

Impeding Acid Corrosion of Mild Steel using *Green Inhibitor*

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ABSTRACT: Natural resources are biodegradable in nature as well as a rich source of active chemical constituents which are well known for their anti-corrosive property for metals. *Lawsonia inermis* leaves are found to be rich in most of these organic compounds in addition to lawsone (2-hydroxy-1, 4-naphthoquinone), gallic acid, ascorbic acid, 1, 4-naphthoquinone, mannitol etc. The inhibitive action and adsorptive behaviour of *Lawsonia inermis* leaves (EELiL) on the industrially used metals, viz. mild steel was explored by employing weight loss measurements in 0.5 M HCl. The inhibition efficiency depends on the mechanical, structural and chemical characteristics of the adsorption layers formed under particular conditions. Kinetic/ Thermodynamic treatment and surface morphological analyses (FT-IR, UV, SEM) were carried out to endorse the inhibitive and adsorptive tendency of the *Lawsonia inermis* leaves. The metal dissolution was mitigated to a very significant extent. Thus, these promising results lead us to explore the applications the green inhibitor in metal surface anodizing and surface coatings.

KEYWORDS: *Lawsonia inermis* leaves, Acid corrosion, Kinetic and Thermodynamic parameters, FT-IR spectroscopy, UV, SEM.

I. INTRODUCTION

Metallic corrosion in aggressive media (like acidic and basic media etc.) causes considerable loss. In order to combat the metallic corrosion, several techniques have been applied. Metals such as iron and copper, brass and aluminium are very useful materials for engineering applications due to their low cost, easy availability, light weight, high thermal and electrical conductivity, and mechanical strength. However, mild steel is severely affected when these exposed to the action of acid in industrial process where acids play an important role in industrial processes like acidizing, pickling, cleaning and decaling. So the use of metal corrosion inhibitors is one of the most appropriate methods for protection of metal against corrosion in acidic media. It has been well established that the organic compounds that contains heteroatoms such as oxygen, nitrogen, sulphur, and phosphorus behaves to be most effective inhibitors as they get adsorbed on the metal surface. Now a day's natural products are used as corrosion inhibitor, because natural products have property of environmental safety, low cost, non toxicity, easily availability and biodegradable also. Moreover green inhibitors are the rich source of active chemical constituents having effective corrosion inhibitive property [1-6]. The ethanolic extract of *Lawsonia inermis* leaves was used to evaluate its anti-corrosive property on corrosion of mild steel in 0.5 M HCl by chemical method. The inhibitive tendency of *Lawsonia inermis* is attributed to its major chemical constituent 'lawsone'(2-hydroxy-1,4-naphthoquinone). Ethanolic extract of the leaves of *Lawsonia inermis* also contains carbohydrates, flavanoids, amino acids / proteins, steroids, saponins, tannins, β - sitosterol, lawsone, gallic acid, ascorbic acid, apigenin, luteolin, 2-methoxy-3-methyl-1,4-naphthoquinone, p-coumaric acid [6, 7-12].

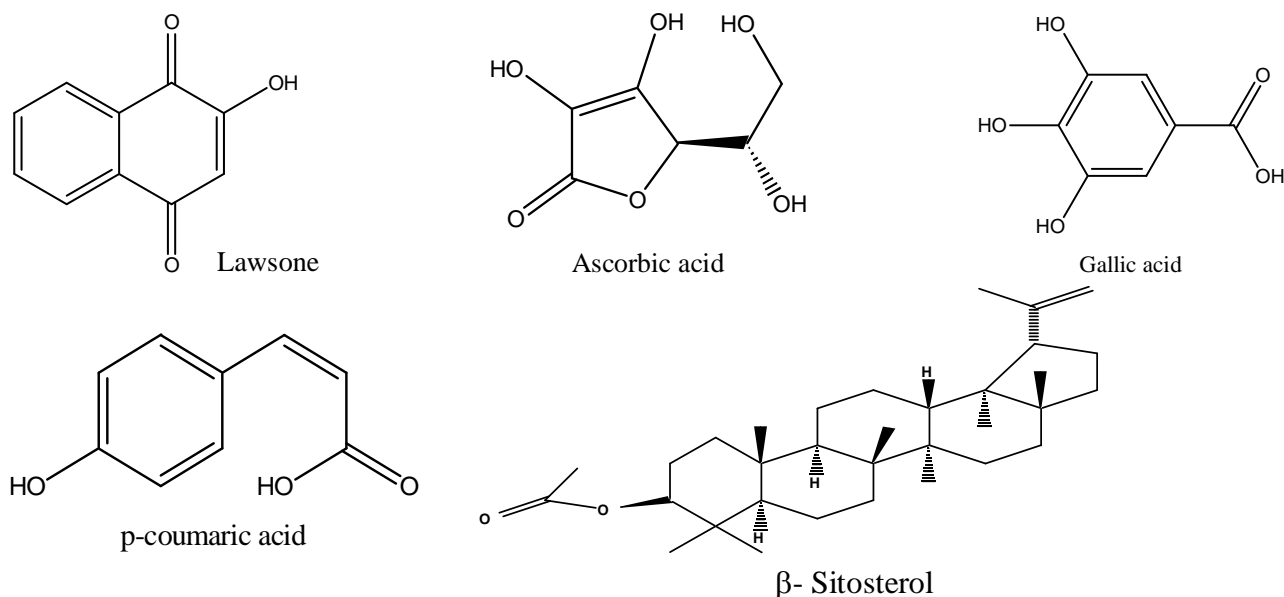


Fig. 1: Some of the major active constituents of *Lawsonia inermis*.

II. EXPERIMENTATION

(A) Preparation of test coupons:

Standard sized coupons of mild steel were used, with a hole of uniform diameter of 0.12mm to facilitate suspension of the coupon in the test solution [13-20]. Prior to experimentation, each coupons was surface treated, dried and stored in desiccators and thereafter, accurate weight of the samples was taken using electronic balance. The XRF studies were carried out at Pay Analytical X-ray fluorescence instrument in the frequency range of 25 KV to assess the exact elemental composition of the test coupons (figures 2). The highest peak in each graph shows the purity of Fe (99.7%) in MS coupons.

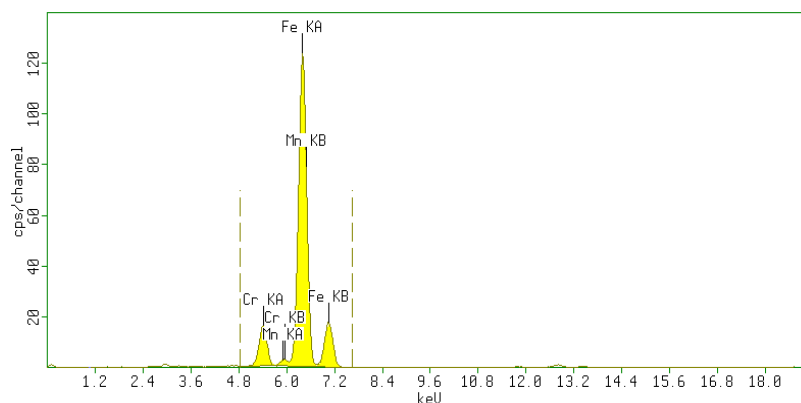


Fig.2: XRF graph of Mild Steel coupon

(B) Preparation of *Lawsonia inermis* extract:

Leaves of *Lawsonia inermis* were air dried in shade, grained and powdered which was soaked with sufficient quantity of distilled ethyl alcohol for desired number of day. After completion of soaking period, the solution was refluxed using Soxhlet. It is distilled and then filtered to remove any suspended impurities. The stock solution of the extract was stored in a clean corked bottle for further use [13-20].

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(C) Preparation of test solutions:

Standard solution of 0.5M HCl was prepared using doubly distilled water. 50 ml of 0.5M HCl was taken in seven separate 100ml beakers labelled as MS-0, MS-1, MS-2, MS-3, MS-4, MS-5, and MS-6. EELiL was added in the order of increasing concentration so as to have 0.0312, 0.156, 0.312, 0.468, 0.78 and 1.092g/L respectively and corrosive medium (blank) in the beaker labelled as MS-0. Pre-weighed MS coupons were suspended in aggressive media without and with inhibitor in different concentrations (0.0312 to 1.092 g/L) for varied immersion period (3 to 72h). After completion of prescribed immersion period, MS coupons were removed, thoroughly washed with double distilled water, dried well in desiccators and then weighed accurately [13-20].

(D) Quantum Chemical Calculations:

Quantum Chemical Analyses were carried out by using the PM3 method of the quantum chemical. The following parameters were evaluated, i.e. the energy of the highest occupied molecular orbital (E_{HOMO}), the energy of the Lowest unoccupied molecular orbital (E_{LUMO}), energy band gap, $\Delta E = E_{\text{HOMO}} - E_{\text{LUMO}}$, binding energy, heat of formation and the dipole moment (μ), point group of molecule [20].

(E) Surface Morphological Analyses:

i. UV-visible Spectroscopy

In order to confirm the possibility of the formation of EELiL-Fe complex, UV-Visible absorption spectra obtained on (8400 Shimadzu, Japan) with help of quartz glass cell on 400-800 nm visible range. Spectra's were recorded for extract of EELiL and MS test coupon dipped in 0.5 M HCl with 1.092 g/L concentration of EELiL.

ii. FT-IR Spectroscopy

The FT-IR spectra of the protective film onto the MS test coupon and EELiL were recorded by employing KBr pellets method using Spectrophotometer (8400 Shimadzu, Japan) in the range of 4000 to 400 cm^{-1} .

iii. SEM Studies

The surface morphology of the coupons without and with different concentrations of EELiL in 0.5 M HCl has also been analysed by employing Scanning Electron Microscope (ZEISS) and Optical microscope (LABOMED).

III. RESULT AND DISCUSSION

The inhibitive effect of additive (inhibitor) at various immersion period and at its different concentrations on acid corrosion of mild steel has been investigated at room temperature. The experimental data were used to evaluate various corrosion parameters (Table 1). It was found that inhibitor very significantly impeded the acid corrosion of MS in its concentration range 0.0312 to 1.092g/L.

i. Effect of exposure period and concentration of EELiL:

The effect of immersion time on inhibition efficiency has been shown in Fig. 5. It has been observed that IE % increases as immersion period was increased and highest IE % (88.26 %) was seen at 72h with highest concentration of inhibitor (1.09 g/L). Similarly, the effect of change in concentration of EELiL on IE % has been shown in Fig. 4, which clearly depict that IE % also increases with increase in concentration of inhibitor. The significant IE % of *Lawsonia inermis* leaves' extract may be attributed due to the formation of a barrier film adsorbed onto the metal surface involving interactions between π electrons, lone pair of O- of inhibitor's active molecules and vacant d-orbitals of both the metal surface atoms [20-26]. The leaves of *Lawsonia inermis* are richly constituted with the major active anti-corrosive compounds, such as, lawsone, gallic acid, tannic acid, ascorbic acids, quinones, alkaloids, apigenin, luteolin, 2-methoxy-3-methyl-1, 4-naphthoquinone, p-coumaric acid, etc. with strong tendency to get adsorbed onto the metal surface thereby forming a protective barrier film. Lawsone (2-hydroxy-1,4-naphthoquinone) is a strong chelating agent which usually forms a chelate ring with metal cation. Therefore, the formation of insoluble complexes, by chelating of the metal cations with the lawsone molecules adsorbed on the metal surface, it is a most probable interpretation of the observed inhibition action of lawsone. The chelating behaviour of lawsone could be the best causes for the high inhibition efficiency in acidic media for mild steel [21-26].

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Table-1: Corrosion parameters for MS in 0.5M HCl without and with different concentrations of *Lawsonia inermis* (EELiL) at 303±K at various exposure time (h).

Time (h)	EELiL Concentration (g/L)	Corrosion parameters				
		Corrosion rate (ρ_{Corr}) (mm y^{-1})	IE %	Functional surface coverage (Θ)	Adsorption Equilibrium Constant (K_{ad})	Gibbs free energy (ΔG)
3	MS0 0.000	17.70				
	MS1 0.031	10.95	38.12	0.38	19.74	-17.63
	MS2 0.156	10.27	41.97	0.42	4.64	-13.98
	MS3 0.312	6.66	62.35	0.62	5.31	-14.32
	MS4 0.468	6.09	65.59	0.66	4.07	-13.66
	MS5 0.78	5.37	68.21	0.68	2.75	-12.67
	MS6 1.09	3.99	77.47	0.77	3.15	-13.01
6	MS0 0.000	14.07	0	0		
	MS1 0.031	8.10	42.43	0.42	23.62	-18.09
	MS2 0.156	7.02	50.10	0.50	6.44	-14.81
	MS3 0.312	4.22	70	0.7	7.48	-15.19
	MS4 0.468	3.96	71.84	0.72	5.45	-14.39
	MS5 0.78	3.97	71.75	0.72	3.26	-13.10
	MS6 1.09	3.41	75.73	0.76	2.86	-12.76
12	MS0 0.000	11.22	0	0		
	MS1 0.031	5.09	54.66	0.55	38.63	-19.32
	MS2 0.156	3.81	66.04	0.66	12.46	-16.47
	MS3 0.312	2.72	75.78	0.76	10.03	-15.93
	MS4 0.468	2.96	73.59	0.74	5.95	-14.61
	MS5 0.78	2.40	78.58	0.79	4.70	-14.02
	MS6 1.09	2.28	79.67	0.80	3.59	-13.34
18	MS0 0.000	10.10	0	0		
	MS1 0.031	4.09	59.47	0.59	47.02	-19.82
	MS2 0.156	2.73	72.95	0.73	17.29	-17.30
	MS3 0.312	2.05	79.71	0.80	12.59	-16.50
	MS4 0.468	1.69	83.23	0.83	10.60	-16.07
	MS5 0.78	1.75	82.64	0.83	6.10	-14.68
	MS6 1.09	1.64	83.77	0.84	4.73	-14.03
24	MS0 0.000	8.58	0	0		
	MS1 0.031	3.25	62.13	0.62	52.59	-20.10
	MS2 0.156	2.32	72.91	0.73	17.25	-17.29
	MS3 0.312	1.71	80.03	0.80	12.85	-16.55
	MS4 0.468	1.41	83.61	0.84	10.90	-16.14
	MS5 0.78	1.41	83.61	0.84	6.54	-14.85
	MS6 1.09	1.29	84.96	0.85	5.17	-14.26
48	MS0 0.000	5.65	0	0		
	MS1 0.031	2.27	59.77	0.60	47.62	-19.85
	MS2 0.156	1.55	72.64	0.73	17.02	-17.26
	MS3 0.312	0.95	83.15	0.83	15.81	-17.07
	MS4 0.468	0.80	85.83	0.86	12.95	-16.57
	MS5 0.78	0.94	83.33	0.83	6.41	-14.80
	MS6 1.09	0.67	88.10	0.88	6.78	-14.94
72	MS0 0.000	3.94	0	0		
	MS1 0.031	1.60	59.50	0.59	47.08	-19.82
	MS2 0.156	1.09	72.40	0.72	16.82	-17.23
	MS3 0.312	0.68	82.73	0.83	15.35	-17.00
	MS4 0.468	0.57	85.51	0.85	12.61	-16.50
	MS5 0.78	0.66	83.31	0.83	6.40	-14.79
	MS6 1.09	0.46	88.26	0.88	6.89	-14.98

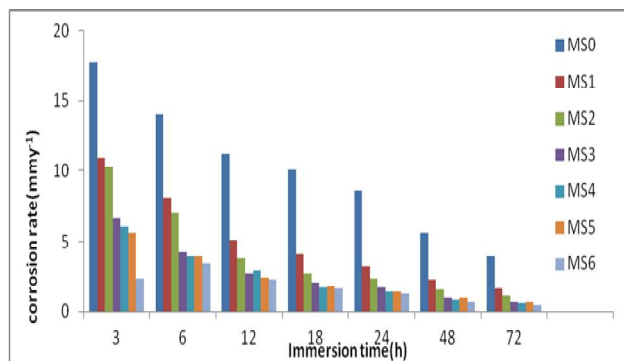


Fig. 3: Corrosion rate vs. Immersion period (h) without and with EELiL.

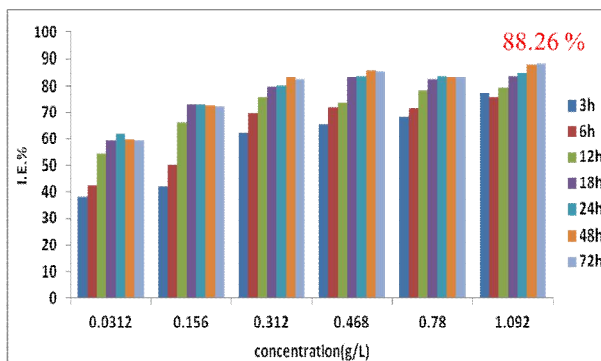


Fig. 4: IE % vs. Concentration (g/L) with EELiL.

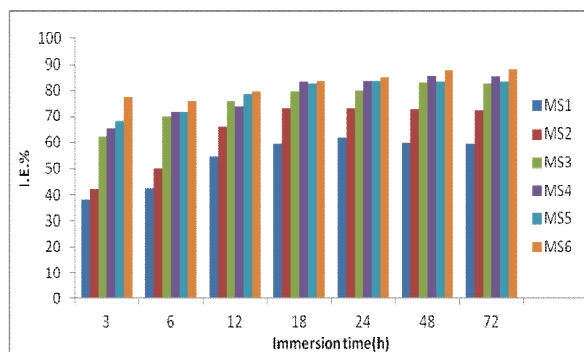


Fig. 5: IE% vs. Immersion period (h) with various concentration of EELiL.

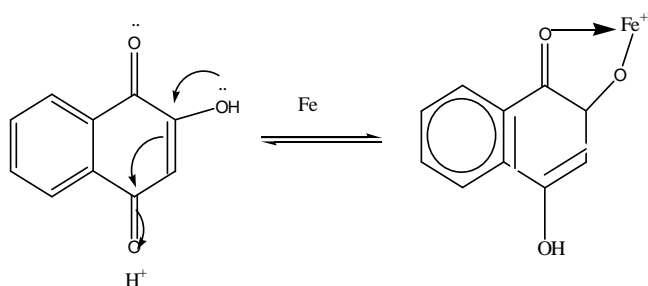


Fig. 6: Proposed adsorptive mechanism for Lawsone on MS coupon.

ii. Adsorption isotherm:

The efficacy of an inhibitor as a successful anti-corrosive agent is mainly dependent on its ability to get adsorbed on the metallic surface. The interaction between the active molecules of the inhibitor and metal surface can be assessed by adsorption isotherm. Nature of adsorption was established by fitting θ values into different adsorption isotherm models.

Table-2: Langmuir adsorption isotherm parameters for EELiL onto MS surface.

S. No.	Immersion Period (h)	Correlation coefficient (R^2)	Slope
1	3	0.960	1.11
2	6	0.996	1.28
3	12	0.999	1.23
4	18	0.999	1.18
5	24	0.999	1.16
6	48	0.998	1.13
7	72	0.998	1.12

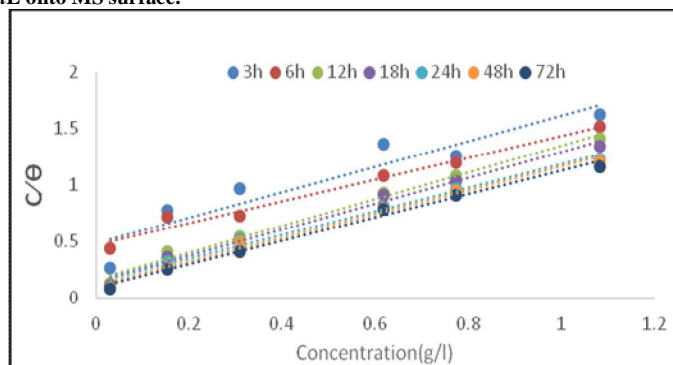


Fig. 7: Plot C/θ vs. C (concentration of EELiL)

Linear correlation was observed between C/θ vs C with slope unit (fig. 7) (table-2) with correlation coefficients (R^2) observed as $0.999 \geq r^2 \geq 0.960$. Langmuir adsorption isotherm was best fitted which is expressed as:

$$(C / \theta) = C + 1 / K_{ad} \quad (1)$$

where, C is the concentration of the inhibitor (EELiL), θ is the fractional surface coverage, and K_{ad} is the adsorption equilibrium constant. Langmuir's isotherm shows that the solid surface contains a fixed number of adsorption sites and each site holds one adsorbed species [27-29].

iii. Quantum chemical analysis:

Quantum chemical calculations have been widely used to study reaction mechanisms as well as it was carried out in order to justify adsorptive and inhibitive mechanism of various inhibitor molecules. Through these calculations, an attempt has been made to evaluate the binding energy of the inhibitor molecules present in ethanolic extract of *Lawsonia inermis* and some independent variables like energy of HOMO, LUMO, and dipole moment etc. were also calculated and tabulated (table-3). The optimized geometry of these active constituents of *Lawsonia inermis* leaves has been depicted in figures 8-10{(A-1), (B-1), (C-1)}.

It is important to focus on the parameters that directly influence the electronic interaction of the inhibitor molecules with the metal surface. E_{HOMO} {figures 8-10 (A-2), (B-2), (C-2)} is always associated with the electron donating ability of the molecule; E_{LUMO} {figures 8-10 (A-3), (B-3), (C-3)} indicates the ability of the molecule to accept electrons; hence these are the acceptor states. High values of E_{HOMO} are likely to indicate a tendency of the molecule to donate electrons to appropriate acceptor molecules with low-energy orbital. The lower the value of E_{LUMO} , the more probable, it is that the molecule would accept electrons. As for the values of ΔE ($E_{LUMO}-E_{HOMO}$) concern; lower values of the energy difference ΔE will cause higher inhibition efficiency because the energy to remove an electron from the last occupied orbital will be low. A larger value of the energy gap indicates low reactivity to a chemical species because the energy gap HOMO and LUMO is related to the softness or hardness of a molecule. A soft molecule is more reactive than a hard molecule because a hard molecule has a larger energy gap and soft has lower energy gap. Likewise, higher values of μ will favour strong interaction of the inhibitor molecules with metal surface. The binding energy of these compounds was found to be negative thus suggest that EELiL is very stable inhibitor. Quantum chemical parameters presented in table-3 confirmed good inhibition performance of active constituents of EELiL as corrosion inhibitor and also confirmed strong interaction between extract molecules and the metal surface and thereby forming protective adsorption layer at mild steel/acid solution interface [30-31].

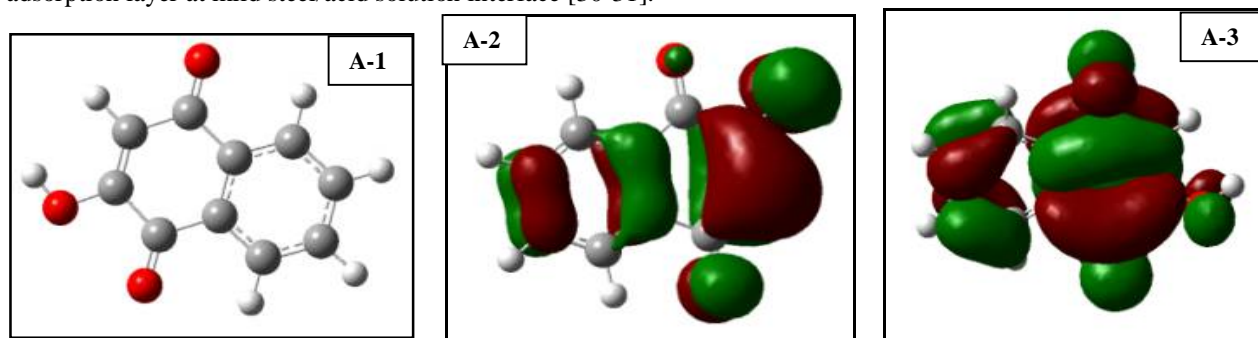


Fig. 8: QCA of Lawsone (one of the major constituents of *Lawsonia inermis*): (A-1) Optimized structures; (A-2) HOMO; (A-3) LUMO.

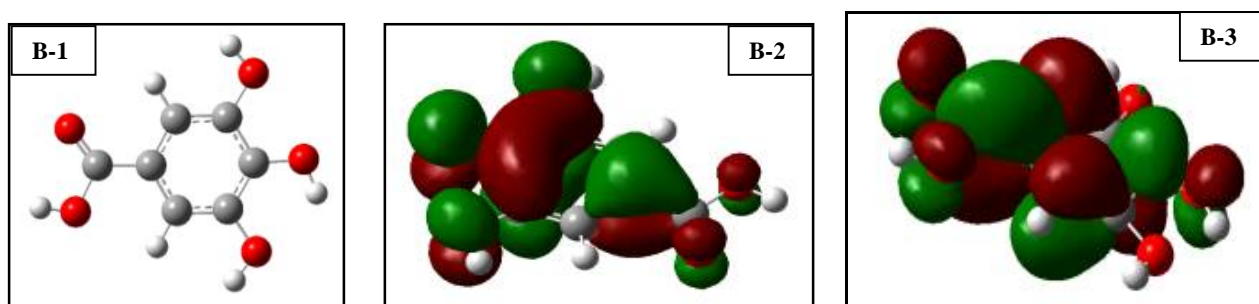


Fig. 9: QCA of Gallic acid (one of the major constituents of *Lawsonia inermis*): (B-1) Optimized structures; (B-2) HOMO; (B-3) LUMO.

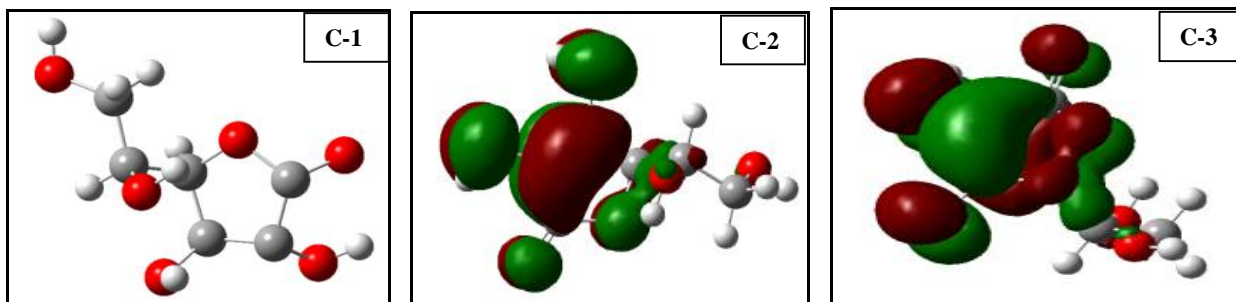


Fig. 10: QCA of Ascorbic acid (one of the major constituents of *Lawsonia inermis*): (C-1) Optimized structures; (C-2) HOMO; (C-3) LUMO.

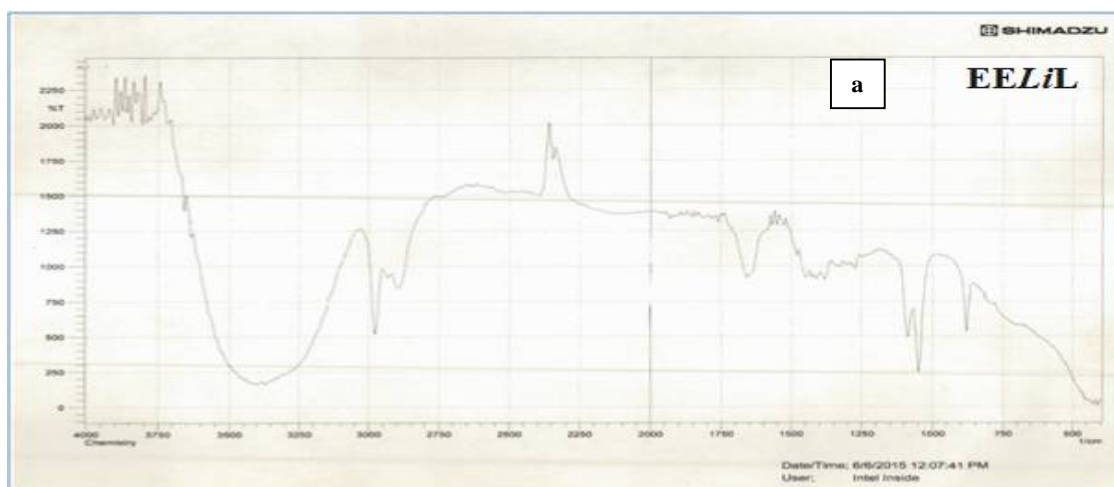
Table-4: Calculated (energies and gradient): Quantum Chemical parameters of Lawsone, Gallic acid, Ascorbic acid (some of the major constituents of *Lawsonia inermis* leaves):

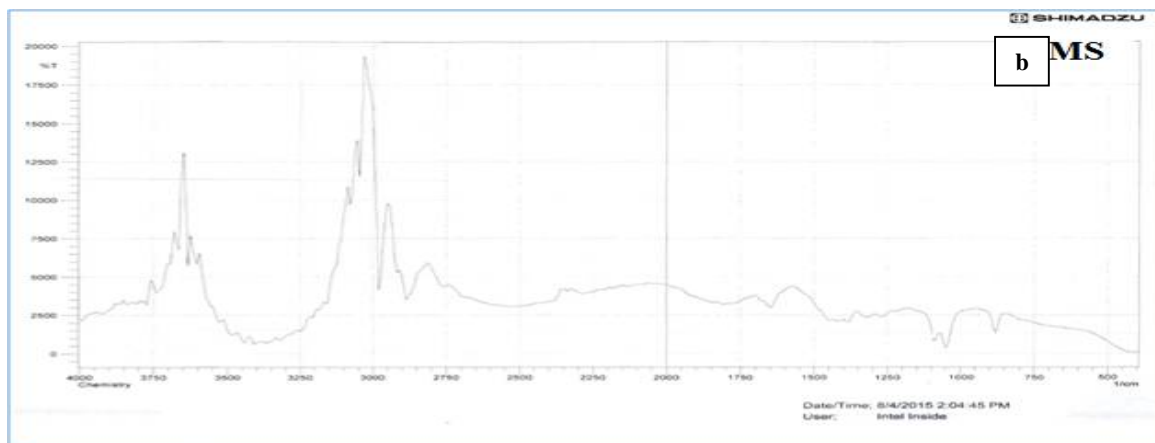
S. No.	Compound	Total energy (kcal/mol)	Dipole moment μ (Debye)	E_{HOMO} (ev)	E_{LUMO} (ev)	$\Delta E = E_{\text{HOMO}} - E_{\text{LUMO}}$ (ev)
1	Lawsone	-383015.36	0.67	-7.46	-3.37	4.080
2	Gallic acid	-405703.53	2.87	-6.34	-1.60	4.730
3	Ascorbic acid	-429729.91	2.20	-8.71	-4.65	4.056

iv Surface morphological analyses:

a. FT-IR Spectroscopy

FT-IR spectra of EELiL as well as the protective films over the surface of MS coupons after 72h immersion in test solution containing 1.092g/L of *Lawsonia inermis* extract have been illustrated in figures 11a and b respectively. The multiplicity of peaks in the spectra for extract implies the presence of a complex mixture of compounds. Again, almost all the peaks observed for *Lawsonia inermis* extract were also noticed in the spectra of protective film of the extract over the MS surface. Some of the peaks for the adsorbed film diminished or even vanished; the peaks with somewhat passive intensities in the adsorbed surface films -C=O stretching around 1710 cm^{-1} , the broad spectrum of -O-H stretching round 3350 cm^{-1} and 3000 cm^{-1} , the -C-O stretch around 1100 cm^{-1} , and -O-H stretch due to secondary alcohol around $1150\text{--}1200\text{ cm}^{-1}$ and bands ranging from $2900\text{ to }3000\text{ cm}^{-1}$ can be attributed to -C-H stretching vibration suggest that these functional groups are directly involved in metal-inhibitor interactions, thus validating the proposed adsorption mechanism of some active chemical constituents of *Lawsonia inermis* onto MS coupons [32-33].





(B)

Fig. 11: FT-IR spectrum for EELiL(A) and the corrosion product on metals in the presence of EELiL extract with 0.5M HCl at room temperature(B)

b. UV-visible Spectroscopy

EELiL contains many anti-corrosive active organic constituents [21-26]. The active organic compound gets dissolved in the solution to interact with the positively charged MS coupon surface. It caused adsorption of inhibitor molecules at metal/solution interface as shows by UV-visible spectroscopy (fig. 12).

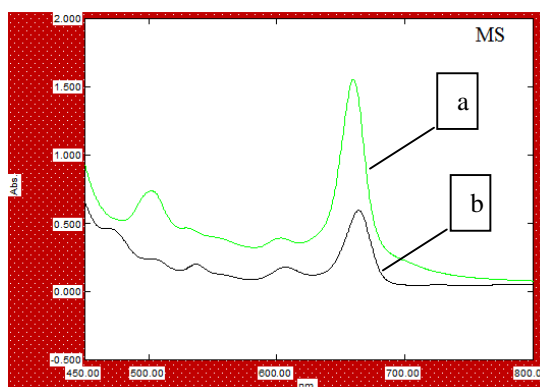


Fig. 12: UV-visible spectra of (a) EELiL and (b) test solution of MS in the presence of EELiL.

UV-Visible absorption spectra were obtained for *Lawsonia inermis* leaves extract and MS coupon immersed in 0.5 M HCl with 1.092 g/L of EELiL (fig. 12 a and b respectively). Ethanolic extract of *Lawsonia inermis* leaves gave absorbance at 1.56 and λ_{max} was obtained in between 600-700 nm. The extract has organic compounds containing conjugated π -complexes and chromophoric groups (a) and a change in position (b) of the absorption (low absorbance 0.57) as well as light change in the position of λ_{max} , was observed after exposed to aggressive medium. However, there was no any significant change in the spectra, but it absorbance was decreased due to possibility of EELiL-Fe complex formation.

c. SEM analysis

Surface morphological studies of MS coupons in uninhibited and inhibited were examined by SEM. The SEM images of MS coupon surfaces were taken, before immersion (surface treated coupon), after 72h immersion in 0.5 M HCl (corroded) and in the presence of 1.09 g/L EELiL (inhibited) (fig 13-a, b, and c respectively). On comparing these SEM images, lots of grooves and roughness was seen on corroded surface (b), while inhibited surface (c) was with less grooves, and covered with a protective film, thus endorsing Fe-EELiL complex formation on metallic surface [34].

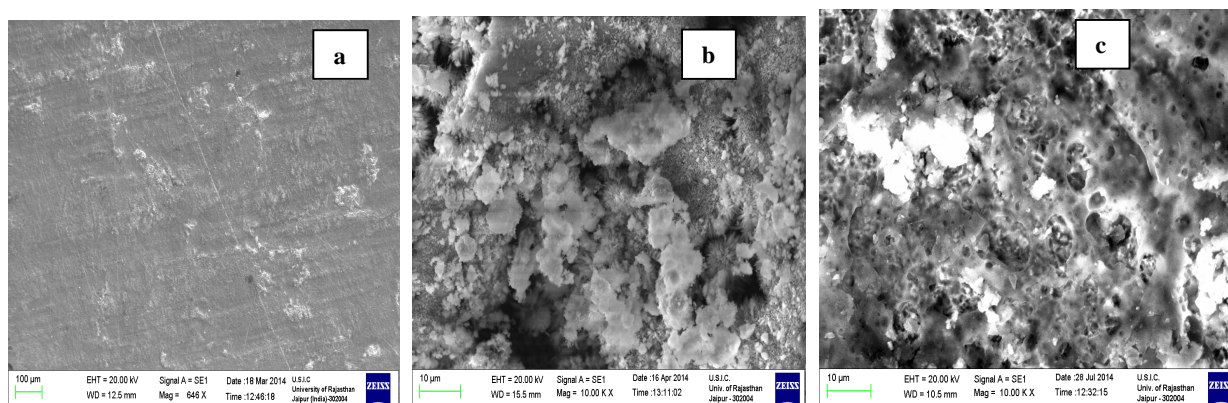


Fig. 13: SEM images of (a) surface treated MS surface; (b) corroded (c) with EELiL (1.09 g/L) in 0.5 M HCl after 72h immersion.

IV. CONCLUSIONS

- A significant inhibitory propensity of ethanolic extract of *Lawsonia inermis* leaves (EELiL) on MS in 0.5 M HCl was observed (88.26 %) at its 1.092 g/L concentration at room temperature.
- The SEM images and optical micrographs validate the formulation of the inhibitor film over the MS surface.
- The nature of adsorption of *Lawsonia inermis* leaves on MS obeys Langmuir adsorption isotherm.
- The high value of adsorption equilibrium constant and the negative value of standard free energy of adsorption suggested the strong adsorption of EELiL over MS surface and that the adsorption is spontaneous.
- The standard free energy of adsorption has been found above -20 kJ mol^{-1} , indicated physical nature of the adsorption.
- The investigations show that inhibitor has the ability to impede acid corrosion of MS, and *Lawsonia inermis* leaves extract can be a good replacement for many toxic chemical inhibitors used for MS in 0.5 M HCl.

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