Inhibition of mild steel corrosion in 0.5 M sulfuric acid by an aqueous extract of leaves of *Tectona grandis L.* plant

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Abstract

Inhibition of corrosion of mild steel in 0.5 M sulfuric acid by an aqueous extract of leaves of *Tectona grandis L.* plant has been investigated by weight loss method. The mechanistic aspects of corrosion inhibition have been studied by electrochemical studies such as potentiodynamic polarization technique and electrochemical impedance spectroscopy (EIS). It is observed that as the concentration of the inhibitor increases the corrosion rate decreases and the inhibition efficiency increases. This is due to adsorption of the molecules of the active ingredients of the extract on the metal surface. A maximum inhibition efficiency of 96.70% is achieved by this inhibitor system. Potentiodynamic polarization technique reveals that the inhibitor system functions as a mixed type of inhibitor, controlling anodic reaction and cathodic reaction to an equal extent. It is noted that, in presence of inhibitor, linear polarization resistance value increases, and corrosion current decreases. Electrochemical impedance studies reveal that a protective film (blanket effect) is formed on the metal surface, since in the presence of inhibitor system the charge transfer resistance value increases and double layer capacitance value decreases. The surface morphology of the protective film has been studied by SEM and AFM. The outcome of the study can be used in pickling industry, where sulfuric acid is used to remove the rust on the mild steel surface.

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Introduction

Numerous corrosion inhibitors have been used in acid media. The results of the study will be used in pickling industry, industrial cleaning and acid de-scaling processes. The ability of a compound to serve as inhibitor depends on its ability to form a compact barrier film and on the nature of adsorption of inhibitor on metal surface. Most of the corrosion inhibitors contain hetero atoms, such as O, N, S and multiple bonds. The electrons on these atoms will coordinate with metal ions on the anodic sites of the metal surface and form metal inhibitor complex as protective layer. Many corrosion inhibitors such as chromates have been discarded due to their toxic nature and due to the objection from environmental scientists. So there is a need to use less toxic (or nontoxic) environmental friendly corrosion inhibitors. The ingredients in natural products can be extracted by simple procedures with low cost. Moreover, the extracts of the natural products are biodegradable in nature. Natural corrosion inhibition and adsorption characteristics of *Tribulus Terrestris* plant extract on aluminium in hydrochloric acid environment has been investigated by Chaudhary and Tak. Potentiodynamic polarization measurements indicated the nature of inhibitor is a mixed type. Impedance studies supported the formation of a protective layer of inhibitor on a metal surface. SEM images confirmed the creation of a protective film over the metal surface [1]. Mofidabadi *et al.* studied steel alloy surface protection against saline attacks *via* the development of Zn(II)–metal–organic networks using *Lemon verbena* leaves extract (LVLE). Synergistic effect is noticed when Ce and zinc ions are added to the leaves extract. FT-IR, UV-Vis and Raman spectra have been employed to analyse the protective film. FE-SEM and AFM have been used to study the surface morphology of the protective film [2]. Mirinioui *et al.* have used an eco-friendly inhibitor, namely, *Dysphania Ambrosioides* essential oil, for mild steel corrosion in hydrochloric and sulfuric acid medium. Thermodynamic parameters and quantum chemical parameters have been measured and discussed [3]. Corrosion mitigation of mild steel in hydrochloric acid solution using grape seed extract has been studied by Marhamati *et al.* The study revealed the formation of more compact corrosion products with improved integrity in the presence of grape seed, which confirmed electrochemical test results. Besides, water droplet contact angle, field-emission scanning electron microscopy coupled with energy dispersive spectroscopy, Fourier transform infrared spectroscopy, Raman spectroscopy, X-ray photoelectron spectroscopy, and atomic force microscopy were utilized to study the surface of mild steel specimens after dipping in acidic solutions [4]. Corrosion inhibition of mild steel by aqueous leaf extract of purple hedge plant has been studied by Mukhopadhyay *et al.* Gas chromatography along with FT-IR and NMR spectroscopy revealed the presence of carbohydrate molecules as major chemical constituents of the freeze dried leaf extract. A plausible mechanism for inhibition properties was proposed on the basis of experimental and theoretical studies [5].

Fouda *et al.* used *Ferula Hermonis* plant extract as safe corrosion inhibitor for zinc in hydrochloric acid solution. The efficiency of *Ferula Hermonis* plant extract, as inhibitor for zinc corrosion in a 1 M HCl solution, has been tested by mass loss (ML), electrochemical measurements (potentiodynamic polarization (PDP), electrochemical impedance spectroscopy (EIS) and electrochemical frequency modulation (EFM)), in addition to surface examination analysis. The outcomes indicated that *F. Hermonis* extract showed good efficiency to Zn corrosion and displayed high inhibition efficiency. The maximum inhibition efficiency approached 90.6% at 300 ppm extract. The surface morphology of the protective film has been analyzed by Fourier transform infrared spectroscopy (FT-IR), atomic force microscopy (AFM) and scanning electron microscopy (SEM) [6]. The aqueous extract synthesized using the biomass, *Ziziphus Spina-Christi*, was applied to protect the surface of aluminium under acidic environment by Natarajan and Al Shibli. The influence of extract concentration, contact time and temperature on the inhibition efficiency has been studied. The biomass-based corrosion inhibitor was characterized using scanning electron microscope and attenuated total reflection techniques [7]. Shahini *et al.* used chamomile flower extract as a green corrosion inhibitor for mild steel in HCl solution. A 98% inhibition efficiency was obtained. The surface morphology of the protective film was analyzed by FE-SEM, EDAX, AFM, and contact angle examinations [8]. The corrosion inhibitory effect of the ecofriendly additive, *Terminalia Catappa* leaf extract, added to soybean oil biodiesel in contact with zinc and carbon steel 1020 was evaluated by Fernandes *et al.* The morphology of the metal plates was analyzed by scanning electron microscopy/dispersive energy spectroscopy, X-ray diffraction and the biodiesel through acidity index, Fourier Transformed Infrared Absorption Spectroscopy. At the end of the tests, carbon steel 1020 suffered no corrosion [9]. The electrochemical behavior of 5083 aluminum alloy in an alkaline solution in the absence and presence of two green additives extracted from *Mentha piperita L* 'MP' and *Lawsonia inermis* 'LI' has been analyzed by Hosseinpour Rokni *et al.* The results of this study can open the way for future investigations to replace the synthetic anti-corrosion additives of Al air-batteries with non-toxic and biodegradable organic ones having similar corrosion inhibition performance without restricting anode discharge currents [10].

The main objective of the present study is to investigate inhibition of corrosion of mild steel in 0.5 M sulfuric acid by an aqueous extract of leaves of *Tectona grandis L.* plant by weight loss method. Another objective is to study the mechanistic aspects of corrosion inhibition by electrochemical studies such as potentiodynamic polarization technique and electrochemical impedance spectroscopy (EIS).

Materials and Methods

The mild steel specimens were made from the same sheet of the following composition: carbon – 0.1%, sulphur – 0.026%, phosphorus – 0.06%, manganese – 0.4% and balance iron. Mild steel specimen of the dimension $1.0\times4.0\times0.2$ cm^3 were polished to mirror finish, degreased with trichloroethylene and used for weight loss and surface examination studies.

Mild steel specimens encapsulated in Teflon with an exposed cross section of 1 cm^2 were used as working electrodes in potentiodynamic polarization studies. The electrode surface was polished to mirror finish and degreased with trichloroethylene. The medium (0.5 M H_2SO_4) was prepared by dilution of analytical grade sulfuric acid with double distilled water.

Inhibitor preparation

An aqueous extract of leaves of *Tectona grandis* plant was prepared by boiling 10 g of shade dried leaves with double distilled water. The suspended impurities were removed by filtration. The solution was made up to 100 ml and used as corrosion inhibitor.

Teak leaf extract (*Tectona grandis L.*) has been shown to have antibacterial activity with specific antibacterial compounds, namely anthratectone, naphthotectone, juglone, rutin, quercetin, gallic acid, ellagic acid, and sitosterol [11]. Teak (*Tectona grandis*) is a tropical hardwood tree species in the *Lamiaceae* family. It is a large, deciduous tree that occurs in mixed hardwood forests.

Weight loss method

Weights of the three polished mild steel specimens were measured before and after immersion in various test solutions (0.5 M sulfuric acid with different extract concentrations) for 3 h. The inhibition efficiencies were calculated from the relation [12–14].

$$IE = [(C_{\mathrm{R1}} - C_{\mathrm{R2}}) / C_{\mathrm{R1}}] \cdot 100\% \tag{1}$$

Where C_{R1} is corrosion rate in the absence of inhibitor and C_{R2} is the corrosion rate in the presence of inhibitor.

Electrochemical study

In the present work, the corrosion resistance of mild steel immersed in various test solutions were measured by Polarization study. Electrochemical measurements were performed in a CHI electrochemical work station with impedance model 660 A.

Polarization study

Polarization studies were carried out in a three electrode cell assembly (Figure 1). A SCE was the reference electrode. Platinum was the counter electrode. Mild steel was the working electrode. From polarization study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes anodic = b_{a}, and cathodic = b_{c}, and LPR (linear polarization resistance) values were measured [15, 16].

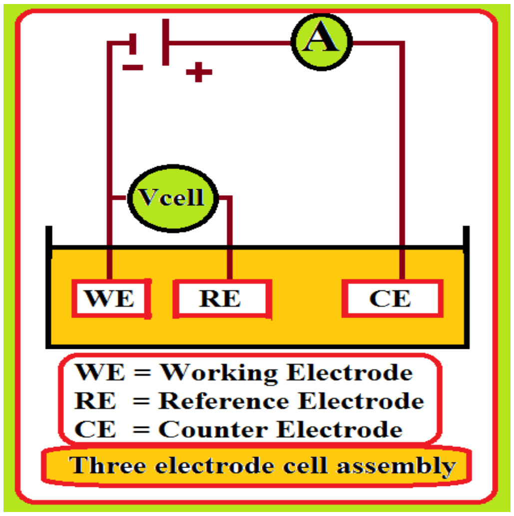

Figure 1. Three electrode cell assembly.

AC Impedance spectra

The same instrument and set-up used for polarization study was used to record AC impedance spectra also. A time interval of 5 to 10 min was given for the system to attain a steady state open circuit model. The real part (Z') and imaginary part ($-Z''$) of the cell impedance were measured in Ohms at various frequencies. AC impedance spectra were recorded with initial E(V) = 0, high frequency ($1-10^5$ Hz), low frequency (1 Hz), amplitude (V) = 0.005 and quiet time (s) = 2. From Nyquist plot the values of charge transfer resistance (R_t) and the double layer capacitance (C_{dl}) values were calculated [17].

Scanning electron microscopy (SEM)

The mild steel specimens immersed in various test solutions for one day were taken out, rinsed with double distilled water, dried and subjected to the surface examination [18]. The surface morphology measurements of the mild steel surface were carried out by scanning electron microscopy (SEM) using Cartizers make model EVO-18.

Atomic force microscopy (AFM)

The mild steel specimens immersed in various test solutions for one day were taken out, rinsed with double distilled water, dried and subjected to the surface examination [19]. The surface morphology measurements of the mild steel surface were carried out by atomic force microscopy (AFM) using SPM Veeco di Innova connected with the software version V7.00 and the scan rate of 0.7 Hz.

Results and Discussion

Weight loss method

Corrosion rates of mild steel in 0.5 M sulfuric acid and the inhibition efficiencies offered by an aqueous extract of leaves of *Tectona grandis* plant (immersion period 3 h) are given in Table 1. It is observed that the inhibitor system offers good inhibition efficiency to mild steel in acid medium. It is observed that as the concentration of the inhibitor increases, the inhibition efficiency increases (Figure 2). This is due to adsorption of the molecules of the active ingredients of the extract on the metal surface. A maximum inhibition efficiency of 96.70% is achieved by this inhibitor system [20, 21].

Table 1. Inhibition efficiency of aqueous leaves extract of *Tectona grandis* plant on the corrosion of mild steel in 0.5 M H_2SO_4 at room temperature (303 K).

Concentration of inhibitor (mL)	Corrosion rate (mdd)	Inhibition efficiency (%)
0	328.13	–
2	78.47	76.09
4	49.93	84.78

Concentration of inhibitor (mL)	Corrosion rate (mdd)	Inhibition efficiency (%)
6	32.10	90.22
8	17.83	94.60
10	10.70	96.70

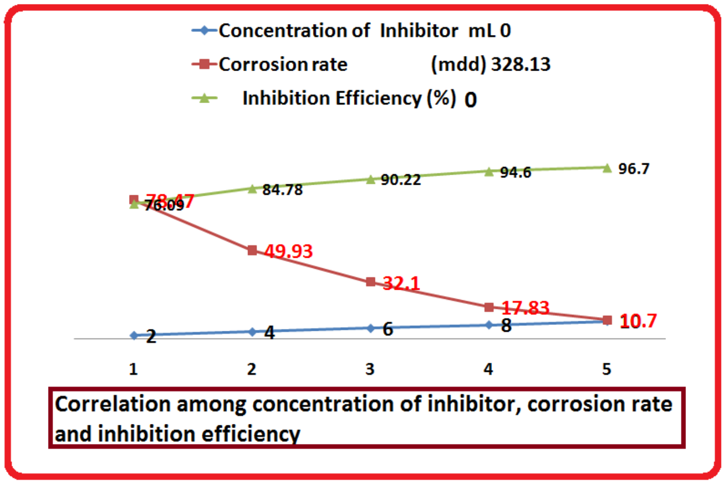

Figure 2. Correlation among concentration of inhibitor, corrosion rate and inhibition efficiency (IE%).

Electrochemical studies

Electrochemical studies such as polarization study and AC impedance spectra have been employed in corrosion inhibition study. When corrosion resistance increases because of blocking effect (Blanket effect) of adsorbed inhibitor molecules on the metal surfaces, the following observations are noted: linear polarization resistance (LPR) value increases and corrosion current (I_{corr}) decreases (Figure 3). Charge transfer resistance increases, impedance increases, phase angle increases and double layer capacitance decreases.

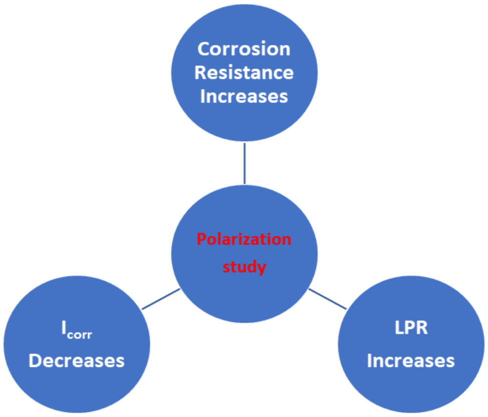

Figure 3. Correlation among corrosion parameters.

Polarization Study

The polarization curves of mild immersed in 0.5 M H_2SO_4 in the absence and presence of inhibitor are shown in Figure 4 and the corrosion parameters are given in Table 2. The electrodes were immersed in the test solutions for 30 min to reach the steady state potential.

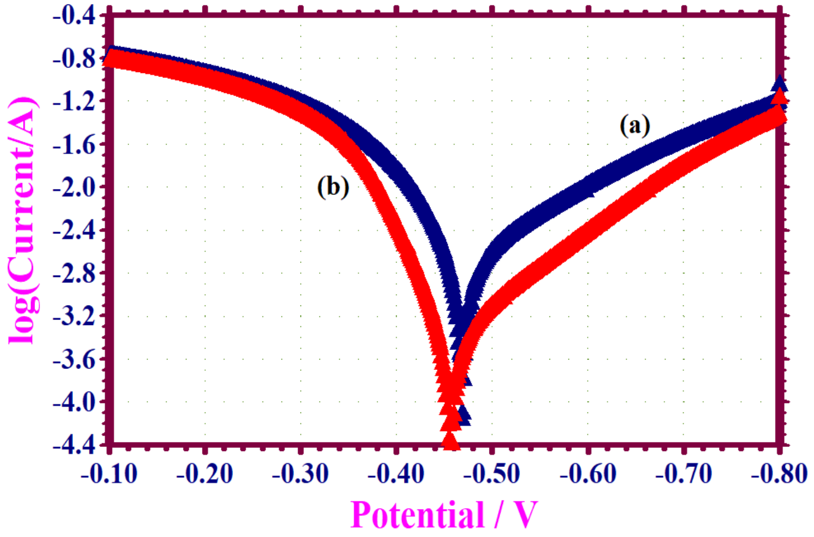

Figure 4. Potentiodynamic curves for corrosion of mild steel in 0.5 M H_2SO_4 (a) in 0.5 M H_2SO_4 (b) in 0.5 M H_2SO_4 + inhibitor extract.

Table 2. Potentiodynamic polarization parameters of mild steel in 0.5 M H_2SO_4 in the absence and presence of aqueous extract of *Tectona grandis* plant leaves.

Concentration of the aqueous extract of TGPL (mL)	**E_{corr}, mV/SCE**	**Tafel slope**		**I_{corr}, A/cm^2**	**LPR, Ohm·cm^2**
		b_a, mV/dec	**b_c, mV/dec**		
0	–468	127	185	$3.459\cdot10^{-3}$	9.5
10	–457	78	150	$5.478\cdot10^{-4}$	40.6

It is observed from Table 2 that when mild steel is immersed in 0.5 M H_2SO_4, the corrosion potential is 468 mV *vs.* SCE, the LPR value is 9.5 Ohm·cm^2 and corrosion current value is $3.459\cdot10^{-3}$ A/cm^2. It is inferred from the Table 2 that in presence of inhibitor, the corrosion potential is shifted from –468 to –457 mV *vs.* SCE. In the presence of inhibitor, there is a very small shift in corrosion potential value when compared with the blank. This shift is less than 50 mV. Hence it is inferred that the inhibitor system functions as a mixed type of inhibitor, controlling anodic reaction and cathodic reaction to an equal extent [22–26]. The LPR value increases (Table 2) from 9.5 to 40.6 Ohm·cm^2. The corrosion current decreases from $3.459\cdot10^{-3}$ to $5.478\cdot10^{-4}$ A/cm^2. These observations confirm that a protective film is formed on the metal surface. This controls the corrosion of metal.

Analysis of results of AC impedance spectra

AC impedance spectra (electrochemical impedance spectra) have been used to confirm the formation of protective film on the metal surface. If a protective film is formed on the metal surface, charge transfer resistance (R_t) increases, double layer capacitance value (C_{dl}) decreases and impedance [log (Z/Ohm)] value increases. The AC impedance spectra of mild steel immersed in 0.5 M H_2SO_4 in presence and absence of inhibitor (extract of leaves) are shown in Figure 5 (Nyquist plots) and Figures 6 and 7 (Bode plots).

AC impedance spectra of mild steel immersed in 0.5 M H_2SO_4 in the absence and presence of aqueous extract of *Tectona grandis* plant leaves inhibitor are shown in Figure 5 (Nyquist plots) and Figures 6 and 7 (Bode plots).

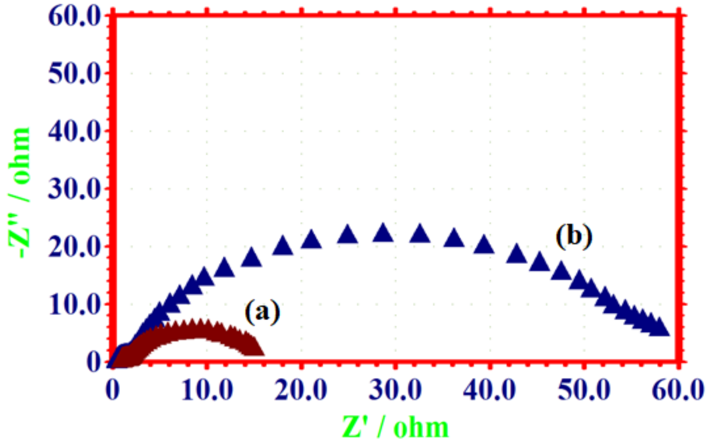

Figure 5. AC impedance spectra of mild steel immersed in 0.5 M H_2SO_4 in the absence and presence of aqueous extract of *Tectona grandis* plant leaves inhibitor (Nyquist plots) (a). Mild steel in 0.5 M H_2SO_4 without inhibitor (b); mild steel in 0.5 M H_2SO_4 with 10% aqueous extract of *Tectona grandis* plant leaves.

Table 3. Electrochemical impedance parameters from Nyquist plots for the corrosion of mild steel with aqueous extract of *Tectona grandis* plant leaves in 0.5 M H_2SO_4.

Concentration of the aqueous leaves extract of TGP (mL)	**Nyquist plot**		**Impedance, log(Z/Ohm)**
	R_t, Ohm·cm^2	**C_{dl}, F/cm^2**	
0	13.88	$1.1859 \cdot 10^{-7}$	1.172
10	59.26	$4.6710 \cdot 10^{-8}$	1.789

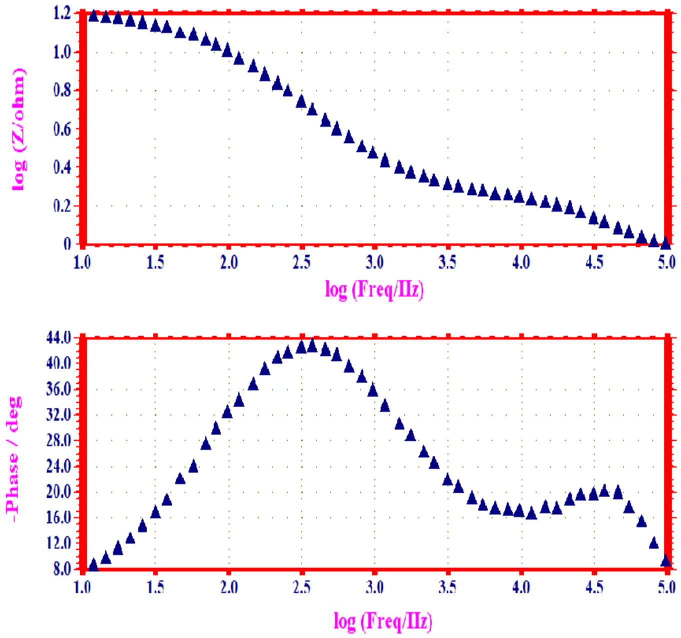

Figure 6. AC impedance spectra (Bode plot) of mild steel immersed in 0.5 M H_2SO_4.

The AC impedance parameters namely charge transfer resistance (R_t) and double layer capacitance derived from Nyquist plots are given in Table 3. The impedance values derived from Bode plots are also given in Table 3. It is observed that when the inhibitor (extract of leaves) is added, the charge transfer resistance value increases from 13.88 Ohm·cm^2 to 59.26 Ohm·cm^2. The C_{dl} value decreases from $1.1859\cdot10^{-7}$ F/cm^2 to $4.6710\cdot10^{-8}$ F/cm^2. The impedance value increases from 1.172 to 1.789 [28–30]. These results lead to the conclusion that a protective film is formed on the metal surface.

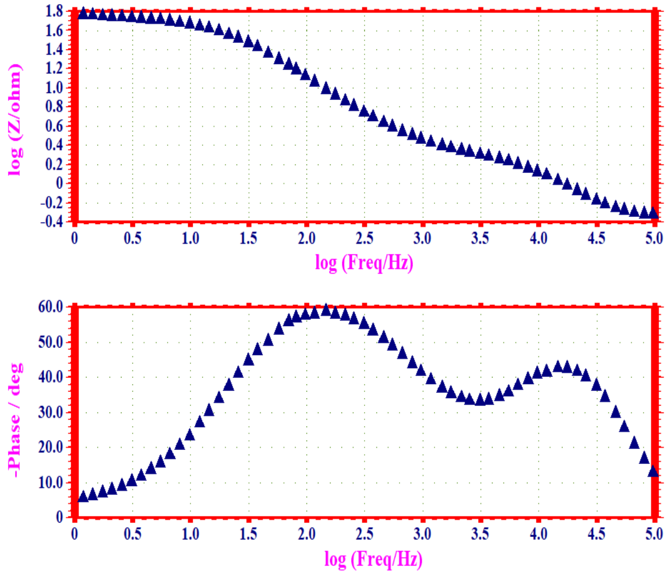

Figure 7. AC impedance spectra (Bode plot) of mild steel immersed in 0.5 M H_2SO_4 with aqueous leaves extract of *Tectona grandis* plant.

Analysis of SEM

The SEM images of various surfaces are shown in Figure 8. The SEM image of polished mild steel is shown in Figure 8(a). The SEM image of polished mild steel immersed in 0.5 M H_2SO_4 (corrosive medium) is shown in Figure 8(b). The SEM image of the polished mild steel immersed in 0.5 M H_2SO_4 (corrosive medium) and inhibitor system is shown in Figure 8(c). The SEM image of polished mild steel is smooth. The SEM image of the mild steel in corrosive medium is found to be rough and the pits are visible on the mild steel surface. The SEM image of the mild steel in corrosive medium and inhibitor system is smooth due to the formation of protective film [31, 32]. Thus SEM study is useful to know the smoothness of the protective film.

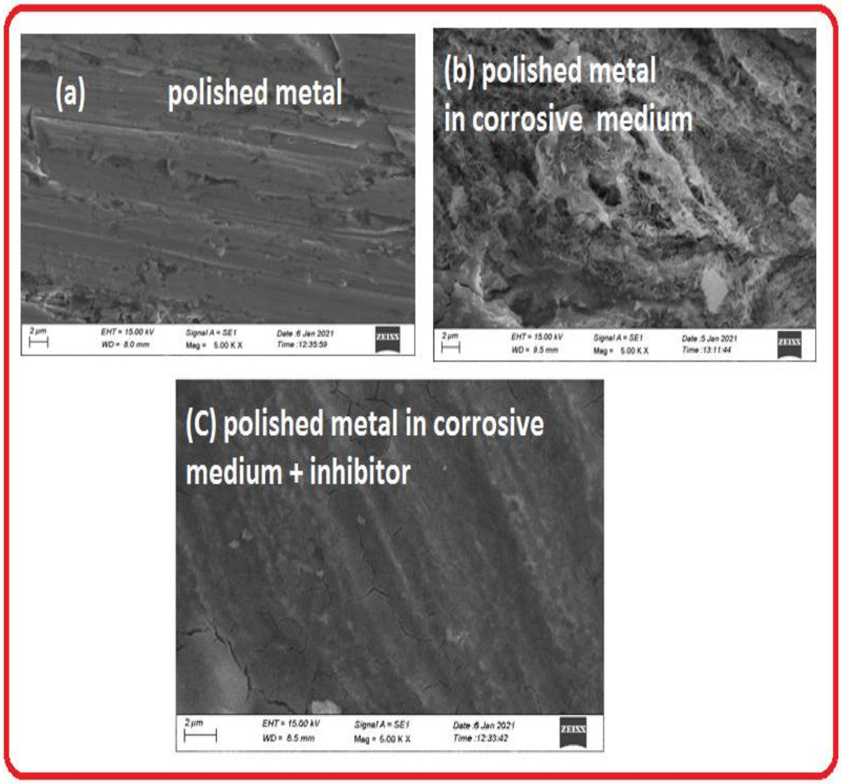

Figure 8. SEM images of various metal surfaces. (a) polished metal (b) polished metal in corrosive medium (c) polished metal in corrosive medium+inhibitor (extract of leaves).

Atomic Force Microscopic Studies

The two dimensional AFM images of polished metal surface, corroded surface (immersed in 0.5 M H_2SO_4) and the film protected metal (0.5 M H_2SO_4+inhibitor, extract of leaves) are shown in Figure 9a, Figure 9b and Figure 9c. The corresponding three dimensional images are shown in Figure 10a, Figure 10b and Figure 10c. The AFM parameters RMS (R_q) roughness (nm), average (R_a) roughness (nm) and maximum peak-to-valley height (nm) were calculated. These values are given in Table 4, for polished mild steel, mild steel immersed in 0.5 M H_2SO_4 and mild steel immersed in inhibitor system.

Table 4. AFM parameters of mild steel surface in the presence and absence of inhibitor (extract of leaves) system.

Sample	RMS (R_q) roughness (nm)	Average (R_a) roughness (nm)	Maximum peak-to-valley height (nm)
Polished MS	222.09	180.64	1558.8
MS immersed in 0.5 M H_2SO_4	423.88	343.05	2763.4
MS immersed in 0.5 M H_2SO_4 and 10 ml of extract	75.939	47.076	1911.6

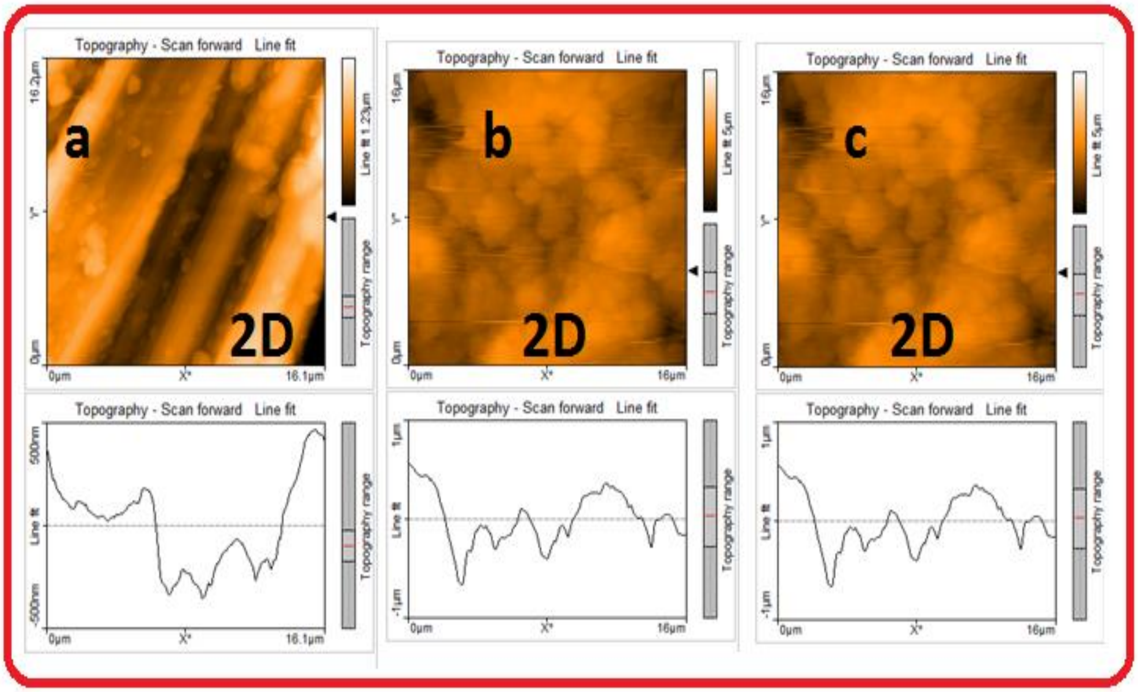

Figure 9. Two dimensional AFM images (a) polished MS; (b) MS immersed in 0.5 M H_2SO_4; (c) MS immersed in 0.5 M H_2SO_4 containing inhibitor (leaves) system.

It is observed from the Table 4 that the RMS roughness of polished mild steel is 222.09 nm. The average roughness is 180.64 nm. The maximum peak-to-valley height is 1558.8 nm. Analysis of Table 4 reveals that the RMS roughness value of mild steel immersed in in 0.5 M H_2SO_4 (corrosive medium) is higher than that of the polished mild steel. This is due to corrosion. Thick nano film is formed on the metal surface. Similar is the case of average roughness and maximum peak-to-valley height. For the metal immersed in inhibitor system, the RMS roughness is less than that of the metal immersed in corrosive medium. Similar is the case of average roughness and maximum-peak-to-valley height [33–35]. This indicates that a protective film of nano scale is formed on the metal surface. This film protects the metal from corrosion.

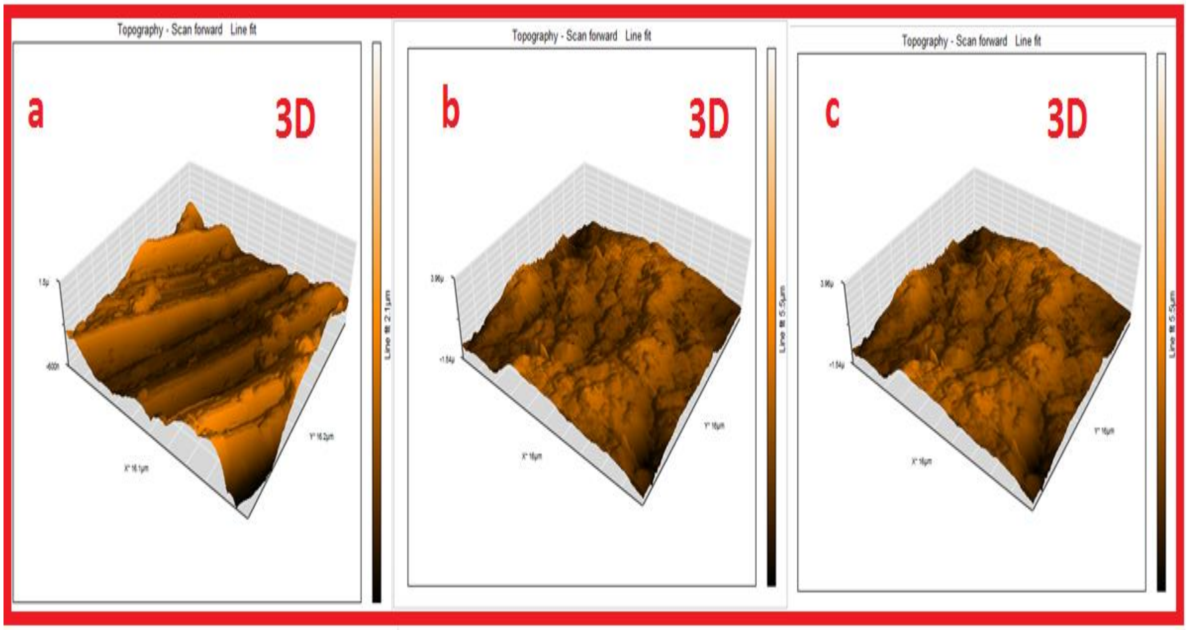

Figure 10. Three dimensional AFM images of the surface (a) Polished MS; (b) MS immersed in 0.5 M H_2SO_4; (c) MS immersed in 0.5 M H_2SO_4 containing inhibitor (leaves) system.

Conclusion

- In this study, the anti-corrosive properties of an aqueous extract of leaves of *Tectona grandis L.* plant have been tested against the mild steel in 0.5 M H_2SO_4 (corrosive medium).
- Decrease in corrosion rate and increase in inhibition efficiency of mild steel in 0.5 M H_2SO_4 medium was observed with the increase in concentration of extract of leaves. The maximum inhibition efficiency of 96.70% was achieved for 10 ml of leaves of *Tectona grandis* plant.
- The potentiodynamic polarization studies conclude that leaves of *Tectona grandis* plant performed as a mixed type of inhibitor.
- According to electrochemical impedance spectroscopy, there was an increase in polarization resistance (R_t) and decrease in double layer capacitance (C_{dl}). This behavior was found to be due to the formation of dense protective layer on the metal/electrolyte surface.
- Furthermore, microscopic studies such as SEM and AFM have indicated the presence of smooth surface in case of inhibited mild steel when compared to the uninhibited samples.
- The investigated aqueous extract of leaves of *Tectona grandis* plant acted as an efficient corrosive inhibitor of mild steel in 0.5 M H_2SO_4 medium.
- The outcome of the study may find application in pickling industry.

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