

# Eco-friendly Inhibition by Weed (*Bidens biternata*) Extract towards Acid Corrosion of AA6063

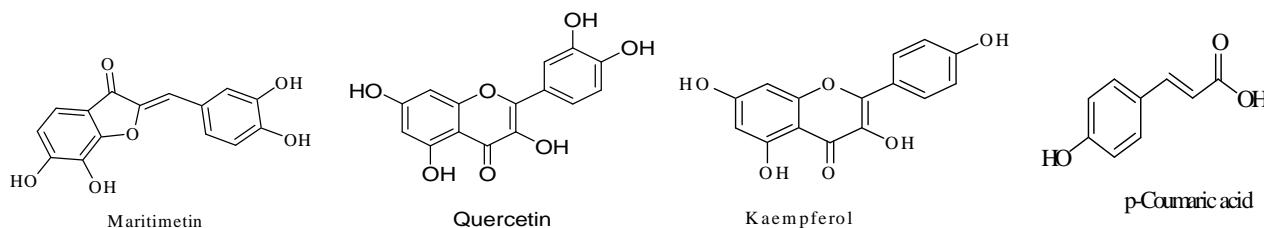
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**Abstract:** With the view of extracting gold out of waste, the ethanolic extract of weed (*Bidens biternata*) (EEBb) was explored for its inhibitory efficacy towards acid corrosion of AA6063 (aluminium alloy) in 0.5 M HCl. The experimentations were carried out employing chemical method at room and elevated temperatures and various corrosion parameters were calculated. The inhibitory efficacy was found to be 88.09 % at 1.04 g/L of EEBb. The adsorption of the inhibitor onto the metal surface was further endorsed by carrying out Quantum Chemical Analysis (QCA), spectroscopic (UV-Visible Spectroscopy) and surface morphological studies (SEM). The adsorptive nature of EEBb was investigated and found to best fitted in Langmuir adsorption isotherm. The high inhibition efficiency was attributed to the adsorption of active molecules leading to the formation of a protective layer on the AA6063 surface. The results were very promising and indicate that toxic weed *Bidens biternata* can be very effectively to combat acid corrosion of aluminium. Moreover, these can be a good replacement for most of the hazardous chemicals used to inhibit metal corrosion.

**Keywords:** Acid Corrosion, Kinetic and Thermodynamic Parameters, QCA, UV-Visible Spectroscopy, SEM.

## I. INTRODUCTION:

Metals and alloys which are widely used in day to day life are more or less susceptible to different types of corrosion due to their exposure to environment [1-2]. Thus considerable efforts are deployed to minimize the corrosion of a metal. In present trends use of inhibitors is very common. As many synthetic inhibitors have proved to be hazardous to mankind and environment hence researchers have developed their interest towards the use of natural products as Green corrosion inhibitors as they are environmentally safe, less toxic, environment friendly and readily available [3-7]. Thus our research interest is directed towards the use of weed plant species as corrosion inhibitor. *Bidens biternata* is a rich source of chemicals such as quercetin, maritimetin, kaempferol, p-coumaric acid, triacontanoic acid, stigmasterol and carbohydrate- Z-6-O-(6"-propionyl-β-D-glucopyranosyl), 6, 7, 3, 4-tetrahydro-xyaaron, 4-O (2"-O-acetyl-6"-P-coumaroyl-β-D-glucopyranosyl) [8-10]. Structure of some of the constituents illustrated below clearly shows that these species contain moieties responsible for inhibitive action. Some of these constituent are reported as efficient inhibitor to combat metal corrosion [11]. Thus, *Bidens biternata* was investigated for mitigation of corrosion of industrially used metal viz. Aluminium in 0.5 M HCl at room as well as elevated temperature.



## II. EXPERIMENTATION AND METHODOLOGY

### CHEMICAL MEASUREMENTS:

Chemical measurement (weight loss) method was employed to explore the inhibitive propensity of the ethanolic extract of *B. biternata* (EEBb). As reported earlier, the loss in weight of coupons on exposure in 0.5 M HCl for a constant period (without and with additives) was evaluated using Adair Dutt microbalance. The weight loss data were used to evaluate various corrosion parameters. To estimate the optimum concentration of the inhibitor the experiments were carried out at its different concentrations at various exposure time at room as well as elevated temperatures (293 to 353±1 K) under thermostatic conditions.

**TEST COUPON PREPARATION:**

Industrially used AA6063 (aluminium alloy) coupons were used with composition purity as: 97.6% Al, 1.3% Mn, 0.87% Fe, 0.11% Cu, 0.11% Zn analyzed by XRF [12-13]. Rectangular sized (3x2.4x0.16) cm<sup>3</sup> test coupons were prepared and each coupon was surface treated prior to experimentation as per standard procedures [12-13].

**ETHANOLIC EXTRACT OF *BIDENS BITERNATA* (EEBb):**

The standard procedure was adopted to prepare ethanolic extract of *Bidens biternata* (EEBb) [2, 12-13]. The extracted amount of plant material was found to be 3.2 g/L.

**PREPARATION OF TEST SOLUTIONS:**

Standard electrolyte solutions (0.5 M HCl) were prepared using AnalR grade HCl (pKa  $1 \times 10^{-7}$ ) in triply distilled water. The EEBb was added in the order of increasing concentration so as to have 0.032, 0.14, 0.26, 0.44, 0.80, and 1.04 g/L respectively in six beakers marked A0 to A6. No extract added to A0 beaker.

**QUANTUM CHEMICAL ANALYSIS:**

Quantum chemical analysis was performed with the Hyperchem software package 7.0. The molecular structures of the neutral species were geometrically optimized using the density functional theory (DFT)/MOPAC 6.0 PM3 method. Various quantum chemical parameters were computed.

**SPECTROPHOTOMETRIC ANALYSIS:**

**UV-VISIBLE SPECTROSCOPY:**

UV-Visible Spectrophotometer in 400 to 800 nm visible range (Shimadzu 1700) was used in order to confirm the formation of protective film of EEBb over metal coupon.

**SURFACE MORPHOLOGICAL ANALYSIS:**

**SCANNING ELECTRON MICROSCOPY (SEM):**

The adsorptive tendency of the EEBb was further assessed by analyzing the surface morphology of the corroded and inhibited coupons by the help of ZEISS-Scanning Electron Microscope (SEM) in the range 10  $\mu$  with the magnification 1000X.

**III. RESULTS AND DISCUSSION:**

**(i) EFFECT OF TEMPERATURE CHANGE ON VARIOUS CORROSION PARAMETERS**

Experiments were performed to study the impact of inhibitor concentration on the corrosion of Al in 0.5 M HCl for 72 h immersion period at room as well as elevated temperature. The experimental data were used to evaluate various corrosion and adsorptive parameters which were tabulated in the **Table 1**.

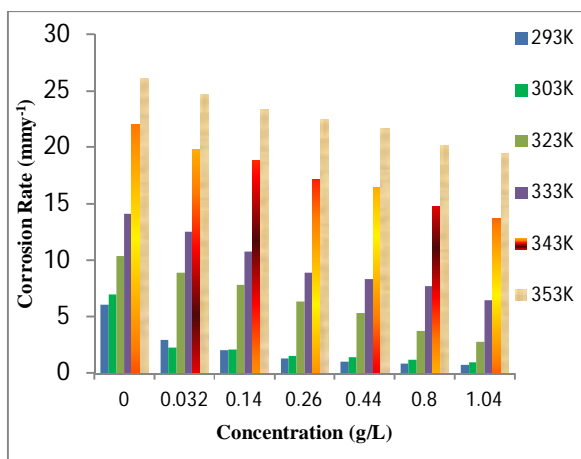


Figure 1: Corrosion rate vs. concentration of EEBb at different temperature (293 to 353±1 K).

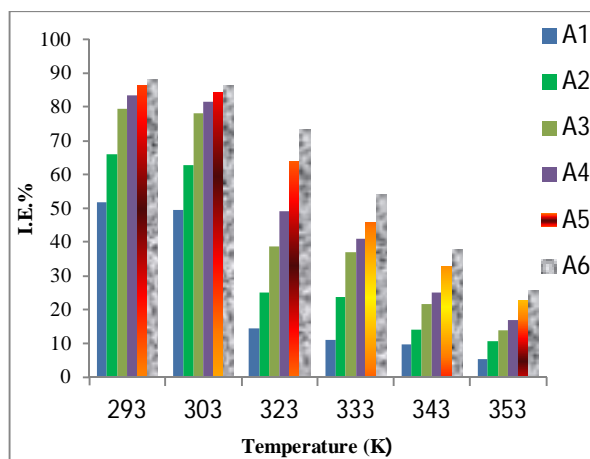


Figure 2: I.E. (%) vs. concentration of EEBb at different temperatures (293 to 353±1 K)

**Table 1:** Corrosion Parameters of Acid Corrosion of AA6063 without and with different concentrations of EEBb at 72h immersion period at different temperatures (293 to 353 K).

Temperature (K)	EEBb Concentration (g/L)	Corrosion parameters at 72 h			
		Corrosion rate (pcorr) (mmy <sup>-1</sup> )	Inhibition Efficiency (IE %)	Fractional Surface coverage (θ)	Adsorption Equilibrium Constant (K <sub>ad</sub> )
293	A0 0.0	6.090	-	-	-
	A1 0.032	2.933	51.83	0.5183	33.63
	A2 0.14	2.067	66.05	0.6605	13.89
	A3 0.26	1.257	79.35	0.7935	14.78
	A4 0.44	1.005	83.48	0.8348	11.48
	A5 0.80	0.838	86.23	0.8623	7.83
	<b>A6 1.04</b>	<b>0.726</b>	<b>88.07</b>	<b>0.8807</b>	<b>7.10</b>
303	A0 0.0	6.929	-	-	-
	A1 0.032	3.492	49.59	0.4959	63.64
	A2 0.14	2.570	62.90	0.6290	16.25
	A3 0.26	1.508	78.22	0.7822	13.88
	A4 0.44	1.285	81.45	0.8145	9.04
	A5 0.80	1.089	84.27	0.8427	6.34
	<b>A6 1.04</b>	<b>0.949</b>	<b>86.29</b>	<b>0.8629</b>	<b>6.29</b>
323	A0 0.0	10.39	-	-	-
	A1 0.032	8.884	14.51	0.1451	5.30
	A2 0.14	7.795	25	0.25	2.38
	A3 0.26	6.370	38.70	0.3870	2.42
	A4 0.44	5.280	49.19	0.4919	2.20
	A5 0.80	3.743	63.97	0.6397	2.22
	<b>A6 1.04</b>	<b>2.766</b>	<b>73.38</b>	<b>0.7338</b>	<b>2.65</b>
333	A0 0.0	14.10	-	-	-
	A1 0.032	12.54	11.08	0.1108	3.89
	A2 0.14	10.75	23.76	0.2376	2.22
	A3 0.26	8.884	37.02	0.3702	2.26
	A4 0.44	8.326	40.99	0.4099	1.57
	A5 0.80	7.655	45.74	0.4574	1.05
	<b>A6 1.04</b>	<b>6.482</b>	<b>54.05</b>	<b>0.5405</b>	<b>1.13</b>
343	A0 0.0	21.93	-	-	-
	A1 0.032	19.78	9.808	0.0980	3.39
	A2 0.14	18.83	14.14	0.1414	1.17
	A3 0.26	17.18	21.65	0.2165	1.06
	A4 0.44	16.42	25.09	0.2509	0.76
	A5 0.80	14.72	32.86	0.3286	0.61
	<b>A6 1.04</b>	<b>13.66</b>	<b>37.70</b>	<b>0.3770</b>	<b>0.58</b>
353	A0 0.0	26.12	-	-	-
	A1 0.032	24.72	5.347	0.0534	1.76
	A2 0.14	23.32	10.69	0.1069	0.85
	A3 0.26	22.49	13.90	0.1390	0.62
	A4 0.44	21.68	17.00	0.1700	0.46
	A5 0.80	20.20	22.67	0.2267	0.36
	<b>A6 1.04</b>	<b>19.41</b>	<b>25.66</b>	<b>0.2566</b>	<b>0.33</b>

It is evident from the Fig. 1 that there was progressive decrease in corrosion rate in the presence of extract but at the same time corrosion rate increases with rise in the temperature thus obeys the Arrhenius type reactions. Inhibition efficiency increases with increase in concentration but decreases with the elevation in temperature. The maximum inhibition efficiency (i.e. 88.07% and 86.29%) was found at highest inhibitor concentration (1.04 g/L) at 293 and 303K for 72h immersion period. But with the elevation in temperature from 303 to 353±1 K the IE % has been found to decrease in 0.5M HCl (Fig. 2) indicating that adsorption of EEBb on Al surface to be physioadsorption. At higher temperature the adsorbed film seems to disappear and the aggressive ions responsible for corrosion seems to be dominant as compared to adsorbed inhibitor molecules.

(ii) KINETIC TREATMENT OF WEIGHT LOSS RESULTS

The Kinetic relationship between the corrosion reaction of Al for the uninhibited and inhibited solutions is expressed by the following relationship [14]:

$$\log \rho_{corr} = \log k + B \log C$$

Where  $k$  is the rate constant and equals to  $\rho_{corr}$  at inhibitor concentration of unity;  $B$  is the reaction constant which, in the present case, is a measure for the inhibition effectiveness and  $C$  is the concentration of  $EEBb$  in g/L. From the slopes of the plot of  $\log \rho_{corr}$  vs.  $\log C$ , kinetic parameter  $k$  and  $B$  were calculated and the values have been tabulated in Table-2. The slopes of the lines were observed negative; depicting that  $EEBb$  becomes more effective as its concentration was increased as the corrosion process decrease. At each temperature a linear variation with negative slope was observed, confirming a first order kinetics.

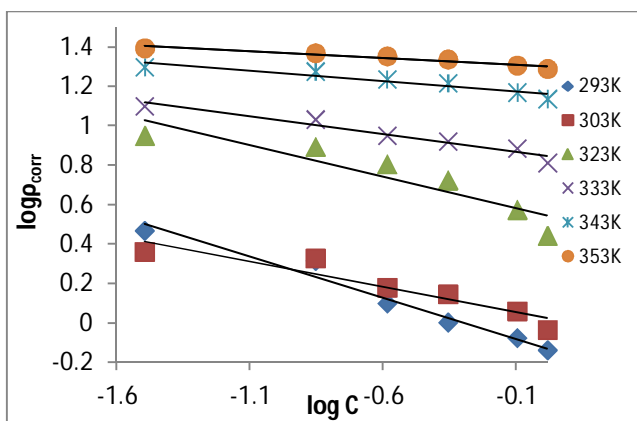


Figure 3:  $\log \rho_{corr}$  vs.  $\log C$  at 72 h at various temperatures.

Table 2: Kinetic Parameters at different temperatures

Temperature (K)	Immersion time (72 h)		
	B	k	R <sup>2</sup>
293	-0.416	00.75	0.967
303	-0.257	01.06	0.886
323	-0.318	03.55	0.842
333	-0.180	07.05	0.947
343	-0.104	14.52	0.890
353	-0.067	20.04	0.960

The temperature dependence of corrosion reaction for aluminium in acidic medium was established plot of  $\log \rho_{corr}$  against  $1/T$  which lead to straight lines as shown in Fig.4 in 0.5 M HCl in absence and presence of different concentrations of  $EEBb$ . Activation energy ( $E_a$ ),  $\Delta H$  and  $\Delta S$  were calculated and tabulated in Table 3.

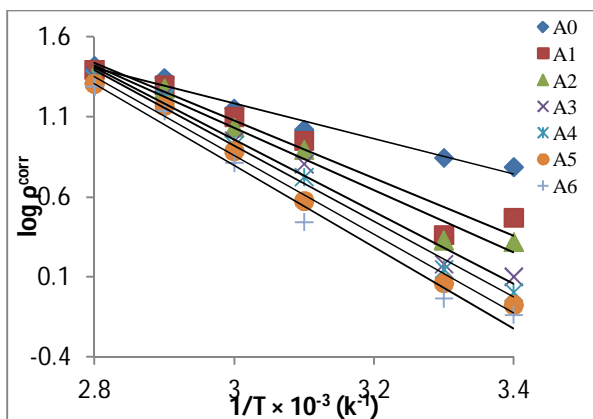


Figure 4:  $\log \rho_{corr}$  vs.  $1/T$  (K) at 72 h.

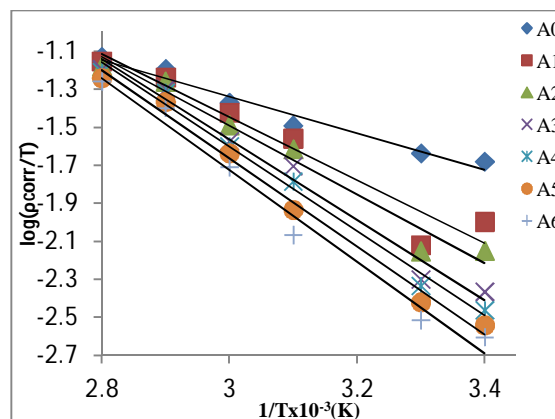


Figure 5:  $\log (\rho_{corr}/T)$  vs.  $1/T$  (K) at 72 h.

The higher values of  $E_a$  in the presence of inhibitor are generally attributed to physisorption mechanism consistent with the formation of an adsorptive film of chemical constituents over metal surface. The absolute values of  $\Delta H^\ddagger$  obtained were lower than  $40 \text{ kJ mol}^{-1}$  for Al which is indication of physisorption. The negative values of entropy suggest that the adsorption process is accompanied by a decrease in the disorder of the system due to adsorption of  $EEBb$  components on the metal surface [15-16].

**Table-3:** Kinetic and thermodynamic parameters of activation

Concentration g/L	72 h		
	$(E_a)(\text{kJ mol}^{-1}) \times 10^{-2}$	$\Delta H (\text{kJ mol}^{-1}) \times 10^{-2}$	$\Delta S (\text{J mol}^{-1} \text{K}^{-1})$
A0 0.00	21.06	-18.38	-167.91
A1 0.032	32.54	-31.78	-130.00
A2 0.14	37.33	-34.46	-122.92
A3 0.26	43.08	-40.40	-105.88
A4 0.44	45.18	-42.50	-100.52
A5 0.80	46.91	-44.42	-96.11
A6 1.04	48.82	-46.14	-91.90

### (III) THERMODYNAMIC PARAMETERS

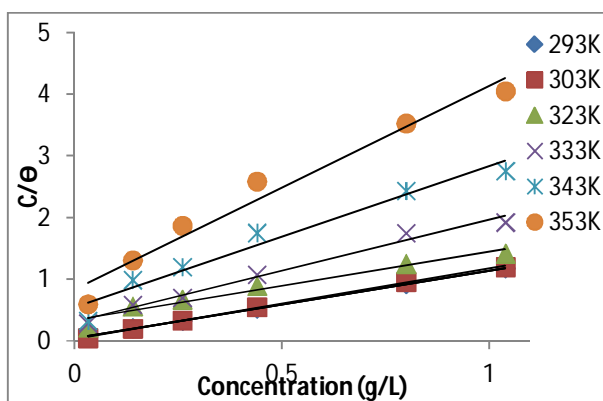
Free energy  $\Delta G_{\text{ads}}^{\circ}$ , enthalpy  $\Delta H_{\text{ads}}^{\circ}$  and entropy  $\Delta S_{\text{ads}}^{\circ}$  for adsorption were calculated, and tabulated in Table 5. The Table 5 reveals that  $\Delta G_{\text{ads}}^{\circ}$  values were in the range (-8.55 to -20.58 kJ/mol) which indicate spontaneous and physical nature of adsorption of the molecule on Al surface. The negative sign of  $\Delta H$  reveals that the adsorption of inhibitor molecules is an exothermic process [17-18].

**Table 5:** Thermodynamic parameters of acid corrosion of AA6063 without and with various concentrations of EEBb at different temperatures (293 to 353±1 K).

Concentration g/L	$\Delta G_{\text{ads}}^{\circ} (\text{kJ mol}^{-1})$						$\Delta H_{\text{ads}}^{\circ} \text{kJ mol}^{-1}$	$\Delta S_{\text{ads}}^{\circ} \text{Jmol}^{-1}\text{K}^{-1}$
	293K	303K	323K	333K	343K	353K		
A0 0.00	0	0	0	0	0	0	-49.3	0.101
A1 0.032	-18.34	-20.58	-15.26	-14.88	-14.94	-13.45	-44.76	0.095
A2 0.140	-16.19	-17.14	-13.11	-13.33	-11.91	-11.32	-47.9	0.105
A3 0.260	-16.34	-16.74	-13.16	-13.37	-11.62	-10.38	-47.88	0.108
A4 0.440	-15.73	-15.66	-12.90	-12.38	-10.67	-9.54	-46.17	0.104
A5 0.800	-14.79	-14.77	-12.92	-11.26	-10.05	-8.84	-46.41	0.105
A6 1.040	-14.55	-14.75	-13.40	-11.46	-9.90	-8.55	-49.3	0.101

### (IV) ADSORPTION ISOTHERM:

Straight lines were obtained for the plot of  $C_{\text{inh}}/\theta$  versus  $C_{\text{inh}}$  with slopes around unity. This suggests that the adsorption of EEBb on the metal surface obeyed Langmuir adsorption isotherm. From the intercepts of the straight line  $C_{\text{inh}}/\theta$ -axis, K values have been calculated. It is based on the assumption that the adsorbed molecule decreases the surface area available for the corrosion reactions to occur.



**Figure 6:** C/θ vs. C at different temperature.

**Table 6:** Adsorption Parameters at different temperatures

Temperature (K)	72 h		Slope
	Correlation coefficient ( $R^2$ )	Adsorption coefficient (K)	
293	0.999	23.80	1.10
303	0.998	30.30	1.13
323	0.959	03.03	1.10
333	0.983	03.33	1.65
343	0.958	01.85	2.27
353	0.967	01.20	3.20

The value of  $R^2$  in the range ( $0.999 \geq r^2 \geq 0.959$ ) is nearly equal to one and slope was also found almost unit at lower temperature, thus indicative that the monolayer of the inhibitor species must have been attached to AA6063 surface without lateral interaction between the adsorbed species. At higher temperature deviation was noticed clearly distinguish the adsorption to be physioadsorption [19].

(V) QUANTUM CHEMICAL ANALYSIS:

To study the relationship between molecular structure and inhibitive effect of the investigated *EEBb*, a quantitative structure and activity relationship method was used. The calculated quantum chemical indices viz.  $E_{HOMO}$ ,  $E_{LUMO}$ ,  $\Delta E$  gap and  $\mu$  of major chemical constituents of *EEBb* has been tabulated in Table 7 and Fig. 7(a-d). The energy of the HOMO is often associated with the capacity of a molecule to donate electrons, whereas LUMO represents the ability of the molecule to accept electrons. Moreover, the gap between the HOMO and LUMO energy levels of the molecules ( $\Delta E$ ) increases (depend upon the cationic/anionic species) the reactivity of the molecule increases leading to increase in the inhibition efficiency of the molecule. For dipole moment ( $\mu$ ), higher values of  $\mu$  will favor strong interaction of the inhibitor molecules with metal surface and lower values favor the accumulation of inhibitor molecules around electrode surface. The heat of formation of the molecule is negative which suggests that the formation of this molecule is spontaneous and it is stable. Thus the results seem to indicate that both the values of the energy band gap,  $\Delta E$  as well as that of the dipole moment,  $\mu$  favours the potential of active constituent quercetin present in *EEBb* as efficient corrosion inhibitor [19-20].

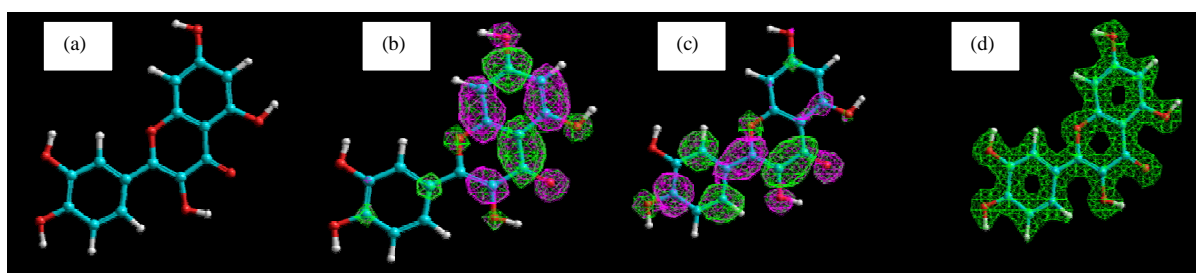


Figure 7(a-d): (a) The optimized structure of Quercetin, (b)  $E_{HOMO}$  (c)  $E_{LUMO}$  (d) 3D structure of total charge density.

Table 7: Quantum Chemical Indices of one of the major chemical constituents (Quercetin) of *Bidens biternata*

Name of the compound	Total Energy (kcal/mol)	Dipole Moment ( $\mu$ ) (Debye)	$E_{HOMO}$ (eV)	$E_{LUMO}$ (eV)	$\Delta E = E_{HOMO} - E_{LUMO}$
Quercetin	-77572.1	1.853	-7.089458	0.4589	6.6305558

(VI) UV-VISIBLE SPECTROSCOPY:

UV-Visible spectra were recorded for crude plant extract and for metal inhibited solution with maximum concentration (1.04 g/L) *EEBb* in 0.5 HCl after 72 h at 303 K. Fig. 8(a-b) indicate that *EEBb* showed main absorption band around 650–700 nm and absorbance at 0.100 which can be assigned to  $\pi \rightarrow \pi^*$  transitions. It was also observed from Fig. that a slight deviation in the position of ( $\pi \rightarrow \pi^*$ ) transition for the metal inhibited solution were observed and the absorbance also increased due to formation *EEBb*-metal complex, thus confirm the possibility of the formation of *EEBb*-metal complex [21].

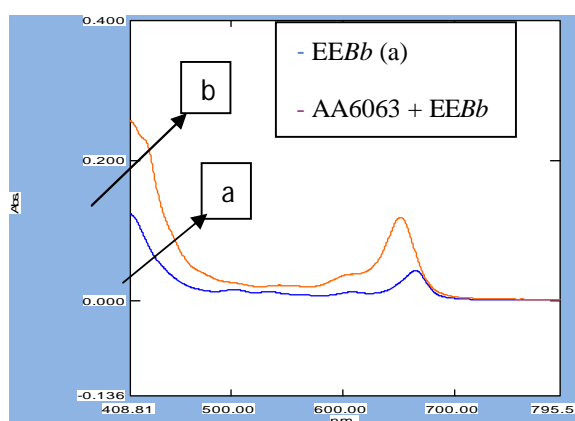


Figure 8: UV-visible spectra of the *EEBb* extract and test solution with *EEBb* in 0.5 M HCl at 72 h immersion at room temperature.

#### (VII) SURFACE MORPHOLOGICAL ANALYSIS (SEM):

SEM images of corroded and inhibited AA6063 surface are shown in fig. 9 (a-b). It can be seen from Fig. 9(a-b) that the metals surface after immersion in uninhibited 0.5 M HCl for 72 h shows a rough surface due to active dissolution in the acidic solution while in the presence of EEBb inhibitor surface appears to be even and smooth as compared to corroded samples due to formation film over metal surface [16-17].

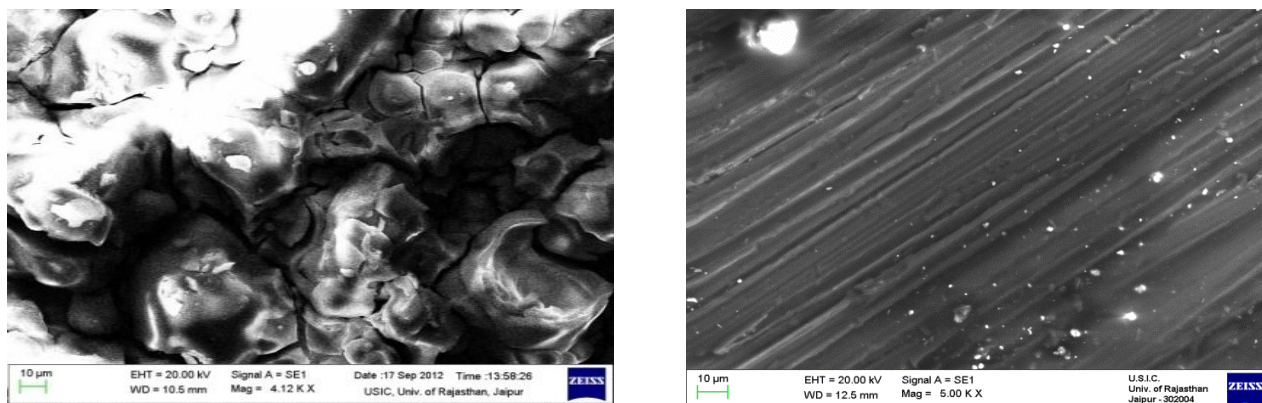


Figure 9: SEM micrographs at 72 h exposure in 0.5 M HCl (a) (Left) corroded surface; (b) (Right) inhibited with EEBb (1.04 g/L).

#### IV. CONCLUSIONS

The maximum inhibition efficiency (IE %) of the ethanolic extract of *Bidens biternata* (EEBb) towards AA6063 in 0.5 M HCl at 303 K was 86.29% at its high concentration (1.04 g/L). With elevation in temperature a considerable decrease in IE% was observed, and IE % was observed to increase at the lower temperature. With the addition of additive IE % was found to be 88% at 293 K temperature. Temperature studies were clearly indicative towards the nature of adsorption and furthermore, at higher temperature, the desorptive tendency of EEBb was clearly observed. Langmuir adsorption isotherm was observed to be best fitted. The values of  $\Delta G^{\circ}$  were close to -20 kJ/mol supporting the spontaneous and physical adsorption of EEBb. Quantum chemical analysis illustrated the adsorptive centers of the inhibitor active constituents. UV-Visible Spectroscopic study indicated the formation of inhibitor EEBb-metal complex thus endorsing the adsorptive nature of the active constituents of EEBb on to aluminium alloy. Surface morphological study by SEM confirmed the formation of a protective film over coupon-surface. The smoothness of the surface of coupons is due to the formation of a compact protective film of metal - EEBb complex on the metal surface thereby inhibiting the corrosion of AA6063 surfaces. It can be concluded that the ethanolic extract of *Bidens biternata* (EEBb) inhibit significantly aluminium alloy (AA6063) in 0.5 M HCl at lower temperature 293K and moderate inhibitory propensity is observed at elevated temperatures. Thus, the best used of a toxic weed, *Bidens biternata*, is to use as an anti-corrosive agent for aluminium alloy (AA6063) (best out of waste). As corrosion inhibitor, it can be a good replacement for many toxic chemicals, thus protecting metal-alloy as well as environment.

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