

Inhibition performance of camel dung extract on the corrosion of Mild Steel in hydrochloric acid

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Abstract. The inhibition action of Camel dung extract on corrosion of Mild steel in 1M and 2M HCl acid solution was studied using weight loss method. Fourier Transform Infrared Spectroscopic Analysis (FTIR) was used to characterize the inhibitor and the corrosion product. The effect of different inhibitor concentrations was studied at 303, 313, 323 and 333K. The result showed that the percentage of inhibition efficiency (% I.E.) increased with increase in the inhibitor concentration and decrease with rising in temperature in all the HCl solution media. The highest I.E. was found 63.04% and 45.94% at 303K and 2.0%v/v inhibitor concentration in 1M and 2M HCl solution respectively. The inhibitive action of the inhibitor is due to the adsorption of inhibitor molecules on the metal surface. The adsorption of the inhibitor on the surface of Mild steel was found to be a physical adsorption mechanism. Activation Energy and Enthalpies governing the adsorption process were found to be increasing with increase in the concentration of the inhibitor.

Keywords: Corrosion Inhibition, Cowdung, Mild steel, HCl

1 Introduction

Corrosion can be explained as the deterioration of metal by chemical attack or reaction with its environment. It is a constant and continuous problem, often difficult to eliminate completely. Prevention would be more practical and achievable than complete elimination. Corrosion processes develop fast after disruption of the protective barrier and are accompanied by a number of reactions that change the composition and properties of both the metal surface and the local environment, for example, formation of oxides, and diffusion of metal cations into the coating matrix, local pH changes, and electrochemical potential [1].

Corrosion inhibitor is a substance which when added in small concentration to an environment, effectively reduces the corrosion rate of a metal exposed to it. Large numbers of organic compounds have been studied and are still being studied to assess their corrosion inhibition potential. However, most of these substances are not only expensive but also possess health and environmental hazards [2] prompting

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the search for their replacement. Plants have been recognized as sources of naturally occurring compounds that are generally referred to as ‘green’ compounds, some with rather complex molecular structures and having a variety of physical, chemical and biological properties. This quest is motivated by the huge cost spent annually on corrosion control and monitoring technologies.

The use of plant source green inhibitors has been very promising in the search for more cost effective and technically efficient substances for corrosion control. Among plants that there exist documented research evidence of their effectiveness as inhibitors are *phyllanthus amarus* [3], *Opuntia* [4], *Mentha pulegium*, *black pepper* and *Datura metel* [5] and *Strychnos nux-vomica* [6], *khillah seeds* [7], *lupine extract* [8], *Gossipium hirsutum* L [9], *Zallouh* [10]. There are also many other plant sources whose inhibition properties are still under investigation.

Despite the success of plant based green inhibitors, there have been a number of concerns raised about their sustainability in the long term particularly plant sources that have other competing domestic, medicinal and industrial uses [11]. It is thus timely for exploration of other potential sources of green inhibitors outside the use of plants.

The use of animal dung as potential source of developing corrosion inhibitor is currently under explored. The potentials of camel dung as corrosion inhibitor of steel in an acidic environment is the focus of this paper. The choice of camel dung was propelled by results obtained from its characterization which show the presence of OH among others. Organic compounds containing nitrogen, sulfur, oxygen, and heterocyclic compounds with a polar functional group and a conjugated double bond have been reported to inhibit mild steel corrosion [12]. The present study assessed the inhibition efficiency of camel dung in hydrochloric acid solutions. The focus on camel dung was influenced by its utilization in manure for farm uses [13]. There is currently no report available in recognized peer reviewed article databases on the corrosion inhibition of camel dung.

2.0 MATERIALS AND METHODS

2.1 Pre-treatment of the mild steel coupon

The sheet of mild steel was measured and found to have a thickness of 0.1cm, it is then mechanically cut into 2cm×2cm. The surface of the mild steels were polished with emery paper, the coupon where then washed with distilled water, degreased in acetone, dried at room temperature and finally preserved.

2.2 Inhibitor preparation

Camel dung used was obtained as being excreted by local Camel from Hauren Shanu Animals market along BUK Road. This was sun dried to reduce the moisture content. The dried camel dung used was pulverized, 25g of the pulverized dung was added to 250ml of 1M HCl. The

mixture was allowed to soak for 3 days, then it was placed in a water bath at 100°C for 2 hours, 30 minutes. The mixtures was cooled and filtered. From the filtrate, 0.25, 0.5, 1.0, 1.5 and 2.0% v/v was prepared as test solution in 1M and 2M HCl .

2.3 Extract characterization

Camel dung extract was characterized using FTIR (Fourier transform infrared spectroscopy).

2.4 Preparation of the HCl solution

Analar grade HCl with percentage purity (36.5%) and a density (1.175g/cm³) supplied by from the chemicals store, was diluted with distilled water for the preparation 1M and 2M HCl.

2.5 Gravimetric measurement

The polished and pre-weighed mild steel coupons were immersed in 250 ml beaker containing 100 ml test solution, maintained at different temperatures in the range of 303–333 K for 1h. After 1h immersion, the specimens were taken from the solution, dried in acetone and then weighed. The weight loss, (in grams), was taken as the difference in the weight of the mild steel coupons before and after immersion in different test solutions (Olusegun *et al.*, 2016). The weight loss was used to calculate the corrosion rate (CR) and the inhibition efficiency (IE).

$$\Delta W = W_1 - W_2 \quad (1)$$

Where W_1 and W_2 are the weight of coupons before and after immersion

$$CR \text{ (gcm}^{-2}\text{h}^{-1}\text{)} = \frac{\Delta W}{At} \quad (2)$$

The degree of surface coverage (Θ) and inhibition efficiency (I.E.) for different concentrations of the inhibitor in acidic media have been evaluated from weight loss experiments using the equations below:

$$\Theta = \left(\frac{W_o - W_i}{W_o} \right) \quad (3)$$

Where W_o and W_i are the weight of the coupon in the absence and presence of inhibitor.

$$I.E. (\%) = \left(\frac{W_o - W_i}{W_o} \right) \times 100 \quad (4)$$

2.6 Effect of Temperature on Corrosion rate and Inhibitor efficiency

To study the effect of temperature on corrosion rate and inhibitor efficiency, the prepared mild steel coupons were completely immersed in 250ml beaker containing test solution without and with different concentrations of camel dung inhibitor at 303, 313, 323 and 333 K for 1h.

2.7 Effect of Inhibitor concentration on Corrosion rate and Inhibitor efficiency

To study the effect of Inhibitor concentration on corrosion rate and inhibitor efficiency, the prepared mild steel coupons were completely immersed in 250ml beaker containing test solution without and with different concentrations of camel dung inhibitor at 303, 313, 323 and 333K for 1h.

3 Results and Discussions

The results of the corrosion parameters (i.e, corrosion rate and inhibitor efficiency), thermodynamics and adsorption parameter of different inhibitor concentration at different temperatures of 303, 313, 323 and 333K in 1M and 2M HCl medium were presented in Table 1-2 respectively and Figure 1-7 represent variation of these parameter with temperature and inhibitor concentration in 1M HCl. While figure 8 and 9 represent the FT-IR Spectra of the inhibitor (Camel dung extract) and the corrosion product respectively.

Table 1. Percentage efficiency of the inhibitor and values of corrosion rate for mild steel in 1M HCl in the absence and presence of Camel Dung Extract at 303-333K for 1hour.

303K			313K		323K		333K	
Inhibitor concentration (%v/v)	Corrosion rate (g/cm ² h)	Inhibitor Efficiency (%)	Corrosion rate (g/cm ² h)	Inhibitor Efficiency (%)	Corrosion rate (g/cm ² h)	Inhibitor Efficiency (%)	Corrosion rate (g/cm ² h)	Inhibitor Efficiency (%)
0.00	0.00230	-----	0.00320	-----	0.00365	-----	0.00428	-----
0.25	0.00195	15.22	0.00295	07.82	0.00345	05.48	0.00413	3.51
0.50	0.00165	28.26	0.00265	17.19	0.00330	09.59	0.00390	8.77
1.00	0.00148	35.83	0.00243	24.22	0.00303	17.12	0.00370	13.45
1.50	0.00130	43.48	0.00225	29.69	0.00288	21.23	0.00348	18.71
2.00	0.00103	63.04	0.00208	39.84	0.00273	25.34	0.00328	23.48

Table 2. Percentage efficiency of the inhibitor and values of corrosion rate for mild steel in 2M HCl in the absence and presence of Camel Dung Extract at 303-333K for 1hour.

Inhibitor concentration (%v/v)	Corrosion rate (g/cm ² h)	Inhibitor Efficiency (%)	Corrosion rate (g/cm ² h)	Inhibitor Efficiency (%)	Corrosion rate (g/cm ² h)	Inhibitor Efficiency (%)	Corrosion rate (g/cm ² h)	Inhibitor Efficiency (%)
0.00	0.00338	----	0.00515	----	0.00673	----	0.0105	----
0.25	0.00300	11.14	0.00460	10.68	0.00613	8.90	0.0099	5.48
0.50	0.00253	25.21	0.00430	16.51	0.00585	13.01	0.0092	12.38
1.00	0.00220	34.83	0.00408	20.87	0.00540	19.70	0.0086	16.67
1.50	0.00203	40.01	0.00385	25.24	0.00513	23.79	0.0081	22.86
2.00	0.00183	45.94	0.00350	32.04	0.00465	30.86	0.0074	29.52

3.1 Effect of concentration of the HCl acid solution

Comparing Table 1 and 2, it was observed that increase in the Hydrochloric Acid concentration increases the corrosion rate of mild steel in the absence and presence of the inhibitor at all the studied temperatures. This observation is due to the fact that the rate of the chemical reaction increases as the concentration of the active species increases. This is attributed to the increase in number of chloride ions at the mild steel surface there by decreasing the inhibition action of the inhibitor (Camel Dung extract). Similar report has been reported by Abdel Gaber *et al.*, [8].

3.2 Effect of inhibitor concentration on corrosion rate and inhibition efficiency

The results from this study as presented in Figure 1 for the plot of corrosion rate against inhibitor concentration for Mild steel in 1M HCl at different temperatures reveals that the corrosion rate of the metal decreases with increase in inhibitor concentrations at all studied temperatures. Inspection of the result further indicates that the protection ability of the inhibitor was concentration dependent. The corrosion rates in the presence of inhibitors were reduced indicating the inhibiting action of the camel dung as inhibitors. This suggests that as the concentration of inhibitors increases, there was an increase in surface coverage of the adsorbed molecules on the mild steel surface which provided a barrier (film) and reduced further corrosion as reported by Olasehinde et al [14].

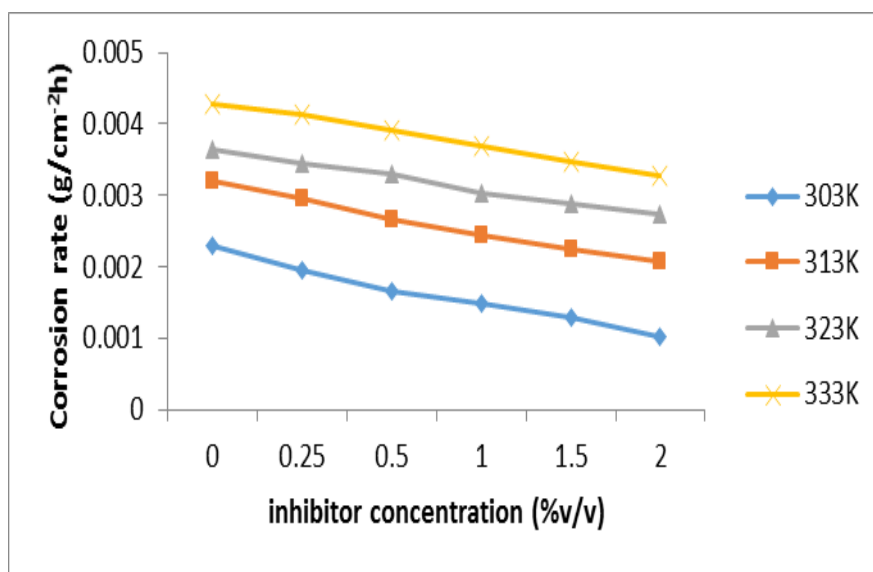


Fig 1. Corrosion Rate with Inhibitor Concentration for mild steel corrosion in 1M HCl.

While the plot of inhibition efficiency against inhibitor concentration shown in Fig. 2 suggest that the surface coverage (Θ) and inhibition efficiency (% I E) were found to increase with increasing inhibitor concentration.. The highest inhibition efficiency was found to be 63.04% at the highest inhibitor concentration (2.0%v/v) at 303 K. This trend was observed due to increase in surface coverage (Θ) of adsorbed specie on surface of the metal as a result of increase in inhibitor concentration. This shows that more inhibitor molecule is adsorbed on the metal surface, thus lead to increase in inhibition efficiency. A similar result was reported by Nnanna *et al.*, [15].

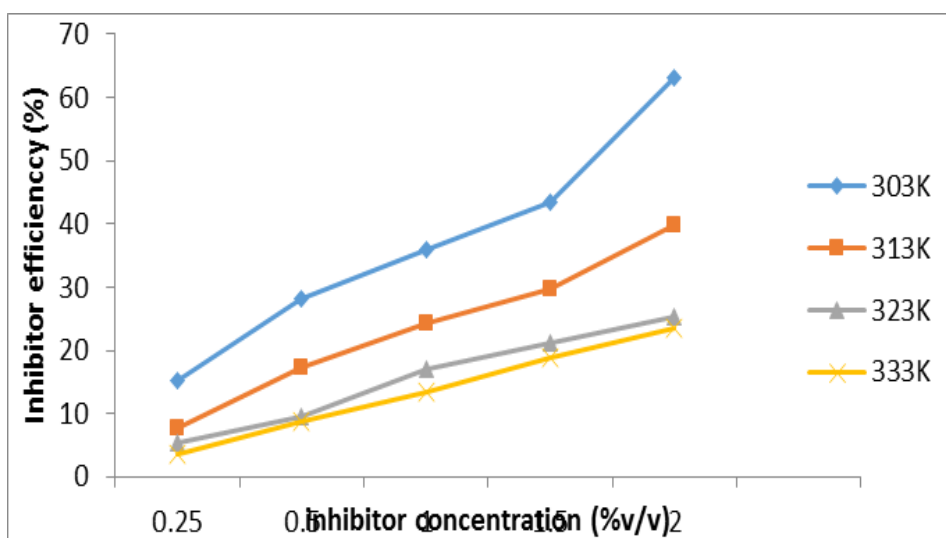


Fig 2. Inhibition Efficiency with Inhibitor Concentration for mild steel Corrosion in 1M HCl at different temperture ranging from 303-333K.

3.3 Effect of Temperature on Corrosion Rate and Inhibition Efficiency

The result of the effect of temperature on the corrosion rate of Mild steel for inhibited and uninhibited solution is presented on Fig. 3 at 303, 313, 323 and 333K respectively. From the result, it can be seen that the rate of inhibited and uninhibited solution increases with increase in temperature. It can be observed from the results that the highest corrosion rate of mild steel was observed at the highest temperature (333K). This observation is due to the fact that chemical reaction rates generally increase with rising temperature [16] Increase in temperature leads to increase in the kinetic energy possessed by the reacting molecules thereby making the molecules to overcome the energy barrier faster [16].

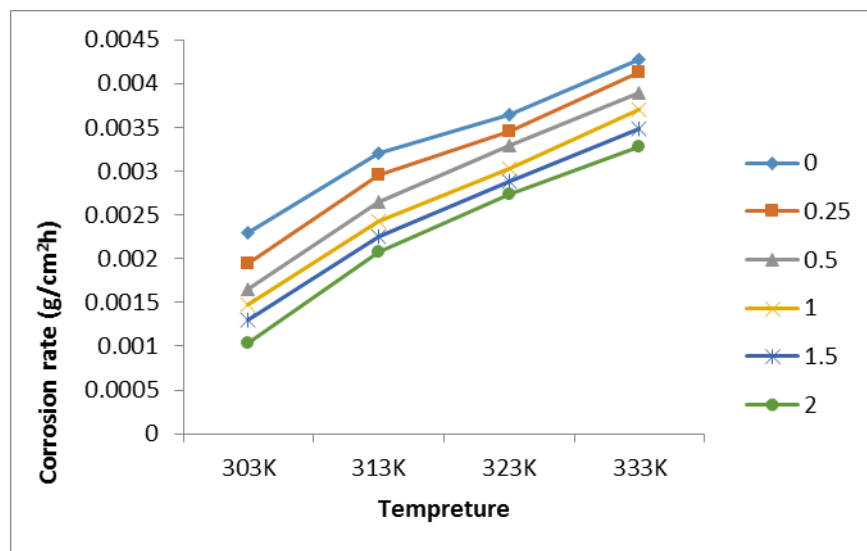


Fig 3. Corrosion Rate with Temperatures for mild steel Corrosion in 1M HCl.

While, The result of the effect of temperature on the inhibitor efficiency of Mild steel for inhibited solution was presented on Fig. 4 at 303, 313, 323 and 333K respectively . From the result it can be seen that inhibition efficiency decreases with increase in temperature. Increase in temperature cause the desorption and destabilization of the inhibitor molecule absorbed on the metal surface and thus results in decrease of inhibition efficiency. Similar result was reported by Mohamed *et al.*,[17].

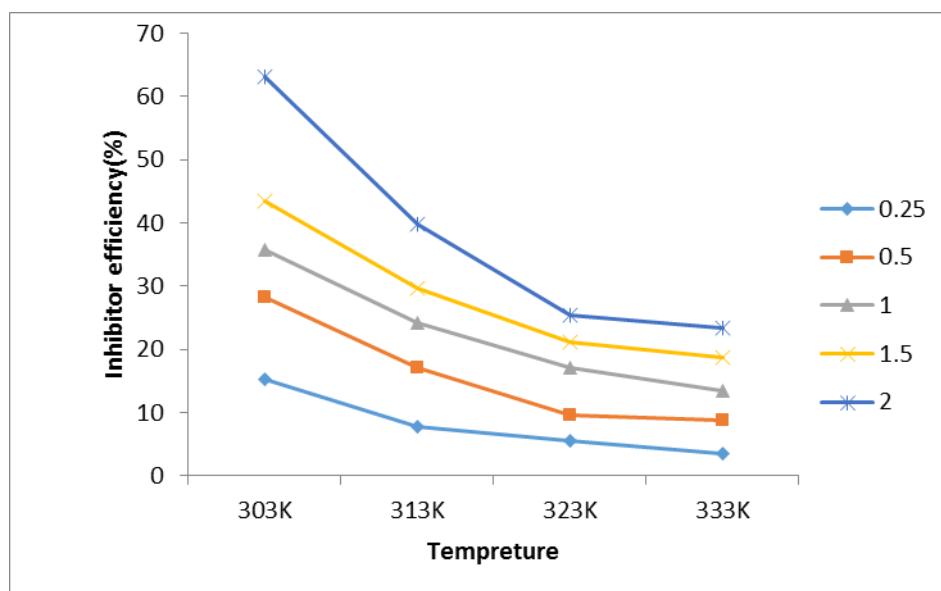


Figure 4. Inhibitor efficiency with temperatures for mild steel in 1M HCl.

3.4 Thermodynamics study

3.4.1 Determination of Activation Energy (E_a)

In order to explain clearly the inhibitive property of camel dung and the dependence of the temperature on the corrosion rate, the activation energy (E_a) for the corrosion process in the absence and presence of the camel dung inhibitor was calculated from Arrhenius type plot according to the equation given below;

$$\ln(C_R) = B - \frac{E_a}{RT} \quad (5)$$

Where E_a is the apparent activation corrosion energy, R is the gas constant, T is the absolute temperature and B is a constant.

Values of apparent activation energy for the corrosion of mild steel corrosion without and with various concentrations of inhibitor are shown in Table 3.

These values were determined from the slope of $\ln CR$ versus $\frac{1}{T}$ plots presented in Figure 5. The results of activation energy from Table 3 showed that the value of E_a for inhibited solution is higher than that for uninhibited solution, suggesting that dissolution of mild steel is slow in the presence of the inhibitors. It is known from Arrhenius equation that the higher E_a values lead to the lower corrosion rate, this is due to the formation of a film on the mild steel surface serving as an energy barrier for Mild steel corrosion [18].

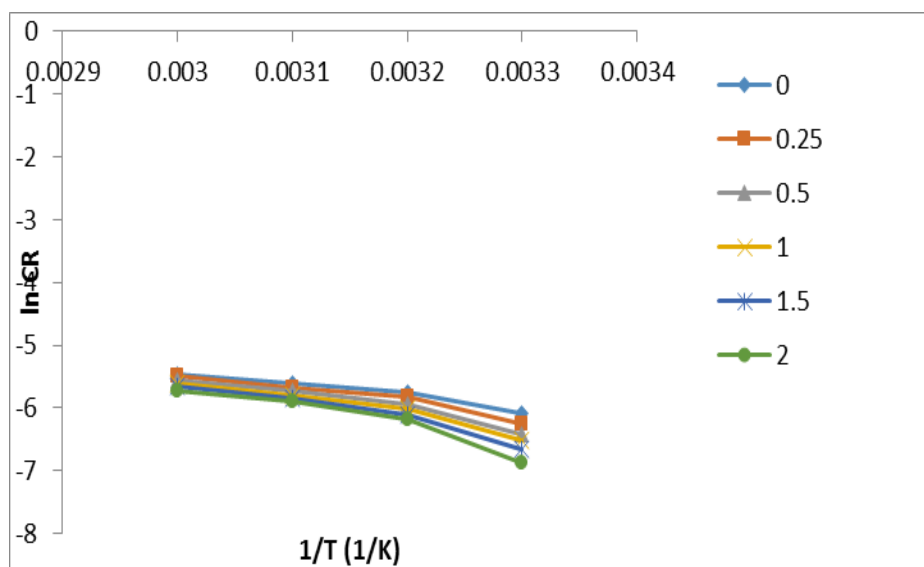


Fig. 5. Arrhenius plot for mild steel in 1M HCl in the absence and presence of the inhibitor.

From the result of activation energy, it can be seen that the Activation Energy in uninhibited solution was 16.6 kJ/mol which increased to 31.1 kJ/mol in the presence of 2.0%v/v inhibitor concentration. This suggest that the activation energy increases with increase in inhibitor concentration.

3.5 Determination of Enthalpy change (ΔH) and Entropy change (ΔS)

Enthalpy change (ΔH) and entropy change (ΔS) of activation process were evaluated from the transition state equation.

$$\ln \left(\frac{CR}{T} \right) = \ln \left(\frac{R}{Nh} \right) + \left(\frac{\Delta S}{R} \right) - \left(\frac{\Delta H}{RT} \right) \quad (6)$$

Where h is the Planck's constant (6.63×10^{-34} j.s), N is Avogadro number (6.02×10^{23}), ΔS_a is the entropy change of activation and ΔH_a is the enthalpy change of activation.

Figure 6 shows a plot of $\ln \left(\frac{CR}{T} \right)$ against $\left(\frac{1}{T} \right)$, straight line is obtained with a slope of $\left(\frac{-\Delta H}{R} \right)$ and intercept of $\ln \left(\frac{R}{Nh} \right) + \left(\frac{\Delta S}{R} \right)$ from which the value of ΔH_a & ΔS_a are calculated and presented in Table 3.

It can be seen from Table 3 all the values of enthalpies (ΔH_a) are positive and the positive signs of the enthalpies (ΔH_a) reflect the endothermic nature of Mild steel dissolution process. The entropy of activation in presence and absence of the inhibitor was large and negative. This implies that the activated complex in the rate determining step represents association rather than dissociation, indicating that a decrease in disorder takes place, going from reactant to the activated complex [19].

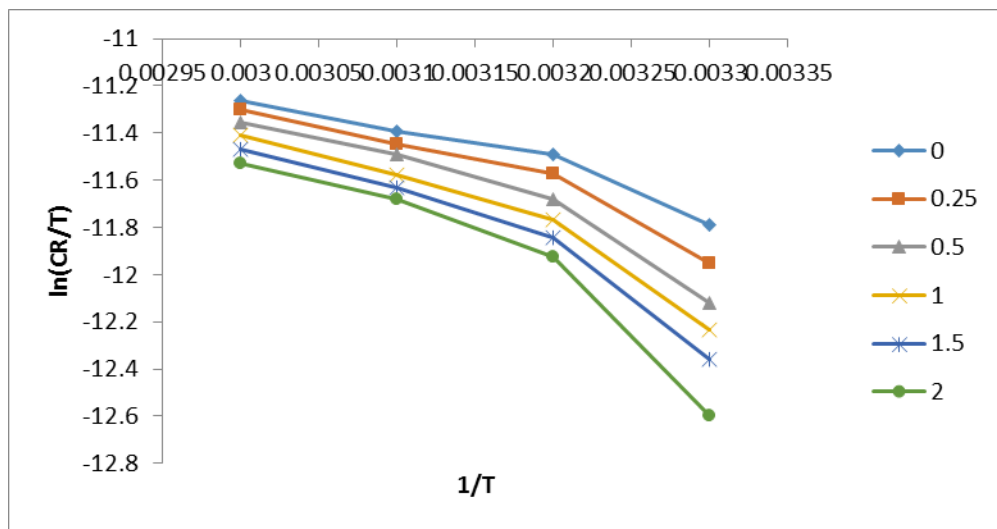


Fig 6. Transition State plot mild steel in 1M HCl in the absence and presence of the inhibitor.

Table 3. Thermodynamics parameters deduced from transition and Arrhenius plot for corrosion of mild steel in 1M HCl at different inhibitor concentration.

Inhibitor concentration (% v/v)	E _a (kJmol ⁻¹)	ΔH (kJmol ⁻¹)	-ΔS (JK ⁻¹ mol ⁻¹)
0.0	16.6	14.0	249.1
0.25	20.0	17.4	239.0
0.50	23.3	20.7	229.4
1.0	24.7	22.15	225.4
1.5	26.6	24.0	220.4
2.0	31.1	28.7	206.5

3.6 Adsorption mechanism

The Langmuir adsorption mathematical relation can be expressed in many forms, but the one utilized in this analysis is as follows:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (7)$$

Where K is adsorption equilibrium constant, in the bulk electrolyte, θ is the degree of surface coverage, and K_{ads} is the adsorption equilibrium constant.

$$\Delta G_{ads} = -RT \ln (55.5 \times K_{ads}) \quad (8)$$

The observed increase in inhibitor efficiency as the inhibitor concentration increases could be attributed to various inhibition mechanisms, the inhibitor could form a thin layer on the metal coupon and partly protect it. Adsorption isotherms are used to understand the mechanism of corrosion inhibition of metals and alloys.

The higher the surface coverage with inhibitor molecules, the fewer sites would be available for corrosion [16].

A plot of $\frac{C}{\theta}$ gave a straight line with slope equals to K_{ads} . The correlation coefficient (R^2) values obtained are presented in Table 4.

Figure 7 shows relationship between $\frac{C}{\theta}$ and C where K (equilibrium constant) is obtained from the intercept of the straight line and it is known that free energy adsorption (ΔG_{ads}) is related to equilibrium constant by the equation 8.

The values of Gibb's free energy of adsorption calculated by using Equation 7 and 8 are all negative as presented in Table 4. The negative values of ΔG_{ads} indicate the spontaneous adsorption of the inhibitor molecules on the mild steel surface. Generally, values of ΔG_{ads} around -20 kJ mol^{-1} or lower are consistent with physical adsorption, while those around -40 kJ mol^{-1} or higher involves chemical adsorption as reported by Toliwal et al., [20-21].

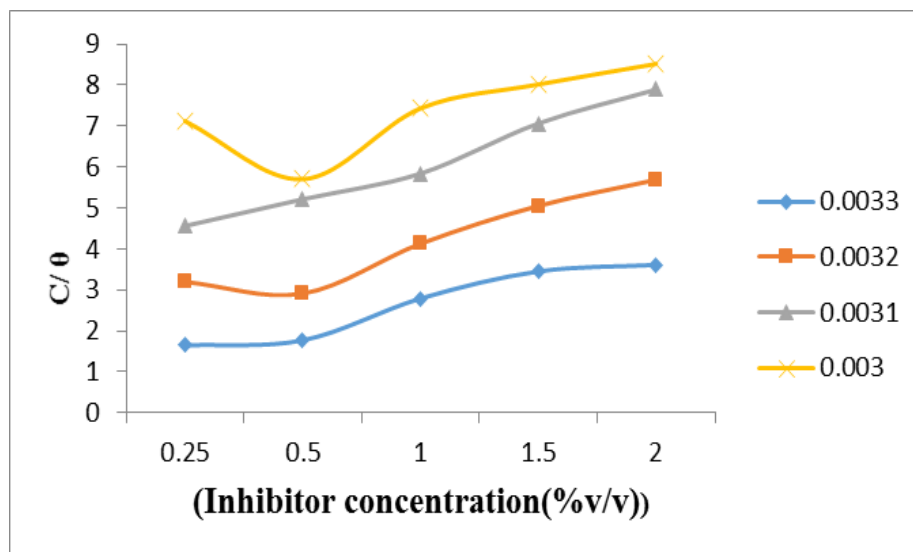


Fig.7. Langmuir adsorption isotherm plot for Mild steel in 1M HCl in the absence and presence of inhibitor at different temperatures range of 303-333K.

Table 4. Adsorption parameters deduced from Langmuir adsorption isotherm for corrosion of mild steel.

Temperature	R ² Values	K _{ads}	ΔG (J mol ⁻¹)
303K	0.9352	1.7803	-11570
313K	0.9037	1.4035	-11330
323K	0.9843	1.1750	-11220
333K	0.5684	1.9585	-12981

3.7 Infrared Spectroscopy Analysis Results

The result presented in Figures 8 and 9 shows the FTIR analysis of the inhibitor (Camel dung extract) and the corrosion product of mild steel in 1M HCl containing the Camel dung inhibitor respectively. The analysis of the inhibitor in fig. 8 shows O-H stretching of alcohol at a peak of 3335cm⁻¹, substituted phenyl stretching(overtone) at a peak of 2113cm⁻¹, and C=C stretching of aromatic compound.

The analysis of the inhibitor in fig. 9 shows monosubstituted phenyl stretching at a peak of 2113 and 2002cm⁻¹, C-H stretching of alkane at a peak of 2918 and 2847cm⁻¹, C=C-H stretching of aromatic compound at a peak of 1652cm⁻¹ and O-H stretching of alcohol.

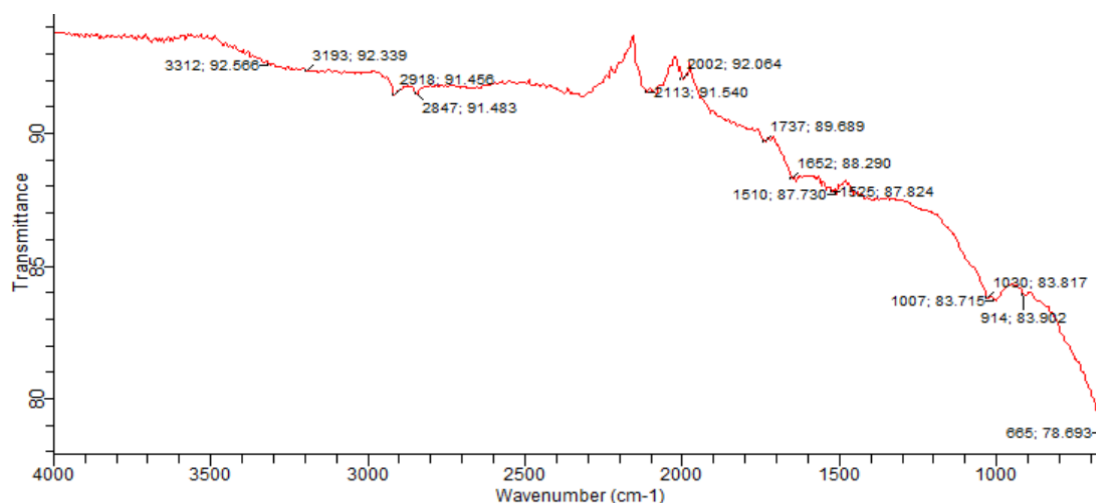


Fig 8. FTIR Spectra of the camel dung inhibitor.

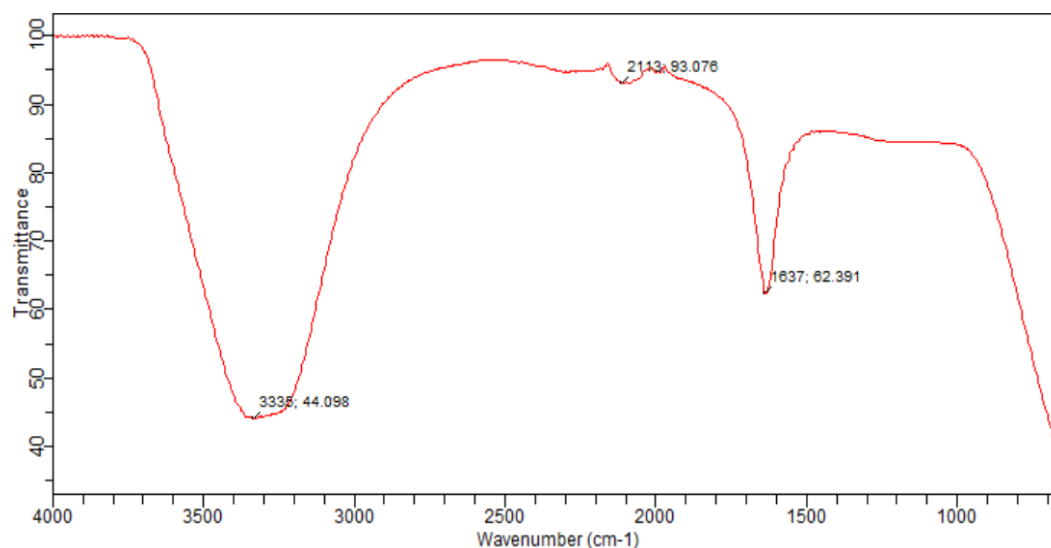


Fig. 9. FTIR Spectra of the corrosion product

Conclusion

The present study shows that Camel dung was found to be a good inhibitor for the corrosion protection of Mild steel in HCl acid solution. Corrosion rate decreases with increasing inhibitor concentration and increases with rising temperature in all the HCl acid solution media. The inhibition efficiency was increased with increase in concentration of the inhibitor and decreases with rises temperature. The adsorption of the inhibitor on the surface of Mild steel was found to be a physical adsorption mechanism.

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