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AlternantheraSessislis as Corrosion inhibitor of Mild Steel in 1N Mineral Acid Medium

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ABSTRACT

The weight loss measurement and electrochemical method studies on mild steel in 1N HCl using AlternantheraSessislis (AS) as inhibitor confirms that the corrosion was decreased by the addition of inhibitor. The adsorption of inhibitor on mild steel obeys Langmuir adsorption isotherm. The Electrochemical measurement shows that AS an act as a mixed type inhibitor and FTIR studies proves that the adsorption of inhibitor inhibits corrosion on mild steel surface.

KEYWORDS: *Corrosion inhibition, AlternantheraSessislis, Mild steel, Electrochemical measurement, Adsorption.*

I. INTRODUCTION

Corrosion processes are responsible for numerous losses mainly in industrial area. It is clear that the best way to combat it is prevention. Among the various methods to avoid or prevent destruction or degradation of metal surface, the corrosion inhibitor / rust preventives are one of most useful in the industry, due to low cost and practice methods [1-4]. Corrosion inhibitor may be used as liquids, powders, oils and concentrates, for application by dipping, brushing, spraying, wiping, slushing or wrapping.

In industries, rust is cleaned by using acid solution, so use of inhibitor is widely method for protection of corrosion. Now-a-days changes are occurred in using of synthetic corrosion inhibitors, due to its toxicity cause various environmental and health problems [5]. Therefore, lot of research has been developed on natural products such as plant extract to obtain environmentally friendly corrosion inhibitors [6].

This paper briefly presents the corrosion inhibition of plant extract of AlternantheraSessislis (AS). AS belongs to the family Amaranthaceae. This plant is commonly known as sessile joy weed or dwarf copperleaf. It is grown in tropical and subtropical regions. In India it is a popular folk medicine in often it is used to treat lung disorders [7]and also have multiple uses for the treatment of burning sensations, diarrhea, skin diseases, dyspepsia, hemorrhoids, liver and spleen diseases, and fever [8]. The AS acts as anti-allergic, anti-bacterial, anti-hyperglycemic, and anti-oxidant [9] agent.

II. EXPERIMENTS

The leaves of AS are collected, cleaned in running water and dried in absence of sunlight. 50g of dried AS leaves are refluxed in 1000ml of 1N HCl (AR grade) for 2hrs. The solution was left as it is overnight, filtered and made up to 1000ml. The made-up solution was used as stock solution (5% acid extract). The different concentrations ranging from 0.05, 0.1, 0.2, 0.4, 0.6, 0.8, and 1.0% are prepared and used to carry out for different studies.

The mild steel specimens with dimension of5 x 2 x 0.2 cm with 0.2 cm diameter hole at the top are used for weight loss measurement. They are smoothened with different grade emery paper, rinsed with double distilled water, cleaned with acetone/ethanol and dried in desicator to remove adsorption of moisture. The weight loss measurement was carried out by immersing the specimen in 100ml of 1N HCl and various inhibitor concentrations ranging from 0.05, 0.1, 0.2, 0.4, 0.6, 0.8, and 1.0% with various time intervals for 0.5, 1, 2, 4, 6, 8, 24, 48 and 72 hrs respectively at 298K. The effect of temperature on mild steel specimen was carried out at different temperature viz, 298K, 313K, 323K, 333K and 343K. Inhibition efficiency was calculated by following equation [10]

$$\mathrm{I.E}(\%) = \frac{W_1 - W_2}{W_1} \times 100 \qquad (1)$$

Where W_1 and W_2 are the weight loss of steel in blank and various concentrations of AS extract respectively. An electrochemical experiment was carried out using electrochemical analyzer (CHI608E instrument) to study the corrosion process at room temperature without any agitation. The three-electrode set up contains mild steel (1 cm^2 exposed area) as working electrode. Ag-AgCl as reference electrode and Platinum electrode as counter electrode. The working electrode was made by coating the mild steel with polyester to create 1 cm^2exposed area for corrosion study. The EIS measurement was carried out in frequency range from 1Hz to 106Hz by applying 0.01V sine wave ac voltage. The charge transfer resistance (R_{ct}) values were obtained from diameter of semicircle of Nyquist plots and double capacitance were calculated by following equation,

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

Where f_{max} is the maximum frequency of Nyquist plot and R_{ct} is charge transfer resistance. The inhibition efficiency is calculated from charge transfer resistance values using following equation R'_{ct}- R^o_{ct}

$$\eta\% = \frac{R'_{ct} - R^{\circ}_{ct}}{R'_{ct}} \times 100 \qquad (3)$$

Where R^o_{ct} and $R'_{ct}$are the charge transfer resistance of blank and various concentrations of AS extract respectively.

The Potentiodynamic polarization measurements is carried out by changing electrode potential from -200 to 200 mV corrosion potential for sweep rate at 10 mVs^{-1}. The values of corrosion current densities (I_{corr}), E_{corr}, anodic slope and cathodic slope are computed. The inhibition efficiency is calculated by following equation.

$$\eta\% = \frac{I^{\circ}_{corr} - I_{corr}}{I^{\circ}_{corr}} \times 100 \qquad (4)$$

Where I^o_{corr} and I_{corr} are corrosion current density values for blank and various concentration of AS extract respectively.

The surface morphology of mild steel in 1N HCl solution for blank and in the presence of inhibitor was studied by SEM analysis.

III. Result and Discussion

A. Weight loss method

Weight loss measurement was studied for mild steel sample in 1N HCL using AS extract as an inhibitor. The studies were carried out for blank and different concentrations of inhibitor (0.5, 0.1, 0.4, 0.6, 0.8 and 1.0%).

B. Effect of inhibitor concentration:

From this study it has been noted that inhibition efficiency raised with raising of inhibition concentration. The 1E reaches maximum value 98.47% at concentration 1.0 ppm of AS extract. It means that AS extract on mild steel sample in 1N HCl was inhibited by adsorption against corrosion and corrosion rate decreases by raise in inhibitor concentration.

C. Effect of immersion time

To study the stability of inhibitor weight loss measurement was carried out for blank and various concentration of inhibitor with different immersion time of 0.5 to 72 h in 1N HCl medium. Figure 1 shows inhibition efficiency of AS extract with various times. It shows that inhibition efficiency raises with raising of immersion period and reaches maximum efficiency 98.47%. It means that maximum adsorption of AS extract on mild steel happened at 72 hrs.

D. Effect of temperature

By weight loss measurements, the effect of temperature on inhibition efficiency of ASextract for various concentration on mild steel in 1N HCl were studied, to know the stability of adsorbed layer of inhibitor on mild steel. The inhibition efficiency and corrosion rate of AS extract for all concentrations on mild steel at different temperature ranging from298 to 343K with 1hr immersion time are tabulated in Table 1. It shows that inhibition efficiency raise with raised in temperature due to increase in rate of dissolution of mild steel and chemical adsorption of inhibitor on the metal surface with temperature [14]. Table 2 shows that corrosion rate increase with raise in temperature for blank solution and slight increase for various concentration of AS extract.

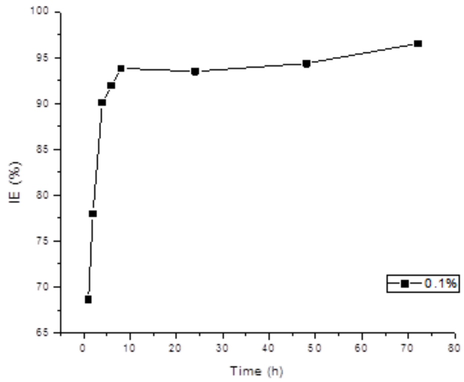


Figure1. Inhibition efficiency of inhibitor Vs Time

Table 1. Inhibition efficiency of mild steel in 1N HCl using Alternantherasessislis extract at 1h immersion time.

Conc. (%)	Inhibition efficiency (%)				
	298K	313K	323K	333K	343K
0.0	-	-	-	-	-
0.05	72.50	80.65	81.55	85.06	84.72
0.1	68.63	81.07	82.02	85.83	89.93
0.2	73.75	85.19	82.18	80.96	83.79
0.4	78.26	84.77	83.75	87.94	82.96
0.6	87.50	87.65	87.38	87.94	83.36
0.8	75.50	88089	94.64	89.96	83.72
1.0	75.50	95.89	96.21	94.03	86.28

Table 2. Corrosion rate of mild steel in 1N HCl using Alternantherasessislis extract for different temperature and concentration.

Conc., (%)	Corrosion rate (mg cm^{-2}h^{-1})				
	298K	313K	323K	333K	343K
0.0	0.0446	0.1356	0.3537	0.8793	1.6795
0.05	0.0139	0.0262	0.0653	0.2193	0.7605
0.1	0.0123	0.0257	0.0636	0.2126	0.6829
0.2	0.0117	0.0201	0.0630	0.1674	0.6081
0.4	0.0047	0.0206	0.0575	0.1060	0.2862
0.6	0.0056	0.0167	0.0446	0.1060	0.2751
0.8	0.0106	0.0151	0.0190	0.091	0.0734
1.0	0.0106	0.0006	0.0134	0.0524	0.2304

Arrhenius equation [15,16,17] gives the relationship between the corrosion rate CR of mild steel sample and temperature.

$$C_R = K \exp(-E_a/RT) \qquad (5)$$

Where E_a is the activation energy. K is the pre-exponential factor; R is the gas constant and T is the absolute temperature. The graph of log $C_R$vs 1/T is shown in Figure 2. From the slope and intercept values, the E_a and K values for blank and various concentrations of AS extract were computed and tabulated in Table 3. This shows that increase in the activation energy (E_a) in presence of AS extract is due to the adsorption occurs in the first stage [18].

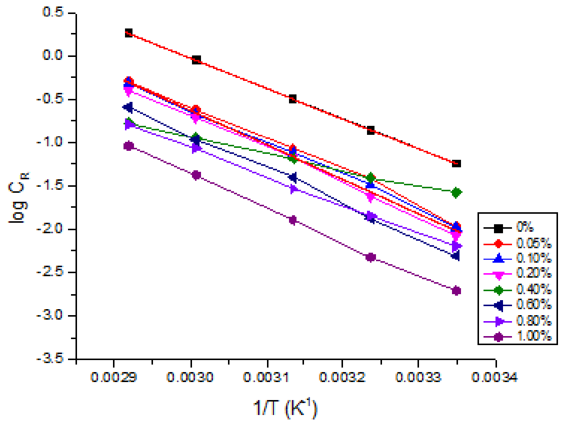


Figure 2.log CR Vs 1/T

Table 3. Activation parameter for mild steel mild steel in 1N HCl using Alternantherasessislis extract different concentration

Conc. (%)	Ea* KJ mol-1	ΔH* KJ mol-1	ΔS* KJ mol-1
0.0	70.20	231.04	-196.89
0.05	76.84	258.10	-196.84
0.1	77.24	260.30	-196.83
0.2	76.08	260.30	-196.85
0.4	36.83	134.54	-197.21
0.6	74.41	258.96	-196.86
0.8	61.95	216.88	-196.99
1.0	75.77	255.58	-197.13

An alternate formulation of Arrhenius equation [19].

$$C_R = RT/Nh \exp(\Delta S^*/R) \exp(-\Delta H^*/RT) \qquad (6)$$

Where h is a Planck constant, N is Avgadros number, ΔS* is the entropy of activation, and ΔH* the enthalpy of activation. The graph of log CR Vs1/T is drawn and ΔS* & ΔH* are calculated from intercept and slope values of the graph respectively are tabulated in Table 3 It shows that ΔH*=+ve values for blank and various concentration of AS extract. It is due to the endothermic nature of mild steel dissolution process [19]. The values of ΔS* is negative for blank and various concentration of AS extract, attributed to the activated complex in rate determining step represents an association rather than dissociation. The corrosion decreases due to adsorption of inhibitor molecule and formation of ordered stable layer of inhibitor on mild steel surface [20, 21].

E. Adsorption isotherm

Adsorption isotherms were studied to know the interaction between inhibitors and mild steel surface and to understand the mechanism of adsorption reaction. For this purpose, the surface(θ) values are calculated using the formula [22]

$$\theta = (W_1 - W_2)/W_1 \qquad (7)$$

Where W_1 and W_2 are the weight loss in absence and presence of inhibitor respectively. Attempts were made to know the best isotherm. The best fit obtained was Langmuir isotherm as suggested by graph C/θvs C shown in Figure3 and slope value is equal to unity, which means that adsorption of the inhibitor molecules obeys the Langmuir's adsorption isotherm as expressed in [23].

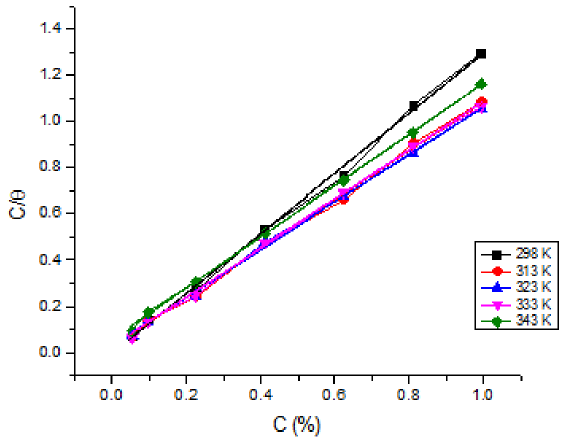


Figure 3. C/θ*vs* C

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (8)$$

Where C is the inhibitor concentration and K_{ads} is equilibrium constant for adsorption/ desorption process of the inhibitor molecules on the metal surface. K_{ads}values were calculated from the intercept of the graph for adsorption process.

The adsorption equilibrium constant, K_{ads} is related to the standard free energy change (ΔG^0_{ads}) by following equation.

$$K_{ads} = \frac{1}{55.5}\exp\left(\frac{-\Delta G_{ads}}{RT}\right) \qquad (9)$$

$$\log K_{ads} = \log\left(\frac{1}{55.5}\right) - \frac{\Delta H_{ads}}{2.303\,RT} + \frac{\Delta S_{ads}}{2.303\,R} \qquad (10)$$

The graph of logK_{ads}vs 1/T, shown in Figure 4. The thermodynamic parameters such as ΔH_{ads}, $\Delta S_{ads}$and ΔG_{ads} are calculated. The ΔH_{ads}, ΔS_{ads}values are calculated from the slope and intercept of the graph and tabulated in Table 4, the negative value of ΔG_{ads} means that the adsorption is spontaneous process, which have strong interaction of inhibitor molecule on mild steel also the value of ΔG_{ads} confirmed that the adsorption process is chemisorptions[24, 25]. The negative value of $\Delta H_{ads}$is found to be -49.47KJmol^{-1}, which further confirmed that the chemical adsorption takes place on mild steel surface. The negative value of ΔS_{ads} means that activated complex is rate determining step represents an association rather than dissociation step. The disordering takes place in going from reactants to metal-adsorbed species reaction complex [26 - 29].

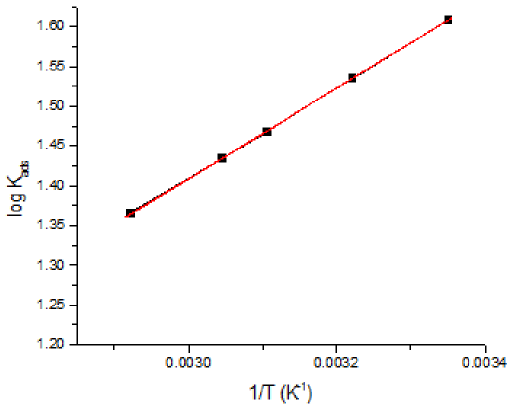


Figure 4.logK_{ads}vs 1/T

Table 4. Thermodynamic parameter for mild steel mild steel in 1N HCl using Alternantherasessislis extract

Inhibitor	ΔH_{ads} KJ mol-1	ΔS_{ads} KJ mol-1	ΔG_{ads} KJ mol-1
Alternantherasessislis	-49.476	-88.69	-23.05

F. Electrochemical impedance studies

The investigation was carried for mild steel in 1N HCl solution for blank and various concentrations of AS extract by EIS at room temperature. The Nyquist plot was obtained for blank and various concentration of ASextract are shown in Figure 5. It shows that depressed semi-circle with the center below the real x axis, and the size of semicircle is increased by raising the concentration of inhibitor, which means that corrosion is mainly due to charge transfer process [30] and film formed is strengthened by addition of extract. A loop formed at low frequency, is due to intermediate product adsorbed such as [$FeCl^-$] in absence and/or ($FeCl^-lnh^+$) in presence of inhibitor [31]. Also, the raising the concentration of AS extract does not alter the style of impedance curves, which means that similar inhibition mechanism is occurred.

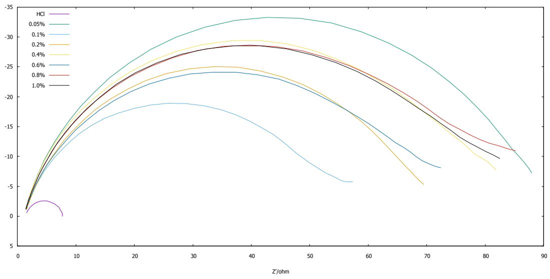


Figure 5. Impedance diagram of mild steel in 1N HCl using Alternantherasessislis extract for different concentration.

The impedance parameters are calculated and tabulated in Table 5. the table shows that R_{ct} values increases and double – layer capacitanceC_{dl}decreases with raise in inhibitor concentration. It is due to the reduction of local dielectric constant and / or increase in the double layer thickness formed at acid / metal interface with adsorption of inhibitor at mild steel surface [32]. As well as increase in charge transfer resistance R_{ct} and inhibition indicates that formation of protective layer at steel /acid interface with raise in concentration of inhibitor.

Table 5. Impedance parameters for mild steel in 1N HCl using Alternantherasessislis extract

Con (%)	Rct(Ωcm2)	Cdl (μF/cm2)	I.E (%)
0.0	6.247	9.911	--
0.05	87.163	0.055	92.83
0.1	55.955	0.1565	88.88
0.2	67.964	0.0937	90.81
0.4	80.257	0.0676	92.22
0.6	71.09	0.0933	91.12
0.8	83.703	0.0666	92.54
1.0	81.197	0.0681	92.30

G. Potentiodynamic polarization studies

The investigation was carried for mild steel in 1N HCl solution for blank and various concentration of AS extract by Potentiodynamic polarization at room temperature. The Tafel polarization curves were obtained for blank and various concentration of AS are shown in Figure6. The Tafel polarization parameters are computed and tabulated in Table 6. The table shows that the addition of inhibitor to acid media affected both cathodic and anodic part of curves. As anode is more polarized, the process of metal dissolution is more inhibited and it also noted that the maximum shift in potential was found as 38 mV, which means that AS inhibitor acts as a mixed type inhibitor in the present work [33, 34]. Also, the Tafel curve shows that the curve shifted to lower current density region and there is no significant change in b_c and b_a values [35, 36]. The increase in inhibition efficiency with raiseof concentration of ASinhibitor indicates that the AS extract acted as very effective corrosion inhibitor for mild steel in 1N HCl solution.

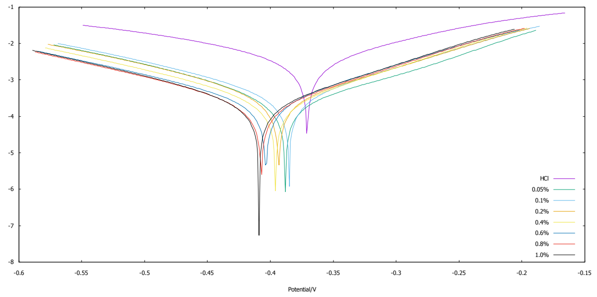


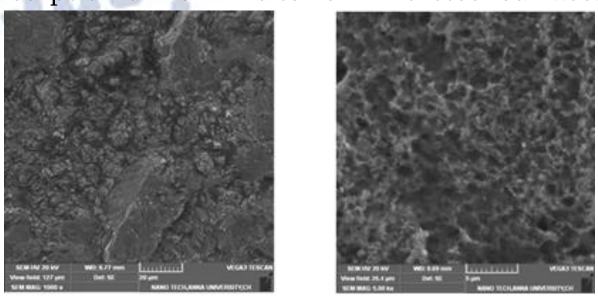
Figure 6. Potentiodynamic Polarization curves for mild steel in 1N HCl using Alternantherasessislis extract for different concentration at 10 mV sweep rate.

Table 6.Potentiodynamic polarization parameters for mild steel in 1N HCl using Alternantherasessislis extract for different concentration at 10mV sweep rate.

Con (%)	Ecorr(mV)	IcorrmA/cm2	b_c	b_a	Linear potential	IE %
0.0	-371	3.579	7.937	6.307	8.5	-
0.05	-388	0.2526	8.899	10.029	90.90	92.94
0.1	-385	0.3420	8.562	10.476	66.70	90.44
0.2	-393	0.3047	8.626	10.429	74.90	91.49
0.4	-396	0.2372	8.536	11.151	93.10	93.37
0.6	-404	0.2292	8.32.	10.779	99.30	93.59
0.8	-407	0.1968	8.465	11.02	113.40	94.50
1.0	-409	0.2040	8.642	11.097	108.00	94.30

H. Scanning Electron Microscope

Figure7 shows the image of SEM for mild steel exposed to 1h in 1N HCl solution (a) blank, (b) AS extract at room temperature.Figure7 (a) clearly shows that surface of mild steel immersed in blank is rough due to high corrosion and Figure7 (b) shows that mild steel immersed in AS inhibitor in smooth surface without any hole, crevice, crack and etc, means that the corrosion is reduced due to adsorption of AS inhibitor on mild steel surface.



(a) (b)

Figure 7. Scanning electron micrograph of mild steel after exposure to (a) 1 N HCl and (b) 0.1% Alternantherasessislis extract.

I. Fourier Transform infrared spectroscopy

The Fourier transform infrared spectroscopy was studied for further confirmation of corrosion inhibition of mild steel in acid media. The FTIR spectroscopy obtained is shown in Figure 8 and spectral peaks obtained are Tabulated in Table 7. It clearly shows that corrosion is retarded by adsorption of inhibitor molecule on mild steel surface, from the prominent peak at 480 cm^{-1} observed for Fe – C stretching.

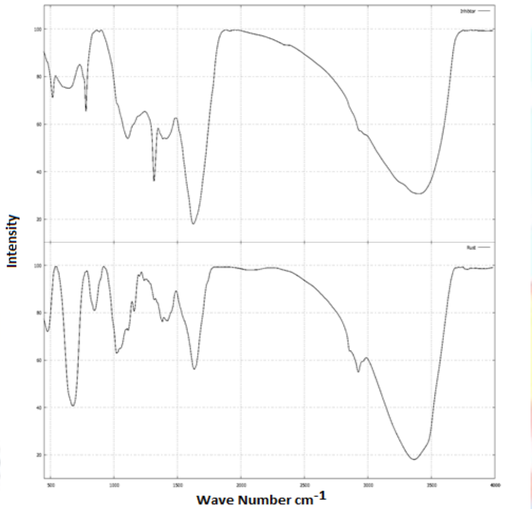


Figure 8.FTIR spectra obtained for Alternantherasessislis extract and Rust powder

Phytochemical studies of AS leaves show that for presence of saponins, tannins, alkaloids, and flavonoids. The structures of compounds are shown in Figure9. Each compound having multiple bonds, which means that structural compound is responsible to hide mild steel surface by adsorption / film formation and prevent corrosion [37]. Thus, AS leaves extract act as an excellent corrosion inhibitor on mild steel in 1N HCl medium.

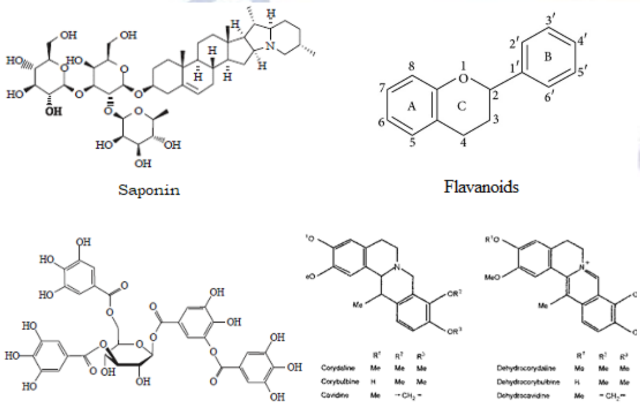


Figure 9. Structure of main constituent of Alternantherasessislis extract.

Table7. FTIR peak assignments for Alternantherasessislis extract and rust

Band assignment	wave number (cm^{-1})	
	Before inhibition	After inhibition
O-H stretching & asymmetric stretching	3405	3369
C-H stretching	---	2927
C=O stretching	---	1711, 1632
C-O/C-H bending	1414	1424
CH3C-H bending	1386	---
O- H stretching	1316	1367
C-O stretching	1109	---
C-Cl stretching	---	848
= C-H bending	780	---
≡ C-H bending	---	681
C-H bending	650	---
C-Br stretching	517	---
Fe-C stretching	---	487

VI. CONCLUSION

From the results obtained from the above studies we conclude that.

- The increase in concentration ofASextract increases the inhibition efficiency of up to 98.47%.
- The corrosion was retarded by chemisorption following Langmuir adsorption isotherm.
- The electrochemical studies prove that Alternantherasessislis extract acts as amixed type inhibitor.
- FTIR and SEM analysis showed that the corrosion is prevented by formation of Alternantherasessislis extract film on mild steel surface.

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