

Inhibitive effect of *Bauhinia tomentosa* leaf extract on acid corrosion of mild steel

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Abstract : Corrosion inhibitive properties of *Bauhinia tomentosa* leaf extract (BTLE) in 1N H₂SO₄ was investigated by weight loss method and electrochemical techniques. Inhibition efficiency of BTLE was found to increase with increasing concentration. The effect of temperature on the corrosion inhibition of mild steel in the temperature range of 30 - 60 °C was carried out. Polarization measurements revealed that the BTLE acted as a mixed type inhibitor. Nyquist plots showed that on increasing the BTLE concentration, the charge transfer resistance increased and double layer capacitance decreased. The adsorption of BTLE on mild steel obeyed the Temkin isotherm. The maximum inhibition efficiency was found (86.63 % in H₂SO₄) at 30 °C in the presence of 700 ppm of BTLE. The adsorption of BTLE on mild steel surface was characterized by UV-visible and SEM studies.

Keywords : *Bauhinia tomentosa*, SEM, FT-IR, Corrosion inhibition.

1. Introduction

Mild steel is employed widely in a variety of industries. But the main problem of using mild steel is its dissolution in acidic solutions. In various industrial processes, acid solutions are commonly used for removal of rust and scale. Use of organic inhibitors in these processes to prevent metal dissolution is very common [1–3]. Most of the well-known acid inhibitors are organic compounds containing nitrogen, sulfur, oxygen, heterocyclic compounds with a polar functional group and conjugated double bond [4–7]. These compounds are adsorbed on the metallic surface and block the active corrosion sites [8]. Most of the synthetic chemicals are costly, toxic to both human being and the environment.

The use of these natural compounds extracted from leaves or seeds as corrosion inhibitors have been reported by several authors [9–16]. Subramania et al. [17] studied the corrosion inhibition of leaf extracts of curry leaves, henna leaves as well as extracts from seeds of Jack fruit and tamarind on mild steel corrosion in acidic solutions. Other than the plant extracts, pure organic compounds extracted from natural products such as piperine [18], Ellagic acid, tannic acid [19], tryptamine [20], caffeine [21], Pennyroyal oil [22], amino acids [23] and caffeic acid [24] have also been used for inhibition of corrosion. The genus of *H. suaveolens* is known to contain significant amount of volatile oils, starch, protein, tannins, saponins, alkaloids, flavonoids and glycosides [25, 26]. The leaf of the plant is biodegradable and a renewable material. The aim of this study was to investigate the corrosion of mild steel in 1 N H₂SO₄ in the presence of *H. Bauhinia tomentosa* leaf extract by

mass loss, potentiodynamic polarization, electrochemical impedance, FT-IR and SEM. In addition, thermodynamic and kinetic data were also evaluated.

2. Experimental

2.1. Preparation of the specimens

Composition of mild steel specimen was C: 0.13, Si: 0.18, P: 0.39, S: 0.04, Cu: 0.025 and balance Fe. The specimens were mechanically cut into sizes with 2.5 cm × 2.5 cm × 0.4 cm dimensions and abraded with different emery papers up to 4/0 grades. Each specimen was degreased by washing with acetone, dried at room temperature and preserved in moisture-free desiccators. All chemicals and reagents are Analar grade. The concentration of test solution (1N H₂SO₄) was prepared by using triple-distilled water and AR grade hydrochloric acid.

2.2. Extraction of *Bauhinia tomentosa* leaf extract

BT leaves were collected in and around Madurai, India. The leaves were dried, grain and soaked in bidistilled water for 6 h. After 6 h, the crude extracts were boiled, cooled and triple filtered. The amount of plant material extracted into solution was quantified by comparing the weight of dried residue with the initial weight of the dried plant material before extraction. From the respective stock solutions, test solution was prepared in the concentration range from 550 ppm to 700 ppm.

2.3. Mass loss method

Mass loss method was conducted at different temperatures in the range 303 – 333 K for 2 h in 1N H₂SO₄. The specimens were immersed in 100 ml of the respective inhibitor and the test solution in a thermostated bath. The specimens were weighed before and after immersion. The difference in weight was taken as the weight loss of mild steel. From the weight loss (ΔW), Corrosion rate (CR) and the percentage of inhibition efficiency (IE %) were calculated using the following equation :

$$CR \text{ (mpy)} = 534 \times \Delta W / DAT \quad (1)$$

$$IE \% = (W_0 - W_1) / W_0 \times 100 \quad (2)$$

Here $\Delta W = (W_b - W_a)$, Where W_b and W_a are the specimen weights before and after immersion in the tested solution, W_0 and W_1 are the weight loss of mild steel in the absence and presence of inhibitor respectively, D is the density of the iron (g/cm^3), A is the area of the specimen in inch^2 and T is the period of immersion in hours.

2.4. Electrochemical Impedance Spectroscopy measurements (EIS)

Tafel polarization curves and Nyquist impedance curves were recorded using H and CHI electrochemical workstation impedance analyzer model CHI 604D. A cell containing three compartments for electrode was used. The working polished mild steel electrode with exposed area of 0.5 cm² was immersed in a test solution. A platinum electrode and saturated calomel electrode were used as the counter and the reference electrode respectively. Before each potentiodynamic polarization (Tafel) and electrochemical impedance spectroscopy (EIS) experiment, the electrode was allowed to corrode freely and its open-circuit potential was recorded. Potentiodynamic polarization curves were recorded from -300 to +300 mV (SCE verses OCP) with a scan rate of 1 mV s⁻¹. Electrochemical impedance spectroscopy measurements were performed in the frequency range of 0.1 Hz to 100 K Hz. All electrochemical measurements were studied at 303 K using 100 ml of electrolyte (1N H₂SO₄) in stationary condition. Each experiment was repeated in triplicate to check the reproducibility of the data.

2.5 Surface analysis studies

2.5.1. UV-Visible spectroscopic studies

The phytochemical constituents of BTLE treated with 1N H₂SO₄ and after 2 h mild steel immersion were analyzed using JASCOV 32 spectrophotometer. The absorption spectra of these solutions were determined with test solution as a reference.

2.5.2. SEM analysis

The mild steel specimens were immersed in acid solutions in the absence and presence of optimum concentration of inhibitor for a period of 2 h. After 2 h, the specimens were taken out and dried. The nature of the surface film formed on the surface of the mild steel specimen was examined by using a JEOL (JSM 6390) scanning electron microscope.

3. Results and Discussion

3.1. Weight loss measurements

Based on the weight loss measurements, corrosion rate (CR) and inhibition efficiency (IE %) for various concentrations of BTLE, after 2 hour of immersion at the temperatures 303 K, 313 K, 323 K, 333 K in 1N H₂SO₄ are given in Table1. The following equation is used to determine the inhibition efficiency (IE %):

$$IE\% = (CR_0 - CR_{inh})/CR_0 \times 100$$

where CR₀ and CR_{inh} are the corrosion rates of steel without and with the additive respectively. Table 1 give the inhibition efficiencies calculated from the weight loss measurements of different concentration of BTLE in 1N H₂SO₄.

Table 1. The dependence of inhibition efficiency of BTLE from its concentration in the corrosion of mild steel in 1N H₂SO₄

Acid solution	Temperature (K)	Concentration (ppm)	Corrosion Rate (mpy)	θ	Inhibition Efficiency (%)
H ₂ SO ₄	303	0	0.464		
		550	0.168	0.6379	63.79
		600	0.101	0.7823	78.23
		650	0.088	0.8103	81.03
		700	0.062	0.8663	86.63
	313	0	1.001		
		550	0.393	0.6071	60.71
		600	0.331	0.6685	66.85
		650	0.265	0.7352	73.52
		700	0.244	0.7562	75.62
	323	0	1.633		
		550	0.828	0.4929	49.29
		600	0.675	0.5864	58.64
		650	0.452	0.7232	72.32
		700	0.418	0.744	74.4
	333	0	2.418		
		550	2.112	0.1265	12.65
		600	1.724	0.287	28.7
		650	0.9749	0.5968	59.68
		700	0.8694	0.6404	64.04

The inhibition efficiency increases with increasing concentration of the inhibitors in acid media. The maximum inhibition efficiency was obtained (86.63 % in 1N H₂SO₄) at 303 K.

3.2. Thermodynamic activation parameters

The apparent activation energy E_a^* , the enthalpy of activation ΔH^* and the entropy of activation ΔS^* for the corrosion of mild steel samples in 1N H₂SO₄ solution in the absence and presence of different concentrations of BTLE were calculated from Arrhenius equation:

$$CR = A \exp (-E_a^*/RT)$$

And the transition state equation:

$$CR = RT/Nh \exp (\Delta S^*/R) \exp (\Delta H^*/RT)$$

where CR is the corrosion rate, A is the frequency factor, h Planck's constant, N is the Avogadro's number and R is the universal gas constant. A plot of $\log CR/T$ versus $1/T$ gives a straight line with a slope of $\Delta H^*/2.303R$ and intercepts of $\log R/Nh + \Delta S^*/2.303$, Figure 1 represents the plot of $\log CR$ vs $1/T$ in 1N H₂SO₄ and Figure 2 represents the plot of $\log CR/T$ vs $1/T$ in 1N H₂SO₄.

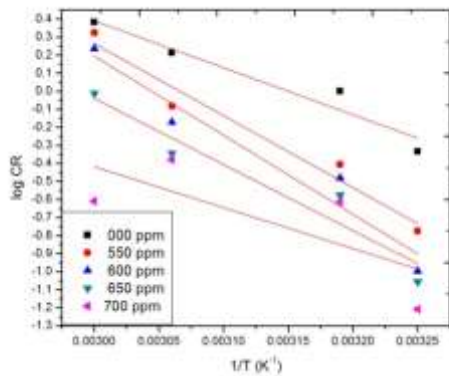


Figure 1: Arrhenius plot of $\log CR$ versus $1/T$ at different concentrations of BTLE.

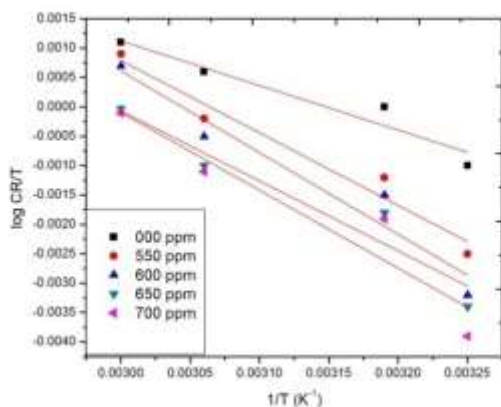


Figure 2: Transition state plots $\log CR/T$ versus $1/T$ at different concentrations of BTLE.

The calculated values of the apparent activation energy (E_a), activation enthalpies (ΔH^*) and the entropy of activation (ΔS^*) are given in Table 2.

Table 2: Corrosion kinetic parameters for mild steel in 1N H₂SO₄ in the absence and presence of different concentrations of BTLE.

C _{inh} (ppm)	E _a (K J/mol)	-ΔH* (K J/mol)	-ΔS* (J/mol/K)
0	49.86	145.36	197.13
550	75.40	236.27	196.86
600	88.48	266.33	196.76
650	70.23	228.23	196.90
700	76.45	254.08	196.82

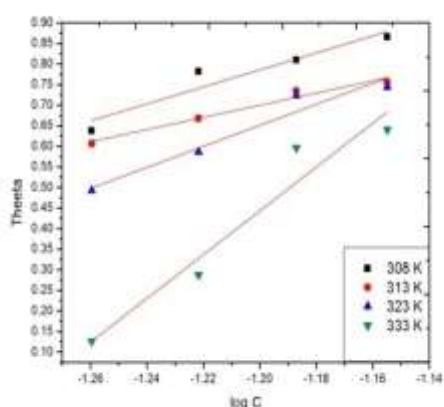
It is clear that the presence of tested compounds increases the activation energy and consequently decreases the corrosion rate of mild steel dissolution by making a barrier to mass and charge transfer by their adsorption on the mild steel surface. The values of ΔH* reflect the endothermic reaction of corrosion process. This suggests a strong adsorption of BTLE on the mild steel surface. The values of ΔS* in the absence and presence of tested compounds are negative which indicates that the activated complex in the rate determining step represents an association rather than dissociation [27]. This means that the activated molecules are in a higher order state than that at the initial state.

3.3. Adsorption isotherm

The adsorption characteristics of the studied inhibitors were investigated by fitting data obtained for the degree of surface coverage into different adsorption isotherms including Langmuir, Temkin, Frumkin, Freundlich, Florry Huggins, Bockris-Swinkel adsorption isotherms. However, the best fit indicates that the adsorption of BTLE on a mild steel surface is best described by the Temkin adsorption model, which is in good agreement with the equation:

$$\text{Log}(C/\theta) = \text{log K} + \text{log C}$$

where C is the concentration of the inhibitors in the bulk electrolyte and K is the equilibrium constant of adsorption. Figure 3 presents the temkin isotherms for the adsorption of the studied BTLE in 1N H₂SO₄. Values of adsorption parameters deduced from the isotherms are presented in Table 3. From the results obtained, the slopes and R² values for the plots are closer to unity, indicating that the adsorption of the studied BTLE is consistent with the Temkin adsorption model [28].

**Figure 3: Temkin adsorption isotherm for mild steel in 1N H₂SO₄ containing different concentrations of BTLE at 303-333 K.**

The equilibrium constant of adsorption deduced from the Temkin adsorption isotherm is related to the free energy of adsorption of the inhibitors as follows:

$$\Delta G_{\text{ads}}^0 = -RT \times \ln(55.5 K)$$

where K is the equilibrium constant of adsorption of water, ΔG_{ads}^0 is the free energy of adsorption, R is the gas constant and T is the temperature in Kelvin. Table 3 displays the equilibrium constants, interaction parameter and free energies for adsorption of various inhibitor concentrations. If the calculated ΔG_{ads} are negative and less than threshold value of -20 KJ/mol, it confirms that the adsorption of inhibitors on mild steel is spontaneous and that the physical adsorption mechanism is applicable [29-31].

Table 3: Temkin adsorption parameters and free energy of adsorption of BTLE as inhibitor on the surface of mild steel.

Temp (K)	K_{ads}	$-\Delta G_{\text{ads}}^0$ (K J/mol)	R^2
303	306	24.94	0.911
313	403	26.06	0.971
323	268	25.79	0.949
333	146	24.91	0.941

From Table 3, it is seen that all free energy values are negative indicating spontaneous adsorption of the inhibitors on mild steel surface. The range of free energy values are around 20 KJ mol⁻¹ indicating that the nature of adsorption is physical one for BTLE inhibitor in $1\text{N H}_2\text{SO}_4$.

3.4. Electrochemical impedance spectroscopy (EIS)

The corrosion of mild steel immersed in acid solution in the presence and absence of BTLE was investigated by EIS at 303 K after exposure period of 2 h. Figure 4 show Nyquist plots for mild steel in $1\text{N H}_2\text{SO}_4$ in the presence and absence of BTLE inhibitor in various concentrations. The impedance diagram (Nyquist) contains a depressed semicircle with centre under the real axis. Such behavior is characteristic for solid electrode which might be the result of surface roughness, dislocations, distribution of the active sites or adsorption of the inhibitor molecules [32].

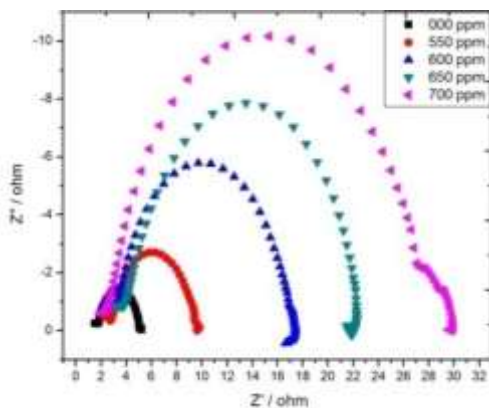


Figure 4 : Nyquist plots for mild steel immersed in $1\text{N H}_2\text{SO}_4$ (n) (inset) and different concentrations of BTLE at 303 K.

In $1\text{N H}_2\text{SO}_4$ solutions, the impedance spectra exhibit one single capacitive loop, which indicates that the corrosion of mild steel is mainly controlled by the charge transfer process. In $1\text{N H}_2\text{SO}_4$ acid solutions inhibition efficiency was increased with increasing the concentration of BTLE, [33] which indicates the strengthening of protective layer formed on the electrode surface and decreased the extent of dissolution reaction by BTLE molecules [34,35]. BTLE showed maximum inhibition efficiency (88.47% in H_2SO_4 at 700 ppm) at 303 K. After increasing the concentration of BTLE, no further change in charge transfer resistance and inhibition efficiency was observed. The impedance parameters such as R_s , R_{ct} and C_{dl} derived from Nyquist plots are given in Table 4. The inhibition efficiencies for each concentration were calculated using the formula:

$$\text{IE}\% = (R_{\text{ct}(\text{inh})} - R_{\text{ct}}) / R_{\text{ct}(\text{inh})} \times 100$$

where R_{ct} and $R_{ct(inh)}$ are charge transfer resistance in the absence and presence of inhibitor respectively. From the table, it is clear that charge transfer values increases and C_{dl} values decreases after the addition of inhibitor.

Table 4: Impedance parameters, rest potential and inhibition efficiency in the corrosion of mild steel in 1N H₂SO₄

Acid solution	C_{inh} (ppm)	R_s (Ω cm ⁻²)	R_{ct} (Ω cm ⁻²)	C_{dl} (F/cm ²)	% IE
H ₂ SO ₄	0	1.639	3.497	3.2×10^{-2}	
	550	1.651	8.386	7.1×10^{-3}	58.29
	600	1.338	16.684	1.6×10^{-3}	79.03
	650	2.378	20.102	1.0×10^{-3}	82.85
	700	0.955	30.348	5.1×10^{-4}	88.47

The decrease in C_{dl} values may be due to the adsorption of inhibitor on mild steel surface leading to the formation of protective film on mild steel surface [36, 37].

3.5. Polarization curve

Figure 5 shows potentiodynamic polarization curves of mild steel in 1N H₂SO₄ in the absence and presence of BTLE. It is understood that both anodic and cathodic reactions of mild steel corrosion are suppressed in the presence of BTLE in 1N H₂SO₄ and the suppression effect increases with the increase in the concentration of BTLE. Electrochemical kinetic parameters, i.e. corrosion potential (E_{corr}), cathodic and anodic Tafel slopes (β_c and β_a) and corrosion current density (I_{corr}), obtained from Tafel method are presented in Table 5. The inhibition efficiencies of BTLE in 1N H₂SO₄ is also given in Table 1. The inhibition efficiency is defined as:

$$IE\% = (I_{corr} - I_{corr(inh)})/I_{corr} \times 100$$

where I_{corr} and $I_{corr(inh)}$ are the corrosion current density values without and with inhibitor respectively.

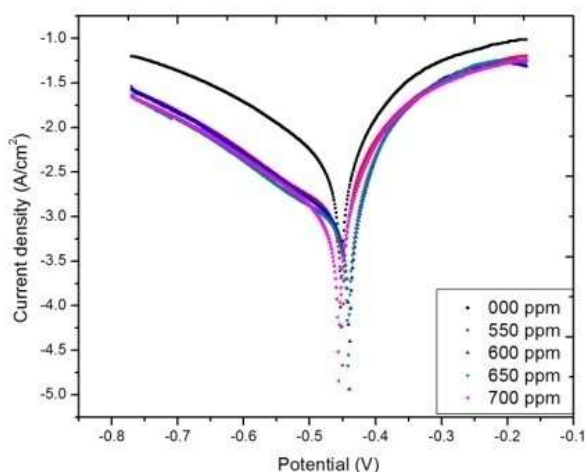


Figure 5 : Tafel plots of mild steel immersed in 1N H₂SO₄ with and without BTLE.

The decrease of both the anodic and cathodic current densities indicates that the inhibitor suppresses both the anodic and cathodic reactions.

Table 5: Potentiodynamic polarization parameters in the corrosion for mild steel in 1N H₂SO₄

Acid solution	C _{inh} (ppm)	E _{corr} (mV)	I _{corr} (μA cm ⁻²)	β _c (mV/decade)	β _a (mV/decade)	R _p (ohm cm ²)	% IE
H ₂ SO ₄	0	-445	1005	208.7	160.8	4	
	550	-450	300	200.5	143.9	12	70.14
	600	-440	182	200.9	134.3	19	81.89
	650	-442	172	207.7	127.2	20	82.88
	700	-456	159	194.2	128.4	21	84.17

This phenomenon may be due to the presence of BTLE on mild steel surface [38, 39].

The inhibitors change the values of β_a to greater extent. This suggests that the anodic reaction was controlled predominantly than cathodic reactions at all concentrations. This behaviour reveals that the inhibitor acts as by nearly blocking the reaction sites of the metal surface with changing the anodic and cathodic reaction mechanism [40]. The overall results suggest that the investigated inhibitor acts as mixed-type inhibitor [41, 42]. It is observed that the inhibition efficiency increases with increasing concentration of the inhibitors. Highest inhibition efficiency reaches a maximum value of 84.17 % in 1N H₂SO₄ at 700 ppm.

These results permit to conclude that:

- ☆ I_{corr} decreases with increasing inhibitor concentration, and addition of BTLE does not change the values of E_{corr} and β_c.
- ☆ IE increases with inhibitor concentration reaching a maximum value at 700 ppm of BTLE in 1N H₂SO₄.
- ☆ All the results obtained from weight loss, polarization and EIS studies are in good agreement.

3.6. Scanning electron microscopic studies

Figures.6 a and 6b show the SEM images of mild steel surface after immersion in 1N H₂SO₄ in the presence of BTLE for 2 h. After 2 h, The specimens were taken out and dried. SEM images showed that the surface of the inhibited of mild steel is formed more uniformly than the uninhibited sample. This examination indicated that the corrosion rate was reduced in the presence of inhibitors. This might be due to the adsorption of inhibitor molecules on the metal surface as a protective layer [43].

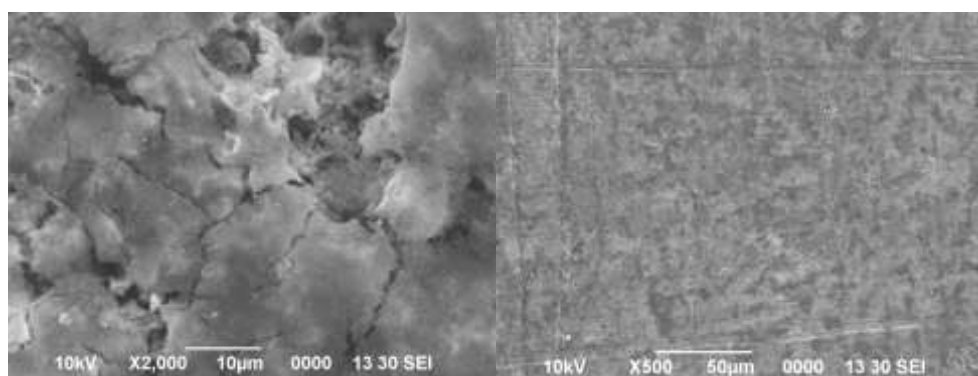


Figure 6: a) SEM images of mild steel surface after immersion of 1N H₂SO₄ with the absence of BTLE. b) SEM images of mild steel surface after immersion of 1N H₂SO₄ with the Presence of BTLE (in 700 ppm).

3.6. UV-Visible Spectroscopy

Corrosion inhibition of mild steel in acidic solution by BT leaves extract can be explained on the basis of molecular adsorption. It contains many organic components as per information provided in literature [44]. It is quite complex to separate every compound present in the extract. In order to confirm the possibility of the formation of BTLE-Fe complex, UV-Visible absorption spectra obtained for crude plant extract is shown in

Figure.7b and spectrum of mild steel immersed for 2 h, in solution containing 700 ppm concentration of BTLE in 1N H₂SO₄ is depicted Figure.7b. The electronic absorption spectra of BTLE crude extract display a main visible band at 279 and 203 nm. From Figure.7a, it was noted that the absorption spectra shifted to 308 and 206 nm. Change in position of the absorption maximum and or change in the value of absorbance indicate the formation of a complex between two species in solution [45]. On the basis of this fact, it can be concluded that the phytochemical components present in the BT leaves extract were adsorbed on the mild steel surface.

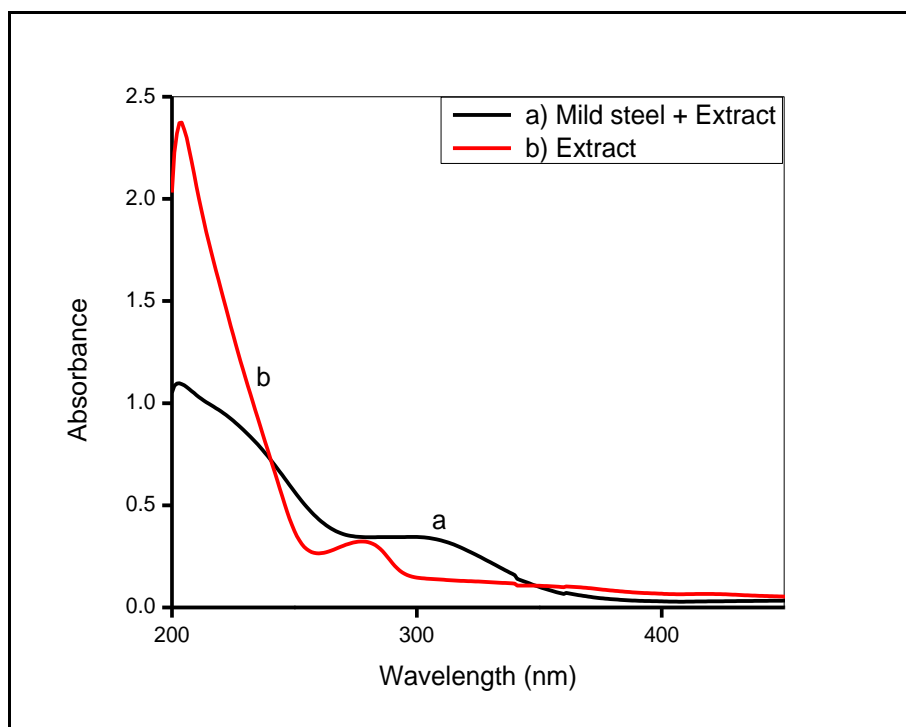


Figure.7. UV-visible spectra of the solution containing 1N H₂SO₄ before (b) and after the mild steel immersion in inhibitor (a)

Conclusion

The inhibition efficiency BTLE on mild steel in 1N H₂SO₄ increased with increasing the concentration of BTLE but decreased with increasing temperature. EIS measurement revealed that charge transfer increased with increase in concentration of BTLE, indicating that the corrosion inhibition increased with increase in concentrations. Tafel polarization measurements revealed that the BTLE behaves as mixed type inhibitor. The adsorption of the inhibitor obeyed the Temkin adsorption isotherm at all investigated temperatures. The adsorption was spontaneous and the inhibition of corrosion by BTLE was due to the formation of physical adsorption on the mild steel surface. Protective film formation against acid attack was confirmed by UV and SEM analysis. The results obtained from weight loss, polarization and impedance measurements are in good agreement.

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