Anti-Corrosive Propensity of *Alstonia boonei* towards Copper in 0.5 M HCl

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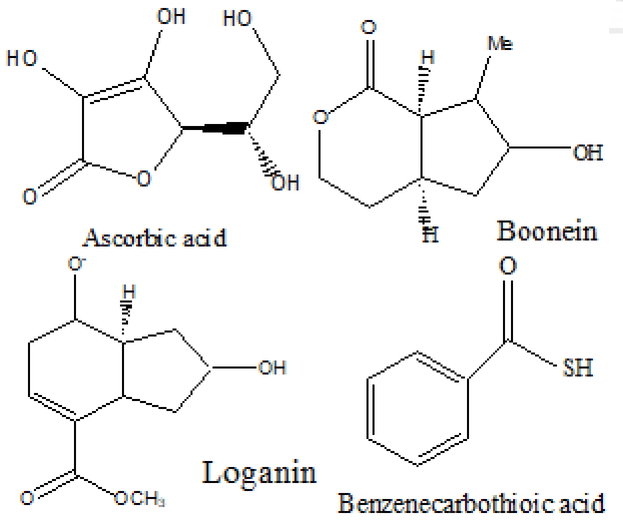
Abstract: ***Inhibitive efficacy of Alstonia boonei leaves extract to combat acid corrosion of copper at the room temperature (30°C) was investigated using chemical method. Various corrosion parameters, viz. corrosion rate (ρ_{corr}), inhibition efficiency (%IE), adsorption equilibrium constant (K_{ad}), degree of fractional surface coverage (θ) were evaluated using experimental data. Maximum inhibition efficacy was observed as 93.2% at 1.081g/L of Alstonia boonei extract and its adsorptive propensity was assessed by Quantum Chemical Analysis. The characterization techniques, viz. FT-IR, UV-Vis spectroscopy and surface morphological analysis tool (SEM) were used to endorse the results. The entire study shows that the Alstonia booei extract can be a promising greener anti-corrosive agent for copper in acid media***.

Keywords: Copper corrosion, *Alstonia boonei*, QCA, SEM.

1. Introduction

Copper metal has significant industrial applications besides household products. Copper, comparatively an inactive metal, gets slowly corroded by air and water in the presence of weak acids [1-4]. Copper dissolution in chloride solutions is very important in the electro-polishing and electro-machining industries. Due to these reasons attention has focused on the behavior of copper in chloride solutions [5].

A number of inorganic and heterocyclic compounds act as effective corrosion inhibitors towards metallic corrosion. These compounds can absorb on metallic surface and shows good anti-corrosive activity. However, these synthesized compounds are highly toxic and hazards to our environment [5-7] thus raising safety and environmental concern globally. Natural resources are great chemical factory of chemicals required to inhibit the metallic-corrosion processes. Because plants are biodegradable, readily available, nontoxic and environmental friendly they are widely used as corrosion inhibitors for protection of metal in acid and alkaline environment [5-15].



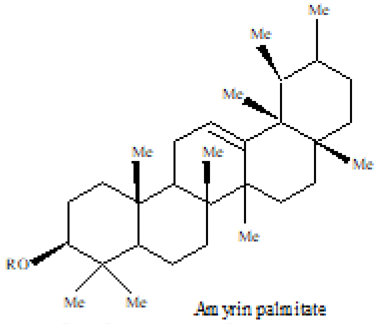


Figure 1: Some of the major constituents of *Alstonia boonei*.

Alstonia boonei species contains various classes of compounds such as ascorbic acid, boonein acid, alkaloids, flavonoids, glycosides, phenolic acids, steroids, amino acids, terpenoids, lipids, saponins, anthriquinone derivatives, volatile oils, coumarins, organic acids [16-22] and most of these chemicals possess anti-corrosive property. An attempt is made to investigate the potentiality of *Alstonia boonei* leaves extract to impede copper corrosion in 0.5 M HCl at room temperature using chemical method.

2. Methodology

The investigations were made employing chemical method as reported earlier [5, 8-11, 13]. Each specimen was surface treated prior to each investigation and loss in weight of test coupon was recorded using an analytical balance (Adair Dutt 205 ACS).

2.1 Preparation of specimens

The specimens were prepared as per procedure reported earlier [5, 7-11, 13]. Sheet was mechanically press-cut into different rectangular coupons each of dimensions (3 cm x 2.4 cm x 0.16 cm) with a hole about 0.12 mm diameter drilled at one end for free suspension. The composition, (Mn) 0.51%, (Fe) 0.19%, (Cu) 93.6%, (Zn) 5.7%, was studied by XRF analysis (figure 2).

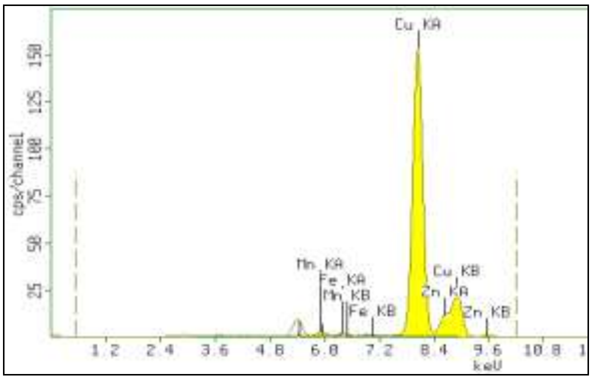


Figure 2: XRF analysis of copper coupon.

2.2 Preparation of *Alstonia boonei* leaves extract

Shade dried leaves of *Alstonia boonei* were accurately weighed, finely powdered and soaked in distilled ethanol in a properly corked container for a stipulated time period. Later, it was refluxed, concentrated, filtered and stored in a clean corked bottle for further use [5, 7-11, 13].

2.3 Preparation of test solutions

AnalR grade reagents were used to prepare the test solutions. The electrolyte (0.5 M HCl) was prepared using bi-distilled water. Seven separate 100 ml beakers, each consisting 50 ml electrolyte solution, were labeled as C0, C1, C2, C3, C4, C5 and C6. *Alstonia boonei* leaves extract was added to these beakers in order of increasing concentration: 0.00, 0309, 0.154, 0.309, 0.618, 0.772, and 1.0815 g/l respectively.

2.4 Quantum Chemical Analysis (QCA)

PM3 method of the quantum chemical Package MOPAC 6.0 of Hyperchem 7.5 was used to carry out QCA. E_{HOMO}, E_{LUMO}, ΔE, binding energy, heat of formation and the dipole moment (μ) were assessed.

2.5 Surface Morphological Studies

2.5.1 UV-visible Spectroscopy

UV-Visible absorption spectra obtained on 8400 Shimadzu, Japan, with help of quartz glass cell in 400-800 nm visible range. The test solutions, after exposure of coupons for 72 h in aggressive solutions containing *Alstonia boonei* leaves extract, were analyzed spectroscopically.

2.5.2 FT-IR Spectroscopy

FT-IR spectrometer (8400 Shimadzu, Japan) (range from 4000 to 400 cm^{-1}) was used for spectroscopic investigations. FT-IR spectrum of the protective film adsorbed onto the coupon surface was analyzed.

2.5.3 SEM

Surface morphological analysis of the coupon-surface was studied by carrying out ZEISS-SEM.

3. Results and Discussion

Anti-corrosive propensity of *Alstonia boonei* leaves extract towards copper was investigated at various exposure period (3 to 72h) and concentrations (0.0309, 0.154, 0.309, 0.618, 0.772 and 1.0815 g/l) at room temperature. The experimental data obtained were used to evaluate various corrosion parameters, viz., corrosion rate (ρ_{corr}) (mmy^{-1}), inhibition efficiency (IE%), fractional surface coverage (θ), adsorption equilibrium constant (K_{ad}) etc. and tabulated (table 1).

Table 1: Corrosion parameters for Copper without and with *Alstonia boonei* leaves extract in 0.5 M HCl at 30 ^{0}C.

Time (h)	***Inhibitor conc (g/l)***	***Corrosion parameters***				
		ρ_{Corr} $x10^{-3}$	***IE %***	***θ *10^2***	***K_{ad}***	***ΔG***
3	C-0 0.0000	1.46	-	-	-	-
	C-1 0.0309	1.28	12.00	12.00	4.41	-13.86
	C-2 0.154	1.17	20.00	20.00	1.62	-11.34
	C-3 0.309	0.99	32.00	32.00	1.52	-11.18
	C-4 0.618	0.80	45.33	45.33	1.34	-10.86
	C-5 0.772	0.56	61.33	61.33	2.05	-11.93
	C-6 1.0815	**0.49**	**66.67**	**66.67**	**1.85**	**11.67**
6	C-0 0.0000	1.36	-	-	-	-
	C-1 0.0309	1.26	7.14	7.14	2.49	-12.42
	C-2 0.154	1.07	21.43	21.43	1.77	-11.56
	C-3 0.309	0.78	42.86	42.86	2.43	-12.35
	C-4 0.618	0.58	57.14	57.14	2.16	-12.06
	C-5 0.772	0.49	64.29	64.29	2.33	-12.25
	C-6 1.0815	**0.39**	**71.43**	**71.43**	**2.31**	**-12.23**
12	C-0 0.0000	1.02	-	-	-	-
	C-1 0.0309	0.78	23.81	23.81	10.11	-15.95
	C-2 0.154	0.63	38.09	38.09	4.00	-13.60
	C-3 0.309	0.44	57.14	57.14	4.31	-13.80
	C-4 0.618	0.34	66.67	66.67	3.24	-13.08
	C-5 0.772	0.29	71.43	71.43	3.24	-13.08
	C-6 1.0815	**0.24**	**76.67**	**76.67**	**3.04**	**-12.92**
18	C-0 0.0000	0.90	-	-	-	-
	C-1 0.0309	0.65	28.57	28.57	12.94	-16.57
	C-2 0.154	0.52	42.86	42.86	4.87	-14.11
	C-3 0.309	0.36	60.71	60.71	5.00	-14.17
	C-4 0.618	0.29	67.86	67.86	3.42	-13.21
	C-5 0.772	0.23	75.00	75.00	3.89	-13.54
	C-6 1.0815	**0.18**	**80.71**	**80.71**	**3.87**	**-13.53**
24	C-0 0.0000	1.22	-	-	-	-
	C-1 0.0309	0.80	34.00	34.00	16.67	-17.21
	C-2 0.154	0.63	48.00	48.00	5.99	-14.63
	C-3 0.309	0.49	60.00	60.00	4.85	-14.10
	C-4 0.618	0.29	76.00	76.00	15.12	-14.23
	C-5 0.772	0.24	80.00	80.00	5.18	-14.26
	C-6 1.0815	**0.15**	**88.00**	**88.00**	**6.78**	**-14.94**
48	C-0 0.0000	1.86	-	-	-	-
	C-1 0.0309	1.20	35.29	35.29	17.65	-17.35
	C-2 0.154	0.79	57.52	57.52	8.79	-15.59
	C-3 0.309	0.70	62.09	62.09	5.30	-14.32
	C-4 0.618	0.41	77.78	77.78	5.66	-14.49
	C-5 0.772	0.34	81.70	81.70	5.78	-14.54
	C-6 1.0815	**0.19**	**89.54**	**89.54**	**7.92**	**-15.33**
72	C-0 0.0000	2.03	-	-	-	-
	C-1 0.0309	1.26	37.60	37.60	19.50	-17.60
	C-2 0.154	0.78	61.60	61.60	10.41	-16.02
	C-3 0.309	0.49	75.60	75.60	10.03	-15.92
	C-4 0.618	0.42	79.20	79.20	6.16	-14.70
	C-5 0.772	0.31	84.80	84.80	7.23	-15.10
	C-6 1.0815	**0.14**	**93.20**	**93.20**	**12.68**	**-16.52**

It is very clearly depicted from data analysis that the corrosion rate of copper varies linearly with time and gets reduced in the presence of the inhibitor compared to the free acid solution. Moreover, low concentration of *Alstonia boonei* is not enough to get adsorbed on to the surface of corroding metal at lower exposure time period. At lower concentration and small immersion period irregular trends were observed, this is may be due to pH effect or many other electrochemical reactions at lower immersion period. The inhibition efficacy (IE %) of *Alstonia boonei* was observed to increase with increase in its concentration and maximum (93.20%) was observed at 1.0815 g/l at 72h exposure time (figure 3 and table 1). This may be attributed to the adsorption of active molecules over to coupon surface and covering maximum exposed surface so as to reduce the available reaction area on to the metal surface. High inhibition efficiency of *Alstonia boonei* at its higher concentration may also be due to the synergistic action of all the active chemical constituents present in it. Most of the constituents of *Alstonia boonei* contain hydroxyl aromatic compounds, aromatic rings, and heteroatoms (oxygen, nitrogen) which enhance its adsorptive property onto coupon surface, thereby, increasing the charge transfer resistance of the anodic dissolution of copper [5,7, 14]. The adsorption mechanism for a given inhibitor depends on such factors as the nature of metal and corrosive medium, and concentration of the inhibitor as well as the functional groups presents in its molecule, since different groups are adsorbed to different extents [6-7, 14].

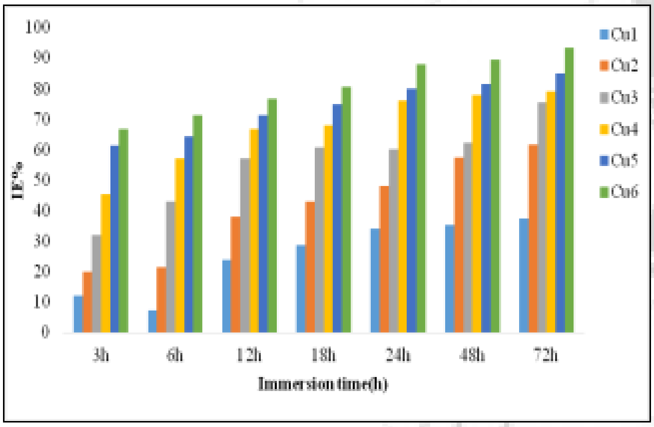


Figure 3: IE% of inhibitor at its various concentrations (g/l) and at different immersion period (h) at 30 ^{0}C temperature

3.1 Adsorption isotherm

The inhibition mechanism involves blocking of copper surface by inhibitor molecules via adsorption. In general, the phenomenon of adsorption is influenced by the nature of metal and chemical structure of inhibitor. The adsorption isotherm is a very useful insights into the mechanism of corrosion inhibition, thus it is necessary to determine empirically which adsorption isotherm fits best to the surface coverage in order to use the corrosion rate measurements to calculate the thermodynamic parameters pertaining to inhibitor adsorption. Different adsorption models were considered for the best fit of experimental data. Langmuir adsorption isotherm best described the adsorption characteristics of extract of *Alstonia boonei*. Langmuir adsorption isotherm is mathematically expressed as:

$$C/\theta = 1/K_{ads} + C$$

where θ is the degree of surface coverage, C the molar inhibitor concentration in the bulk solution and K_{ads} is the equilibrium constant of the process of adsorption.

Plot of C/ θ versus C yield straight line (figure 4) which proves that Langmuir adsorption isotherm is obeyed over the range of concentration studied. The correlation coefficient, slopes, obtained from Langmuir isotherm plots are tabulated in Table-2. Furthermore, the ΔG values, as evaluated from the experimental data (table 1), clearly illustrate physisorption of the active molecules over the copper coupon surface thus protecting the dissolution of the sheet.

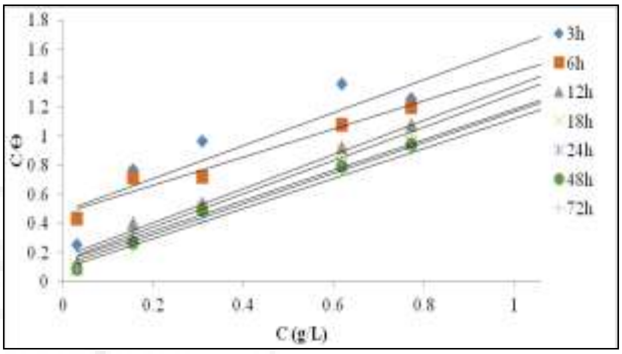


Figure 4: C/θ vs C at different immersion period.

Table 2: Langmuir adsorption isotherm parameters at 72h exposure periods at various concentrations of *Alstonia boonei*

Immersion Period (h)	*Correlation coefficient R^2*	*Slope*
3	0.875	1.135
6	0.978	0.969
12	0.991	1.178
18	0.988	1.145
24	0.986	1.051
48	0.988	1.055
72	0.992	1.037

3.2 Quantum chemical analysis (QCA)

To assess adsorption and inhibitory mechanism of the active constituents of *Alstonia boonei*, QCA was carried out. On reviewing literature, it was found that ascorbic acid, boonein and benzocarbothioic acid are the most active molecules present in *Alstonia boonei* and these individually also show adsorptive propensity [16-22]. Thus, quantum chemical studies of these molecules were carried out (figures 5-7).

It is important to focus on the parameters that directly influence the electronic interaction of the inhibitor molecules with the metal surface. These are mainly: E_{HOMO}, E_{LUMO}, ΔE (E_{LUMO}-E_{HOMO}), and dipole moment μ [23-26]. The values of calculated quantum chemical parameters, viz., E_{HOMO}, E_{LUMO}, ΔE (E_{LUMO}-E_{HOMO}) and μ of ascorbic acid, boonein and benzocarbothioic acid are tabulated in Table 3. Higher values of the E_{HOMO} facilitate adsorption (and therefore inhibition) by influencing the transport process through the adsorbed layer. Therefore, the energy of the E_{LUMO} indicates the ability of the molecule to accept electrons; hence these are the acceptor states. 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. Higher values of the dipole moment (μ) will favour strong interaction of the inhibitor molecules with metal surface and lower values favour the accumulation of inhibitor molecules around electrode surface [23-26]. Quantum chemical parameters of these active molecules of *Alstonia boonei* (table 3) confirm strong interaction of these molecules with the metal surface and thereby forming protective adsorption layer at copper /acid solution interface [23-26].

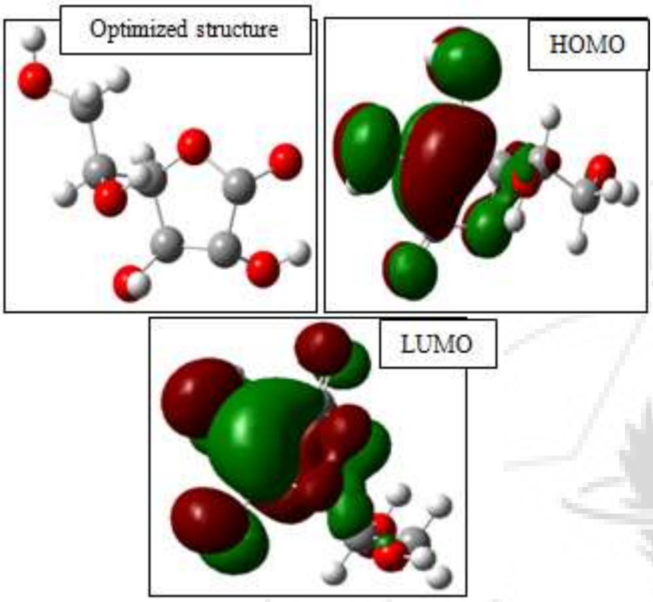


Figure 5: Optimized structure, HOMO, LUMO of ascorbic acid, one of the active molecules of *Alstonia boonei*.

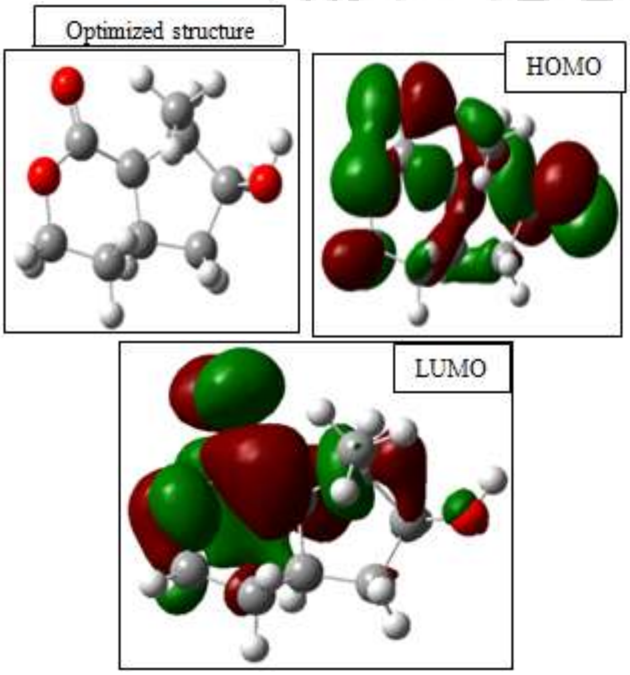


Figure 6: Optimized structure, HOMO, LUMO of boonein, one of the active molecules of *Alstonia boonei*.

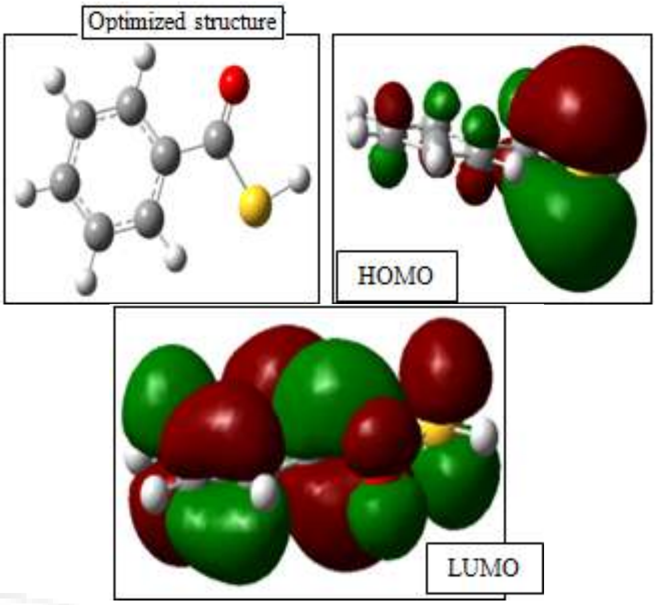


Figure 7: Optimized structure, HOMO, LUMO of benzocarbothioic acid, one of the active molecules of *Alstonia boonei*.

Table 3: Calculated QCA parameters of the major constituents of *Alstonia boonei*, viz., ascorbic acid, boonein and benzocarbothioic acid.

Molecules	Total energy (kcal/mol)	Point group	μ Debye	Energy HOMO (ev)	Energy LUMO (ev)	ΔE(ev) = (HOMO-LUMO)
Ascorbic acid	-429729.91	C1	2.20	-8.71	-4.65	4.06
Boonein	-362137.01	C1	3.97	-7.35	-0.46	6.88
Benzocarbothioic acid	-466741.97	C1	2.57	-8.60	-6.07	2.53

3.3 Spectroscopic Analysis:

3.3.1 UV-visible Spectroscopy

Inhibitory propensity of an inhibitor can be very well elucidated on the basis of molecular adsorption [5, 7-10, 25]. *Alstonia boonei* is a mixture of variety of organic compounds which cannot be easily separated. UV-Visible absorption spectra were obtained for *Alstonia boonei* leaves extract and that of copper immersed in 0.5 M HCl with 1.081 g/L *Alstonia boonei* (figure 8). Main absorption band (figure 8-a) around 660-700nm and absorbance 0.4 were seen which can be seen due to π to π^* transitions. When extract is adsorbed at metallic coupon, a weak band slightly downward around 650 to 690nm was appeared. The absorbance peak for Cu around 0.1was observed.

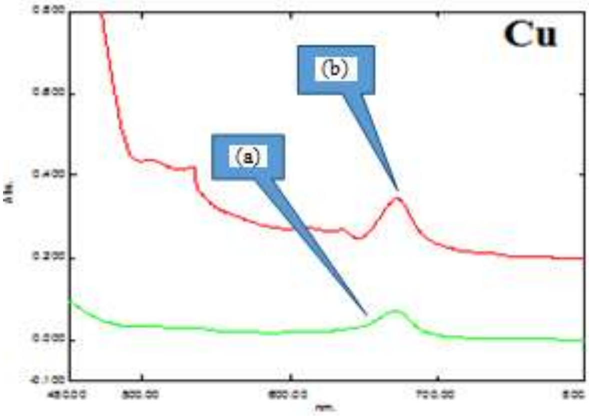


Figure 8: UV-Vis spectra of: (a) *Alstonia boonei* leaves extract (b) test solution of copper coupon in the presence of *Alstonia boonei* extract in 0.5M HCl

A change in position of the absorption maxima is seen in figure 8, which indicates the formation of a complex between two species in solution thus confirm the possibility of the formation of *Alstonia boonei* active molecules+Cu complex**.** Hence confirming adsorption of active organic compounds present in *Alstonia boonei* onto the copper-metal surface resulting in combating copper dissolution [5, 7-10, 25].

3.3.2 FT-IR Spectroscopy

The FT-IR was conducted on 8400 Shimadzu, Japan FT-IR spectrometer in the IR range from 4000 to 400 cm^{-1} to endorse the adsorption of *Alstonia boonei* on copper coupon surface. The FT-IR spectra of vacuum dried *Alstonia boonei* leaves extract was compared with the spectra of its adsorbed film over metal surface immersed in 0.5 M HCl for 72h (figures 9-10). The comparative study of FT-IR spectra reveals the changes in the peaks after adsorption of *Alstonia boonei* on metal surface; certain peaks disappeared completely and some shifted to higher frequencies region, justifying the adsorption phenomenon taking place over the copper coupon surface [27-29].

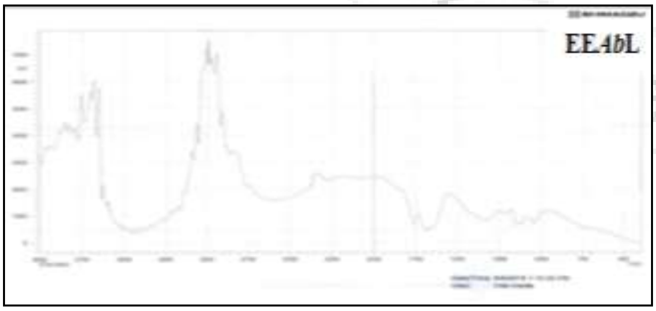


Figure 9: FT-IR spectra of *Alstonia boonei* leaves extract

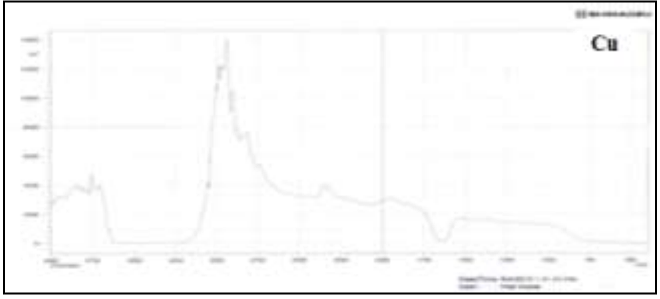


Figure 10: FT-IR spectra of the *Alstonia boonei* leaves extract-film adsorbed onto the copper coupon surface

3.4 Surface morphological studies (SEM)

The adsorptive propensity of the active molecules of *Alstonia boonei* leaves extract was further established by carrying out surface morphological studies, viz., SEM. SEM images of copper coupons in various conditions were carried out (figure 11) (a) surface treated pre-exposed to aggressive media; (b) after immersion in 0.5 M HCl without inhibitor for 72h; and (c) after immersion in 0.5 M HCl containing *Alstonia boonei* leaves extract for 72h. The SEM micrographs clearly illustrate that the surface of samples dipped in corrosive media is highly damaged with severe pits and cracks as more active sites are available for dissolution in corroding media while in presence of the inhibitor i.e. *Alstonia boonei* leaves extract, the coupon surface appears to be smooth and clear with lesser number of cavities as compared to the samples: blank/exposed to aggressive media [5, 7-13, 26, 30]. Thus, the surface morphological analysis we can conclude that active molecules of the inhibitor (*Alstonia boonei* leaves extract) gets adsorbed onto the copper surface thereby forming a thin film/layer which protects the metal surface dissolution in corrosive environment.

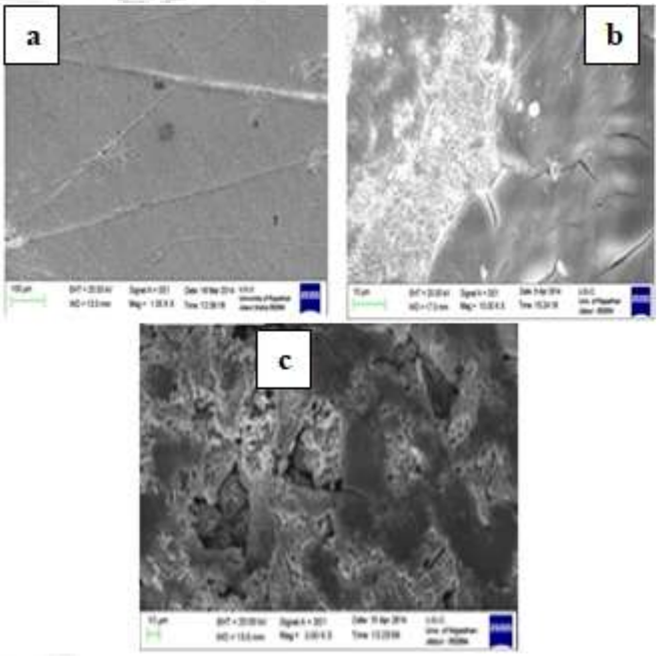


Figure 11: SEM micrographs of copper coupons: (a surface treated pre-exposed to aggressive media (b) after immersion in 0.5 M HCl without inhibitor for 72h; and (c) after immersion in 0.5 M HCl containing *Alstonia boonei* leaves extract for 72h.

4. Conclusions

- A remarkably excellent inhibition efficacy was observed with *Alstonia boonei* leaves extract as inhibitor towards copper corrosion in 0.5 M HCl at room temperature.
- *Alstonia boonei* leaves consists of rich phytochemicals, viz. ascorbic acid, boonein acid, alkaloids, flavonoids, glycosides, phenolic acids, steroids, amino acids, terpenoids, lipids, saponins, coumarins, benzocarbothioic acid etc., each of these compounds has strong adsorptive propensity which is also endorsed by quantum chemical studies.

- IE % increases with increase in concentration of *Alstonia boonei* leaves extract and observed maximum inhibition efficacy (93.2%) at 1.081g/l concentration of the inhibitor.
- The active phytochemical constituents present in *Alstonia boonei* leaves extract block the active sites of the surface of copper coupon by its physical adsorption.
- Langmuir adsorption isotherm was observed as best fitted to the experimental data.
- Spectroscopic studies, viz., FT-IR and UV-Vis, strongly recommend the occurrence of adsorption phenomenon of the active constituents of *Alstonia boonei* leaves onto the metal surface.
- Quantum chemical and surface morphological analyses clearly reveal the adsorptive propensity of the active molecules of *Alstonia boonei* leaves extract.
- SEM images clearly illustrate the surface of copper coupon is being protected by the inhibitor molecules present along with aggressive media.
- *Alstonia boonei* leaves extract can be used to combat acid corrosion of copper at room temperature.
- *Alstonia boonei* is an eco-friendly, low cost and readily available effective corrosion inhibitor which can conserve environment, energy as well as economy of the country.

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