



## **Inhibitive action of Bombax Malabricum leaves extract on the Corrosion of Mild steel in 1N HCl Medium**

**P. R. Sivakumar, K. Vishalakshi and A. P. Srikanth\***

\*PG & Research Department of Chemistry, Government Arts College, Coimbatore, TN, **INDIA**

Email: [apsrikanth8@gmail.com](mailto:apsrikanth8@gmail.com)

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### **ABSTRACT**

*The inhibition of the corrosion of mild steel in hydrochloric acid solution by the leaves Extract of Bombax Malabricum (BM) has been studied using weight loss, electrochemical impedance spectroscopy and Potentiodynamic polarization techniques. Inhibition was found to increase with increasing concentration of the extract. Results indicate that Bombax Malabricum leaves Extract was an efficient natural corrosion inhibitor in the acidic solution. Polarization measurements showed that the studied inhibitor acts as mixed type inhibitor in 1N HCl acid with significant reduction of cathodic and anodic current densities. The electrochemical impedance study further confirmed the formation of an adsorbed film on the mild steel. The SEM morphology of the absorbed protective film on the mild steel surface has confirmed the high performance of inhibitive effect of the plant extract.*

**Keywords:** Bombax Malabricum, Mild Steel, Corrosion Inhibition, SEM.

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### **INTRODUCTION**

Acid solutions are generally used for the removal of rust and scale in several industrial processes. Hydrochloric acid is often used as a pickling acid for steel and its alloys [1]. Mild steel is employed widely in most industries due to its low cost and availability in ease for the fabrication of various reaction vessels such as cooling tower tanks, pipelines etc [2]. Inhibitor are substances which retards the cathodic processes and / or the anodic processes, that inhibitors function in one or more ways to control corrosion: by adsorption of a thin film onto the surface of a corroding material, by inducing the formation of a thick corrosion product, or changing the characteristic of the environment resulting in reduced aggressiveness. Inhibitors are generally used in these processes to control the metal dissolution. Acid inhibitors are essentially used in metal finishing industries, acidizing of oil wells, cleaning of boilers and heat exchangers [3].

Corrosion of metal is a serious environmental problem that has been given adequate attention in the oil and gas industries because, during industrial processes such as acid cleaning and etching, metal surface are often made to come in contact with acidic medium, indicating that the use of inhibitors is necessary. Although there are numerous options for controlling the corrosion of metal, the use of inhibitors is one of the best methods of protecting metals against corrosion. An inhibitor can be chosen from compounds that

have hetero atoms in their aromatic ring systems or synthesized from cheap raw material. However, the problem of finding an inhibitor that has little or no impact on the environment has attracted numerous researchers in recent times. Green or safe corrosion inhibitors are biodegradable and do not contain heavy metal or other toxic compounds [4- 8]. Most green corrosion inhibitors are obtained from ethanol, aqueous, acid, methanol, or formaldehyde extract of plants materials. The successful use of naturally occurring substance to inhibit the corrosion of metal in acidic and alkaline environments has been reported by some research groups [9-26]. However, the use of aqueous extract of BM plants as a green inhibitor has not been reported elsewhere. Therefore the objective of the present study is aimed at investigating inhibitive and adsorption properties of aqueous extracts of BM leaves for the corrosion of mild steel in 1 N HCl solution.

## MATERIALS AND METHODS

**Preparation of mild steel specimen:** Mild steel strips were mechanically cut into strips of size 4 cm x 2 cm x 0.1cm containing the composition of C- 0.030 %, Mn- 0.169 %, Si- 0.015 %, P- 0.031 %, S - 0.029 %, Cr- 0.029 %, Ni- 0.030 %, Mb- 0.016 %, Cu- 0.017 % and the remainder Fe and provided with a hole of uniform diameter to facilitate suspension of the strips in the test solution for weight loss method. For electrochemical studies, mild steel strips of the same composition but with an exposed area of 1cm<sup>2</sup> were used. Mild steel strips were polished by using emery paper of 400, 600, 800, 1000 and 1200 grade, subsequently degreased with acetone and finally washed with deionized water, and stored in the desiccator. Accurate weight of the metal was taken using four digital electronic balances (Shimadzu ay 220).

**Preparation of the plant extract:** The leaves of the medicinal plants BM were taken and cut into small pieces, and dried in room temperature and ground well into powder. 10g of the powder was refluxed in 150 mL distilled water and kept overnight. The refluxed solution was then filtered carefully, the filtrate volume was made up to 250 mL using double distilled water which was the stock solution, and the concentration of the stock solution was expressed in terms of ppm. From the stock solution, 5-20 ppm concentration of the extract was prepared using double distilled water. Similar kind of preparation has been reported in studies using aqueous plant extract in the recent years [27].

**Weight loss method:** Mild steel specimens were immersed in 200 ml of 1N HCl solution without and with various concentrations of the inhibitors using glass hooks and rods for a predetermined time period (24 h) at room temperature. The weights of the specimens before and after immersion were determined using four digit electronic balance (Shimadzu ay220 model). From the weight loss measurements, the corrosion rate was calculated using the following relationship.

$$CR \text{ (mmpy)} = \frac{K \times \text{Weight Loss}}{D \times A \times t \text{ (in hours)}} \quad (1)$$

Where, K = 8.76 x 10<sup>4</sup> (constant), D is density in gm/cm<sup>3</sup> (7.86), W is weight loss in grams and A is area in cm<sup>2</sup>.

The inhibition efficiency (%) was calculated using equation (2)

$$IE \% = \frac{W_0 - W_i}{W_0} \times 100 \quad (2)$$

Where, W<sub>0</sub> and W<sub>i</sub> are the weight loss in the absence and presence of the inhibitor respectively.

**FTIR measurements:** FTIR spectra were recorded in a Bruker ALPHA 8400 S spectrophotometer. The film was carefully removed, mixed thoroughly with KBr made into pellets and FTIR spectra were recorded.

**Potentiodynamic polarization methods:** Potentiodynamic polarization measurements were carried out using CHI660E electrochemical analyzer. The polarization measurements were made to evaluate the corrosion current, corrosion potential from Tafel slope. Experiment were carried out in a conventional three electrode cell assembly with mild steel specimen of 1cm<sup>2</sup> area which was exposed and the rest being

covered with red lacquer, a rectangular was used as working electrode, a rectangular Pt foil as the counter electrode and a saturated calomel electrode as reference electrode. A time interval of 15 minutes was given for each experiment to attain the steady state open circuit potential. The polarization was carried from a cathodic potential of -800 mV (vs SCE) to an anodic potential of -200 mV (vs SCE) at a sweep rate of 1 mV per second. From the polarization curves, Tafel slopes, corrosion potential, and corrosion current were calculated. The inhibitor efficiency was calculated using the formula:

$$IE \% = \frac{I_{\text{Corr}} - I_{*\text{Corr}}}{I_{\text{Corr}}} \times 100 \quad (3)$$

Where  $I_{\text{corr}}$  and  $I_{*\text{corr}}$  are corrosion current in the absence and presence of inhibitors.

**Electrochemical impedance method:** The electrochemical AC-Impedance measurements were also performed using CHI660E electrochemical analyzer. Experiments were carried out in a conventional three electrode cell assembly as that used for potentiodynamic polarization studies. A sine wave with amplitude of 10 mV was superimposed on the steady state open circuit potential. The real part ( $Z'$ ) and the imaginary part ( $Z''$ ) were measured at various frequencies in the range of 100 KHz to 10 MHz. A plot of  $Z'$  versus  $Z''$  was made. From the plot, the charge transfer resistance ( $R_{\text{ct}}$ ) was calculated, and the double layer capacitance ( $C_{\text{dl}}$ ) was then calculated using formula:

$$C_{\text{dl}} = \frac{1}{2\pi} f_{\text{max}} R_{\text{ct}} \quad (4)$$

Where  $R_{\text{ct}}$  is charge transfer resistance, and  $C_{\text{dl}}$  is double layer capacitance.

$$IE\% = \frac{R_{\text{ct}} - R_{\text{ct}}^0}{R_{\text{ct}}} \times 100 \quad (5)$$

Where,  $R_{\text{ct}}$  and  $R_{\text{ct}}^0$  are the charge transfer resistance values in the inhibited and uninhibited solution respectively.

**Phytochemical analysis:** Phytochemical screening were performed to assess the qualitative chemical composition of the different samples of plants extract using commonly employed precipitation and coloration reactions to identify the major secondary metabolites like alkaloids, flavonoids, glycosides, proteins, phenolic compounds, saponins, starch, steroids, tannins and terpenoids.

**Effect of temperature:** The polished and pre – weighed specimens were suspended in 100 mL of the test solution without and with the addition of various concentration of the flower extract for 1h in the temperature range of 303 – 323K using water thermostats. The specimens were removed from the test solution after 3 h and washed with distilled water, dried and weighed. The inhibition efficiency was calculated from the weight loss.

**Scanning electron microscopy:** The mild steel specimen immersed in blank and in the inhibitor solution for a period of one day was removed, rinsed with double distilled water, dried and observed in a scanning electron microscope to examine the surface morphology. The surface morphology measurements of mild steel were examined using (JEOL) computer controlled scanning electron microscope.

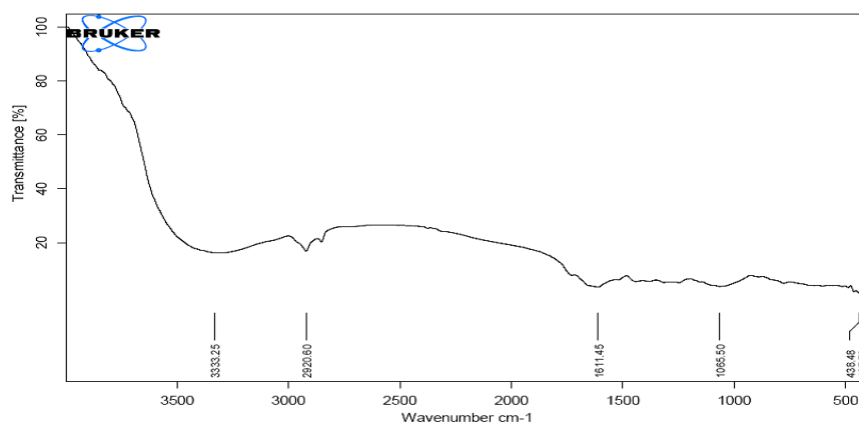
## RESULTS AND DISCUSSION

**Weight loss methods:** The weight loss studies were done in 1N hydrochloric acid in the absence and presence of various concentration of the plant extracts ranging from 5 to 20 ppm. Using the weight loss data, corrosion rate, inhibition efficiency, and the optimum concentration of the extract have been calculated. The corrosion parameters obtained in the weight loss method are given in table 1. It was observed from the table that the rise in concentration of BM leave extract, on the corrosion rate of mild steel in 1N HCl solution was decreased and the inhibition efficiency increased from 88.04 % to 96.09% up to 20 ppm. It indicates that 20 ppm is the optimum concentration to get maximum corrosion protection for mild steel in 1N HCl solution using BM leaves extract.

**Table 1** Data from Weight Loss Method for MS corroding in 1 N HCl solutions at various concentrations of BM leaves extract.

Conc. (ppm)	Weight loss (g)	Corrosion rate (mmpy)	Inhibition Efficiency (%)
Blank	0.3557	206.47	*
5	0.0455	026.41	88.04
10	0.0380	022.57	90.01
15	0.0346	020.00	90.91
20	0.0134	007.77	96.48

**FTIR measurement:** FTIR spectrum of BM leaves extract was shown in fig.2. Original absorption band at  $3333\text{ cm}^{-1}$  (associated hydroxyl) was overlapped by the broad stretching mode of N-H. The  $2920\text{ cm}^{-1}$  band is stretching mode of  $\text{CH}_3$ . The peak at  $1611\text{ cm}^{-1}$  could be assigned to stretching mode of C=O. The bands at  $1065\text{ cm}^{-1}$  was due to the symmetric stretching mode of C-O. This showed that the extracts contained mixture of compounds, ie alkaloids, phenol, and tannins compounds. The peak at  $1096.64\text{ cm}^{-1}$  is due to ring oxygen atom. This conform the presence of mild steel BM leaves extract complex on the metal surface. Mild steel has co-ordinated with the O – atom of the OH group, -C = O group and the ring oxygen atom. On the basis of the result, it can be said that BM leaves extract contain Nitrogen and Oxygen (N-H, C=N, C-N, O-H, C=O, C-O) in various functional group and aromatic ring , which make this extract attractive for being used as inhibitor. This result confirmed the adsorption of inhibitor on the surface of the mild steel [28].

**Fig. 1:** FTIR Spectrum of BM leaves extract

**Potentiodynamic polarization methods:** Potentiodynamic polarization curves obtained for mild steel in 1N HCl solution at various concentration of BM extract ranging from 5ppm to 20 ppm are shown in fig.2 and the data obtained are given in table 2. It can be observed that the addition of BM extract at all the studied concentration decreased the anodic and cathodic current densities and resulted in significant decline in the  $I_{\text{corr}}$ . This indicates that BM extracts shifted to smaller  $I_{\text{corr}}$  values in both anodic and cathodic branches of the curve, thus, acting as a mixed type inhibitor [29-30] and the decrease is more pronounced with the increase in the inhibitor concentration. By comparing polarization curves in the absence and in the presence of various concentrations of BM extracts, it was observed that, increase in concentration of the inhibitor shift the corrosion potential( $E_{\text{corr}}$ ) in the positive direction and reduces both anodic and cathodic process.

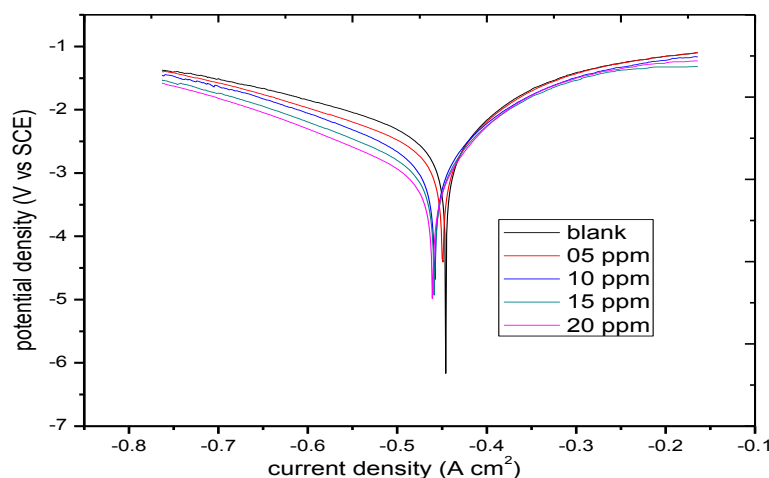


Fig.2 Potentiodynamic polarization (Tafel) curves for mild steel in 1N HCl solution in the absence and presence of different concentration of BM extracts.

Table 2. Electrochemical parameter from polarization measurement and calculated values of inhibition efficiency of BM leaves extracts.

Conc.of BM extract (ppm)	$E_{corr}/$ (mV/ SCE)	$I_{corr}/$ (mA/cm <sup>2</sup> )	$b_c$ (mV/dec.)	$b_a$ (mV/dec.)	LPR Ohm*cm <sup>2</sup>	IE (%)
Blank	-0.446	$3.781 \times 10^{-3}$	204	133	9.2	*
5	-0.449	$2.857 \times 10^{-3}$	194	125	11.6	9.44
10	-0.458	$1.947 \times 10^{-3}$	173	120	15.8	48.50
15	-0.459	$1.506 \times 10^{-3}$	171	117	20.1	60.16
20	-0.461	$1.107 \times 10^{-3}$	167	107	25.7	70.72

**Electrochemical impedance methods:** Fig.3. shows the effect of the inhibitors concentration on the impedance behaviour of mild steel in 1N HCl solution and the results are given in table 3. It was observed from the figure that the impedance spectra showed a single semicircle and as the concentration of inhibitor increases diameter of the semicircle increases. It was evident from the results that BM extracts inhibited the corrosion of mild steel in 1N HCl at all concentration used, and the inhibition efficiency increased continuously with increasing concentration at room temperature, and the maximum inhibition efficiency of 70.72% was observed at 20 ppm of BM leaves extract (shown in table.3). The results indicate that the  $R_{ct}$  significantly increase with increase in concentration of inhibitor and  $C_{dl}$  tends to decrease. This decrease in  $C_{dl}$  may probably due to decrease in local dielectric constant and / or an increase in the thickness of a protective layer at electrode surface which enhance the corrosion resistance of the mild steel [31-35]. The increase in  $R_{ct}$  values is attributed to the formation of protective film at the metal-solution interface [36-45]. These observations suggest that BM leaves extracts function by adsorption at the metal surface thereby causing decrease in  $C_{dl}$  values and increase in  $R_{ct}$  values.

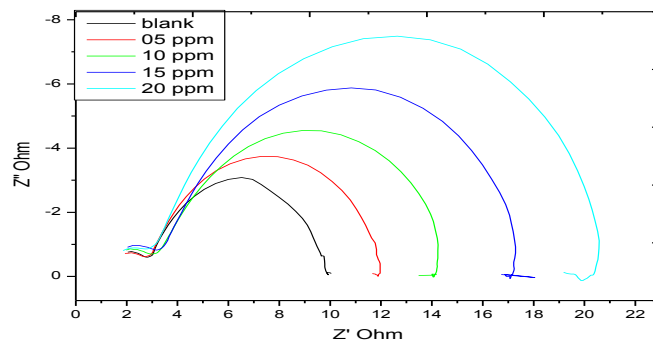


Fig. 3 Nyquist plots for mild steel in 1N HCl acid solution without and with presence of different concentration of BM extract

Table 3. Impedance parameter for mild steel in 1N HCl acid solution in the absence and presence of varied concentration of inhibitor

Conc. of BM extract (ppm)	Rct (ohm cm <sup>2</sup> )	Cdl ( $\mu\text{F}/\text{cm}^2$ )	IE (%)
Blank	8.272	$7.85 \times 10^{-3}$	*
5	10.930	$1.246 \times 10^{-4}$	24.31
10	14.518	$1.004 \times 10^{-4}$	43.02
15	16.333	$4.437 \times 10^{-5}$	49.35
20	19.150	$1.465 \times 10^{-4}$	56.80

**Phytochemical Screening Method:** Phytochemical screening was carried out on the aqueous extracts freshly prepared to the common phytochemical methods described by Harborne [39]. The findings of the phytochemical screening of the aerial parts aqueous extract are shown in table 4.

Table 4. Phytochemical screening test of extract of BM leaves

Phytochemical test	Aqueous extract
Alkaloids	+
Carbohydrates	+
Diterpenes	+
Saponins	+
Phytosterols	-
Tannins	+
Flavanoids	+
Phenol	+
Glycosides	-
Amino acids	+

(+) Presence

(-) Absence

**Scanning Electron Microscopy (SEM):** The morphologies of the mild steel immersed in 1N HCl solution in the absence and presence of the optimum concentration of inhibitor for 24 h are shown in Fig. 4. 4(a) shows that the mild steel surface before the specimen was immersed in the acid solution is absolutely free of any pits and cracks; however, small scratches are clearly visible due to the abrading treatment. Fig. 4(b) shows the appearances of the smooth mild steel surface after the inhibitor was added solution. As evidence in Fig. 4b the rate of corrosion is diminished, and the smooth surface appears as a result of the formation of a protective film on the metal surface; this film was responsible for corrosion inhibition.

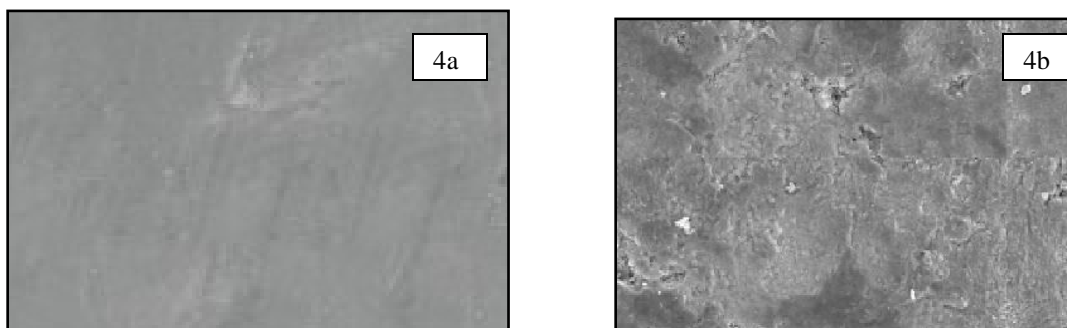


Fig 4. SEM image of the surface of mild steel after immersion for 24 hours in 1N HCl solution in the (4a) absence and (4b) presence of optimum concentration of the BM plants leaves extract

**Effect of temperature:** To assess the effect of temperature on corrosion and corrosion inhibition process, gravimetric experiments were performed at different temperature (303-323K) in the absence and presence of various concentration of the inhibitor during 3 hours of immersion. The results are given in Table 5.

Table 5. IE at various Temperatures

Concentration of BM extract (ppm)	IE (%)		
	303K	313K	323K
5	49.40	44.90	42.00
10	62.50	57.57	47.09
15	72.90	70.62	69.86
20	75.18	<b>77.89</b>	75.66

**Adsorption isotherm:** The adsorption isotherm is processes which are used to investigate the mode of adsorption and its characteristic of inhibitor on the metal surface. In our present study the Temkin adsorption isotherm is investigated. The straight line in figure 5 clearly indicated that the inhibitor obey Temkin adsorption isotherm.

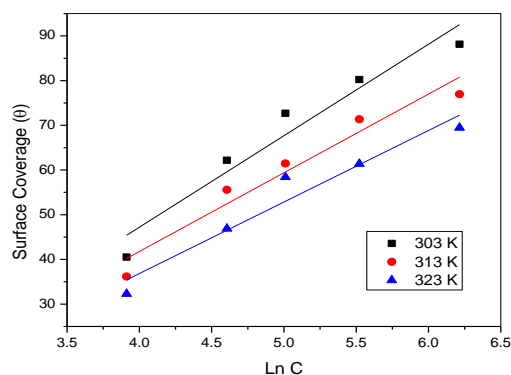


Figure 5 Temkin adsorption isotherm plot for mild steel in 1N HCl containing BM leaves extract

## APPLICATIONS

The BM leaves extract exhibit excellent corrosion resistance to the mild steel in HCl medium. The BM inhibitor that has little or no impact on the environment i.e. the inhibitor is eco friendly, safe and some extent good corrosion resistance with temperature. Therefore this inhibitor can be used for the corrosion protection of mild steel in acid medium for various industries.

## CONCLUSIONS

The aqueous extracts of BM leaves act as good inhibitor for the corrosion of mild steel in 1N HCl solution. Electrochemical polarization results indicated that the BM leaves extracts act as a mixed type inhibitor and the impedance results showed that the corrosion of mild steel is mainly controlled by a charge transfer process and the presence of BM leaves extracts in acid solution does not alter the mechanism of mild dissolution.

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#### AUTHORS' ADDRESSES

1. **P. R. Sivakumar**

PG & Research Department of Chemistry,  
Government Arts College (Autonomous), Coimbatore 641 018, Tamilnadu, India.  
E-mail: shivarashee@gmail.com, Ph: +919944713951

2. **K. Vishalakshi**

PG & Research Department of Chemistry,  
Government Arts College (Autonomous), Coimbatore 641 018, Tamilnadu, India.  
E-mail: vishalikaarthik@gmail.com, Ph: +917092340192

3. **Dr. A.P.Srikanth**

Assistant Professor, PG & Research Department of Chemistry,  
Government Arts College (Autonomous), Coimbatore 641 018, Tamilnadu, India.  
E-mail: apsrikanth8@gmail.com, Ph: +91 98842 30206 / +91 96778 64111