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COMPARISON OF THE CORROSION INHIBITION EFFICIENCIES OF MILD STEEL IN DIFFERENT ACIDIC MEDIUMS USING COMMIPHORA CAUDATA PLANT EXTRACT

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ABSTRACT

The inhibition of corrosion of mild steel using commiphora caudata in 1MHCl and $H_2\ SO_4$ acidic mediums by weight loss and electrochemical technique was investigated.. The inhibition efficiency was found to vary with temperature, time and CC concentration .The result was found that the corrosion inhibition behavior of CC is greater in hydrochloric acid than in sulphuric acid. The adsorption of the inhibitor on the MS surface is in conformity with Langmuir adsorption isotherm. The inhibition efficiencies are increased with an increase in the concentration and temperature of the acid. Thermodynamic parameters revealed that the adsorption of inhibitor onto the mild steel surface was spontaneous process. The polarization curves showed that CC extract is a mixed-type inhibitor in both acidic media.

Keywords: Commiphora Caudata, Mild Steel, Langmuir Adsorption, Polarization Studies

I. INTRODUCTION

Mild steel is extensively used in various industries like sugar, food, leather, petrochemical, paper and textile industries. Acids are used to remove the undesirable scales and rust in several industrial processes, including pickling and descaling operations[1-3]. The corrosion inhibitors are one of the most effective and economical methods to protect metal from corrosion in acid media [4] .The organic compounds have been used to control the corrosion, especially those containing nitrogen, oxygen, sulphur, phosphorus, and multiple bonds or aromatic rings in their structures [5-6] .Those are toxic in nature and pollute the environment. Hence use of natural products which are eco-friendly, low cost, readily available and non toxic corrosion inhibiting additives. Some investigators studied that the plant extracts and are readily available and renewable source for a wide range of corrosion inhibitors [7-9]. These inhibitors are biodegradable and rich source of organic compounds which have high corrosion inhibition efficiency. The present study is aimed to investigate the inhibitive and adsorption properties of CC extracts on mild steel corrosion in 1 M HCl and 1 M H_2SO_4 using weight loss and electrochemical methods.

II. MATERIALS AND METHODS

2.1 Materials

The steel was mechanically cut to coupons , polished and stored in a desiccators prior to use.

2.2 Extract Preparation

Commiphora Caudata leaves were collected from in and around Erode city of India. The Commiphora Caudata leaves were air-dried and ground to make powder. The mixture of 5 g dried powder and 100 ml 1M HCl were taken in RB flask and refluxed for 3 hours and kept overnight. Next day it was filtered and the filtrate stored in standard flask. Similarly another extract was prepared in 1M H2SO4 solution.

2.3 Weight Loss Studies

The weight loss studies were carried out by immersing pre- weighed steel coupons in 100 ml each of blank 1M HCl and 1M H2SO4 and test solutions of various concentrations of extract at 303, 313, 323 and 333 K in a thermostat controlled water bath. The specimens were taken out, air dried and weighed..From this study ,the weight loss, inhibition efficiency(IE) and corrosion rate were determined. The inhibition efficiency was calculated using the following formula[10]

$$\text{IE \%} = \frac{\text{Wu} - W_I}{\text{Wu}} \times 100 \qquad (1)$$

$$\text{Corrosion rate (CR)} = 534 \times W / \text{DAT} \quad \text{mpy} \qquad (2)$$

Wu –weight loss in the absence of inhibitor W_I–weight loss in the presence of inhibitor , mpy - mils per year, W - Weight loss in mg, D - Density in g/cm2 (7.9 g/cm2 for mild steel), A - Area in square inch and T - immersion time in hours.

2.4 Electrochemical Studies

Electrochemical Studies were carried out by using a platinum electrode and a saturated calomel electrode (SCE) were used as auxiliary and reference electrodes, respectively, while the working electrode comprised of mild steel specimen. The three-electrode set up was immersed in acid solutions both in the absence and presence of inhibitor and allowed to attain a stable open circuit potential. The corrosion potential (Ecorr), corrosion current (Icorr), anodic Tafel slope (βa) and cathodic Tafel slope (βc) were obtained by extrapolation of anodic and cathodic regions of the Tafel plots. The inhibition efficiency (IEp) values were calculated from Icorr values using the following equation[11]

Tafel method

$$\text{IE\%} = \frac{[I_{corr}(b) - I_{corr}(I)]}{I_{corr}(b)} \times 100 \qquad (3)$$

Where , $I_{corr}(I)$ and $I_{corr}(b)$ Corrosion current with and without inhibitor

LPR method

$$\text{IE\%} = \frac{[R_p(b) - R_p(I)]}{R_p(b)} \times 100 \qquad (4)$$

Where , R_p (I) and R_p (b) – Resistant polarization with and without inhibitor

III. RESULTS AND DISCUSSION

3.1.Phytochemical Screening

The results obtained from preliminary study of phytochemical constituents of extract are Alkaloids, Terpenoids, Phenols , Carbohydrate and Protein[12-15].

Table 1: Phytochemical screening of extract of CC

Extract	Alkaloid	Terpenoid	Glycoside	Flavanoid	Saponin	Carbohydrate	Phenol	Protein
HCl	+	-	+	+	+	+	-	-
H_2SO4	+	+	-	-	-	++	++	++

3.2 Weight Loss Measurements

Table 2 showed that the weight loss increased with increase in exposure time. Also,the addition of commiphora Caudata extract from HCl and H_2SO_4 solutions resulted in a significant reduction in the weight loss of the mild steel in comparison with the blank solution. The Commiphora Caudata extract from H2SO4 solution could not reduce the weight loss considerably as that of Commiphora Caudata extract from HCl solution. The anions of acids Cl^- or $SO4^{2-}$ adsorb physically on the positively charged metal surface, giving rise to a negative charge on the metal surface. Then the organic cations are physically attracted to the anions layer which is formed on the metal surface [16]. According to the smallest anion (Cl−) attracted faster than the biggest one ($SO4^{2-}$) on to the metal surface leading to good inhibitor performance in HCl. The decrease in weight loss in the presence of the Commiphora Caudata extract has been attributed to the adsorption of phytochemical constituents (Table 1) which are present in the extract.

Table 2 Influence of concentration of CC extract on the corrosion of MS in 1M HCl and 1M H_2SO_4

Inhibitor Conc. (%v/v)	IE % of HCl extract of CC						IE % of H2SO4 extract of CC					
	1/2hr	2hrs	4hrs	6hrs	8hrs	24hrs	1/2hr	2hrs	4hrs	6hrs	8hrs	24hrs
0.1	38.33	70.38	78.82	87.77	78.3	76.6	36.3	46.8	51.5	58.2	57.2	53.0
0.2	46.67	73.81	79.93	90.36	83.2	78.2	44.6	53.3	62.0	67.3	65.9	59.8
0.3	48.33	78.46	83.24	90.78	84.7	79.6	49.5	56.9	67.4	72.2	70.9	61.1
0.4	53.33	81.15	84.35	91.81	87.1	81.6	55.4	61.3	68.1	77.4	74.8	66.3
0.6	69.67	86.54	88.60	93.01	90.2	85.3	62.3	65.8	75.1	87.4	79.1	71.2
0.7	73.63	88.85	93.55	94.58	92.4	87.5	65.4	69.2	80.0	90.1	88.1	75.7
0.9	60.50	88.04	88.58	93.37	88.9	84.9	64.2	67.6	78.5	89.8	87.7	73.7

The maximum inhibition efficiency of 94.58% and90.1 % was noticed at a concentration 0.7% of the inhibitor in1M HCl and 1M H_2SO_4 medium. Since more adsorption takes place on the metal surface, the inhibitor efficiency increases with an increase in immersion time. It was found that the optimum inhibition efficiency is reached in 6 hours and further increase in time did not cause any appreciable change in the performance of the inhibitor [17-18]The inhibition efficiency was found to decrease with the immersion time was increased from 6 h to 24 h. This may be because, prolonged immersion may result in desorption of the inhibitor molecules from the mild steel surface[19-20].

3.3 Effect of Temperature

The influence of temperature on acid extracts of CC in 1 M HCl and 1M H_2SO_4 was investigated in the temperature range, 303K-343K, for ½ an hour of immersion and the results are presented in Figure 1 and Figure 2 .It indicated that, as the temperature increases from 303K to 343K, the IE also increased[16]. The increase in IE was from 73.63 % to 90.17% for 0.7%concentration of 1 M HCl and85.6 % for 0.7% concentration of 1M H_2SO_4 at 333 K. Further rise in temperature results in slight depletion of inhibitor efficiency [21-24]. From the data, it can be inferred that the protective layer formed on MS surface, due to adsorption of plant extract, was stable up to 333 K and after that there may be desorption of plant extract at 343 K..

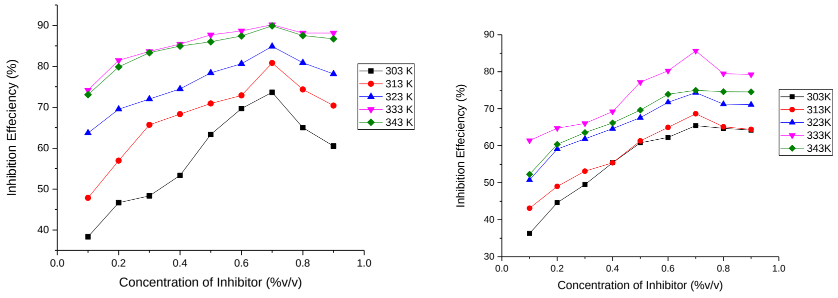


Figure 1 Variation of inhibition efficiency of CC extract, at different concentrations, on mild steel in a) 1M HCl b) 1M H_2SO_4at different temperatures

3.4 Thermodynamic Consideration

3.4.1 Activation Energy (E_a)

The inhibitive properties of the inhibitor and the temperature dependence on the corrosion rate, the apparent activation energy (E_a) for the corrosion process in the absence and presence of inhibitor were evaluated from Arrhenius equation.From the 3, it is evident that the addition of extract leading to an abrupt decrease in the apparent activation energy (E_a) to a value less than that of the uninhibited solution followed by the monotonous decrease with an increase in inhibitor concentration indicates that the inhibitory action of on mild steel in1M HCl and 1M H_2SO_4 occurs via chemical adsorption[25-27]. Activation energy (E_a) value for blank is 65.39kJ/mol and 0.7% concentration of the inhibitor in 1M HCl for 42.93kJ/mol and in 1M H_2SO_4 blank is 59.58 kJ/mol and 33.98 kJ/mol for 0.7% concentration of the inhibitor in 1M H_2SO_4.Figure 3a & b shows Arrhenius plot of the corrosion rate of mild steel in 1M HCl and 1M H_2SO_4 in the presence and absence of extract at different temperatures.

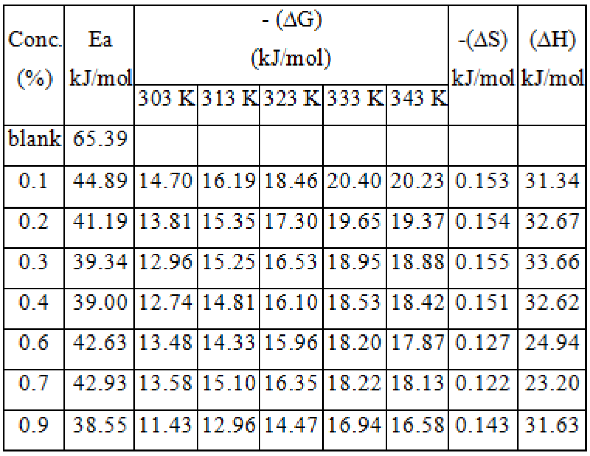
3.4.2 Change in Free Energy of Adsorption

The value of enthalpy of adsorption ΔH_{ads} and entropy of adsorption ΔS_{ads} are obtained from the basic thermodynamic equation.

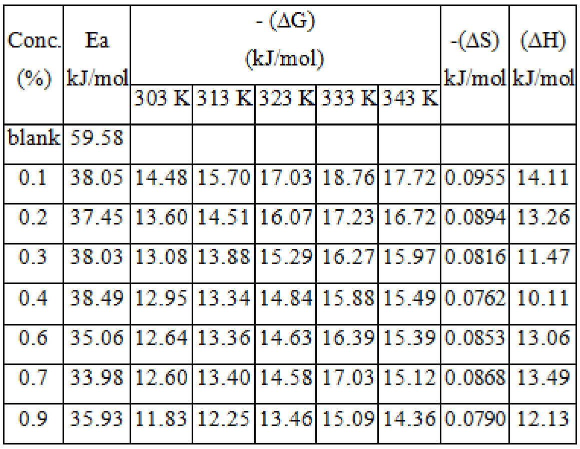
$$\Delta G_{ads} = \Delta H_{ads} - T\Delta S_{ads} \qquad (5)$$

The slope (ΔS_{ads}) and intercept (ΔH_{ads}) obtained from the plot -ΔG_{ads} Vs T was depicted in fig 4 a & b for extract of CC leaves in 1M HCl and1M H_2SO_4 .Table 3 shows the calculated values of activation energy (E_a), free energy of adsorption (ΔG_{ads}), the enthalpy of adsorption (ΔH) and the entropy of adsorption (ΔS) for mild steel in 1M HCl and1M H_2SO_4 with and without extract. The negative value of free energy of adsorption (ΔG_{ads}) indicates the spontaneous adsorption of the inhibitor. The positive values of enthalpy indicate the endothermic reaction suggesting that a high temperature favours the complexation process and the same is in good agreement with the increasing stability with temperature [28]. The negative values of ΔSact pointed to a greater order produced during the process of activation. This can be achieved by the formation of activated complex and represents association or fixation with consequent loss in the degrees of reedom of the system during the process [29]. This also supports the assumption of chemical adsorption.

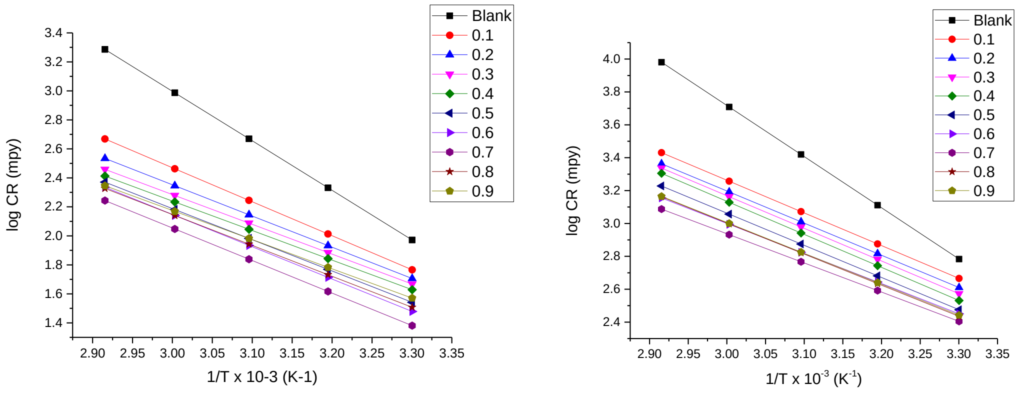
Table 3 Thermodynamic data for mild steel in of **1M** HCl **and 1M H_2SO_4 in the presence and absence of acid extract CC at 303 K to 343 K**



Conc. (%)	Ea kJ/mol	- (ΔG) (kJ/mol) 303 K	313 K	323 K	333 K	343 K	-(ΔS) kJ/mol	(ΔH) kJ/mol
blank	65.39							
0.1	44.89	14.70	16.19	18.46	20.40	20.23	0.153	31.34
0.2	41.19	13.81	15.35	17.30	19.65	19.37	0.154	32.67
0.3	39.34	12.96	15.25	16.53	18.95	18.88	0.155	33.66
0.4	39.00	12.74	14.81	16.10	18.53	18.42	0.151	32.62
0.6	42.63	13.48	14.33	15.96	18.20	17.87	0.127	24.94
0.7	42.93	13.58	15.10	16.35	18.22	18.13	0.122	23.20
0.9	38.55	11.43	12.96	14.47	16.94	16.58	0.143	31.63

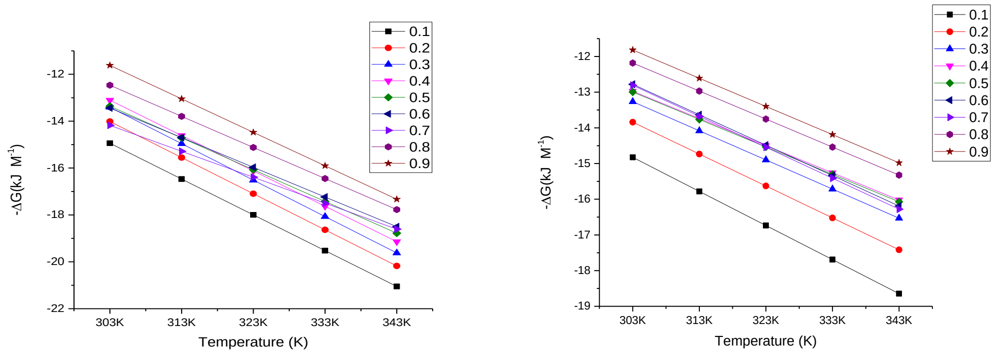


Conc. (%)	Ea kJ/mol	- (ΔG) (kJ/mol) 303 K	313 K	323 K	333 K	343 K	-(ΔS) kJ/mol	(ΔH) kJ/mol
blank	59.58							
0.1	38.05	14.48	15.70	17.03	18.76	17.72	0.0955	14.11
0.2	37.45	13.60	14.51	16.07	17.23	16.72	0.0894	13.26
0.3	38.03	13.08	13.88	15.29	16.27	15.97	0.0816	11.47
0.4	38.49	12.95	13.34	14.84	15.88	15.49	0.0762	10.11
0.6	35.06	12.64	13.36	14.63	16.39	15.39	0.0853	13.06
0.7	33.98	12.60	13.40	14.58	17.03	15.12	0.0868	13.49
0.9	35.93	11.83	12.25	13.46	15.09	14.36	0.0790	12.13



a **b**

Figure 3 Arrhenius Plot for the Dissolution of Mild Steel in a)1M HCl b) 1M H_2SO_4 with and without CC Extract at Various Temperatures



a **b**

Figure 4 ΔG_{ads} as a function of temperature in the presence of CC extract in a) 1M HCl b)1MH_2SO_4

3.4.3 Adsorption Consideration

Since corrosion inhibition is related to the adsorption of the inhibitor molecules on the metal surface, the surface coverage θ of the adsorption process was calculated using the equation:

$$\theta = IE / 100 \quad (6)$$

In order to find out the adsorption isotherm for the current study, attempts were made to fit the data with various isotherms viz,Langmuir,Temkin and freundlich isotherms. Langmuir isotherms give the best fit with the correlation co efficient almost near to (0.9998)unity.Thus a plot of C/θ and C should be a straight line. This is indicative the adsorption follows Langmuir adsorption isotherm. Langmuir adsorption isotherm could be represented as:

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

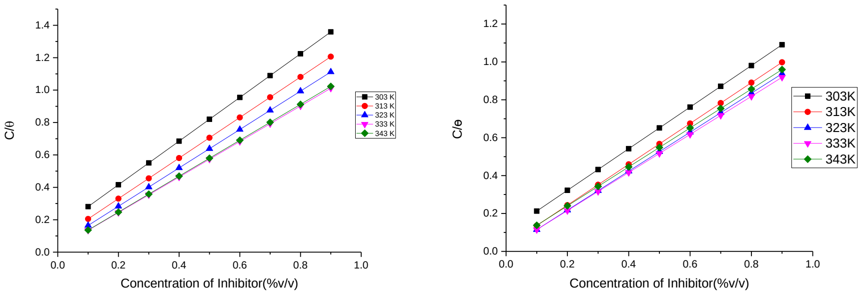


Figure 5 Langmuir Adsorption is Otherm plot for the Different Concentrations of CC Extract on the Mild Steel in a)1M HCl b) 1M H_2SO_4

3.5 Potentiodynamic Polarisation

Electrochemical measurements were carried out to study the nature of inhibitor, mode of action and mechanism of reaction. The effect of the extract on the electrochemical behavior of mild steel in 1M HCl and 1M H_2SO_4 solutions in the absence and presence of inhibitor was studied and the electrochemical parameters such as corrosion potential (Ecorr), corrosion current density (Icorr),anodic and Cathodic Tafel slopes (ba and bc), resistance polarization (Rp) and percentage of the inhibition efficiency are listed in the Tables 5and 6. Ecorr values are centered and the addition of extract in 1M HCl and 1M H_2SO_4 solution did not show much significant change in Ecorr suggesting that the plant extract control the corrosion by controlling both anodic and cathodic reactions. This indicates that the inhibition of corrosion of mild steel in 1M HCl and 1M H_2SO_4 for the extract is under mixed control [30]. The Ecorr values are also shifted to the more negative side with an increase in the inhibitor concentration. Icorr values of the inhibited acids are lower than that of the uninhibited acid, which indicates that increase in corrosion inhibition property is due to increase in blocked fraction by adsorption of inhibitor molecules on the electrode surface [31]. Thus as the concentration increases, there is a regular decrease in Icorr, showing that extract of *commiphora caudate* in 1M HCl and 1M H_2SO_4 act as a very good inhibitor for mild steel. Ecorr suggests that the inhibitor is a mixed type inhibitor [32]. This observation suggests that the plant extract consists of various organic compounds like phenols, flavonoids, sterols, amino acids, all of which might act by specific mechanism and the overall effect may be a mixed mode of inhibition [33]. The IE values were found to increase with increase in the concentration of the extract. The maximum linear inhibition efficiency at an optimum concentration of 0.7% v/v of cc extract in 1M HCl and 1M H_2SO_4were found to be 70.34%and 63.69% respectively.

Table 5 Electrochemical Polarization (Tafel) Parameters for the Corrosion of Mild Steel in 1M HCl Containing with and without CCextract

Con. (%)	$-E_{corr}$ (mV)	I_{corr}		b_a	b_c	R_p	
		(mA/Cm^2)	IE (%)	(mV/dec)	(mV/dec)	(Ωcm^2)	IE(%)
blank	502.9	0.412	-	83	125	67.03	
0.2	487.1	0.175	57.48	66	138	140.5	52.29
0.4	486.6	0.136	67.11	68	133	183.4	63.45
0.7	484.7	0.122	70.34	67	136	204.3	67.19

Table 6 Electrochemical Polarization (Tafel) Parameters for the Corrosion of Mild Steel in 1M H_2SO_4 Containing with and without CC extract at room Temperature

conc. (%)	$-E_{corr}$ (mV)	I $_{corr}$		b_a (mV/dec)	b_c (mV/dec)	Rp	
		(mA/cm^2)	IE (%)			(Ωcm^2)	IE (%)
Blank	522.90	0.896		81	119	29.81	
0.2	502.40	0.429	52.11	62	137	55.32	46.11
0.4	493.10	0.377	57.90	65	148	66.15	54.94
0.7	487.90	0.325	63.69	60	157	73.68	59.54

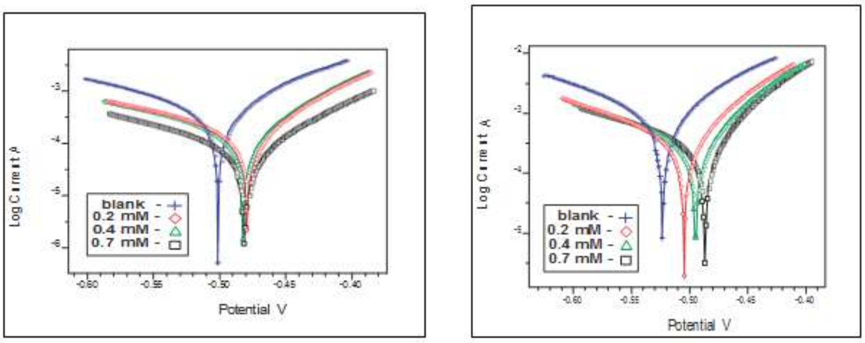


Figure 6 :Potentiodynamic polarization curves for the different concentrations of CC extract on the mild steel in a)1M HCl b) 1M H_2SO_4

3.6 Electrochemical Impedance Measurement

The impedance parameters for mild steel in 1M HCl and1M H2SO4 with and without CC extract is given in Table 7. The decrease in Cdl values indicates that the adsorption of phytochemical constituents in the acid extracts of CC on the mild steel surface. Decrease in C_{dl}, which can result from a decrease in local dielectric constant and an increase in the thickness of the electrical double layer, suggests that the inhibitor functions by adsorption at the metal-solution interface[34]. The increase in Rct values with an increase in inhibitor concentration is the result of an increase in the surface coverage by the inhibitor molecules. The increased surface coverage led to an increase in inhibitor efficiency.The maximum inhibition efficiency obtained for CC extract is 73.09% for 1M HCl and 66.99 % for 1M H_2SO_4 at 0.7% concentration. From the data it is clear that Cdl values decrease with the increase in inhibitor concentrations due to the adsorption of the phytochemical constituents of the plant extract replacing water at the metal-solution interface.The Nyquist plots (Figure 7) are semi circle in shape corresponding to a charge transfer controlled process. There was a gradual increase in the diameter of each semicircle of the Nyquist plot due to increase in the number of inhibitive molecules in the extract when the concentration was raised from 0.1 to 0.7% v/v. This suggested that the formed inhibitive film was strengthened by addition of plant extract. This may be due to formation and gradual improvement of the barrier layer of the inhibitive molecules (IE) and as a result the acid corrosion rate of mild steel gradually decreased [35].

Table 7 Electrochemical Impedance Parameters for Mild Steel in 1M HCl and 1M H_2SO_4 Containing Different Concentrations of CC extract

Conc. (%)	HCl			H_2SO_4		
	R_{ct} (Ωcm^2)	C_{dl} $\mu F/cm^2$	IE (%)	R_{ct} (Ωcm^2)	C_{dl} $\mu F/cm^2$	IE (%)
blank	25.66	53.14		19.15	35.82	
0.2	53.52	49.14	52.06	30.55	31.36	37.31
0.4	70.62	43.36	63.67	41.14	31.88	53.45
0.7	95.33	40.96	73.09	58.01	28.03	66.99

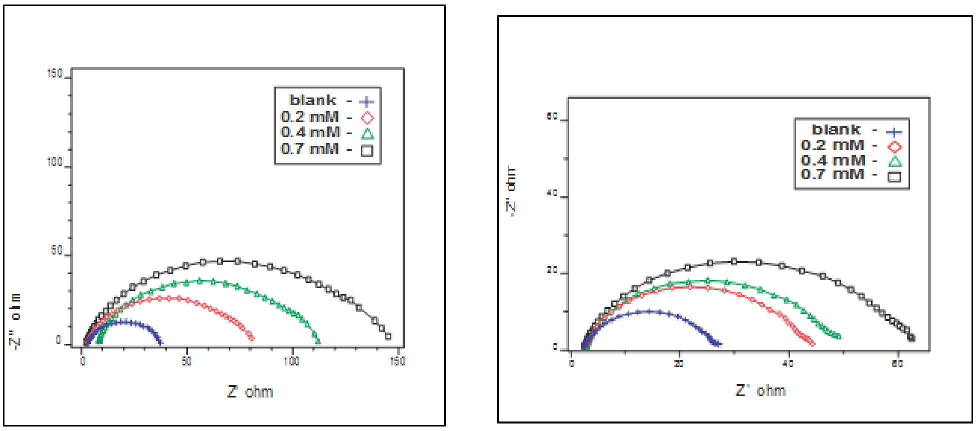


Figure 7 Impedance Diagram for Mild Steel in a)1M HCl b) 1M H_2SO_4 in the Presence and Absence of Different Concentrations of CC Extract

IV. CONCLUSIONS

The results showed that the inhibition efficiency increased with increase in concentration of the inhibitor and temperature in both acid solutions. The chemical adsorption mechanism of the inhibition process was in conformation with the data obtained, and Langmuir adsorption were best fitted into the obtained results. The calculated activation energy values also conformed to a chemical adsorption mechanism. The negative values of free energy of adsorption of inhibitor indicate the spontaneity of adsorption. In Polarization studies indicate that the CC inhibitor act as a mixed type inhibitor. The results obtained from weight loss and electrochemical studies are found to agree very well with each other. Comparative analyses of the results from both acid solutions suggest that phyto constituents in the extract may be responsible for the observed inhibitive behavior.

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