

Ethanol Stem Extract of *Costus Lucanusianus* as a Novel Ecofriendly Corrosion Inhibitor for Mild Steel and Aluminium in HCl

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ABSTRACT

Corrosion inhibition of ethanol stem extract of *Costus lucanusianus* (ESECL) was studied on mild steel and aluminium by weight loss method. The constituents of the extract identified by Gas Chromatography-Mass Spectroscopy (GC-MS) were forty one (41) compounds with 13-Octadecenal: (Z)-(14.39 %) and 9-Octadecenoic acid, 12-hydroxy-, methyl ester : [R-(Z)]- (11.49 %) as the major compounds. Weight loss method revealed that as the concentration of inhibitor increased, the corrosion rate decreased and inhibition efficiency increased. Maximum corrosion inhibition efficiencies of 91% and 85% were obtained in 1 g/l concentration at low temperatures for mild steel and aluminium, respectively. The adsorption followed physisorption mechanism. Positive values of ΔH suggested that the process was endothermic, and negative values of ΔS suggested a decrease in the disorderliness of the system from thermodynamic approach. The adsorption model showed that Freundlich adsorption isotherm was the best fit for the process.

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Introduction

Corrosion is the deterioration of metals or alloys when they are attacked by their environment. Corrosion degrades the useful properties of materials and structures including strength, appearance and permeability to liquid and gases (Oyewole et al., 2021).

Corrosion inhibitors are chemicals that react with a metallic surface or the environment this surface is exposed to, giving the surface a certain level of protection. The nature of the corrosion inhibitor depends on the materials being protected, which are most commonly metal objects, and the corrosive agent(s) to be neutralized. The corrosive agents are generally oxygen, hydrogen sulphide, and carbon dioxide. Some corrosion inhibitors form a passivating coating on the surface by chemisorption. The suitability of any given chemical for a task in hand depends on many factors, including their operating temperature. Geethamani (2019) highlighted that inhibition usually results from adsorption of corrosion inhibitors onto metals, the presence of surface charge on the metal, effect of functional group and structure and the interaction between adsorbed inhibitor species among other.

Green corrosion inhibitors are biodegradable and do not contain heavy metals or other toxic compounds. Natural-based corrosion inhibitors have gained great research interest: thanks to their low cost and higher performance (Zaher et al., 2021). Some research groups have reported the successful use of naturally occurring substances to inhibit the corrosion of metals in acidic and alkaline environment. (Rani and Basu, 2012) Plant extracts have the potential to replace synthetic organic and inorganic inhibitors given their success story in literature review and as such, this study explores the inhibition potential of ethanol stem extract of *Costus lucanusianus*.

Materials and Methods

Sample extraction and preparation

C. lucanusianus stem was collected, washed to remove debris and dried at room temperature. It was then milled into powder and completely soaked in ethanol solution for 72 hours. After the extraction, it was filtered. The filtration of the mixture was done firstly using cheesecloth, thereafter by Whatman grade 1 filter paper. The filtrate was subjected to evaporation using Stuart rotary evaporator (Re 300) to remove the solvent. 1 g/l, 0.7 g/l, 0.5g/l, 0.2g/l concentrations of the extract were prepared in 1.0 M HCl.

Gas Chromatography-Mass Spectroscopy (GC-MS)

The sample was analysed using Agilent technologies 7890A GC and 5977B MSD. The experimental conditions were set as follows: Hp 5-MS capillary standard non-polar column; Dimension: 30 m; ID: 0.25 mm; Film thickness: 0.5 μ m. Flow rate of mobile phase (carrier gas: He) was set at 1.0 ml/min. Oven temperature was raised from 298 K to 313 K at 278 K/min, and injection volume was 1 μ l. Samples dissolved in methanol were fully scanned at the range of 40-650 m/z and the results were compared by using NIST mass spectral library search programme.

Weight loss measurement

Mild steel and aluminium (4 \times 4 cm dimensions) used for this research were polished with emery paper (100-1200 grade), was degreased with ethanol and dried with acetone. After taking the initial weight of the coupons, they were dipped into various concentrations of the acid solution prepared at 303 K to 333 K. Coupons were retrieved after two (2) hours, washed with distilled water, scrubbed with bristle brush, rinsed with ethanol, dried in acetone and reweighed at two (2) hours interval progressively for 10 hours.

Using Equation 1, the weight loss was taken to be the difference between the initial weight and the weight of the coupons at 2 hours intervals.

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$$\Delta W = (W_0 - W_1) \text{ g} \quad \text{Equation 1}$$

The corrosion rate (CR), surface coverage (Θ) and inhibition efficiency (% IE) were computed using the Equations 2 – 4, respectively

$$\text{CR} = \frac{\Delta W}{S t} (\text{g/cm}^2 \text{hr}) \quad \text{Equation 2}$$

$$\Theta = \frac{\text{CR}_0 - \text{CR}_1}{\text{CR}_0} \quad \text{Equation 3}$$

$$\% \text{ IE} = \frac{\text{CR}_0 - \text{CR}_1}{\text{CR}_0} \times 100 \quad \text{Equation 4}$$

Where ΔW is the weight loss, S is the total surface area of the coupons, t is the corrosion time (10hrs), CR_0 is the corrosion rate in the absence of inhibitor, while CR_1 is the corrosion rate in the presence of inhibitor.

Results and Discussion

Gas Chromatography-Mass Spectroscopy (GC-MS)

The 41 identified compounds with their retention time, percentage area, compound name, CAS number and structures of ESECL are listed in Table 1. 13-Octadecenal: (Z)-(14.39 %) and 9-Octadecenoic acid, 12-hydroxy-, methyl ester: [R-(Z)]- (11.49 %) are the abundant component of ESECL. Double bonds and heteroatoms are found in the structures of the identified compounds contain double bonds and oxygen, which suggests the use of the stem extract of *C. lucanusianus* as corrosion inhibitor.

Weight loss analysis

Weight loss test is one of the most straightforward methods to investigate the degree of metal corrosion in acidic solution (Li *et al.*, 2021). The immersion time is an important parameter in assessing the stability of corrosion inhibitive properties of compounds (Vorobyova and Skiba, 2021). The corrosion rate and inhibition efficiency of mild steel and aluminium in HCl with and without ESECL are given in Table 2. The data shows that corrosion rate was highest in the blank solution. However, upon the addition of various concentrations of the inhibitor, corrosion rate reduced, indicating that the corrosion has been inhibited (Ituen *et al.*, 2021). The inhibition efficiency increased as the concentration of the inhibitors increased. According to the results, the maximum inhibition efficiency of 91 % and 85 % was obtained at 1 g/l concentration at low temperatures for mild steel and aluminium respectively, implying that the ESECL exhibited a good anticorrosion effect.

Effect of temperature and activation parameters

Considering the well-known effect of temperature on most chemical reactions, a better understanding of the corrosion inhibition process can be achieved by studying the effect of temperature on the inhibition performance of tested inhibitor (Zaher *et al.*, 2021). Figure 1 shows the effect of temperature on the corrosion rate investigated using weight loss experiments at 303 K, 313 K, 323 K and 333 K. Increase in temperature, increased the corrosion rate of the metals. Nevertheless, in the optimal extract concentration of 1 g/l, maximum inhibition efficiency of 91% and 85 % at 313 K and 303 K were obtained for mild steel and aluminium respectively.

The relationship between the corrosion rate (CR) of metals in acidic media and temperature (T) is often expressed by the Arrhenius equation (Obi-egbedi *et al.*, 2012)

$$\log \text{CR} = \log A - \frac{E_a}{2.303RT} \quad \text{Equation 5}$$

Where CR signifies the corrosion rate of mild steel and aluminium determined from the weight loss experiment, A signifies the pre-exponential constant, E_a signifies the apparent activation energy, R signifies the universal gas constant and T signifies the temperature,

The plot of $\log \text{CR}$ against $10^3/T$ for mild steel and aluminium in 1 M HCl in the absence and presence of different concentrations of ESECL is presented in Figure 2. The calculated activation energies are given in Table 3. E_a in the inhibited solution is higher than the blank solution, indicating that the corrosion reaction of mild steel and aluminium was inhibited by ESECL and it also indicated that the energy barrier was increased, thereby emphasizing on the electrostatic character of the inhibitor's adsorption on the mild steel and aluminium surfaces (Obot and Obi-Egbedi, 2008)

Thermodynamic parameters

Transition state equation (Equation) gives the thermodynamic parameters

$$\log \frac{\text{CR}}{T} = \log \left(\frac{R}{N h} \right) + \left(\frac{\Delta S}{2.303R} \right) - \left(\frac{\Delta H}{2.303RT} \right) \quad \text{Equation 6}$$

Where CR signifies the corrosion rate of mild steel and aluminium determined from the thermo-gravimetric experiment, R signifies the universal gas constant, T signifies the temperature, N signifies the Avogadro's number, h signifies Planck's constant, ΔH signifies enthalpy change and ΔS signifies entropy change.

ΔH (enthalpy change) and ΔS (entropy change) were calculated from the data obtained from the plot of $\log \text{CR}/T$ versus $10^3/T$ (Figure 3) for mild steel and aluminium in 1 M HCl in the absence and presence of different concentrations of ESECL. The data presented in Table 3 shows positive values of ΔH suggesting that mild steel and aluminium dissolution processes are endothermic in nature, and ΔH values increased with increased concentration of ESECL (Saraswat *et al.*, 2020). Negative values of ΔS indicated a decrease in the disorderliness of the system during the corrosion process.

Adsorption consideration

The basic information on the interaction between the extract and metal surfaces is provided by adsorption isotherms. These were investigated using Langmuir, Temkin, Freundlich and El-Awady isotherms. All these isotherms can be expressed using the following equations respectively (Ituen and Udo, 2012):

$$\frac{C}{\theta} = \frac{1}{K_{\text{ads}}} + C \quad \text{Equation 7}$$

$$\theta = -\frac{1}{2a} \log C - \frac{1}{2a} \log K \quad \text{Equation 8}$$

$$\log \theta = \log K + n \log C \quad \text{Equation 9}$$

$$\log \frac{\theta}{1-\theta} = \log K^* + y \log C \quad \text{Equation 10}$$

Where C is the concentration, K_{ads} is the adsorption equilibrium constant, a is the molecular interaction parameter used to predict the nature of interactions in the adsorbed layer and Θ is the surface coverage (Ituen *et al.*, 2017). The coverage degree (θ) of the surface could be plotted as a function of the concentration of inhibitor tested and the results were made compatible with different isotherm models of adsorption (Jesudoss *et al.*, 2020). The Freundlich adsorption isotherm provided the best fit. Figure 4 shows the linear plots of $\log \Theta$ against $\log C$ for Freundlich adsorption isotherm using $\log \theta = \log K + n \log C$, where n and $\log K$ gives the slope and intercept respectively.

The correlation coefficient obtained from the plots (Figure 4) which is in the range ($0.9472 \leq R^2 \leq 0.9793$) for mild steel and ($0.7315 \leq R^2 \leq 0.9833$) for aluminium as shown in table 4.

The free energy of adsorption ΔG_{ads}^0 was obtained as follows (Fouda *et al.*, 2021):

$$K_{ads} = \frac{1}{55.5} \exp \frac{-\Delta G_{ads}^0}{RT} \quad \text{Equation 11}$$

The calculated ΔG_{ads}^0 values are tabulated in table 4. The negative values of free energy of adsorption show that the adsorption of ESECL on mild steel and aluminium surfaces was spontaneous (Tan *et al.*, 2021) (Khamis *et al.*, 2001; Ituen *et al.*, 2017). The mechanism of adsorption was physisorption since the value of $\Delta G_{ads} \leq -20 \text{ KJmol}^{-1}$ (Oguzie *et al.*, 2010; El-Awady *et al.*, 2008; El-Sherif *et al.*, 2011).

Conclusion

Ethanol stem extract of *Costus lucanusianus* (ESECL) characterized by GC-MS investigated as an inhibitor for mild steel and aluminium corrosion in HCl using weight loss measurement yielded maximum inhibition efficiencies of 91% and 85 % at 313 K and 303 K for mild steel and aluminium, respectively. The inhibition efficiency of the

extract increased with increase in concentration of ESECL and decreased with increase in temperature. 13-Octadecenal, (Z)- and 9-Octadecenoic acid, 12-hydroxy-, methyl ester, [R-(Z)]- were identified as the major compounds principally accountable for the inhibition because of their high percentage areas of 14.39 and 11.49, respectively.

E_a in the inhibited solution is higher than the blank solution, indicating physisorption. Positive values of ΔH suggested that the process was endothermic and negative values of ΔS indicated a decrease in the disorderliness of the system from thermodynamic approach. The adsorption model showed that Freundlich adsorption isotherm was the best fit for the adsorption process. The negative values of free energy of adsorption showed that the adsorption of ESECL on mild steel and aluminium surfaces was spontaneous

Table 1. Identified components of ESECL

S/N	RT	Area %	Library/ID	CAS	Structure
1	5.2371	-2.49	7-Hexadecenoic acid, methyl ester, (Z)-	056875-67-3	
2	7.2328	1.23	Benzofuran, 2,3-dihydro-	000496-16-2	
3	13.0791	2.51	Dodecanoic acid, methyl ester	000111-82-0	
4	15.0406	3.29	Dodecanoic acid, ethyl ester	000106-33-2	
5	18.2788	0.76	Undecanoic acid, methyl ester	001731-86-8	
6	19.839	0.81	Ethyl tridecanoate	028267-29-0	
7	20.988	0.36	13-Pentacosanone	002123-19-5	
8	22.6451	1.72	Hexadecanoic acid, methyl ester	000112-39-0	
9	24.0063	0.93	Hexadecanoic acid, ethyl ester	000628-97-7	
10	25.2534	7.03	13-Hexyloxacyclotridec-10-en-2-one	127062-51-5	
11	25.9105	0.97	8,11-Octadecadienoic acid, methyl ester	056599-58-7	
12	26.0377	1.64	11-Octadecenoic acid, methyl ester, (Z)-	001937-63-9	
13	26.5491	0.55	Heptadecanoic acid, 14-methyl-, methyl ester	002490-23-5	
14	27.031	1.21	Linoleic acid ethyl ester	000544-35-4	
15	27.1603	0.75	9-Oxabicyclo[6.1.0]nonane	000286-62-4	
16	27.2753	1.91	cis-7,cis-11-Hexadecadien-1-yl acetate	052207-99-5	
17	27.7819	6.17	Eicosanoic acid	000506-30-9	
18	27.9128	1.16	9,12-Octadecadienoic acid (Z,Z)-	000060-33-3	
19	28.23	5.43	9,12-Octadecadienoic acid (Z,Z)-	000060-33-3	
20	28.4554	3.38	9,12-Octadecadienoic acid (Z,Z)-	000060-33-3	
21	28.8215	6.83	Cyclopropaneoctanal, 2-octyl-	056196-06-6	
22	29.2142	3.63	9,12-Octadecadienoyl chloride, (Z,Z)-	007459-33-8	
23	29.543	11.49	9-Octadecenoic acid, 12-hydroxy-, methyl ester, [R-(Z)]-	000141-24-2	
24	30.6462	1.43	2-Methyl-Z,Z-3,13-octadecadienol	1000130-90-5	
25	31.7064	0.32	2-Methyl-Z,Z-3,13-octadecadienol	1000130-90-5	
26	31.8	0.42	9,17-Octadecadienal, (Z)-	056554-35-9	
27	32.554	7.37	12-Methyl-E,E-2,13-octadecadien-1-ol	1000130-90-4	
28	32.9814	1.34	3-Methyl-4-(phenylthio)-2-prop-2-enyl-2,5-dihydrothiophene 1,1-dioxide	1000305-61-3	

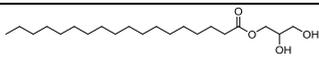
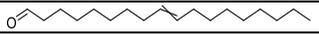
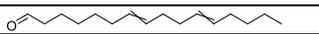
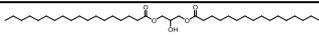
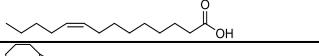
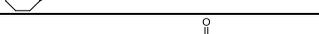
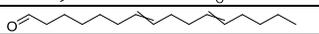
29	33.3479	6.74	Octadecanoic acid, 2,3-dihydroxypropyl ester	000123-94-4	
30	34.7863	0.33	9-Octadecenal	005090-41-5	
31	34.9038	0.35	2-Methyl-Z,Z-3,13-octadecadienol	1000130-90-5	
32	35.4853	0.80	7,11-Hexadecadienal	1000130-85-7	
33	35.6431	1.21	7,11-Hexadecadienal	1000130-85-7	
34	36.131	14.39	13-Octadecenal, (Z)-	058594-45-9	
35	36.5033	2.02	Octadecanoic acid, 2-hydroxy-1,3-propanediyl ester	000504-40-5	
36	37.362	0.44	Myristoleic acid	000544-64-9	
37	37.5207	0.42	9-Oxabicyclo[6.1.0]nonane	000286-62-4	
38	37.8008	0.37	E-11-Hexadecenoic acid, ethyl ester	1000245-71-9	
39	38.2045	0.53	7,11-Hexadecadienal	1000130-85-7	
40	38.3114	0.05	Oleic Acid	000112-80-1	
41	38.4975	0.22	9-Oxabicyclo[6.1.0]nonane	000286-62-4	

Table 2. Calculated Values Of Corrosion Rate (Cr), Degree Of Surface Coverage (Θ) And Inhibition Efficiency (% Ie) Of Mild Steel And Aluminium At 303 K To 333 K In The Presence And Absence Of Esecl.

Conc. (g/L)	303 K			313 K			323 K			333 K		
	CR g/cm ² h×10 ⁻³	Θ	%IE	CR g/cm ² h×10 ⁻³	Θ	%I E	CR g/cm ² h×10 ⁻³	Θ	%IE	CR g/cm ² h×10 ⁻³	Θ	%IE
	MILD STEEL											
Blank	1.91	-	-	9.80	-	-	13.90	-	-	14.52	-	-
0.1	1.35	0.29	29	6.58	0.33	33	11.27	0.19	19	13.90	0.04	4
0.2	1.27	0.33	33	4.77	0.51	51	9.51	0.32	32	13.14	0.09	9
0.5	0.79	0.59	59	1.38	0.86	86	8.20	0.41	41	11.50	0.21	21
0.7	0.46	0.76	76	1.27	0.87	87	5.84	0.58	58	9.41	0.35	35
1	0.31	0.84	84	0.87	0.91	91	2.96	0.79	79	3.76	0.74	74
	ALUMINIUM											
Blank	4.61	-	-	5.84	-	-	9.48	-	-	10.51	-	-
0.1	4.31	0.06	6	5.70	0.03	3	8.83	0.07	7	9.46	0.10	10
0.2	4.29	0.07	7	5.46	0.07	7	8.62	0.09	9	9.44	0.10	10
0.5	3.47	0.25	25	4.80	0.18	18	8.40	0.11	11	8.31	0.21	21
0.7	1.84	0.60	60	4.74	0.19	19	7.40	0.22	22	8.17	0.22	22
1	0.71	0.85	85	3.73	0.36	36	2.94	0.69	69	2.61	0.75	75

Table 3. Activation parameters from Transition State equation for mild steel in different concentrations of ESECL at 303 K to 333 K

Coupons	Conc. (g/L)	Activation parameters		
		E _a (J/mol)	ΔH (J/mol)	ΔS (J/mol.K)
Mild steel	Blank	23.26	50.9462	-125.3273
	0.1	27.19	60.0039	-98.8651
	0.2	27.78	61.3595	-95.8576
	0.5	35.46	79.0581	-44.2690
	0.7	38.20	85.3512	-26.8594
	1	31.47	69.8659	-80.5645
Aluminium	Blank	10.68	21.9722	-217.3008
	0.1	10.10	20.6430	-222.0482
	0.2	10.19	20.8568	-221.5252
	0.5	11.49	23.8362	-213.1687
	0.7	17.75	38.2667	-169.3555
	1	13.30	28.0051	-208.4675

Table 4. Adsorption parameters from Freundlich isotherm for mild steel and aluminium in different concentrations of ESECL at 303 K to 333 K

Inhibitor	Temp (K)	Adsorption parameters		
		K _{ads} (mol/l)	ΔG (kJ/mol)	R ²
Mild steel	303 K	0.9309	-4.0905	0.9675
	313 K	1.0096	-4.5829	0.9472
	323 K	0.8663	-4.0578	0.9595
	333 K	0.7969	-3.8486	0.9793
Aluminium	303 K	0.8862	-3.8942	0.9367
	313 K	0.6205	-2.8164	0.9833
	323 K	0.6462	-3.0271	0.7315
	333 K	0.6950	-3.3561	0.7627

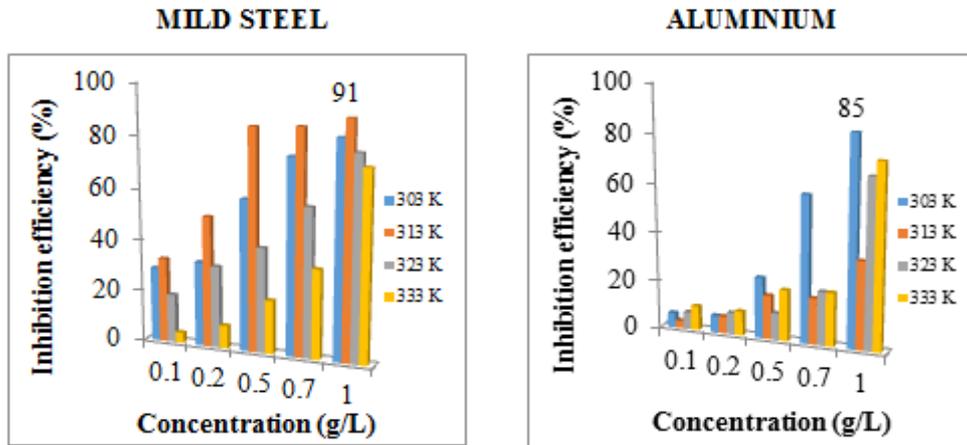


Figure 1. Variation of inhibition efficiencies of ESECL with concentration and temperature for mild steel and aluminium

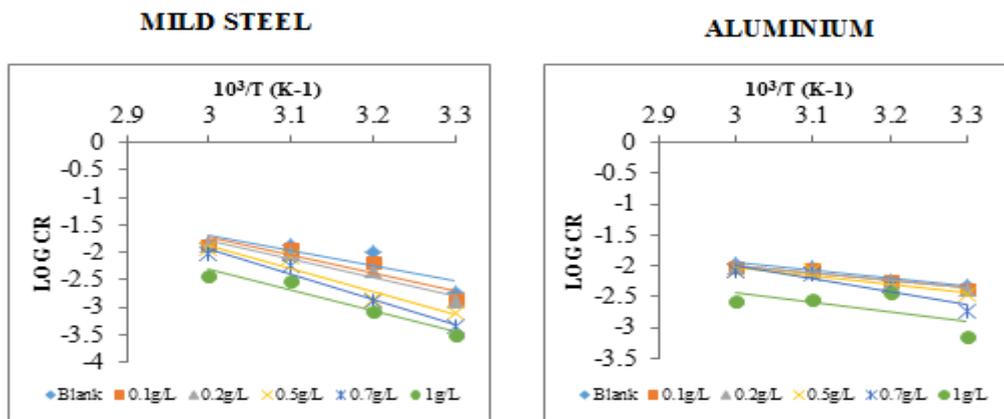


Figure 2. Arrhenius plot for mild steel and aluminium in different concentrations of ESECL

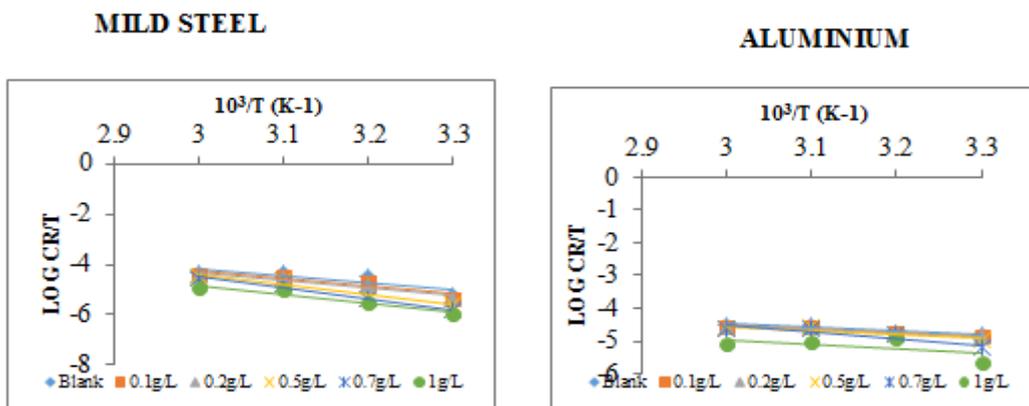


Figure 3. Transition state plot for mild steel and aluminium in different concentrations of ESECL.

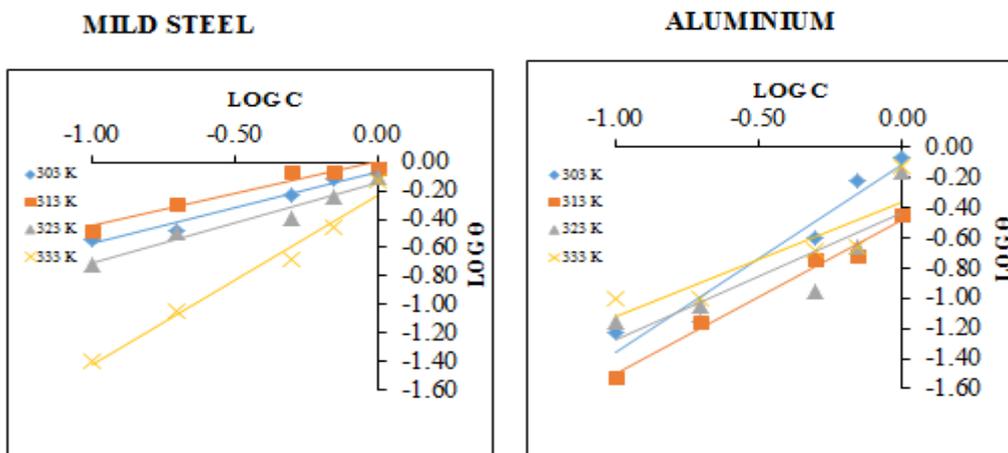


Figure 4. Freundlich adsorption isotherm plot (C/θ versus C) for mild steel and aluminium.

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