Available online at www.derpharmachemica.com

ISSN 0975-413X
CODEN (USA): PCHHAX

Der PharmaChemica, 2016, 8(19):548-553
(http://derpharmachemica.com/archive.html)

Analysis of Corrosion Resistance Behavior of Green Inhibitors on Mild Steel in 1N HCl Medium Using Electrochemical Techniques

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ABSTRACT

The use of Balsamodendron Caudatum (BC) leaves extract as mild steel corrosion inhibitor in 1N HCl medium was investigated using weight loss, potentiodynamic polarization methods and Electrochemical Impedance Spectroscopy and characteristics analysis by Fourier Transform Infra-Red (FTIR). Obtained data from weight loss polarization methods, impedance suggested that the value of inhibition efficiency (% IE) was proportional to added inhibitor concentration. Tafelconstants data indicated that BC extract can act as mixed type inhibitors. The surface morphology was determined using Scanning Electron Microscopy.

Keywords: Corrosion inhibition, Mild steel, Electrochemical techniques, SEM.

INTRODUCTION

Mild steel has been extensively used for many mechanical and structural engineering purposes. Corrosion of mild steel has an enormous economic impact. The understanding of the corrosion problem and the solution to tackle this problem is a very active field of research. Generally acidic solutions are used as pickling agents for iron and steel, chemical cleaning of scale in metallurgy, acidizing in oil recovery, and other petrochemical process [1-2]. The known hazardous effect of most synthetic corrosion inhibitors is the motivation for the use of some natural products. The use of chemical inhibitors has been limited because of the environmental threat. Recently, due to environmental regulations, plant extracts have become important because they are environmentally acceptable, inexpensive, readily available and renewable sources of materials, and ecologically acceptable [3-7]. Plant products are organic in nature and some of the constituents including tannins, organic and amino acids, alkaloids, flavonoids, pigments, carbohydrates, etc., by simple procedures with low cost are known to exhibit inhibiting action [8-10]. The successful use of naturally occurring substance to inhibit the corrosion of metal in various medium has been reported by our research groups such as MeliaA zederach, Mimusops Elengi Linn, Madhuca Longifolia, etc., [11-14].

Therefore, in the present study the inhibition performance of Balsamodendron Caudatum leaves, on mild steel in the presence of HCl was examined using various techniques.

MATERIALS AND METHODS

2.1 Preparation of Leaves Extract

The leaves of Balsamodendron Caudatum plant were collected, cut into small pieces, and they were shade dried in room temperature for few days and ground well into powder.25g of powder was refluxed in 200 ml Ethyl alcohol for 3 hours and kept overnight. The refluxed was then filtered carefully; the filtrate volume was made up to 500 ml.

2.2 Preparation of Electrodes

The working electrode was mild steel (MS), which was cut from a square mild steel plate. The chemical composition of mild steel strips was (C-0.030%, Mn-0.169%, Si-0.015%, P-0.O31%, S-0.029%, Cr-0.029%, Ni-0.030%, Mb-0.016%, Cu-0.017%), and the reminder Fe, were mechanically cut into 4cm x 2cm x 0.1cm and were used for weight loss studies. For electrochemical studies, the mild steel strips of the same composition but with an exposed area of 1cm^2 were used.

3. METHODS

3.1 Weight Loss Method

The simplicity and reliability of the measurement offered by the weight loss method is such that the technique forms the baseline method of measurement in many corrosion monitoring programs [15-22].Mild steel specimens were immersed in 200 ml of 1N HCl solution containing various concentration of the inhibitor in the absence and presence of mild steel for 24 hours. The weights of the specimens before and after immersion werecalculated for inhibition efficiency.

$$\text{Corrosion Rate (mmpy)} = \frac{\text{K x Weight loss}}{\text{D x A x t (in hours)}} \qquad 1$$

Where, K = 8.76x10^4 (constant), D is density in gm/cm^3 (7.86), W is weight loss in grams and A is area in cm^2.

$$\text{IE\%} = \frac{W_0 - W_i}{W_0} X100 \qquad 2$$

Where, W_0 and $W_i$are the weight loss in the absence and presence of the inhibitor.

3.2 Fourier Transform Infrared (FTIR) Spectrum

FTIR testing was conducted to determine the characteristics of the existing group on BC extract. From these characteristics, it can be known that the functional group usually used as an inhibitor, such as alcohol group, alkaloids group and free compound (N, O, P and S). FTIR spectra were recorded with BRUKER ALPHA 8400S spectrometer. The film was carefully removed, thoroughly mixed with KBr and it was made into pellets, then the FTIR spectra were recorded.

3.3 Potentiodynamic Polarization Method

The electrochemical studies were made using a three-electrode cell assembly at room temperature. The mild steel specimen of 1cm^2 was the working electrode, platinum electrode was used as an auxillary electrode and standard calomel electrode was used as reference electrode. Potentiodynamic polarization measurements were carried out using IVIUM electrochemical work station analyzer. A time interval of 10-15 minutes was given for each experiment to attain the study state open circuit potential. The polarization was carried out from cathodic potential to anodic potential at a sweep rate of 1 mV S^{-1}.

$$\text{IE\%} = \frac{I_{Corr} - I^*_{Corr}}{I_{Corr}} X100 \qquad 3$$

Where I_{corr} and I^*_{corr} are corrosion current in the absence and presence of inhibitors.

3.4 Electrochemical Impedance Method

The electrochemical AC-impedance experiments were carried out in three cells assembly as that used for potentiodynamic polarization studies. 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.

$C_{dl} = 1/2\pi f_{max} Rct$ 4

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

$$\text{IE\%} = \frac{R_{ct} - R^0_{ct}}{R_{ct}} \text{X100} \qquad 5$$

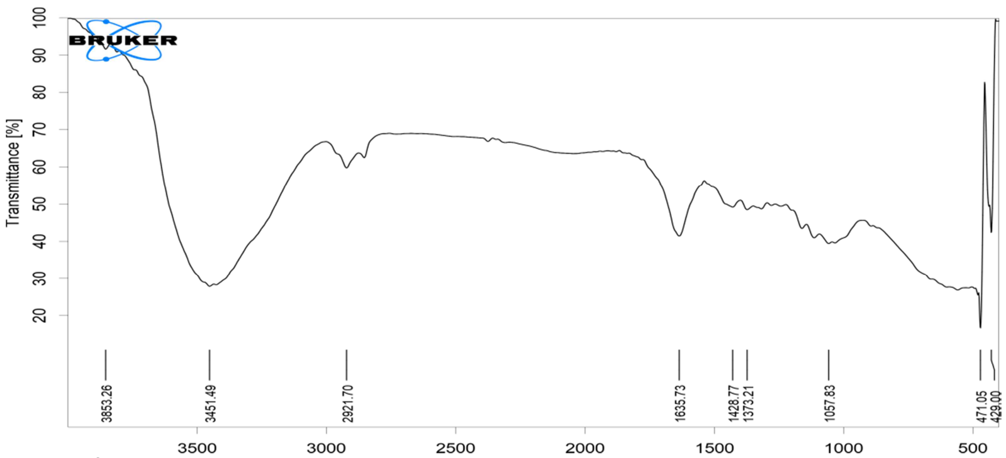
Where R_{ct} and R^0_{ct} are the charge resistance values in the inhibited and uninhibited solution.

3.5 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

Wavenumber cm-1

Figure. 1 FT-IR spectrum of Balsamodendron Caudatum leaves extract



4.1 FT-IR spectrum of BC
FT-IR spectrum was recorded for BC leaves extract in order to confirm the presence of various compounds which contributed in effective working of the inhibitor is shown in Figure. 1. A strong peak obtained at 3853 cm^{-1}, which corresponds to O-H group. The broad peak obtained at 3451 cm^{-1} can be assigned to N-H stretching. Another peak obtained at 2921 cm^{-1} may be strong C-H stretching vibration. Adsorption peak obtained at 1635cm^{-1} may be due to stretching of –C=O band. The peaks observed at 1428 cm^{-1} may be –C-H- bending vibration. Few peaks could also be observed at 1373 cm^{-1}, 1057 cm^{-}1, which correspond to C-H and C-O stretching vibration.

4.2 Weight Loss Method
The weight loss method was done with different concentrations of BC extract ranging from 5 to 20 v/v for mild steel in 1N HCl, and the corresponding values of inhibition efficiency and corrosion rate are given in Table. 1. It was observed from the table that the corrosion rate decreased and thus the inhibition efficiency increases with increasing concentration of BC extract. The maximum inhibition efficiency of about 77.93was achieved at 15 v/v of BC extract. This result indicated that BC extract could act as an excellent corrosion inhibitor.

Table. 1 Data from Weight Loss Method for MS corroding in 1N HCl solutions at various concentrations of BCextract

Conc. of BC leaves Extract (v/v)	Corrosion Rate (mmpy)	Inhibition Efficiency (%)
Blank	0.0011	*
5	0.0007	37.74
10	0.0124	53.33
15	0.0110	77.93
20	0.0104	76.60

4.3 Potentiodynamic polarization Studies
The corrosion inhibition efficiency of BC extracts of various concentrations was measured using the polarization technique and the results are summarized in Table 2. To evaluate the various concentrations of BC extracts on the

electrochemical behavior of mild steel in 1N HCl solution, cathodic and anodic polarization curves were carried out with the aid of potentiostat. Fig.2. Shows the polarization curves of various concentrations of BC extract.

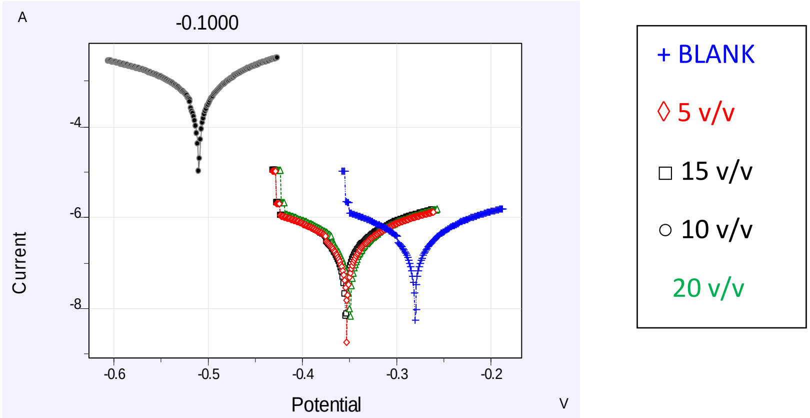


Figure. 2 Potentiodynamic polarization curves for mild steel in 1N HCl solution in the absence and presence of different concentrations of BCleaves extract

The electrochemical parameters corrosion potential (E_{corr}), the corrosion current density (I_{corr}), inhibition efficiency (IE%), anodic (ba) and cathodic (bc) Tafel slopes obtained from the polarization measurements were listed in Table 2. The data exhibited that, the corrosion current density (I_{corr}) decreases, and the inhibition efficiency increases as the concentration of the inhibitor is increased. These results suggest that retardation of the electrodes processes occurs, at both cathodic and anodic sites, as a result of coverage of these sites by surfactant molecule. The increase of inhibitor efficiency with increasing the concentration can be interpreted on the basis the adsorption amount and the coverage of surfactant molecule, increases with increasing concentration [23].

Table.2 Potentiodynamic polarization parameter for mild steel in 1N HCl solution containing various concentrations of BC leaves extract

Conc. of BC leaves extract (v/v)	E_{corr} (mV) vs. SCE	I_{corr} (mA cm^{-2})	CR (mmpy)	b_c (mV/dec)	b_a (mV/dec)	IE (%)
Blank	-0.504	6.95 X 10-4	2.273	0.128	0.087	*
5	-0.2795	3.60 X 10-7	0.0011	0.102	0.107	48.20
10	-0.3477	3.49X 10-7	0.0011	0.104	0.107	49.78
15	-0.3499	3.15X 10-7	0.0010	0.106	0.106	54.67
20	-0.3521	3.36 X 10-7	0.0011	0.105	0.106	51.65

4.4 Electrochemical Impedance spectroscopy (EIS) Studies

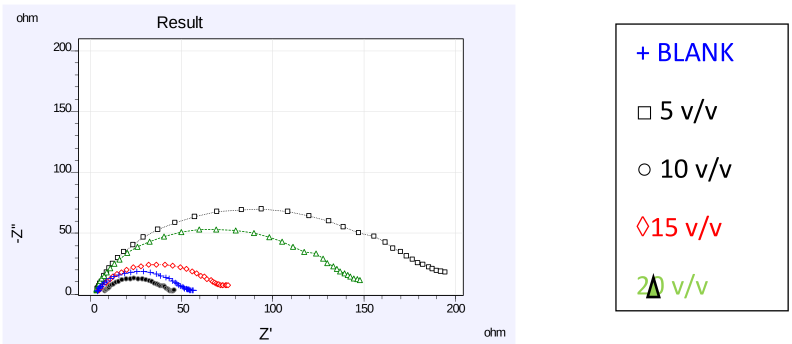


Figure. 3Nyquist plots of mild steel immersed in 1N HCl in absence and presence of different concentrations of BC leaves extract

EIS diagrams for mild steel in 1N HCl obtained in the absence and presence of BC extract at various concentrations (5–20 v/v)are shown in Fig. 3. Where it can be concluded that the Nyquist diagrams is recorded at the corrosion potential and represents a slight semicircle at high frequencies followed at the lower frequencies. The parameter deduced from the fit of Nyquist diagram for 1N HCl medium containing various concentration of BC leaves extracts are given in Table 3.

Table 3 shows the EIS data where the C_{dl} values decreases and the R_{ct} values increases with the increase of the BC extract. This increase is due to the gradual replacement of water molecules by the adsorption of the inhibitor molecules on the electrode surface, and decreasing the extent of dissolution reaction. The decrease in the C_{dl} can result from the decrease of the local dielectric constant and/or from the increase of thickness of the electrical double layer [24] suggested that the inhibitor molecules function by adsorption at the electrode/solution interface.

Table. 3 The electrochemical parameters (EIS) for mild steel corrosion rate in 1N HCl solution in different concentrations of BC leaves extract

Conc. of BC leaves extract(v/v)	C_{dl} (μF cm^{-2})	b_c (mV/dec)	b_a (mV/dec)	R_{ct} (Ω cm^2)	IE (%)
Blank	1.57 X 10^5	0.087	0.128	-0.2070	*
5	1.79 X 10^{-6}	0.106	0.106	-0.0061	39.93
10	1.52 X 10^{-6}	0.104	0.107	-0.0034	61.88
15	1.45 X 10^{-6}	0.102	0.107	-0.0022	95.09
20	2.34 X 10^{-6}	0.105	0.106	-0.0040	52.75

4.5 Scanning Electron Microscopy (SEM)

SEM images of the surface of mild steel in 1N HCl without and with optimum concentration of inhibitor are recorded are shown in Fig 4A and 4B. There is much difference occur in both images. Fig. 4A is for uninhibited mild steel surface in 1N HCl which is highly damaged and more roughness occur in it. After adding inhibitor smoothness occurs in Fig. 4B due to the formation of protective layer on mild steel surface.

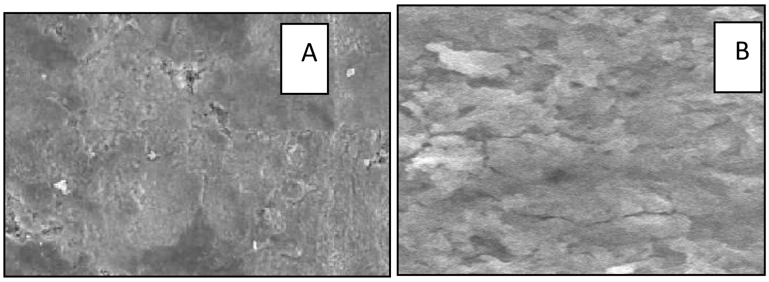


Figure.4 SEM image of the surface of mild steel after immersion for 24 hrs in 1N HCl solution. (A) in the absence of inhibitor (B) in the presence of 20 v/vBC leaves extract

4.6 Phytochemical Screening

Phytochemical screening of the aerial parts of plant's powder (alcoholic) extract was tested in order to find the presence of various chemical constituent included Alkaloids, Carbohydrates, Reducing sugars, Flavonoids, Glycosides, Saponins, Triterpenoids& Steroids, Tannins and Phenolic Compounds the results are listed in Table. 4

Table.4 Phytochemical screening of leaves extract of BC

S.No	Phytochemical constituents	Alcoholic extract of BC leaves
1	Alkaloids	+
2	Carbohydrates	+
3	Reducing Sugars	+
4	Flavanoids	-
5	Glycosides	-
6	Saponins	+
7	Triterpenoids& Steroids	-
8	Tannins & Phenolic Compoundnds	-

+ is Present *- is absent*

CONCLUSION

From the present study, it is concluded that BC leaves extract can be used as efficient corrosion pickling inhibitor on mild steel in 1 N HCl acid. The use of BC plant as corrosion inhibitor is environmentally safe, nontoxic, ecofriendly, cost effective and easily available. The extracts of BC plants showed maximum efficiency of 77.93% at the optimum concentration of 15ppm for one day immersion time at room temperature. Results obtained in non-electrochemical methods have good agreement with the electrochemical methods. Polarization studies showed the BC plants extracts act as a mixed type inhibitor on the metal surface. SEM examination showed that there was improvement in the surface morphology of the as corroded inhibited mild steel compared to uninhibited samples.

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