. The results of electrochemical and weightloss studies indicated that Colocid acid could act as a potential inhibitor for mild steel in acid media. It has been observed that the inhibition efficiency increases with inhibitor concentration and the inhibition efficiency of Colocid acid dye for the corrosion of mild steel in 1.0 M HCl solution is larger than that in 0.5 M H_2SO_4 solution.Potentiodynamic polarization studies showed that the dye molecule is of mixed type inhibitor. The adsorption of the compound on the mild steel surface obeyed Langmuir adsorption isotherm. The potential of zero charge was measured to find corrosion inhibition mechanism in these media.

Keywords: *Colocid acid dye, Corrosion inhibition, Electrochemical Impedance Spectroscopy, Adsorption isotherm, Potential of zero charge.*

1. Introduction

The reaction of metals with their environment causes corrosion. The corrosion behavior of metal is due to its natural tendency to combine with chemical elements in their environment to return to its lowest energy state. The phenomenon corrosion is always linked with an environment. The corrosion behavior of a material cannot be described unless the environment in which the material is to be exposed is identified. Similarly, the corrosivity or aggressiveness of an environment cannot be described unless the material that is to be exposed to that environment is identified[1]. The general relation existing among the rate of corrosion, the corrosivity of the environment, and the corrosion resistance of a material is[1]:

$$\frac{\text{corrosivity of the environment}}{\text{corrosion resis tan ce of the metal}} \simeq \text{rate of corrosive attack} \quad (1)$$

Mild steel is one of the most widely used material in many industries. Excessive corrosion attack is known to occur on mild steel surfaces deployed in service in aqueous acidic environments[2]. In industries acid solutions are often used for cleaning, descaling and pickling of steel structures[3]. Moreover, metal corrosion in acid medium is a serious environmental problem in the oil, fertilizer, metallurgical and other industries[4]. Hence, corrosion control becomes an essential issue from application point of view and it has been reported that inhibitors are needed to be used which act as a barrier to reduce the aggressiveness of the environments against the corrosion attack[5,6]. Organic compounds, which contain polar functional groups with nitrogen, oxygen, and/or sulfur in conjugated systems in their molecules, are generally used as corrosion inhibitors for the reduction of corrosion rate of metals in acid media[7-13]. Studies on corrosion inhibition performance of dyes have shown that organic dyes are quiet effective in retarding the corrosion of mild steel and aluminium in acid or base environments[14-24]. Hence, an attempt has been made to use an organic dye, as corrosion inhibitor for corrosion of mild steel in acid media and thus, the present study describes corrosion inhibition performance of an organic dye, Colocid acid (Figure 1), on corrosion of mild steel in acid media using weight loss, electrochemical impedance and potentiodynamic polarization methods. From the structure of this dye molecule it can be seen that it satisfies almost all the properties of a good corrosion inhibitor. It has large molecular surface area, many aromatic benzenoid rings through which the π electrons can interact with metal d orbitals, hetero atoms such as N, O and S - which are more electron pair donating atoms to the vacant d orbitals of surface Fe atoms. The corrosion inhibition mechanism of the dye is also explained using adsorption isotherm and potential of zero charge studies in acid media.

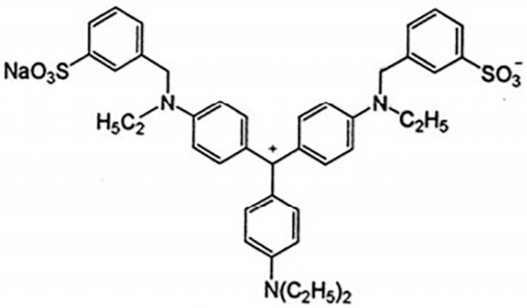

Figure 1. Structure of Colocid acid dye

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2. Materials and Methods

2.1 Materials

Mild steel specimens of dimensions 2.5 cmX1.0 cmX0.1 cm with the composition 0.084% C, 0.037% Mn, 0.026% P, 0.021% S and rest being Fe were used for weight loss studies. The specimens were polished well with the emery sheets of grade 1/0 to 5/0 and then washed thoroughly in double distilled water. Finally, the specimens were degreased with acetone and immersed in acid solutions after weighing. A Teflon coated cylindrical mild steel rod having the same composition of mild steel specimen with an exposed area of 0.1963 cm^2 was used for electrochemical studies. Colocid acid purchased from ZAUBA was used as such without further purification. All the acid solutions used for studies are of analytical grade and double distilled water was used for preparing all solutions.

2.2 Weight loss measurements

According to ASTM procedure weight loss studies were carried out[25]. Mild steel specimens in triplicate were immersed in 100 mL of the acid solutions with and without inhibitor for two hours and the average weight loss of three specimens were used to calculate the corrosion inhibition efficiency. The weight loss studies have been carried out at room temperature (300±1K). The corrosion inhibition efficiency was calculated using the expression;

$$IE\% = \frac{w_1 - w_2}{w_1} x100 \qquad (2)$$

Where, W_1 and W_2 are weight loss of mild steel specimens in the absence and presence of inhibitor respectively in the acid media.

2.3 Electrochemical impedance measurements

Electrochemical Impedance measurements were carried out using a potentiostat "GAMRY REFERENCE 3000". The impedance spectra were recorded using a three electrode set up, in which a Pt foil served as counter electrode and Saturated Calomel Electrode served as reference electrode. A Teflon coated cylindrical mild steel rod was used as working electrode. Before recording impedance spectra, a stabilization period of 45 minutes was given after immersing the working electrode in acid solutions, to attain the stationary corrosion potential. The impedance spectra were recorded at the AC sinusoidal potential of amplitude 10 mV in the frequency range 100 kHz - 100 mHz. The double layer capacitance and charge transfer resistance values were evaluated from the impedance spectra using standard procedures[26]. The corrosion inhibition efficiency was calculated using the expression;

$$IE\% = \frac{R'_{ct} - R_{ct}}{R'_{ct}} x100 \qquad (3)$$

Where R'_{ct} and R_{ct} are charge transfer resistance values in the presence and absence of inhibitor respectively in the acid media.

2.4 Potentiodynamic polarization measurements

Potentiodynamic polarization measurements had been carried out using the same cell setup employed in Electrochemical Impedance studies. The potentials were swept primarily from more negative potential than E_{OCP} (Open Circuit Potential) to more positive potential than E_{OCP} through equilibrium corrosion potential at a scan rate of 1.67 mV/s. The inhibition efficiency of the dye on the corrosion of mild steel in acid media was calculated employing the formula,

$$IE\% = \frac{i_{corr} - i'_{corr}}{i_{corr}} x100 \qquad (4)$$

Where, i_{corr} and i'_{corr} are corrosion current density values in the absence and presence of inhibitor respectively in the acid media.

2.5 Measurement of potential of zero charge

The electrochemical impedance spectra were recorded for blank and inhibited acid solutions at the fixed frequency (100 Hz) at different applied DC potentials. The double layer capacitance values so obtained are then plotted against applied DC potential to find potential of zero charge (PZC).

2.6 Scanning Electron Micrograph

Surface morphologies of corroded metal surfaces in the acid media in the presence and absence of inhibitor molecules were studied, using a digital Scanning Electron Microscope model SU6600 with an accelerating voltage of 22.0 kV, at a scan speed-Slow 5 and calibration scan speed of 25. All micrographs of the specimen were taken at a magnification of 500X. Before taking SEM images the mild steel specimen were immersed in acid solutions with and without inhibitor for two hours.

3. Results and Discussion

3.1 Results of weight loss measurement

The corrosion inhibition efficiency values of inhibitor in acid media,calculated from the weight loss data, are presented in Table 1. The corrosion inhibition efficiency increased with inhibitor concentration in the acid media. This implies that there is an interaction between inhibitor molecules and metal surface in acid media and led to adsorption of inhibitor onto the metal surface. The increase in inhibitor efficiency with inhibitor concentration can be attributed to the increase in adsorption with inhibitor concentration in the acid media. Adsorption of inhibitor molecules onto the metal surface separates the metal surface from corrosive media[27] and reduce corrosion rate.

Table 1. Corrosion inhibition Efficiency values calculated from the weight loss measurements

Medium	Inhibitor concentration (ppm)	Corrosion rate (mpy)	Inhibition efficiency (IE%)
1MHCl	Blank	169.0	-
	5	99.37	41.2
	10	89.40	47.1
	20	79.26	53.1
	50	65.40	61.3
	100	32.11	81.0
	200	26.87	84.1
	400	20.52	87.8
	600	18.08	89.3
0.5M H_2SO_4	Blank	522.7	-
	5	395.7	24.3
	10	226.8	56.6
	20	192.8	63.1
	50	167.7	67.9
	100	125.9	75.9
	200	91.47	82.5
	400	83.11	84.1
	600	68.47	86.9

3.2 Electrochemical impedance spectroscopic studies

The corrosion response of mild steel in the presence and absence of the inhibitor in 1.0M HCl and 0.5M H_2SO_4 solutions have been investigated using electrochemical impedance spectroscopy at 27 ± 1 ^oC and the corresponding Nyquist plots are shown in Figure 2.

The Nyquist plots obtained are simple semi circles, free from inductive loops. The simple - R(CR) - model best describes metal/solution interface. However the complex planes obtained are depressed semi circles, indicating a non-ideal capacity behavior of this interface[28,29]. This capacitance dispersion at the interfaces can be attributed to the surface roughness, the degree of polycrystallinity, the chemical inhomogeneities and anion adsorption[28,30]. A precise modeling for this situation is done by introducing a constant phase element (CPE) in the place of capacitor[28,30] and the corresponding equivalent circuit is shown in Figure 3.

The electrochemical impedance parameters obtained for the corrosion of mild steel in these two acid media in the presence and absence of the inhibitor by using the approximated equivalent circuits are shown in Table 2. From this table it is seen that with increasing inhibitor concentration both in 1.0M HCl and 0.5M H_2SO_4 acid media, the R_{ct} values increased while the values of C_{dl} decreases which can be calculated using the expression,

$$C_{dl} = Y_0 \omega^{n-1} \tag{5}$$

where 'ω' is the angular frequency at the maximum value of the imaginary part of impedance spectrum. The increase in the R_{ct} values with an increase in inhibitor concentration is the result of an increase in the surface coverage of the inhibitor molecules; the increased surface coverage led to an increase in inhibitor efficiency[29]. The decrease in C_{dl} values with the increase in inhibitor concentration is due to the adsorption of inhibitor molecules at the metal - solution interface with the replacement of water molecules, which led to the decrease in local dielectric constant and/or an increase in the thickness of electrical double layer[31]. Hence the decrease in C_{dl} values caused by the gradual displacement of H_2O molecules by the adsorption of inhibitor dye molecule at the metal-solution interface decreases the extent of the corrosion[32].

3.3 Potentiodynamic polarization studies

The potentiodynamic polarization curves obtained for mild steel in 1M HCl and 0.5M H_2SO_4 solutions in the absence and presence of inhibitor are shown in Figure 4a and 4b respectively.

From these figures it is clear that the addition of inhibitor to the corrosive media does not bring about significant changes in the cathodic and anodic slopes. The electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}) and cathodic and anodic tafel slopes are presented in Table 3.

From Figure 4 it is clear that the addition of inhibitor to the corrosive media shifts both anodic and cathodic curves to a greater extent towards lower current density values compared to blank acid solutions. In both the media the corrosion potential is also shifted to more negative with respect to uninhibited solution to smaller extent with the addition of inhibitor. This indicates the decrease in rate of hydrogen evolution on the mild steel surface due to the adsorption of dye molecules onto the metal surface[28,33]. The shift in corrosion potential is not much larger with the addition of inhibitor. All these indicate that the inhibitor is of mixed type. While scanning the potential in the negative direction (cathodic region) the metal surface is negatively charged and it could attract the positively charged inhibitor molecules. When the potential is scanned in the positive direction the metal surface is positively charged that could attract the negatively charged ions namely anions from acid molecules and this anion can act as a bridge between metal surface and protonated positively charged dye molecules.

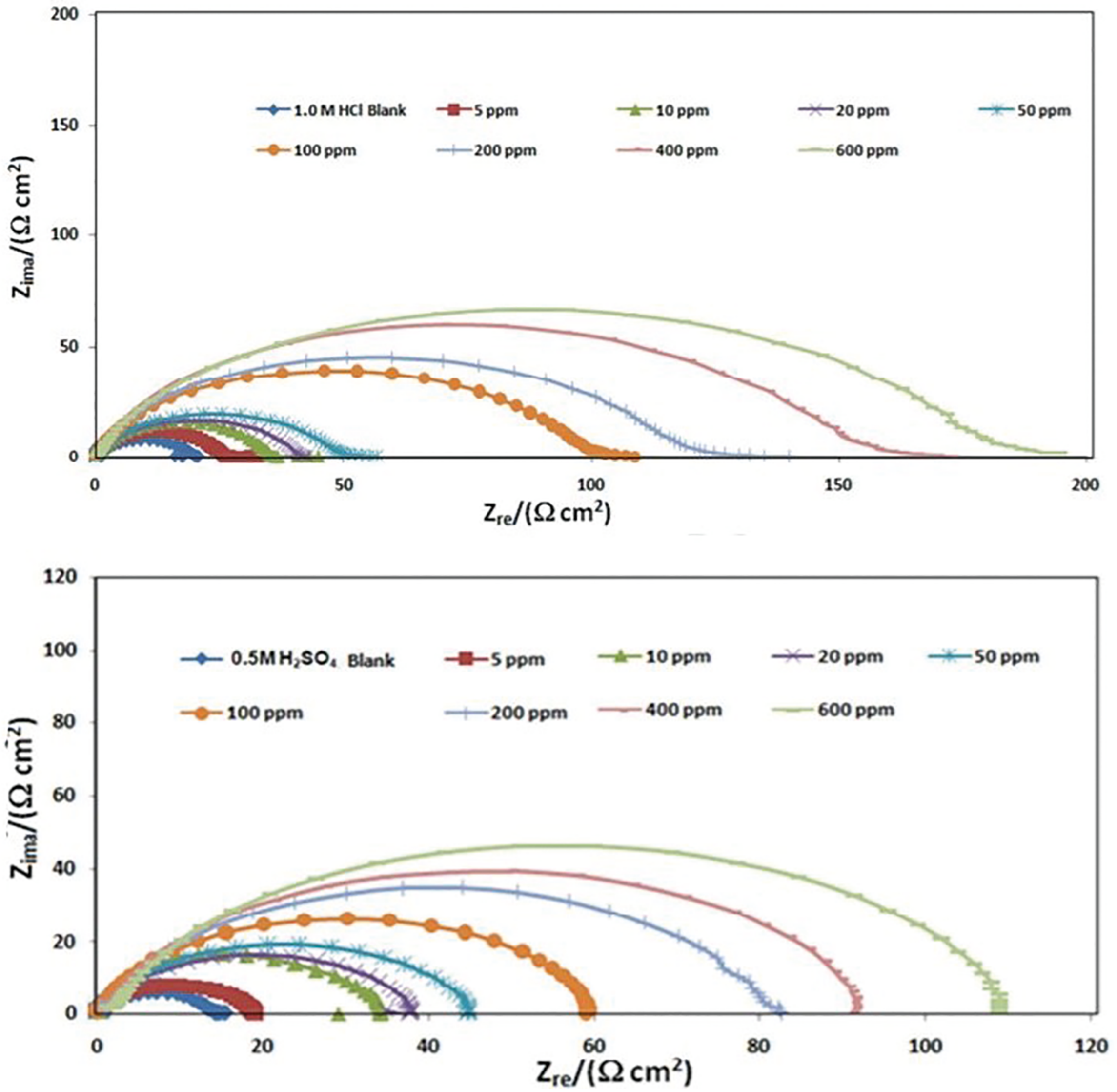

Figure 2. The Nyquist plots for MS electrode obtained in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the absence and presence of Colocid dye at various concentrations

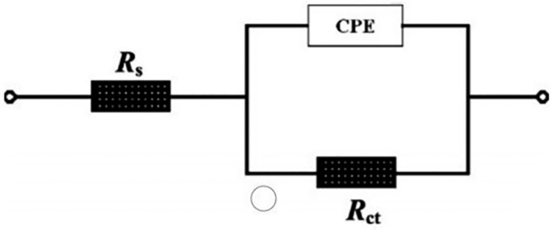

Figure 3. Equivalent circuit for the mild steel surface-corrosive media interface

Table 2. Electrochemical impedance parameters for MS in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the presence and absence of inhibitor.

Medium	Inhibitor concentration (ppm)	Rct Ωcm^2	$Y_0(x10^{-6})\ \Omega^{-1}cm^{-2}$	n	Inhibitor Efficiency
1.0 MHCl	Blank	18.8	262	0.970	-
	5	32.2	253	0.938	41.6
	10	35.7	205	0.940	47.3
	20	40.1	176	0.967	52.9
	50	48.8	169	0.968	61.5
	100	100	143	0.966	81.2
	200	120	127	0.974	84.4
	400	142	112	0.978	86.8
	600	189	101	0.969	90.1
0.5M H_2SO_4	Blank	14.6	145	0.902	-
	5	19.5	132	0.911	25.2
	10	34.2	114	0.920	57.3
	20	38.9	102	0.922	62.5
	50	43.8	95.1	0.923	66.7
	100	60.3	87.3	0.918	75.8
	200	83.4	72.4	0.965	82.5
	400	90.7	66.1	0.988	83.9
	600	110	51.8	0.976	86.7

3.4 Potential of zero charge and the inhibition mechanism

The dependence of double layer capacitance on the applied dc potential is graphically represented in Figure 5 in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the absence and presence of inhibitor. The values PZC and E_{OCP} (open circuit potential) for mild steel in the inhibited and uninhibited solutions of 1.0M HCl and 0.5M H_2SO_4 are shown in Table 4. The surface charge of the mild steel at the open circuit potential can be calculated using the equation,

$$E_r = E_{OCP} - E_{PZC} \qquad (6)$$

where E_r - Antropor's "rational" corrosion potential[34].

While measuring impedance or electrical properties of a system using EIS, the region of analysis from solution side to metal surface can be reached by decreasing frequency, i.e. lower the frequency closer the region to metal surface. Thus, the impedance measured at 100 Hz frequency, will correspond to the region of metal solution interface. The surface charge of the mild steel at OCP was found to be positive in the inhibited and uninhibited 1.0M HCl and 0.5M H_2SO_4 solutions with respect to PZC.

The hetero atoms such as N present in the inhibitor molecules can get protonated and will have positive charge. These positively charged inhibitor molecules can't directly approach the positively charged metal surface due to electrostatic repulsive forces. However, the anions (Cl^- and SO_4^{2-} ions) derived from the acids can directly get attached to the metal surface[28]. This indicates the adsorption of anions onto the metal surface by electrostatic attraction towards the metal surface to form first adsorption layer on the metal surface. The positively charged inhibitor molecules get attached to metal surface through the chloride and sulphate bridges.

3.5 Adsorption isotherm

The adsorption of dye molecules at the metal solution interface minimizes the corrosion rate and it is considered as a substitution adsorption process where an organic compound from the aqueous media displaces the water molecules associated with the surface (H_2O_{ads}).

$$Org_{sol} + xH_2O_{ads} \leftrightarrow Org_{ads} + xH_2O \qquad (7)$$

where 'x' is the number of water molecules replaced by the adsorption of one inhibitor molecule. The mechanism of adsorption of organic molecule on the metal surface in the corrosive media can be established using surface coverage and inhibitor efficiency.

In the present study, various adsorption isotherms were tested and it was found that the adsorption of inhibitor on the mild steel surface in acid media follows Langmuir adsorption isotherm (Figure 6), given by the expression[35]

$$C_{inh}/\theta = C_{inh} + 1/K_{ads} \qquad (8)$$

Where C_{inh} is the concentration of the inhibitor, θ is the surface coverage and K_{ads} is the equilibrium constant. The value of K_{ads} is determined from the plot of C_{inh} vs C_{inh}/θ at

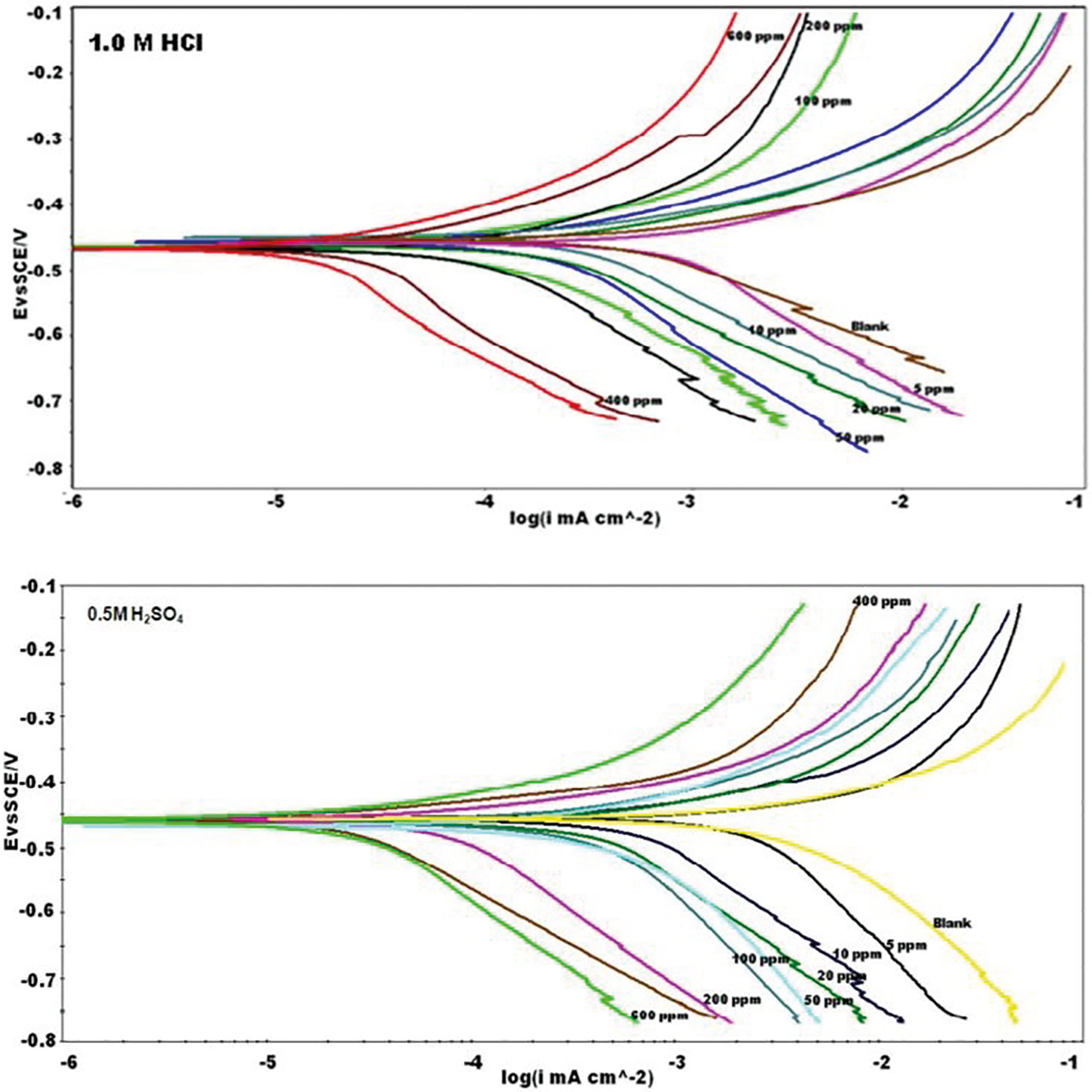

Figure 4. Potentiodynamic polarization plots recorded for MS electrode in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the absence and presence of Colocid dye at various concentrations

constant temperature, which is used to calculate the value of the standard free energy of adsorption using the following expression[28]

$$K_{ads} = 1/55.5 \exp(-\Delta G/RT) \quad (9)$$

The plots obtained were linear in both the media with the correlation coefficients greater than 0.99. The calculated values of ΔG_{ads} at room temperature in 1.0 M HCl and 0.5 M H_2SO_4 solutions are -27.73 kJ mol^{-1} and -27.98 kJ mol^{-1} respectively. The negative values of ΔG_{ads} show the spontaneous adsorption of dye molecules on the metal surface. In general the value of $-\Delta G_{ads}$ less than and around 20 $kJmol^{-1}$ represents physisorption, while the value greater than 40$kJmol^{-1}$ represents chemisorptions[28]. If the value of $-\Delta G_{ads}$ lies between 20 $kJmol^{-1}$ and 40 $kJmol^{-1}$, then it represents both physisorption and chemisorptions[28]. Thus in the present study the dye molecules get adsorbed on the metal surface by both by physisorption and chemisorption in the acid media studies.

3.6 Surface morphology - SEM Analysis

The adsorption of inhibitor molecules onto the metal surface greatly reduces the corrosive attack of the environment. Figure 7 and 8 show the surface morphologies of Iron metal in the presence and absence of inhibitor molecules in the corrosive media. From these images it can be seen that the corrosive nature of sulphuric acid is larger than that of hydrochloric acid. Presence of inhibitor greatly reduced the pits on the metal surface in both the acid environments.

Table 3. Electrochemical parameters for MS in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the presence and absence of inhibitor from potentiodynamic polarization curves.

Medium	Inhibitor Concentration (ppm)	E_{corr}(mV)	I_{corr}(mA cm^{-2})	β_c	β_a	Inhibitor efficiency (IE %)
1.0 M HCl	Blank	-452	1.21	192	141	-
	5	-449	0.716	`198	147	40.8
	10	-442	0.642	201	144	46.9
	20	-458	0.567	199	149	53.1
	50	-453	0.474	203	145	60.8
	100	-460	0.216	207	148	82.1
	200	-456	0.199	198	152	83.5
	400	-464	0.166	202	157	86.2
	600	-469	0.129	207	143	89.4
0.5 M H_2SO_4	Blank	-458	2.93	227	155	-
	5	-463	2.165	218	161	26.1
	10	-465	1.266	212	152	56.8
	20	-468	1.089	215	151	62.8
	50	-472	1.01	220	157	65.7
	100	-475	0.738	216	152	74.8
	200	-461	0.545	219	156	81.4
	400	-463	0.501	224	161	82.9
	600	-459	0.407	229	167	86.1

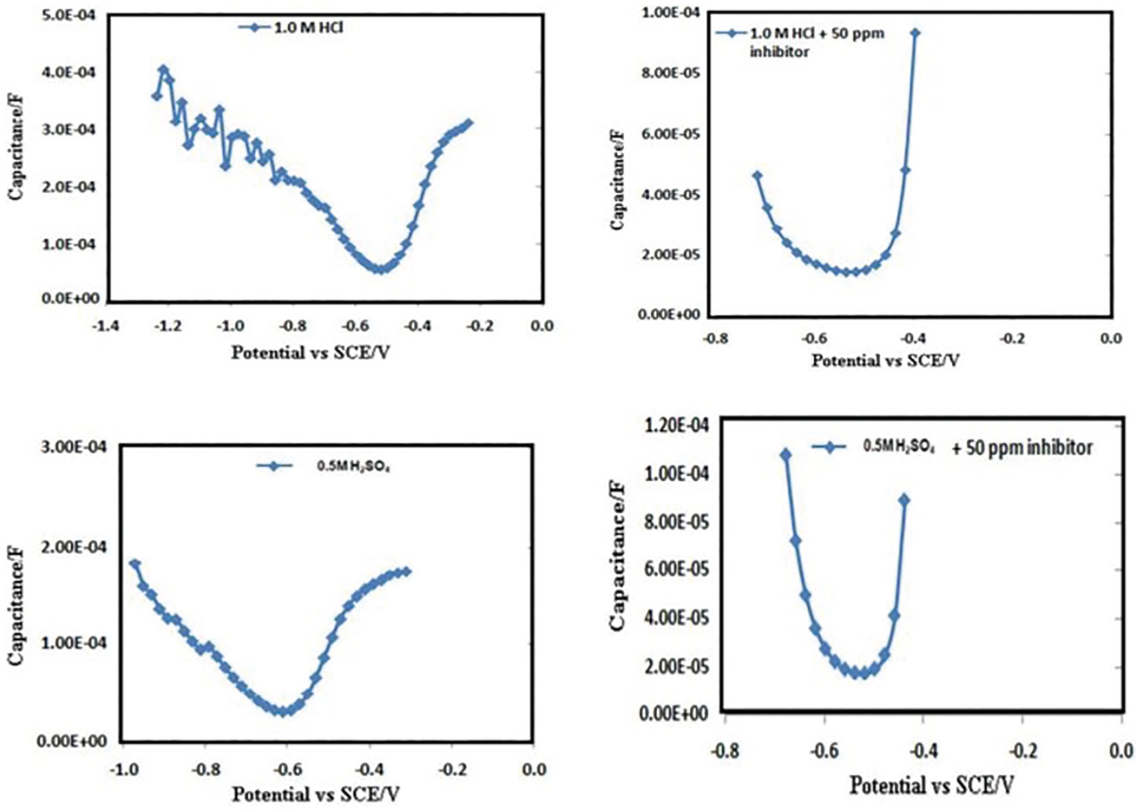

Figure 5. Plot of differential capacitance vs. applied electrode potential in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the absence and presence of Colocid dye

Table 4. Excess charge on MS electrode in 1.0 M HCl and 0.5 M H_2SO_4 solutions in the presence and absence of inhibitor.

Medium	E_{ocp}(mV)	PZC (mV)	Excess Charge
1.0 M HCl	-452	-470	+
1M HCl + 50 ppm of inhibitor	-462	-510	+
0.5M H_2SO_4	-458	-600	+
0.5MH_2SO_4+ 50 ppm of inhibitor	-460	-580	+

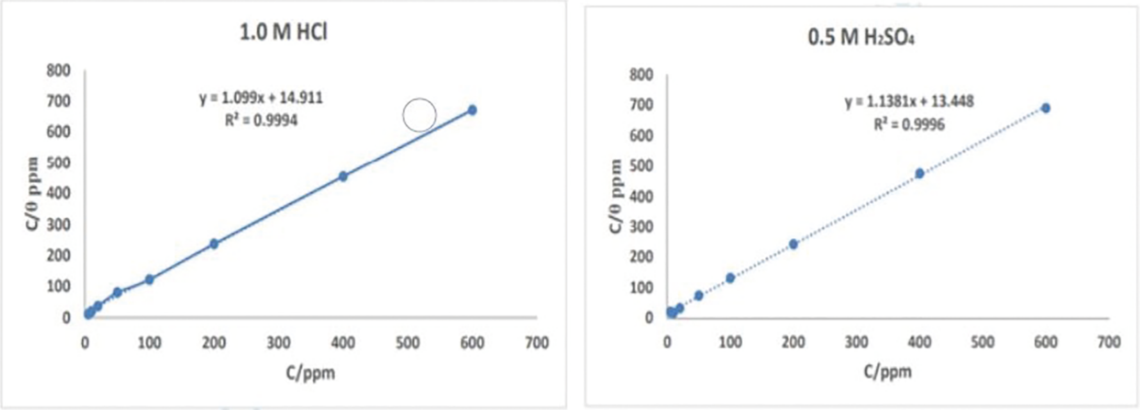

Figure 6. Langmuir adsorption isotherm for the adsorption of Colocid dye onto MS surface in 1.0 M HCl and 0.5 M H_2SO_4 solutions

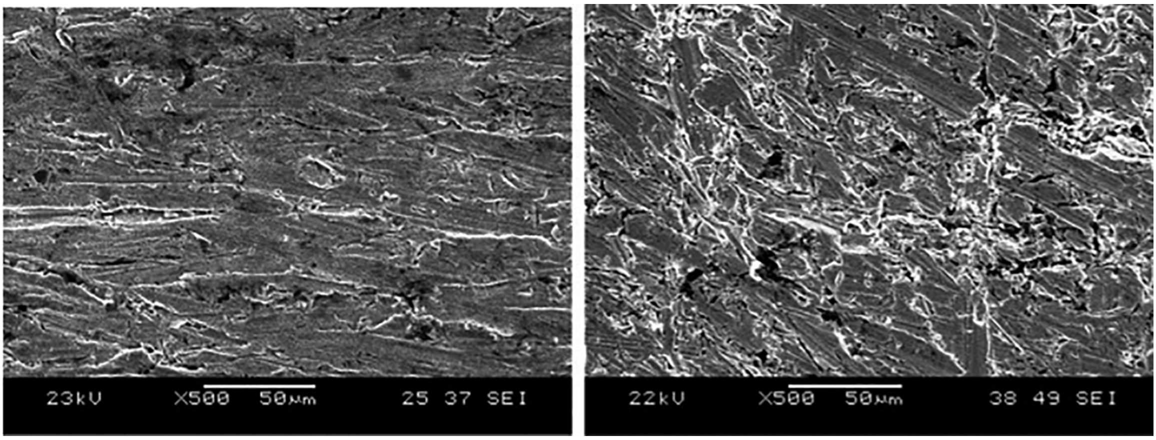

Figure 7. SEM Monographs of Iron specimen in 1.0 M HCl (7a) and 0.5 M H_2SO_4 (7b) solutions in the absence of inhibitor

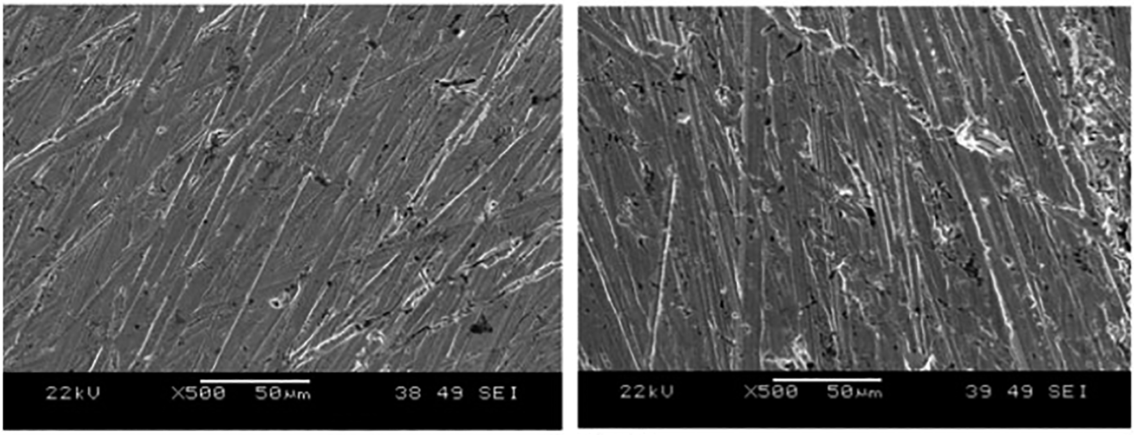

Figure.8a

Figure.8b

Figure 8. SEM Monographs of Iron specimen in 1.0 M HCl (8a) and 0.5 M H_2SO_4 (8b) in the presence of inhibitor caption

4. Conclusion

The corrosion inhibition performance of Colocid dye on the corrosion of mild steel was compared in two acid media (1.0 M HCl and 0.5 M H_2SO_4) using weight loss and electrochemical methods. The following are the important conclusions drawn from the present study.

i In both the acid media with increase in inhibitor concentrations, the inhibition efficiency increases.
ii The maximum inhibition efficiency was observed at 600 ppm concentration of inhibitor in both the acid media.
iii The dye molecules exhibited higher corrosion inhibition efficiency in 1.0M HCl solution compared to 0.5 M H_2SO_4 solution due to the higher bridging capacity of chloride ions than sulphate ions.
iv The potentiodynamic polarization curve indicates that the dye molecule acts as a mixed type inhibitor.
v The adsorption of dye molecules onto the metal surface follows Langmuir adsorption isotherm.
vi The negative values of ΔG indicated the spontaneous adsorption on the metal surface.
vii Studies on PZC and adsorption isotherm showed that the adsorption mechanism involves both physisorption and chemisorption in 1.0 M HCl and 0.5M H_2SO_4 solutions.

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