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Inhibition Effect of *Bombax ceiba* Flower Extract as Green Corrosion Inhibitor of Mild Steel in 0.5 M H_2SO_4 Medium

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The search for low cost, biodegradable and easily available green corrosion inhibitors against metal corrosion has incited the study of the inhibition potentiality and adsorption behaviour of *Bombax ceiba* flowers extract for mild steel in 0.5 M H_2SO_4 medium. Weight loss, potentiodynamic polarization and scanning electron microscopy techniques were employed for the analysis. Inhibition efficiency and various thermodynamic parameters such as activation energy (E_a), adsorptive equilibrium constant (K_{ads}) and free energy of adsorption (ΔG_{ads}) were obtained using weight loss measurements data. *Bombax ceiba* flower extract constituents inhibited the mild steel corrosion process by formation of protective layer on surface follows the Langmuir adsorption isotherm. The results of electro-chemical analysis directed the mixed-type behaviour of investigated inhibitor. Weight loss measurements showed the 90.84 % inhibition efficiency of *Bombax ceiba* flower extract at optimum concentration of inhibitor at 298 K. SEM analysis shows the remarkable upgraded surface of the mild steel strips with the inhibitor in 0.5 M H_2SO_4 medium.

Keywords: Adsorption, Mild steel, Corrosion inhibitor, *Bombax ceiba*.

INTRODUCTION

The metal corrosion recognized as a significant challenge of modern civilization. Hence the development of corrosion control techniques and measures remains a topic of important academic and industrial concern [1]. Additional costs required for the investigation, renovation and replacement of various equipment damage done by corrosion engenders. Corrosion cannot be completely stopped, but it can be significantly reduced using various approaches, such as blending production fluids, alloying, upgrading the metals, and using corrosion inhibitors [2]. In various industries metal cleaning process involving pickling, chemical cleaning, descaling and acid oil-well acidizing is one of the stimulating topic of current research, for the corrosion inhibition using environmental friendly inhibitors in various acidic media, containing H_2SO_4 or/and HCl [3,4]. Since these acidic solutions are aggressive in nature hence inhibitors are frequently used to prevent the corrosive attack of acids on metal surface. Various organic inhibitors have been synthesized and used to inhibit metal corrosion in acidic solutions. Some of these organic inhibitors are expensive, toxic, hazardous and harmful for human beings and environment as well [5-7]. The toxicity of organic inhibitors may manifest either during the synthesis of the organic molecules or when it used. This attract investigators to find the naturally occurring low-cost and non-hazardous corrosion inhibitors [8]. Plant products, such as, leaves, barks, seeds, fruits and their peels are a rich source of organic compounds. Thus, the use of natural sources of compounds is not only low-cost but also environmental friendly [9-11].The extract of leaves, flowers, bark and seeds have been widely reported as green corrosion inhibitors *e.g.*, *Aquilaria crassna* [12], *Alstonia angustifolia* var. *latifolia* [13], *Mentha rotundifolia* [14], Halfabar (*Cymbopogon proximus*), Chamomile (*Chamaemelum mixtum L.*), Kidney bean (*Phaseolus vulgaris* L.), black cumin (*Nigella sativa* L.) [15], *Telferia occide-ntalis*, *Occimum viridis*, *Hibiscus sabdariffa* and *Azadirachta indica* as well as seeds extracts of *Garcinia kola* [16].

In the present investigation, *Bombax ceiba* flower extract is used as corrosion inhibitor on mild steel in 0.5 M H_2SO_4 medium. *Bombax ceiba* is an important medicinal plant widely distributed in India and commonly known as silk cotton tree which belongs to family Bombacaceae. Leaves, bark, fruits and flowers of this plant is used as folk medicine in India [17]. It is used in tradition system of medicine and exhibits antiangiogenic activity [18], hypotensive, hypoglycaemic activity [19], antimicrobial and antibacterial activity [20], and diuretic activity [21]. Bombax ceiba flowers have been shown to contain the free β-sitosterol, β-D-glucoside of β-sitosterol, hentriacontanol, hentriacontane, traces of an essential oil, quercetin and kaempferol [22]. The objective of the present investigation is to utilize experimental techniques such as potentiodynamic polarization, weight loss and SEM in 0.5 M H_2SO_4 medium to gain insight into the adsorption mechanism of *Bombax ceiba* flower extract components on the mild steel surfaces.

EXPERIMENTAL

Preparation of materials and corrosive solutions: Mild steel specimens with compositions (wt. %) of C = 0.202, S = 0.045, O = 0.027, P = 0.039, Mn = 0.441, Cr = 0.031 and Fe = 98.96 % were used for the weight loss and electrochemical studies. The strips were scratched using different grit sizes emery papers. These specimens were washed carefully with double distilled water, degreased with acetone and dried at room temperature than kept in desiccator. The aggressive solution (0.5 M H_2SO_4) was prepared from AR grade concentrated acid (Merck) using double distilled water obtained from Elix essential 10 millipore water purifier. Concentration range 0.2-0.8 g L^{-1} of *Bombax ceiba* flower extract (BCFE) were used for each experiment.

Preparation of inhibitor solution: Inhibitor solutions were prepared as per standard procedure as described earlier [23].

Electrochemical measurements: Potentiodynamic polarization was performed using a conventional three electrode system consisting of platinum as the counter electrode, a mild steel working electrode (WE) with an exposed area of 1 cm^2, and a saturated calomel electrode (SCE) as the reference electrode. To establish a steady state open circuit potential (OCP) a 30 min period was allowed as a period of stabilization before performing all the electrochemical measurements. Gamry applications include EIS 300 for EIS measurements and software DC105 for corrosion and Echem Analyst version 5.50 software packages for data fitting. Potentiodynamic polarization analyses were performed in 0.5 M H_2SO_4 medium containing different concentrations of *Bombax ceiba* flower extract by changing the electrode potential automatically from −250 to +250 mV *vs*. OCP at a scan rate of 1 mV s^{-1}. Corrosion current density (i_{corr}) was determined from the intercept of extrapolated anodic and cathodic Tafel lines at the corrosion potential (E_{corr}). All the experiments have been conducted at room temperature.

Weight loss measurements: Weight loss measurements were performed on mild steel strips by immersing it in the inhibited and uninhibited solution of different concentrations of *Bombax ceiba* flower extract (BCFE) at different temperatures ranging from 298 to 328K for 4 h in 0.5 M H_2SO_4 medium. METTLER TOLEDO electronic balance (sensitivity up to 0.0001 g) was used for weighed the mild steel specimens. The corrosion rate (ρ) in mg cm^{-2} h^{-1} of inhibitor was determined using the following eqn [24]:

$$\rho = \frac{\Delta W}{At} \tag{1}$$

where, W is weight loss of mild steel specimen (mg), A is the total area of the specimen (cm^2) and t is the exposure time (h). Inhibition efficiency (% IE) of *Bombax ceiba* flower extract (BCFE) was calculated using following the eqn. [24]:

$$\text{Inhibition efficiency (IE, \%)} = \left(\frac{\rho^1 - \rho^2}{\rho^1}\right) \times 100 \tag{2}$$

where ρ^1 and ρ^2 are the corrosion rates of the mild steel strips in the absence and presence of inhibitor, respectively.

Surface analysis: The mild steel strips were immersed in 100 mL of 0.5 M H_2SO_4 solution in absence and presence of optimum concentration of *Bombax ceiba* flower extract for 4 h. After 4 h, the strips were removed and washed with double distilled water, dried in hot air oven for few minutes. These strips were examined for their surface morphology by using scanning electron microscope (SEM) (ZEISS EVO SEM 18 model Oxford).

RESULTS AND DISCUSSION

Potentiodynamic polarization measurements: Fig. 1 represents the potentiodynamic polarization curves (cathodic and anodic) for mild steel in 0.5 M H_2SO_4 medium at room temperature in absence and presence of various concentrations of *Bombax ceiba* flower extract and corresponding parameters derived from the curves are given in Table-1.The curves showed that the addition of inhibitor to the blank solution has modest influence on both cathodic and anodic half reactions, although anodic influence appeared much pronounced. The minor shift in E_{corr} value (15 mV) in inhibited solution as compared to E_{corr} value in uninhibited solution, indicating that *Bombax ceiba* flower extract acts as mixed type inhibitor [25]. It is also clear from Table-1 that β_a and β_c values for the different concentrations of inhibitor increased slightly compared to uninhibited solution and are nearly same indicating that inhibitor molecules simultaneously modified both cathodic and anodic reactions. This results also support the mixed-type nature of inhibitor. The corrosion inhibition efficiency of *Bombax ceiba* flower extracts was calculated using the following eqn. [26]:

$$\eta_p\,(\%) = \frac{i_{corr} - i_{corr\,(inh)}}{i_{corr}} \times 100 \tag{3}$$

where, $i_{corr\,(inh)}$ and i_{corr} are the corrosion current density values of mild steel with and without inhibitors, respectively.

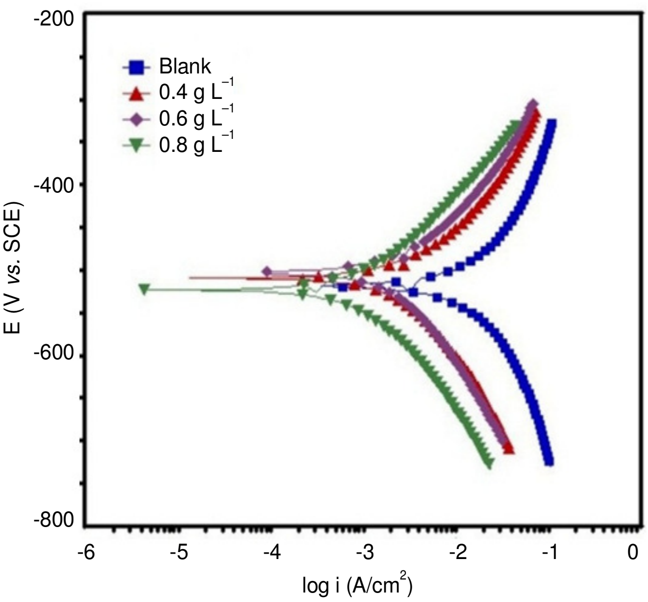

Fig. 1. Tafel polarization behaviour of mild steel in 0.5 M H_2SO_4 medium with different concentrations of inhibitor at room temperature

TABLE-1
DATA FROM THE POTENTIODYNAMIC POLARIZATION ANALYSIS FOR MILD STEEL IN 0.5 M H_2SO_4 WITH VARIOUS CONCENTRATION OF INHIBITOR AT ROOM TEMPERATURE

Inhibitor	Concentration of inhibitor (g L^{-1})	Tafel polarization parameters					
		i_{corr} (mA/cm^2)	E_{corr} (mV/SCE)	β_a (mV/dec)	β_c (mV/dec)	θ	η (%)
Bombax ceiba flower extracts	Blank	28.18	-517	257.9	304.2	-	-
	0.4	6.377	-510	110.8	191.3	0.773	77.37
	0.6	3.281	-504	124.4	222.5	0.883	88.35
	0.8	1.044	-522	106.7	130.5	0.962	96.29

Weight loss measurements: The effect of *Bombax ceiba* flower extracts towards the mild steel corrosion inhibition in 0.5 M H_2SO_4 corrosive solution was followed using weight loss measurements at various concentrations of inhibitor at studied temperatures. The results obtained from weight loss measurements are presented in Table-2. It shows that the corrosion rates of mild steel increase in inhibited and uninhibited solutions of inhibitor as temperature increases. A remarkable decrease in mild steel corrosion rate and increase in inhibition efficiency were obtained in the presence of *Bombax ceiba* flower extract (BCFE) in compared with blank solution. Also, inhibition efficiency increases with increase in the concentration of plant extract but decreases with increasing temperature both in the presence and absence of BCFE. It is apparent from Table-2 that the corrosion rates of mild steel is decreased from 1.147 to 0.105 mg cm^{-2} h^{-1} (298 K) and 5.891 to 1.689 mg cm^{-2} h^{-1} (328 K) on the addition of optimum concentration of BCFE. Also optimum inhibition efficiency of 90.84 % was obtained at 0.8 g L^{-1} concentration of inhibitor at 298 K. This reveals that BCFE is good inhibitor of mild steel corrosion in 0.5 M H_2SO_4 medium.

Adsorption isotherm: The adsorption behaviour of inhibitor molecules on mild steel surfaces provides some insight into their inhibition mechanism. The degree of surface coverage (θ) calculated from the weight loss technique, is related to the inhibitor concentration (C). Some adsorption isotherms were tested in order to determine the adsorption isotherm and the best fit was obtained with the Langmuir adsorption isotherm following the relation given in eqn. 4 [27]:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \tag{4}$$

where C is the inhibitor concentration and K_{ads} is the adsorptive equilibrium constant. K_{ads} values indicates the binding ability of the inhibitor molecules to metal surfaces. Fig. 2 represents the relationship between C/θ and C at studied temperatures. K_{ads} can be calculated from the intercept on the C/θ axis according to eqn. 4. With the help of K_{ads} values, the Gibbs free energy of adsorption (ΔG_{ads}) can be calculated by using the eqn. 5 [24]:

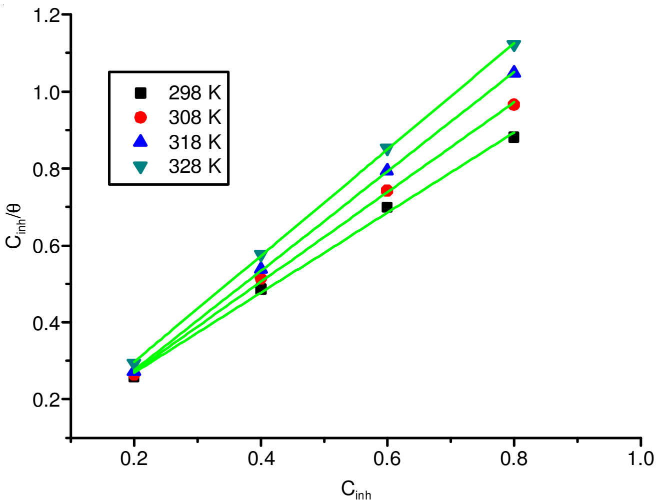

Fig. 2. Linear fitting of *Bombax ceiba* flower extract to Langmuir adsorption isotherm at studied temperatures at various inhibitor concentrations

$$\Delta G_{ads} = -RT \ln (55.5\ K_{ads}) \tag{5}$$

where R = universal gas constant, 55.5 = water concentration and T = absolute temperature. The average value of free energy of adsorption (ΔG_{ads}) was found to be -19.55 kJ mol^{-1}, which indicates that the inhibitor molecules spontaneously adsorbed onto the metal surface in 0.5 M H_2SO_4 medium. Several studies suggested that values of ΔG_{ads} around -40 kJ mol^{-1} directed the chemisorption phenomenon involves dative bond while those ΔG_{ads} around -20 kJ mol^{-1} or lower are associated with the physisorption which involves the electrostatic interaction between the inhibitor molecules and metal [27]. ΔG_{ads} values are given in units of kJ mol^{-1} because the molecular masses of the extract constituents are unknown [28]. Table-3 reveals that the values of ΔG_{ads} lie between -16.89 to -21.42 kJ mol^{-1} (< 20 kJ mol^{-1}) indicating the physisorption nature of studied inhibitor on the metal surfaces in acidic solution.

SEM: The SEM microphotographs of mild steel surface in 0.5 M H_2SO_4 medium are shown in Fig. 3. Fig. 3a represents the some abrading scratches and smooth surface before immersion in aggressive solution. The SEM image (Fig. 3b) revealed the

TABLE-2
CORROSION PARAMETERS OBTAINED FROM WEIGHT LOSS TECHNIQUE FOR MILD STEEL IN 0.5 M H_2SO_4 CONTAINING DIFFERENT CONCENTRATIONS OF *Bombax ceiba* FLOWER EXTRACTS

Inhibitor	Conc. (g L^{-1})	Corrosion rate ρ, (mg cm^{-2} h^{-1})				Inhibition Efficiency (IE, %)			
		298 K	308 K	318 K	328 K	298 K	308 K	318 K	328 K
Bombax ceiba flower extracts	0.0	1.147	2.196	4.511	5.891	–	–	–	–
	0.2	0.259	0.541	1.198	1.887	77.41	75.36	73.44	67.96
	0.4	0.202	0.486	1.167	1.805	82.38	77.86	74.12	69.36
	0.6	0.164	0.423	1.104	1.738	85.70	80.73	75.52	70.49
	0.8	0.105	0.377	1.065	1.689	90.84	82.83	76.39	71.32

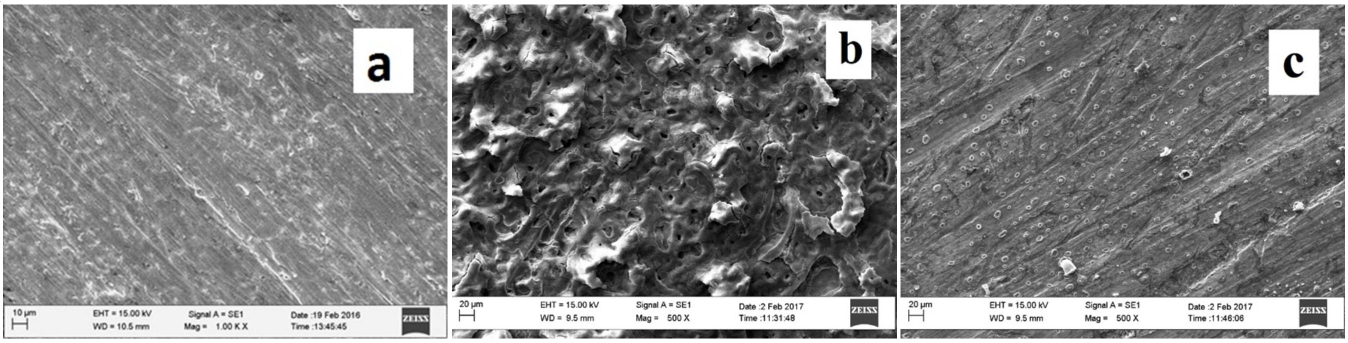

Fig. 3. SEM images of mild steel (a) mild steel specimen (b) mild steel immersed in 0.5 M H_2SO_4 and mild steel immersed in 0.5 M H_2SO_4 in the presence of *Bombax ceiba* flower extract

TABLE-3
THERMODYNAMIC PARAMETERS FROM THE LANGMUIR ADSORPTION ISOTHERM FOR *Bombax ceiba* FLOWER EXTRACTS AT VARIOUS TEMPERATURES

Inhibitor	Temp. (K)	Slope	R^2	K_{ads} (L g^{-1})	ΔG_{ads} (kJ mol^{-1})
Bombax ceiba flower extracts	298	1.045	0.996	16.52	-16.89
	308	1.166	0.999	25.64	-18.59
	318	1.291	0.999	57.14	-21.31
	328	1.379	0.999	46.51	-21.42

highly damaged and rough surface in the absence of inhibitor, while Fig. 3c shows the uniform and reduced rough surface due to formation of protective layer that creates a barrier for mass and charge transfer and there is slight evidence of adsorbate presence on the mild steel surface.

Conclusion

The present investigation shows that *Bombax ceiba* flowers extract (BCFE) is a good corrosion inhibitor for mild steel in 0.5 M H_2SO_4 medium. Corrosion rate decreases and inhibition efficiency increases with the inhibitor concentration. The extract inhibits mild steel corrosion by being adsorbed on the mild steel surface and adsorption behaviour of the inhibitor is consistent with the Langmuir adsorption model. Potentiodynamic polarization study revealed that *Bombax ceiba* flowers extract (BCFE) acts as a mixed type inhibitor for mild steel corrosion. SEM analysis further confirmed the inhibitive character of the extract of *bombax ceiba* flowers in 0.5 M H_2SO_4 medium.

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