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Evaluation of the inhibitive action of eco-friendly benign *costus afer* stem extract on the corrosion of mild steel in 5 M HCL solution

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Abstract

The corrosion inhibition performance of the ethanol extracts of *Costus afer* stem (EECAS) on the corrosion of mild steel in 5.0 M HCl solution at temperatures of 303, 313 and 323 K was investigated. The experimental work was performed by the use of weight loss (gravimetric) and hydrogen evolution (gasometric) technique. The results indicate that the plant extracts inhibited the corrosion of mild steel in the acid medium and a maximum inhibition efficiency of 94.8 was recorded at 5.0g/L of *Costus afer* extract. Generally the inhibition efficiency was found to increase with increase in concentration of the plant extracts but decreased with rise in temperature. Adsorption of the extracts on the mild steel coupon was found to obey the Langmuir, Temkin, Frumkin and Freundlich adsorption isotherms. The phenomenon of physical adsorption was proposed from the obtained thermodynamic parameters as the values obtained for E_a. The values are also consistent with the data expected for the mechanism of physical adsorption (<80KJ/mol), free energy of adsorption ($-\Delta G^o_{ad}$) values were more negative than -40KJ/mol.

Keywords: Corrosion inhibition, Costus afer, Adsorption isotherms, Mild steel

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1. Introduction

Industries depend heavily on the use of metals and alloys. One of the most challenging and difficult tasks for industries are the protection of metals from corrosion. Corrosion is a ubiquitous problem that continues to be of great relevance in a wide range of industrial applications and product (Clubley, 1990; Clark and Zoitos, 1992). In this kind of situation, organic and inorganic inhibitors are used to reduce the corrosive action of acids (Cushman and Gardner, 1998). Some of the organic inhibitors available are considered to be very toxic and expensive. Therefore, attention has been focused on 'environment friendly green inhibitors' of plant origin, which are inexpensive, readily available and are renewable sources of materials (Bagotsky, 2006). Although, a number of plants and their phytochemicals have been reported as anticorrosive agents, vast majority of plants have not yet been properly studied for their anti-corrosive activity. For example, of the nearly 300,000 plant species that exist on the earth, only a few (less than 1%) of these plants have been completely studied relative to their anticorrosive activity. Thus, enormous opportunities exist to find out novel, economical and eco-friendly corrosion inhibitors from this outstanding source of natural products (Saratha and Meenashi, 2010). *Costus afer* is a member of the family Costaceae and a specie of the genus Costus. It is known by the common name Bush cane or Ginger lily. The plant is mostly found in swampy areas. The succulent part of the stem is edible while leaves are used to feed animals. *Costus afer* stem has been remedies of many diseases. This present work is aimed at investigating the effect of different concentrations of extracts of *Costus afer* stem on the corrosion of mild steel in HCl solution using gravimetric and gasometric techniques (Uwah et al*.,* 2013).

2. Experimental design

2.1. Preparation of extract of Costus afer stem

Samples Costus afer stem were cut into small pieces dried in a laboratory Oven at 313 K and ground into powder form. 100g of the powdered sample was extracted continually with 250 cm^3 of absolute ethanol in a Soxhlet Extractor for 24 hours after which the solvent was evaporated at low temperature of 50° C. 5g of the ethanol extract was diluted 1litre of 5.0 M HCl solution, kept for 24 hours, filtered and stored. From the stock solution (5 g/L), inhibition test solutions were prepared to obtain 0.5 g/L, 1.0 g/L, 2.0 g/L, 3.0 g/L and 5.0 g/L for weight loss and hydrogen evolution measurements respectively.

2.2. Mild steel specimen

The sheet of Mild Steel used for this study has the following composition: Fe (98.34%), C (0.19%), Si (0.26%), Na (0.64%), S (0.05%), P (0.06%), Ni (0.09%), Cr (0.08%), Mo (0.02%), and Cu (0.27%). The sheet was mechanically press-cut into 0.08 x 4.00 x 5.00 cm coupons. These were polished with different grades (#800, #1000 and #1200) of emery paper, degreased in absolute ethanol, dried in acetone and stored in moisture free desiccator.

2.3. Weight loss measurements

Weight loss measurements were performed on the Mild Steel coupons of dimension 0.08 x 4.00 x 5.00 cm in 5.0 M HCl solutions with and without different concentrations of *Costus afer* extracts. Each mild steel coupon was weighed with a digital analytical balance and then suspended with the aid of glass rods in the acid solution contained in a 100mL beaker. The duration of immersion was 5 hours. After immersion the surface of the specimen (Mild steel coupon) was cleaned with distilled water using a bristled brush to scrub the surface followed by rinsing with absolute ethanol and drying with acetone. Then the sample was weighed again to calculate the weight loss. This process was repeated progressively for 5 hours. The same experiment was carried out in the presence of ethanol extracts of *Costus afer* stem using concentrations of 0.5g/L, 1.0g/L, 2.0g/L, 3.0 g/L and 5.0g/L at 303 K. The Corrosion rate (CR) of the Mild steel was obtained from the slope of the graphical plot the weight loss per surface area against time of exposure (Figure 1). Surface coverage (θ) and Inhibition efficiency (IE%) were calculated using the following Equations 1 and 2.

$$\theta = \frac{CR_o - CE_i}{CR_o} \qquad (1)$$

$$IE\% = \frac{CR_o - CE_i}{CR_o} \times 100 \qquad (2)$$

where CR_o and CR_i are the corrosion rate values in presence and absence of the inhibitor, respectively.

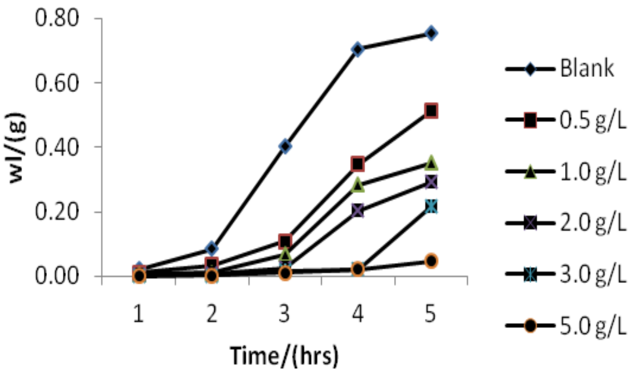

Figure 1. Variation of weight loss against time of immersion for mild steel in 5 M HCl in the absence and presence of different concentrations of *Costus afer* stem extract

2.4. Hydrogen evolution measurements

100 cm^3 of the corrodent (5 M HCl) was introduced into a two-necked flask and the initial volume of the air in the burette noted. Thereafter, a mild steel coupon of dimension 0.08 x 1.20 x 4.00 cm already weighed was dropped into the corrodent and the flask quickly closed. The volume of the hydrogen gas evolved from the corrosion reaction was monitored by volume changes in the level of paraffin oil in the graduated burette of the gasometric apparatus. The reading was taken every minute for 30 minutes. This procedure was repeated for a set of fresh coupons at different concentrations of *Costus afer* stem extracts (0.5g/L, 1.0g/L, 2.0g/L, 3.0 g/L and 5.0g/L). The study was conducted at 303 K, 313 K and 323 K using a thermostat water bath.

3. Results and discussion

3.1. Effect of inhibitor concentration and exposure time

Figures 1and 2 show the variation of weight loss with time and concentration respectively for the corrosion of mild steel in 5 M HCl containing various concentrations of *Costus afer* stem extracts. From Figure 1 it is evident that weight loss of mild steel increases with increases in the period of contact indicating that the rate of corrosion of mild steel in HCl solution increases with increase in the period of contact (Uwah et al., 2012; Singh et al., 2012; Parameswari et al., 2012; Zuo et al., 2004; Benali et al., 2013; Chaieb et al., 2005). However, weight loss of mild steel was also found to decrease with increase in the concentration of *Costus afer* stem extract indicating that the extract retarded the rate of corrosion of mild steel in solutions of HCl (El-Etre and El-Tantawy, 2006; El-Etre, 2003; Bagotsky, 2006; Asher and Singh, 2009) as indicated in Table 1. θ and % IE increased with increase in EECAS concentration to a maximum of 0.9477 and 94.7% respectively, this indicates that the corrosion of the metal has been inhibited and a larger fraction of the surface is protected against acidic attack at high inhibitor concentrations according to Oguzie et al., (2010) and El-Etre et al., (2003).

The maximum value of 94.7 % obtained at 5 g/L of EECAS as shown in Table 1indicates that EECAS is a good inhibitor for the corrosion of mild steel in 5 M HCl solution.

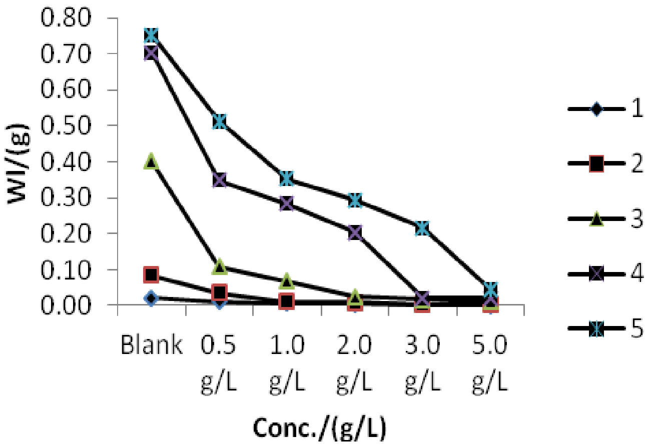

Figure 2. Variation of weight loss with extract concentration of *Costus afer* stem on mild steel in 5 M HCl

Table 1. Calculated values of Corrosion rates (CR), Surface coverage (θ) and Inhibition efficiency (IE) for mild steel coupons in 5 M HCl in the absence (blank) and presence of EECAS at 303 K

	Blank	0.5 g/L	1.0 g/L	2.0 g/L	3.0 g/L	5.0 g/L
CR	5.024	3.178	2.319	1.86	1.078	0.263
θ		0.3674	0.5384	0.6298	0.7854	0.9477
IE%		36.7	53.8	63.0	78.5	94.8

3.2. Effect of temperature

The temperature of the system was varied across the inhibitor concentrations studied from which the activation energy for the corrosion of mild steel in solutions of HCl was evaluated using the Arrhenius equation given by Equation 3 (Oguzie et al., 2010 and Patrick et al., 2006).

$$\ln R_c = \ln A - \frac{E_o}{RT} \qquad (3)$$

where R_c is the corrosion rate, E_a is the apparent effective activation energy, R is the general gas constant, and A is the Arrhenius pre-exponential factor (Singh et al., 2010, 2011,2012; Okafor et al., 2012; Ananda et al., 2005). Calculated values of Activation energy between 303 K and 323 K were obtained from the slope of Figure 3 and presented in Table 2. The values obtained are greater than the value (43.3 KJ/mol) obtained for the blank solution indicating that *Costus afer* stem extract retards the corrosion of mild steel in HCl solutions (El – Etre et al., 2006; Uwah et al., 2012; Ananda and Singh, 2007). Since the Activation energy which the energy required to oxidize metal is increased with inhibitor concentration, it implies that more energy has to be supplied to the system for the corrosion to take thus the observed decrease in corrosion rate. The values are also consistent with the data expected for the mechanism of physical adsorption (<80KJ/mol) according to Oguzie et al., (2010), Patrick et al., (2006) and Okafor et al., (2012). However, the closeness of the calculated values of the activation energy to the threshold value (80KJ/mol) expected for chemisorptions suggest that after physical adsorption, chemisorptions might have taken place according to Okafor et al., (2007). In adsorption process, physical adsorption precedes chemical adsorption (Okafor et al., 2009; Singh et al., 2012).

Thermodynamic parameters; Enthalpy, ΔH_{ads} , Entropy ΔS_{ads} and Heat of adsorption Q_{ads} of *Costus afer* on mild the steel surface was calculated using Equations 4 and 5 (transition state equation), which can be written as follows (Parameswari et al., 2012; Uwah et al., 2012; Singh et al., 2010 and Patrick et al., 2006).

$$Log\frac{CR}{T} = Log\frac{CR}{Nh} + \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \qquad (4)$$

$$Qads = 2.303\ R \left[Log \frac{\theta_2}{1-\theta_2} \right] - \left[Log \frac{\theta_1}{1-\theta_1} \right] \times \left[\frac{T_1 \times T_2}{T_2 - T_1} \right]\ KJmol^{-1} \qquad (5)$$

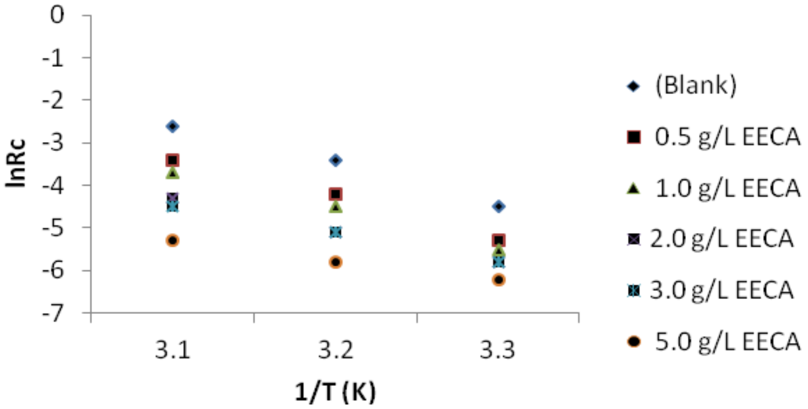

Figure 3. Arrhenius plots for mild steel in 5 M HCl solutions in the absence and presence of ethanol extract of *Costus afer* Stem

were Q_{ads} is the heat of adsorption, R is the universal gas constant, Θ_1 and Θ_2 are the degrees of surface coverage of the inhibitors at temperatures T_1 and T_2 respectively (Oguzie et al., 2010). From Equations 4 and 5 values of log (CR/T) were plotted against 1/T as shown in Figure 4 and from the slope and intercept of the plot, values of enthalpy and entropy of adsorption were calculated as shown in Table 2 (Okafor et al., 2012). From the calculated values of ΔH* (Table 2), it can be deduced that the adsorption of the inhibitor on mild steel surface is exothermic and the reaction becomes less exothermic with increase in inhibitor concentration. The negative values of Q_{ads} indicates that the degree of surface coverage decrease with rise in temperature, supporting the earlier proposed physisorption mechanism for *Costus afer* stem extracts. The negative values for ΔS* shows the nonspontanous dissolution of the mild steel and and the increase in its value suggests decrease in disordering in the rate determining step (El-Etre and El-Tantawy, 2006).

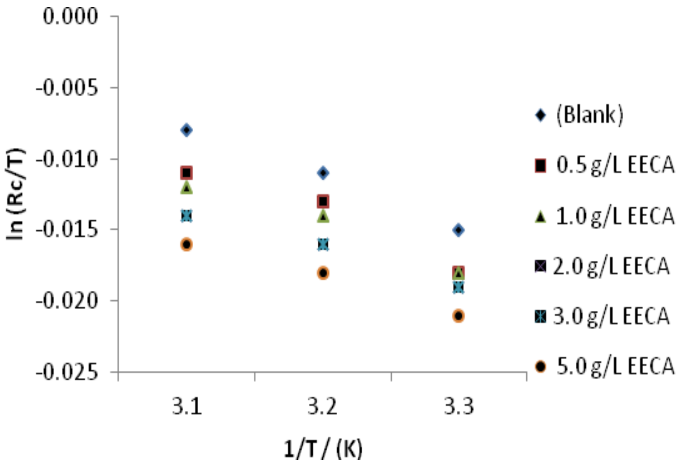

Figure 4. Transition state plots for mild steel in 5 M HCl solutions in the absence and presence of ethanol extract of *Costus afer* Stem

Table 2. Values of activation/thermodynamic parameters for mild steel in 5 M HCl in the absence and presence of the plant extracts

System	E_a (KJ/mol)	ΔH * (KJ/mol)	ΔS* (KJ/mol)	$Q_{ads.}$ (KJ/mol)
5 M HCl (Blank)	9.63	-54.04	-77.80	-
5 M HCl + 0.5 g/L EECAS	37.41	-24.94	-48.70	-354.5
5 M HCl + 1.0 g/L EECAS	54.04	-24.94	-48.70	-133.5
5 M HCl + 2.0 g/L EECAS	65.82	-16.62	-40.38	-373.5
5 M HCl + 3.0 g/L EECAS	70.40	-16.62	-40.38	-495.8
5 M HCl + 5.0 g/L EECAS	74.80	-16.62	-40.38	-715.3

3.3. Adsorption Isotherm consideration

The surface coverage (θ) values for different concentrations of the inhibitors in HCl medium was evaluated from the weight loss data. The data were tested graphically to find a suitable adsorption isotherm to describe the adsorption characteristics of the extracts (Saratha et al., 2009; Singh et al., 2011; Zuo et al., 2004; Benali et al., 2013).

3.3.1. Langmuir Adsorption Isotherm

A plot of Log (Θ/C) against Log C (Figure 5) shows a straight line (R>= 0.9) indicating that adsorption follows the Langmuir adsorption isotherm according to Uwah et al., (2012), Saratha et al., (2009), Oguzie et al., (2010), Okafor et al., (2012) and Chaieb et al., (2005). Saratha et al (2009, p. 5) states that "although these plots are linear, the gradients are never unity, contrary to what is expected for ideal Lamgmuir adsorption isotherm equation. Organic molecules having polar atoms or groups which are adsorbed on the metal surface may interact by mutual repulsion or attraction and this may be advocated as the reason for the departure of the slop values from unity".

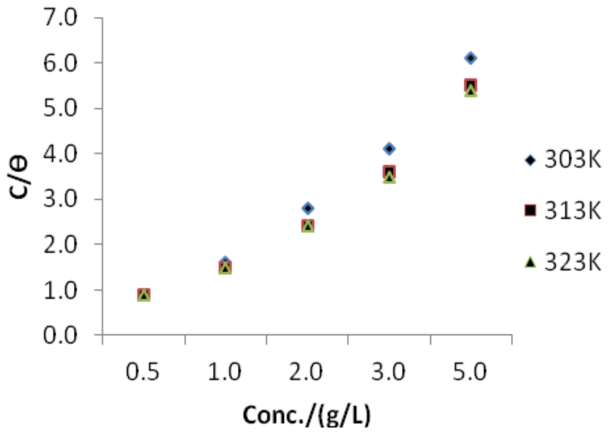

Figure 5. Langmuir adsorption isotherm for EECAS at various temperatures

3.3.2. Temkin Adsorption Isotherm

According to Saratha et al (2009, p. 6) "The number of active sites of the surface occupied by one molecule of the inhibitor is given by the values of (1/y)". A straight line was obtained when the surface coverage was plotted against log C for the inhibitor. This shows that the adsorption obeys a Temkin adsorption isotherm (Figure 6).

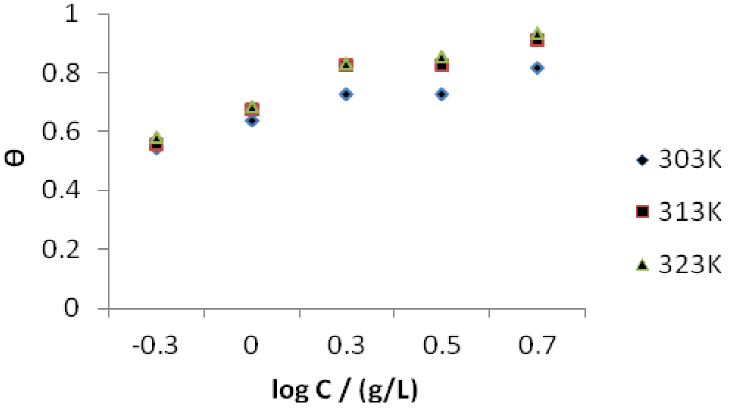

Figure 6. Temkin adsorption isotherm for EECAS at various temperatures

3.3.3. Freunlich Adsorption Isotherm

The plots of Log Θ against Log C (Figure 7) shows that the adsorption of the inhibitor on mild steel surface follows Freundlich isotherm from its linearity.

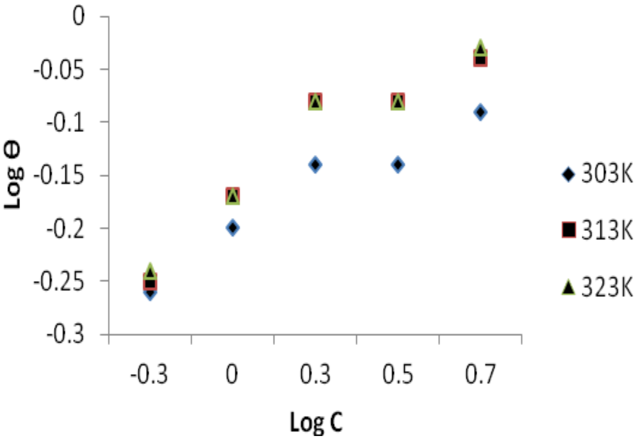

Figure 7. Freundlich adsorption isotherm for EECAS at various temperatures

3.3.4. Frumkin Adsorption Isotherm

The plot of IE against Log C (Figure 8) shows a sigmoidal shape which also demonstrate the fact that the adsorption of the inhibitor on the mild steel obeys the Frumkin isotherm (Saratha et al., 2009 and Patrick et al., 2006).

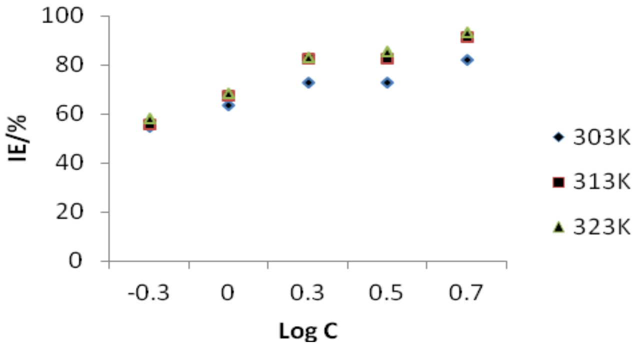

Figure 8. Frumkin adsorption isotherm for EECAS at various temperatures

3.3.5. Flory Huggins Isotherm

This Isotherm is expressed as Equation 6.

$$\text{Log } \Theta/C = \log XK + X\log (1-\Theta) \qquad (6)$$

Where Θ is the degree of surface coverage, X is the number of active sites occupied by one inhibitor molecule or number of one molecule of the adsorbate. The value of $X > 1$, implied that one inhibitor molecule replaces more than one water molecule (Saratha et al., 2009; Okafor et al., 2012). The plot of log (Θ/C) against log (1-Θ) is shown in Figure 9. The results further indicates that the adsorption of the phytochemicals in the inhibitor is spontaneous ($-\Delta G_{ads}$) and this confirms the mechanism of physical adsorption (Table 3).

The results obtained from Langmuirs plot gave R^2 values and slopes that are very close to unity indicating the application of Langmuir isotherm to the adsorption of the inhibitor (Singh et al., 2010 and 2012).

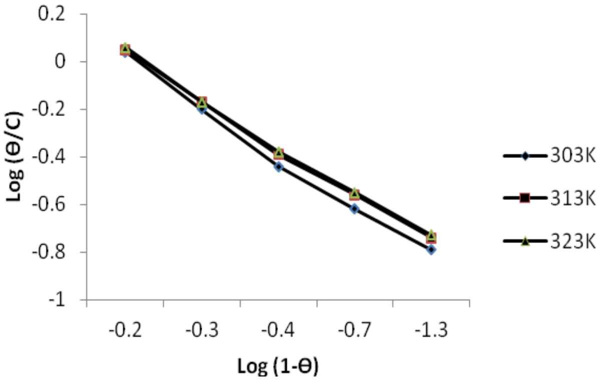

Figure 9. Flory Huggins adsorption isotherm for EECAS at various temperatures

Table 3. Adsorption parameters for mild steel in 5 M HCl solutions containing EECAS at various temperatures

Temperature/(K)	K_{eq} (L/g)	ΔG^*_{ads} (KJ/Mol)	R^2	Slope
303	1.30	-10.78	0.968	1.29
313	1.64	-11.74	0.953	1.13
323	1.79	-12.35	0.953	1.10

4. Conclusion

The natural extract (EECAS) was found to be an effective inhibitor in the acidic medium giving up to 94.8 % efficiency. The % IE increases with EECAS concentration. Adsorption Isotherm models-Langmuir, Temkin, Freundlich, Frumkin and Flory-Huggins isotherms all fit well as evident from the correlation coefficient values ($R \approx 1$ in all cases). The inhibitor acts via a simple adsorption of the phytochemicals present in the extract on the mild steel surface. The adsorption of the extracts on the mild steel surface is spontaneous as is evident from negative ΔG_{ads}.

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