

KINETICS CONSIDERATION OF ETHANOL LEAVES EXTRACT OF COSTUS LUCANUSIANUS AS GREEN CORROSION INHIBITOR FOR MILD STEEL AND ALUMINIUM IN HCL SOLUTION



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ABSTRACT

The chemical composition of ethanol leaves extract of *Costus lucanusianus* (ELECL) was evaluated by Gas Chromatography-Mass Spectroscopy (GC-MS) for corrosion inhibition of mild steel and aluminium in 1 M HCl using weight loss technique. A total of 18 compounds were identified. 11-Octadecenoic acid, methyl ester (30.01%); 9-Octadecenoic acid, 12-hydroxy-, methyl ester, [R-(Z)]- (25.53%); 9,12-Octadecadienoic acid, methyl ester (13.52%); and Hexadecanoic acid, methyl ester (13.14%) were the major counpounds. The weight loss analysis showed ELECL as an effective corrosion inhibitor at low temperatures for mild steel and aluminium. At 1 g/l concentration, the inhibitory action of the extracts attained an inhibition efficiency of 94% and 79% in 1 g/l at 313 K and 303 K for mild steel and aluminium respectively. The extracts inhibitor compounds covered the metal surfaces following Freundlich adsorption isotherm. The enthalpy change showed an endothermic process while the entropy change showed an orderly adsorption of the inhibitor molecules on the metal surfaces.

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Keywords: Corrosion, Inhibition, Gas Chromatography-Mass Spectroscopy, Adsorption Isotherm, Corrosion Kinetics

1. INTRODUCTION

Corrosion is an electrochemical process which generally occurs in the presence of oxygen, aqueous electrolyte solution and moisture. It could be over the entire surface of a metal or alloy, and it occurs electrochemically between two different metallic materials or two points on the surface of alloys of different chemical activity Potgieter (2010). The rate of corrosion is expressed based on the loss per unit time. The rate at which the attack takes place is of prime importance and is usually expressed in one of the two ways: Weight loss per unit area per unit time and decrease in thickness per unit time (that is, rate of penetration or the thickness of metal) Geethamani (2019).

Inhibition is a preventive measure against corrosive attack on metallic materials. One of the extensively studied topics in the field of corrosion is inhibition Geethamani (2019). Corrosion inhibitors are chemical substances added to a liquid or gas, that decrease the corrosion rate or prevent corrosion of the metal, when added in small amounts to the environment in which a metal would corrode. The effectiveness of a corrosion inhibitor depends on fluid composition, quantity of water, and flow regime. A common mechanism for inhibiting corrosion involves formation of a coating, often a passivation layer, which prevents access of the corrosive substance to the metal. However,

corrosion inhibitors are additives to the fluids that surround the metal Guo et al. (2020).

The phenomenon of adsorption is influenced by the nature and surface charge of the metal and chemical structure of inhibitors. Inhibitors often work by adsorbing themselves on the metallic surface to protect the metallic surface by forming a film. Inhibitors are normally distributed from a solution or dispersion. Some are included in a protective coating formulation. According to Rani and Basu (2012), inhibitors decrease or prevent the reaction of the metal with the media. They reduce the corrosion rate by adsorption of ions/molecules onto metal surface, increasing or decreasing the anodic and/or cathodic reaction, decreasing the diffusion rate for reactants to the surface of the metal and decreasing the electrical resistance of the metal surface.

Literature study showed that numerous plant parts extracts including bark, leave, fruit, peel, seed, root, flower, and even entire plant extracts are widely used as corrosion inhibitors. Out of several extracts, leaves extract generally showed reasonably better protection effectiveness at relatively low concentration Alrefaee et al. (2020). Plants are natural, green, renewable, easy to access, and contain many heterocyclic organics, thereby making it one of the most suitable in replacing conventional toxic inhibitors Li et al. (2021). Consequently, plant extracts as green and effective corrosion inhibitors are wildly explored. The mechanism of action for green inhibitors depends on the structure of the active ingredient. The adsorption of natural corrosion inhibitors on metal surfaces is influenced by a number of factors including nature of metal, testing media, chemical structure of inhibitor, nature of the inhibitor constituents, presence of additives, solution temperature, and solution concentration Verma and Quraishi (2016).

Costus lucanusianus (Figure 1) commonly known as monkey sugarcane is a vigorous grower and a medicinal plant used to treat various ailments in tropical Africa. *C. lucanusianus* is very similar to *Costus afer* (bush cane), differing in their hairy leaf sheath. Besides being used as a medicinal plant, it is often used as ornamental for its flowers with showy labellum which last singularly one day only but are continuously produced. In southern Nigeria, *Costus afer* and *C. lucanusianus* produce hybrids. The methanol and n-hexane extracts have been reported to act as inhibitors. However, they contained different constituents Obot et al. (2021a), Obot et al. (2021b). Therefore, this study seeks to investigate the constituents of the ethanol extract and its inhibitory potential on mild steel and aluminium in 1 M HCl.



Figure 1 Costus lucanusianus plant

2. MATERIALS AND METHODS 2.1. SAMPLE COLLECTION PREPARATION

The fresh leave samples of *C. lucanusianus* leave were rinsed and dried at room temperature, crushed and ground into fine powder. Extraction with ethanol was performed. 70 g leaves were soaked in 900ml ethanol for 72 hours and filtered. The filtrate was allowed to evaporate completely 40°C using Stuart Rotatory Evaporator (Re 300) to remove the ethanol solvent. Different weights of the extract were measured and used to prepare five different concentrations (1 g/l, 0.7 g/l, 0.5g/l, 0.2g/l 0.1g/l) in 1 M HCl.

2.2. COUPONS

The experiment was conducted on mild steel with the following composition carbon – 0.17%, silicon – 0.26%, manganese – 0.46%, phosphorus – 0.0047%, sulphur – 0.017%, iron – 98.9%) and pure aluminium metal of the type of AA 1060. The coupons with dimensions 4×4 cm was used for weight loss measurements. For the insertion of hook, a hole was drilled at the top centre of the coupons. The coupons were brushed with series of emery paper of variable grades starting with the course (100) to the finest (1200) grade to obtain a smooth surface free of grooves. Each coupon was degreased by washing with ethanol, dried with acetone and stored in a desiccator.

2.3. GAS CHROMATOGRAPHY-MASS SPECTROSCOPY (GC-MS)

The chromatographic analysis of the ethanol stem extract of *C. lucanusianus* was carried out using Agilent technologies 7890A GC and 5977B MSD. The system was equipped with Hp 5-MS capillary standard non-polar column (30 m × 0.25 mm × 0.5 μ m film thickness). The temperature was set from 25°C to 40°C at 5°C/min and injection volume was 1 μ l. Helium gas was used as a carrier gas with a constant flow rate of 1.0 ml/min. 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.

2.4. WEIGHT LOSS EXPERIMENT

Weight loss experiment was performed to determine the concentration influence of ethanol extracts of *C. lucanusianus* on mild steel and aluminium coupons. the coupons were immersed in a test solution containing different concentrations of the extract. The system was maintained at 303 K, 313 K, 323 K and 333 K using a thermostat water bath. After immersion, the coupons were washed with distilled water, scrubbed with bristled brush to remove the corrosion product, cleansed with ethanol, dried in acetone and reweighed at 2-hours intervals 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

$$\Delta W = (W_0 - W_1) g$$

Equation 1

The corrosion rate (CR), surface coverage (Θ) and inhibition efficiency (%IE) were computed using the Equation 2, Equation 3, Equation 4 respectively

$$CR = \frac{\Delta W}{St} (g/cm^2hr)$$
 Equation 2

Where ΔW is the weight loss, S is the total surface area of the coupons and t is the corrosion time (10hrs).

$$\theta = \frac{CR_0 - CR_1}{CR_0}$$
 Equation 3

% IE =
$$\frac{CR_0 - CR_1}{CR_0} X 100$$
 Equation 4

Where CR_0 is the corrosion rate in the absence of inhibitor, while CR_1 is the corrosion rate in the presence of inhibitor.

3. RESULTS AND DISCUSSION 3.1. GAS CHROMATOGRAPHY-MASS SPECTROSCOPY (GC-MS)

The GC-MS analysis of ELECL showed the presence of 18 compounds compounds. Table 1 presents the identified compounds of ELECL with their retention time, pecentage area, compound name, CAS number and structures.

Table 1 Identified components of ELECL							
S/ N	RT	Area %	Library/ID	CAS	Structure		
1	13.826 8	0.38	Dodecanoic acid, 2-methyl-	002874- 74-0	о Цон		
2	18.433 6	0.6	Methyl tetradecanoate	000124- 10-7	° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° °		
3	22.219	0.71	7-Hexadecenoic acid, methyl ester, (Z)-	056875- 67-3			
4	22.694 7	13.1 4	Hexadecanoic acid, methyl ester	000112- 39-0	0 		
5	25.265 5	1.08	Linoelaidic acid	000506- 21-8	° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° °		
6	25.947 5	13.5 2	9,12- Octadecadienoi c acid, methyl ester	002462- 85-3	° °		
7	26.092 8	30.0 1	11- Octadecenoic acid, methyl ester	052380- 33-3	°		

Kinetics Consideration of Ethanol Leaves Extract of *Costus Lucanusianus* as Green Corrosion Inhibitor for Mild Steel and Aluminium in Hcl Solution

8	26.171 6	1.76	9-Octadecenoic acid (Z)-, methyl ester	000112- 62-9	0
9	26.570 5	6.97	Methyl stearate	000112- 61-8	
10	26.893 1	1.6	9,12- Octadecadienoi c acid (Z, Z)-, methyl ester	000112- 63-0	
11	29.572 5	25.5 3	9-Octadecenoic acid, 12- hydroxy-, methyl ester, [R-(Z)]-	000141- 24-2	
12	29.663 4	0.8	7-Octadecenoic acid, methyl ester	057396- 98-2	° Lor
13	32.474	0.39	9,17- Octadecadienal, (Z)-	056554- 35-9	۵ ۵
14	32.554 7	1.41	2-Methyl-Z, Z- 3,13- octadecadienol	1000130 -90-5	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~
15	36.105 3	1.05	9,17- Octadecadienal, (Z)-	056554- 35-9	//////////////////////////////////////
16	36.131 4	0.88	9,12- Octadecadien- 1-ol, (Z, Z)-	000506- 43-4	HO
17	38.258 6	0.09	9-Octadecenal, (Z)-	002423- 10-1	0~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~
18	38.398 7	0.08	Hexanoic acid, heptadecyl ester	1000282 -83-9	°

The identified compounds contain oxygen as a heteroatom in their structures. This property suggests the use of the leave extract of *C. lucanusianus* as corrosion inhibitor. 11-Octadecenoic acid, methyl ester (30.01%); 9-Octadecenoic acid, 12-hydroxy-, methyl ester, [R-(Z)]- (25.53%); 9,12-Octadecadienoic acid, methyl ester (13.52%); and Hexadecanoic acid, methyl ester (13.14%) was identified as the predominant compounds.

4. WEIGHT LOSS ANALYSIS

The weight loss experiment was carried out to determine ELECL shielding proficiency at consecutively increasing concentrations. The 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 ELECL are tabulated in Table 2.

The data shows that the inhibition process was a concentration dependent one. While the concentration of the extracts increased, the corrosion rate decreased. Therefore, the corrosion inhibition activity of ELECL advanced with concentration; and showed maximum inhibitive effect of 94% and 79% for mild steel and aluminium, respectively at low temperature. The results also suggest larger corrosion protection of the steel surface that is confirmed by the rising value of

surface coverage (θ) Jesudoss et al. (2020). The ability of the extract's natural chemical constituents to adsorb onto the metal surface is mainly the reason for its corrosion resistance ability Zaher et al. (2021). The proportion of surface covered with adsorbed extract molecules determines the amount of corrosion protection. Therefore, as the adsorbed molecules of extract grow on the metal surface, its concentration increases, thereby making the θ value a critical variable since it reflects the percentage of metal substrate surface covered by extract molecules Zaher et al. (2021)

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 ELECL

	303 K			313 K	-	_	323 K		-	333 K	-	
Conc. (g/L)	CR g/cm ² h× 10 ⁻³	θ	%IE	CR g/cm ² h× 10 ⁻³	θ	%IE	CR g/cm ² h× 10 ⁻³	θ	%IE	CR g/cm ² h× 10 ⁻³	θ	%IE
					MI	LD STE	EL					
Blank	1.93	-	-	10.93	-	-	13.96	-	-	17.05	-	-
0.1	1.87	0.03	3	6.61	0.39	39	11.88	0.15	15	16.34	0.04	4
0.2	1.81	0.06	6	6.33	0.42	42	9.9	0.29	29	14.72	0.14	14
0.5	1.35	0.3	30	5	0.54	54	5.69	0.59	59	13.59	0.2	20
0.7	1.17	0.39	39	1.71	0.84	84	5.3	0.62	62	9.39	0.45	45
1	0.62	0.68	68	0.61	0.94	94	2.15	0.85	85	5.4	0.68	68
					AL	JMINIU	M					
Blank	5.35	-	-	8.82	-	-	11.42	-	-	12.28	-	-
0.1	3.27	0.39	39	5.7	0.35	35	9.49	0.17	17	9.52	0.23	23
0.2	3.17	0.41	41	5.42	0.39	39	8.66	0.24	24	9.32	0.24	24
0.5	2.24	0.58	58	5.22	0.41	41	8.1	0.29	29	8.51	0.31	31
0.7	2.03	0.62	62	4.11	0.53	53	7.84	0.31	31	8.53	0.31	31
1	1.13	0.79	79	3.85	0.56	56	5.46	0.52	52	3.98	0.68	68

4.1. EFFECTT OF TEMPERATURE

The weight loss analysis of mild steel and aluminium was studied in the temperature range 303 K – 333 K in inhibited and uninhibited solutions. Figure 2 shows the variation of inhibition efficiencies of ELECL with concentration and temperature for mild steel and aluminium. The inhibition efficiency (%IE) for mild steel and aluminum increased with increasing concentrations of the extract. The maximum inhibition efficiencies (94 % and 79 %) at 1 g/l were attained at 313 K and 303 K for mild steel and aluminium, respectively. This indicates that the extract molecules are adsorbed on metal-solution interface forming a protected layer on the metal surface that hindered its corrosion Fouda et al. (2021). However, decrease in the extract efficiency with increasing temperature indicates desorption of extract molecules on the metal surfaces. This behaviour is consistent with physical adsorption mechanism Ituen et al. (2021)

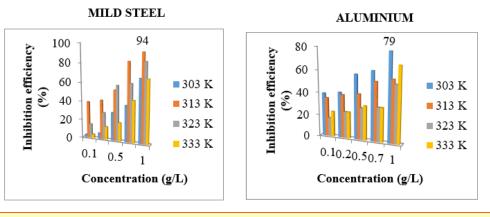


Figure 2 Variation of inhibition efficiencies of ELECL with concentration and temperature for mild steel and aluminium

4.2. MECHANISM OF ADSORPTION

The inhibitor molecules adsorption onto metal surface is divided into chemical and physical adsorption Tan et al. (2021). This process gives the interation details between the inhibitors and metal surfaces. However, while physical adsorption involves interaction between the charged corrosion inhibitor and the charged metal, chemical adsorption involves the formation of coordination bonds with the metal unoccupied orbitals with lone pairs of heteroatoms in the inhibitor molecule. Variety of adsorption isotherms including Langmuir, Temkin, Fruendlich and El-Awady isotherms were used to fit the weight loss data. These isotherms were expressed using the following equations respectively Ituen and Udo (2012):

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C$$
 Equation 5

$$\theta = -\frac{1}{2a}\log C - \frac{1}{2a}\log K$$
 Equation 6

$$\log \theta = \log K + n \log C$$
 Equation 7

$$\log \frac{\theta}{1-\theta} = \log K^* + y \log C$$
 Equation 8

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.

The freundlich adsorption isotherm (Figure 3) provided the best fit and described the adsorption behaviour of the extracts of *C. lucanusianus* presented in Table 3. Investigation of the adsorption characteristics as provided in Table 3, showed that the inhibitor exhibited more of a monolayer chemical adoption Ugi

(2020) as data fit accurately to the isotherm as seen in the correlation coefficient obtained from the plots (Figure 3) which are in the range ($0.8767 \le R^2 \le 0.9904$) for mild steel and ($0.6486 \le R^2 \le 0.9355$) for aluminium.

The value of ΔG_{ads}^0 was obtained by following: Zhang et al. (2018), Li et al. (2018), Fatima et al. (2019)

$$K_{ads} = \frac{1}{55.5} exp \frac{-\Delta G_{ads}^0}{RT}$$
 Equation 9

the negative values of ΔG_{ads}^0 indicate a spontaneous adsorption of the inhibitor molecules on the metal surfaces. The ΔG_{ads}^0 value determines the type of adsorption. When the value is greater than -20 kJ/mol, it reveals that the charged corrosion inhibitor and the charged metal have physical adsorption through electrostatic attraction Sadeghi et al. (2019). When the value is less than -40 kJ/mol, it reveals that the interaction between the inhibitor molecules and the metal surface is chemisorption Biswas et al. (2018). When the value is between -20 kJ/mol and -40 kJ/mol, it reveals that it is a mixed physical and chemical adsdorption process Zhang et al. (2018). From Table 3, it can be seen that the ΔG_{ads}^0 values are greater than -20 kJ/mol at varied temperatures, therefore the mechanism of adsorption followed by ELECL is believed to be physical adsorption.

Table 3 Adsorption parameters from Freundlich isotherm for mild steel and aluminium in different concentrations of ELECL at 303 K to 333 K

Inhibitor	Temp (K)	Adsorption parameters					
		Kads (mol/l)	∆G (kJ/mol)	R ²			
Mild steel	303 K	0.8458	-3.7163	0.9904			
	313 K	0.9409	-4.2708	0.8767			
	323 K	0.9444	-4.4237	0.9836			
	333 K	0.8156	-3.9387	0.9456			
Aluminium	303 K	0.8711	-3.8277	0.9355			
	313 K	0.7632	-3.4642	0.8578			
	323 K	0.6904	-3.2337	0.8695			
	333 K	0.72	-3.4772	0.6486			

MILD STEEL



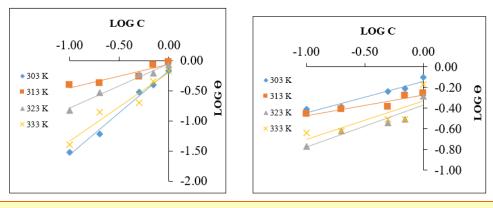


Figure 3 Freundlich adsorption isotherm plot (C/ θ versus C) for mild steel and aluminium

4.3. CORROSION KINETIC ANALYSIS

The Arrhenius and transition state equations was used to consider the kinetic parameters of ELECL adsorption on mild steel and aluminium surfaces. The activation energy Ea, enthalpy and entropy of activation were calculated using the Arrhenius and transition state equations, respectively as shown:

$$\log CR = \log A - \frac{E_a}{2.303RT}$$
 Equation 10

$$\log \frac{CR}{T} = \log \left(\frac{R}{Nh}\right) + \left(\frac{\Delta S}{2.303R}\right) - \left(\frac{\Delta H}{2.303RT}\right)$$
Equation 11

Where CR denotes the corrosion rate of mild steel and aluminium determined from the weight loss experiment, A denotes the pre-exponential constant, E_a denotes the apparent activation energy, R denotes the universal gas constant, T denotes the temperature, N denotes the Avogadro's number, h drenotes Planck's constant, ΔH denotes enthalpy change, and ΔS denotes entropy change.

The activation energies were obtained from the slope of the linear plot of log CR against 10^3 /T for mild steel and aluminium in 1 M HCl in the absence and presence of different concentration of ELECL and the data presented in Table 4 show an increasing trend. An increase in the E_a value indicated that inhibitor molecules physically adsorped on the metal surfaces. The increase in E_a value with the addition of the inhibitor that suggests electrostatic metal-inhibitor interactions about et al. (2021) and suggests the increase of the energy barrier for the corrosion reaction, resulting in reduced rate of corrosion process Verma and Quraishi (2016).

MILD STEEL

ALUMINIUM

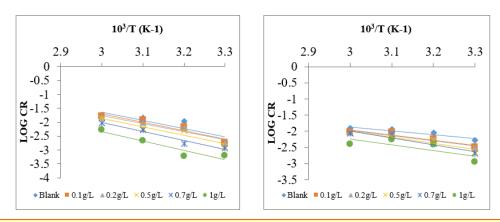


Figure 4 Arrhenius plot for mild steel and aluminium in different concentrations of ELECL

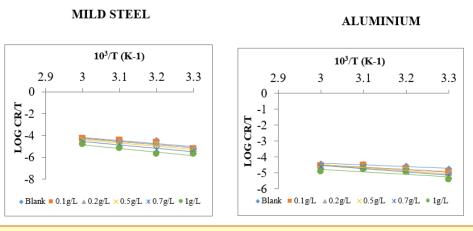
Table 4 Activation parameters from Transition State equation for mild steel in different concentrations of ELECL at 303 K to 333 K $\,$

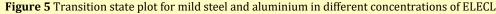
Coupons Conc. (g/L)		Kinetic parameters		
		E _a (J/mol)	ΔH (J/mol	ΔS (J/mol.K)
Mild steel	Blank	24.49	53.7772	-115.8147
	0.1	25.62	56.3949	-109.106

Obot, A. S, Boekom	, E. J, Ita,	, B. N, and	Utam, E. C
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	0.2	24.33	53.4151	-119.2444
	0.5	25.52	56.1602	-113.023
	0.7	26.62	58.6897	-108.4846
	1	28.07	62.0341	-104.4608
Aluminium	Blank	9.93	20.2558	-220.8304
	0.1	13.43	28.305	-198.3267
	0.2	13.38	28.2034	-199.0475
	0.5	16.02	34.2865	-181.0089
	0.7	17.87	38.5365	-168.3864
	1	14.86	31.6131	-193.8787

The data in Table 4 obtained from the transition state plot of log CR/T versus 10^3 /T (Figure 5) for mild steel and aluminium in 1 M HCl in the absence and presence of different concentration of ELECL show positive values of Δ H indicating an endothermic reaction. Thus, implying that the inhibitors have the potential of influencing the incoming energy in the system resulting in higher potential energy and lower kinetic energy, hence breaking up the intermolecular force between the molecules, leading to a slower corrosion reaction rate Srinivasulu and Kasthuri (2017), Gadow and Motawea (2017). The negative values of Δ S indicate a decrease in the disorderliness of the system compared with the blank Azzaoui et al. (2017) and therefore an increased orderliness in the system as a result of an orderly adsorption of the inhibitor molecules freely moving in the bulk solution onto the mild steel and aluminium metal surfaces which often indicates an associative mechanism in which both the molecules of the extracts and ions of the metals form a single activated complex, hence creating a better inhibition Cookey et al. (2018)





CONCLUSION

The Ethanol leave extract of *Costus lucanusianus* (ELECL) was characterized by GC-MS analysis. 11-Octadecenoic acid, methyl ester (30.01%); 9-Octadecenoic acid, 12-hydroxy-, methyl ester, [R-(Z)]- (25.53%); 9,12-Octadecadienoic acid, methyl ester (13.52%); and Hexadecanoic acid, methyl ester (13.14%) was identified as the predominant compounds, largely responsible for the inhibition. The inhibition study was carried out using weight loss method. The inhibition efficiency reached values of 94% and 79% for mild steel and aluminium respectively at 1g/l concenentration. The Freundlich adsorption showed that the adsorption process of

ELECL was physisorption, the enthalpy showed an endothermic process while the entropy values showed an orderly adsorption of the inhibitor molecules onto the metal surfaces.

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