ADSORPTION AND ELECTROCHEMICAL BEHAVIOR OF CYPERUS ROTUNDUS ON OIL AND GAS PIPELINE STEEL IN1.0N HYDROCHLORIC ACID

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Abstract:

The inhibition efficiency of Cyperus Rotundus extract on the corrosion of oil and gas pipeline steel in 1.0N Hydrochloric acid in various concentrations with temperature was investigated by mass loss measurement and electrochemical techniques. The observed results indicate that the inhibition efficiency is increased with increase of inhibitor concentration but decreases with rise in temperature. The inhibitor obeys Langmuir adsorption isotherm. Thermodynamic parameters (Viz: E_a, Q_{ads}, ΔG_{ads}, ΔH and ΔS) revealed the adsorption of the inhibitor is physisorption, exothermic and spontaneous. The inhibition efficiency was found to be 81.95%and 82.01% by impedance and polarization studies respectively. The study also showed that CR extract functioned as a mixed-type corrosion inhibitor in acidic environment.

Keywords: Mass loss, Oil and Gas pipeline, Acid, Green inhibitor, Potentiodynamic polarization, Impedance spectroscopy.

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1. Introduction

In oil and gas industries, the corrosion issues have always been of great importance with consequences similar to those of natural disasters. Corrosion normally occurs in oil and gas pipelines, since pipelines play the role of transporting oil and gas from the well to the processing units and they are exposed to the continuous threat of corrosion. The existence of corrosion is the consequence of chemicals reacting with metal surface and its rate depend on the quality of crude oil, acidic constituents and the environment. Moreover, oil and gas pipeline steel have a high proportion of iron to carbon, which means it is very susceptible to deterioration and are responsible for numerous financial loss primarily in the industrial area. It is obviously the best way to combat is prevention. Among several methods to prevent destruction or deterioration of metal surface, the corrosion inhibition is one of the best-known methods of protection and widely used in industries. This method is adopting righteously due to low cost and practicable one [1-3]. Generally, inhibitors had great acceptance in industries because of its excellent anti-corrosive properties. However, many showed up a secondary effect which harms the surrounding.Thus, the scientific gathering began inquiring for environmentally benign inhibitors, like green inhibitors. Many plant extract such as Aloe veragel, Caesalpinia pulcherrima, Phyllanthus amarus, Pterolobium hexapetalum, Celosia argentea,Cucurbita maxima8, Polyalthia longifolia, Ligularia fischeri, Molasses, Phyllanthus fraternus, Pentaclethra macrophyllaBentham, Antigonon leptopus, Aniba rosaeodora, Rotula aquatica, Nypa fruticans wurmb, Anacardium occidentale gum, Juniperus, Amaranthus cordatus, Citrus aurantifolia, Coconut coir dust, Albizia lebbeck, Hyptis suaveolens,Eugenia Jambolana,Terminalia chebula, Musa paradisica peel, Hibiscus sabdariffa, Raphanus sativus, Ocimumtenuiflorum, Cassiaalata leaves and canna indica[4- 32] exhibit great corrosion hindrance ability towards oil and gas pipeline steel surface. In continuity of our research work, the inhibiting properties of Cyperus Rotundus (CR) extract in 1.0N HCl was studied using mass loss measurement with various temperatures and electrochemical techniques.

2. Materials and Methods

Cyperus Rotundus used as a corrosion inhibitor
Stock solution of Cyperus Rotundus Extract

Cyperus Rotundus(CR) was collected from the Western Ghats in courtallam area and dried under shadow for about 10 days, grained well, then soaked in a solution of ethyl alcohol for about 48 hrs.It was then filtered followed by evaporation in order to remove the solvent completely and the plant extract was collected. From this extract, different concentration of 10 to 1000ppm stock solution was prepared using double distilled water and used throughout our present investigation.

Specimen preparation

Rectangular specimen of oil and gas pipeline steel was mechanically pressed cut to form different coupons, each of dimension exactly 20cm^2 (5x2x2cm)with emery wheel of 80 and 120 and degreased with trichloroethylene, washed with distilled water, cleaned and dried, then stored in desiccators for our present study.

3. Results and Discussion

Effect of Temperature

Dissolution behavior of oil and gas pipeline steel in 1.0N HCl containing various concentration of CR extract at 303 to 333K was studied by mass loss experiments and the observed results are listed in Table-1. Observed results reveal that corrosion rate is decreased with increase of inhibitor concentration and increased with rise in temperature from 303 to 333K because the hydrogen evolution over potential increases. However the surface coverage (θ) and inhibition efficiency is decreased with rise in temperature from 303 to 313 k and then increased slightly at 333k proving that the phytoconstituents are stable even at higher temperature and get strongly adsorbed onto the metal surface. The maximum of 88.40% inhibition efficiency is achieved at 303K and the nature of adsorption will be confirmed by thermodynamic parameters...

Table-1: Corrosion parameters of oil and gas pipeline steel in 1.0N HCl containingdifferent concentration of CR extract at 303 to 333 K.

Con.of inhibitor (ppm)	Corrosion Rate (mmpy)			Inhibition Efficiency (%)		
	303K	313k	333K	303K	313K	333K
0	64.56	81.68	133.05	-	-	-

10	56.42	72.42	116.77	12.61	11.34	12.24
50	47.44	62.60	100.77	26.52	23.36	24.26
100	41.54	55.02	88.70	35.66	32.64	33.33
500	20.77	27.79	44.63	67.83	65.98	66.46
1000	7.49	12.07	17.68	88.40	85.22	86.71

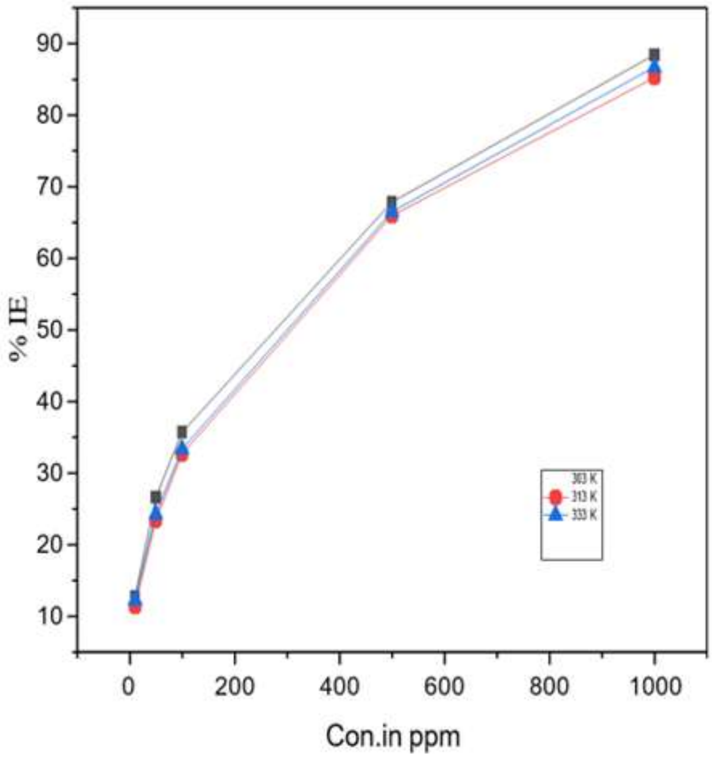


Fig-1. IE diagram of oil and gas pipeline steel in 1.0N HCl comprising CR extract atvarious temperatures

Activation Energy

The relation between corrosion rate and temperature is expressed by Arrhenius equation -1

Log CR (K) = -Ea / 2.303 RT + log A--1

The Arrhenius plot is obtained by plotting log CR against 1/T and the values of E_a and A, (Arrhenius factor) obtained from the straight line are given in table-2.The observed E_a values ranged from 20.2222 to 24.0180 kJ/mol (0- 1000ppm) indicated that there is an attraction between the molecules in CR and the metal surface thereby resist the dissolution of metal surface. Moreover the E_a value is lower than the threshold value of 80 KJ/mol required for chemical adsorption revealed that the adsorption of CR on oil and gas pipeline is physical adsorption.

Table -2: Activation energy (E_a) and heat of adsorption (Q_{ads}) of CR extract on oil andgas pipeline steel in1.0N HCl.

S.No	**Con. of inhibitor (ppm)**	**Reg. coefficient(R^2)**	**E_a ($KJmol^{-1}$)**	**Arrhenius factor (A)**	**Q ads ($KJmol^{-1}$)**
1.	0	0.9999	20.2222	1.86×10^5	--
2.	10	0.9999	20.3412	$1.67.\times10^5$	-9.5153
3.	50	0.9985	21.0680	$1.79.\times10^5$	-13.3287

4.	100	0.9983	21.2142	$1.65.\times10^5$	-10.5867
5.	500	0.9972	21.3902	$0.87. \times10^5$	-06.5917
6.	1000	0.9368	24.0180	0.65×10^5	-21.9905

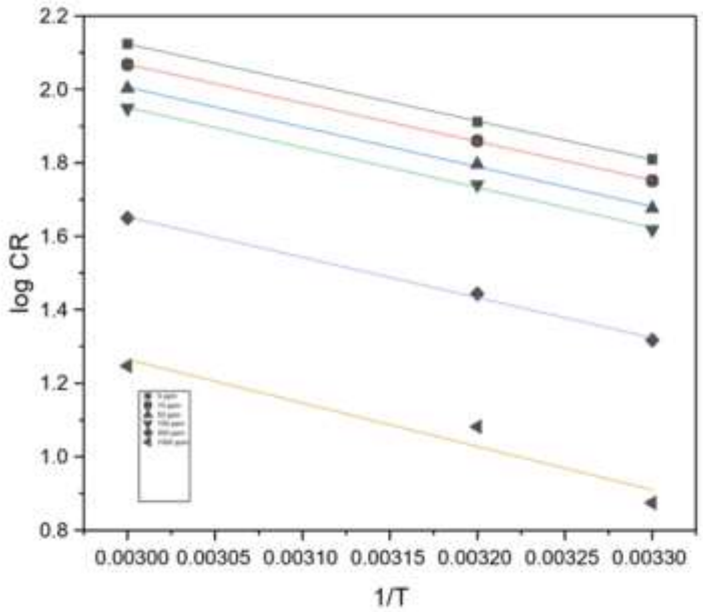


Fig-2. Arrhenius plot of oil and gas pipeline steel in the absence and presence ofdifferent concentration of CR extract

Heat of Adsorption

The heat of adsorption (Q_{ads}) of various concentration of CR extract on oil and gas pipeline steel in 1.0N HCl is calculated using equation (2) and Q_{ads} ranged from -9.5153 to -21.9905 kJ/mol

$$Q_{ads} = 2.303R[\log (\theta_2/1-\theta_2)- \log (\theta_1/1-\theta_1)] \times (T_2T_1/T_2-T_1) \text{-----------} 2$$

Adsorption Studies

Adsorption isotherms are very important in determining the mechanism of corrosion inhibition. The most frequently used isotherms are Langmuir, Temkin, Frumkin, Flory- Huggins, Freundlich, Bockris-Swinkles, Hill-Deboer, Parson"s and El-Awady isotherms.

Langmuir Isotherm

Langmuir adsorption isotherm is expressed according to equation -3,

$$\log C/\theta = \log C - \log K \text{-----------} 3$$

Plotting log (C/θ) against log C gave a linear relationship as shown in fig-3, and the adsorption parameters are presented in table - 3. Average regression co-efficient value (R^2=0.9995) is almost close to unity ($R^2 \leq 1$)and suggested Langmuir adsorption isotherm provides a good model of the sorption system, The values of (ΔG) are however below -20KJ/mol provides an existence of multimolecular layer on the surface of oil and gas pipeline steel surface. The average monolayer capacity (Q_m) obtained from Langmuir equation is 9.573 mg/l

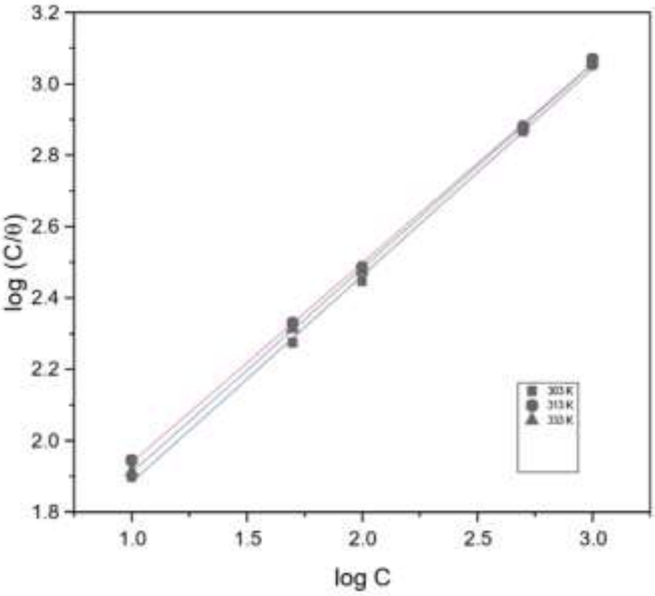


Fig -3. Langmuir isotherm for adsorption of CR extract on the surface of oil and gaspipeline steel

Temkin Isotherm

In Temkin adsorption isotherm, the degree of surface coverage (θ) is related to theinhibitor concentration (C) according to equation - 4,

Exp (-2a θ) =KC-- 4

where K= adsorption equilibrium constant and "a" is an attractive parameter, Rearranging andtaking logarithm on both sides of equation (4) gives equation - 5

θ = (-2.303logk/2a)- (2.303log C/2a) --- 5

The surface coverage (θ) against log Cwas plotted and presented in fig-4 and the adsorption parameters obtained are recorded in table-3. The average regression co-efficient (R^2) is 0.9441 away from unity. However, the values of attractive parameter (a) are positive, indicating that there is no interaction between the adjacent molecules in the adsorbed layer.

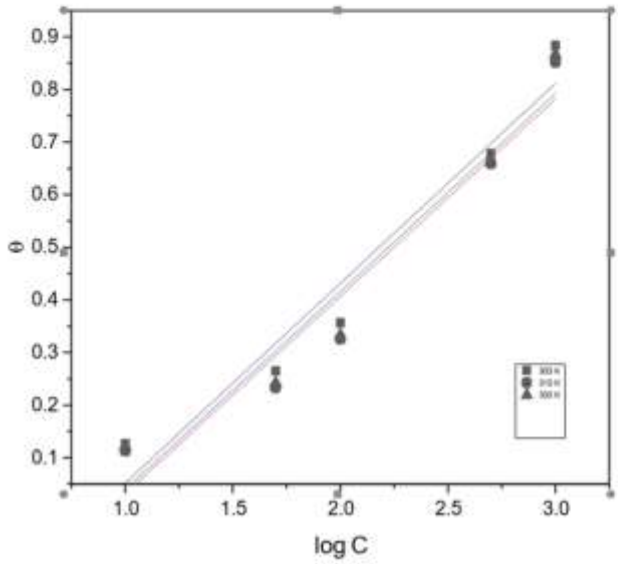


Fig-4. Temkin isotherm for adsorption of CR extracton the surface of oil and gas pipelinesteel.

Flory-Huggins Isotherm

Flory-Huggins adsorption isotherm can be expressed according to equation -6log (θ/C) = log K + xlog (1- θ)--6

A plot of log (θ/C) against log (1- θ) is shown in fig-5, and obtained data are given in table- 3. Theaverage regression co-efficient (R^2) 0.8343 is very far away the unity. The values of the size parameter(x) are positive which indicates that the adsorbed species of CR extract is bulky, since it could displace number of water molecules and occupying adsorption sites on the surface of oil and gas pipeline steel

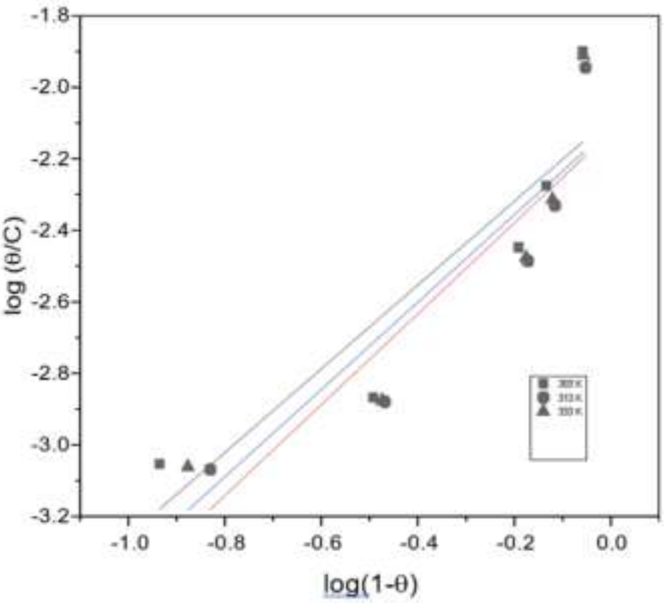


Fig-5. Florry-Huggins isotherm for adsorption of CR extraction the surface of oil and gaspipeline steel

Frumkin Isotherm

Frumkin adsorption isotherm is given by equation - 7

log { [C]* (θ/1- θ)} = 2.303 log K +2αθ------------------------------

where,„k" is the adsorption–desorption constant and „α" is the lateral interaction term describing the interaction in adsorbed layer. The plot of log {[C]* (θ/1- θ)} versus θ is shown in fig-6. The values for Frumkin adsorption parameters are recorded in table-3. The average regression coefficient (R^2= 0.9763) is near to unity and the adsorption parameter „α" are positive suggest that the attractive behavior of the inhibitor on the surface of oil and gas pipeline steel.

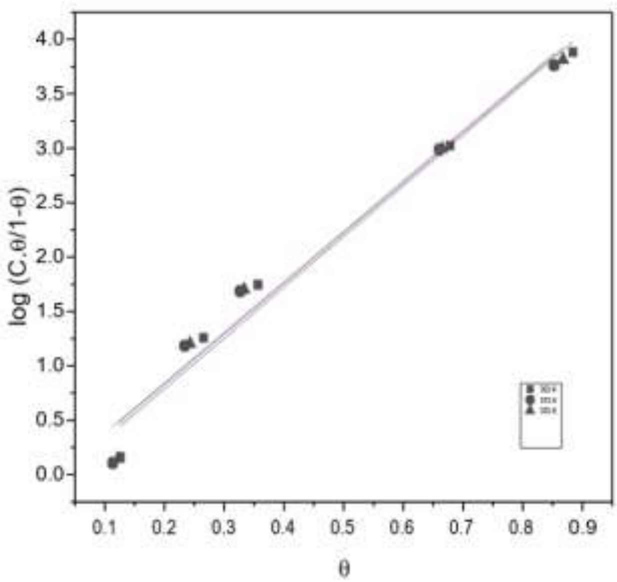


Fig -6. Frumkin isotherm for adsorption of CR extraction the surface of oil and gaspipeline steel

Freundlich Isotherm

The Freundlich adsorption isotherm can also be applied using equation -8

$$\theta = Kc^{1/n} \qquad 8$$

The linear form of Freundlich isotherm equation is as followslog

θ = logK + 1/nlog C--- 9

where, k" is adsorption capacity (L/mg) and „1/n" is adsorption intensity and also indicates the relative distribution of the energy and heterogeneity of the adsorbed sites. This can be obtained by plotting log θ vs. log C and the parameters are shown in table-3. The value of „n" gives an indication on the feasibility of adsorption. It is generally stated that values of „n" in the range 2-10 represent good, 1-2 moderate and less than 1 poor adsorption characteristics. Thus, the adsorption of CR extract as inhibitor on oil and gas pipeline steel is good by physical process, since its "n" value in the range 2.3759 to 2.3370.

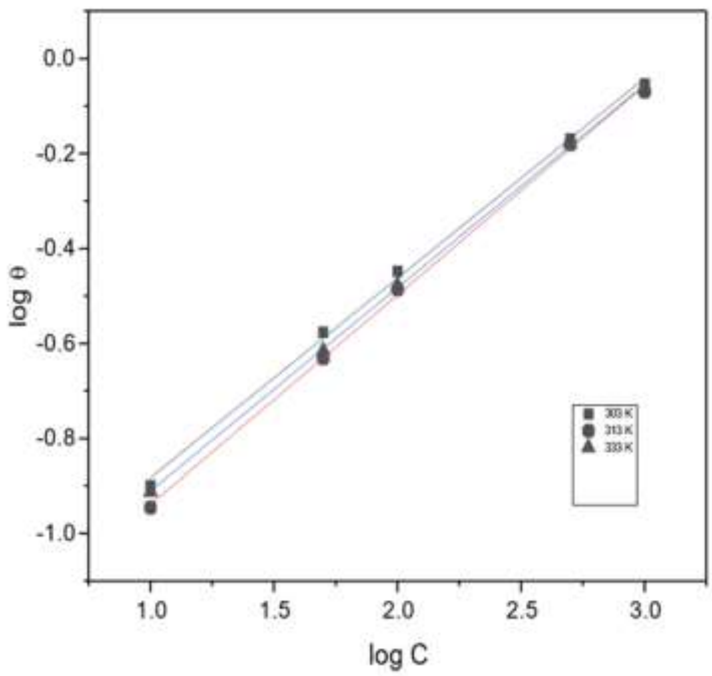


Fig –7. Freundlich isotherm for adsorption of CR extract on the surface of oil and gaspipeline steel

El –Awady Isotherm

The El-Awady adsorption isotherm is given by

$$\log (\theta/1-\theta) = \log K + y \log C \quad \text{--} \ 10$$

where C is the concentration of inhibitor in the bulk solution, „θ" is the degree of surface coverage, K is an El-Awady isotherm constant, equilibrium constant of an adsorption process $K_{ads} = K^{I/Y}$ and y represents the tendency of adsorbate occupying a given active site. Value of 1/y less than unity implies the formation of multilayer onto the metal surface, while the value of 1/y greater than unity reveals that a given inhibitor occupy more than one active site Curve fitting of the data to the thermodynamic/kinetic model [El-Awady] is shown in fig-8. The calculated k_{ads} and 1/y from the El-Awady et al isotherm model is listed in table-3.

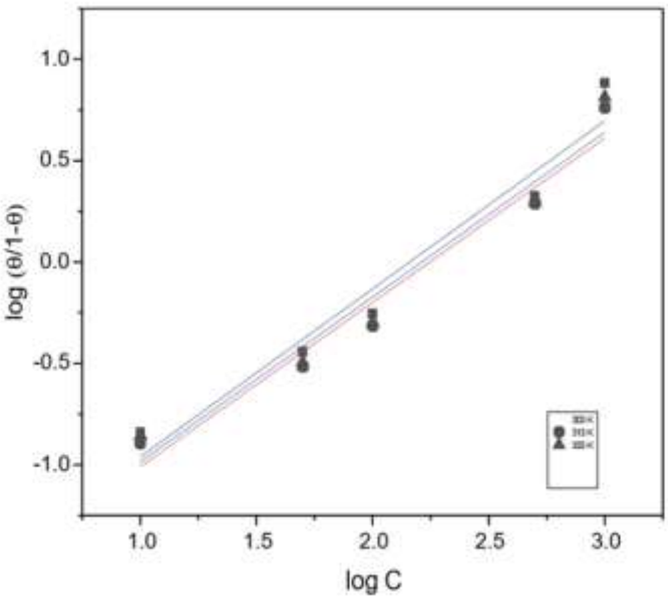


Fig-8. El-Awady isotherm for adsorption of CR extract on the surface ofoil and gaspipeline steel

Table-3: Adsorption parameters of CR extract on the surface of oil and gas pipelinesteel in 1.0N HCl

AdsorptionIsotherm	peraturein K	R^2	K	ΔG_{ads} kJ/mol	VariablesQ_m /a/x/α /n/ 1/y/ k_{ads}
	303	0.9991	7.3866	-10.7893	0.9670
Langmuir	313	0.9995	6.7088	-11.2922	0.9470
	333	0.9998	6.8636	-11.9292	0.9579
					a
	303	0.9497	0.1368	-12.8433	3.0295
Temkin	313	0.9427	0.1218	-13.1736	3.0690
	333	0.9398	0.1275	-14.0590	3.0601
					x
	303	0.8323	0.0823	-10.1675	1.1708
Florry-	313	0.8465	0.0748	-10.4985	1.2703
Huggins	333	0.8241	0.0723	-11.1709	1.2209
					α
	303	0.9805	0.8680	-14.3062	2.3315
Frumkin	313	0.9735	0.9202	-15.4019	2.3196
	333	0.9749	0.9103	-16.2579	2.3157
					n
	303	0.9983	0.0496	-10.4075	2.3759
Freundlich	313	0.9992	0.0417	-10.7035	2.2665
	333	0.9997	0.0459	-11.4140	2.3370
					1/y
	303	0.9546	0.0163	-10.2142	1.2079

El-Awady	313	0.9648	0.0151	-10.5444	1.2341
	333	0.9549	0.0160	-11.2234	1.2311
					K_{ads}
					-2.1601
					-2.2461
					-2.2124

Con. ppm	$-\Delta G_{ads}$ KJ/mol			ΔH_{ads} KJ/mol	ΔS_{ads} KJ/mol
	303 K	313 K	333 K		
10	22.62	23.05	24.77	-0.2133	0.0736
50	20.87	21.12	22.61	-2.4808	0.0602
100	20.21	20.53	21.93	-2.2263	0.0590
500	19.52	19.94	21.28	-1.3232	0.0598
1000	21.01	20.98	22.66	-2.8365	0.0591

Table-4: Thermodynamic parameters of adsorption of CR extract on the surfaceof oil and gas pipeline steel

Thermodynamic Parameters of adsorption

Thermodynamic parameters play an important role in determining the inhibition mechanism. The free energy of adsorption ($-\Delta G_{ads}$) characterize the interaction of adsorbed molecules and the metal surface which was calculated using the relation-11

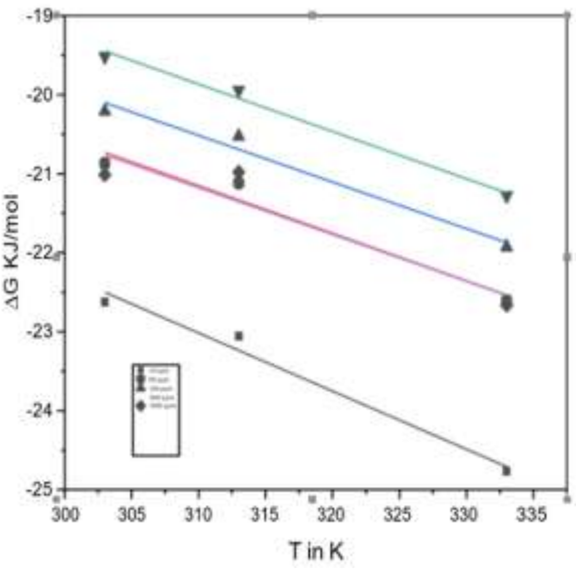
$-\Delta G_{ads} = 2.303RT(1.74 + \log(\theta/1-\theta) - \log C)$ --------------------------------- 11

where „C" is the concentration of the inhibitor in % (i.e. v/V) Then the change in enthalpy and entropy were calculated using Gibbs-Helmholtz equation -12

$\Delta G_{ads} = \Delta H - T\Delta S$ --- 12

The calculated ΔG_{ads}, ΔH and ΔS are shown in table-4. The negative values of ΔG_{ads} ensure the adsorption process and stability of adsorbed layer onto the metal surface. The stability of adsorbed layer increases with increase in temperature which is clearly shown from the increase in change in free energy with rise in temperature. The calculated $\Delta G_{ads}$is found to be negative less than 40 KJ/mol indicating that the adsorption of phytoconstituents on the metal surface is spontaneous and are adsorbed by a strong physical adsorption

Fig-9 a plot of Gibbs free energy versus Temperature



a.

Thermodynamic parameters of dissolution

The another form of transition state equation derived from Arrhenius equation (4)is shownbelow -13

$$CR = RT/Nh \exp(\Delta S/R) \exp(-\Delta H/RT) \quad \text{------------------------------ } 13$$

A plot of log (CR/T) vs. 1/T gave a straight line shown in Fig-10 with a slope (−ΔH/R) and an intercept [log(R/Nh)) + (ΔS/R)], from which the values of ΔS and ΔH were calculated and listed in table-5. The positive values of ΔH depicts that the nature of the dissolution processin acid medium is endothermic. The decreases in positive values of entropy (ΔS) with increase in inhibitor concentration indicating a decrease in disorderness on going from reactants to the activated complex.

Table - 5: Thermodynamic parameters of oil and gas pipeline steel in 1.0N HClwith various concentration of CR extract

S.No	Con. ppm	ΔH (kJ mol^{-1})	ΔS (J $k^{-1}mol^{-1}$)
1	0	7.59	82.83×10^{-3}
3	10	7.60	82.78×10^{-3}
4	50	7.87	82.81×10^{-3}
5	100	7.93	82.78×10^{-3}
6	500	7.98	82.50×10^{-3}
7	1000	8.71	82.38×10^{-3}

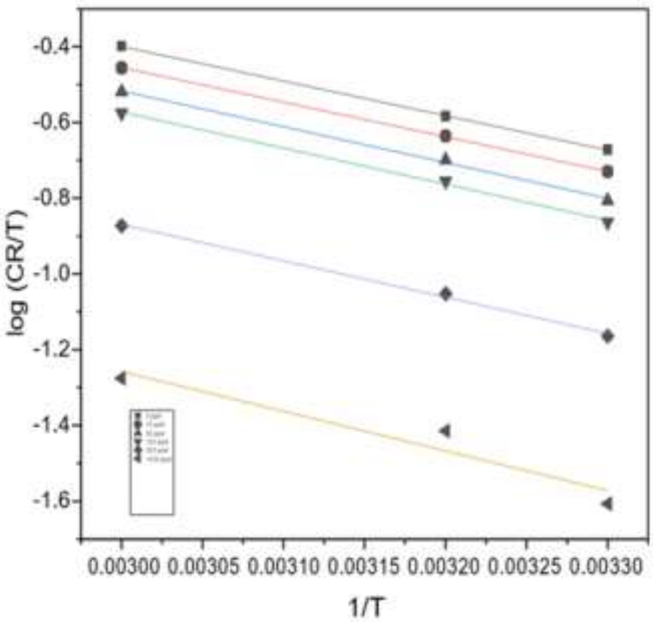
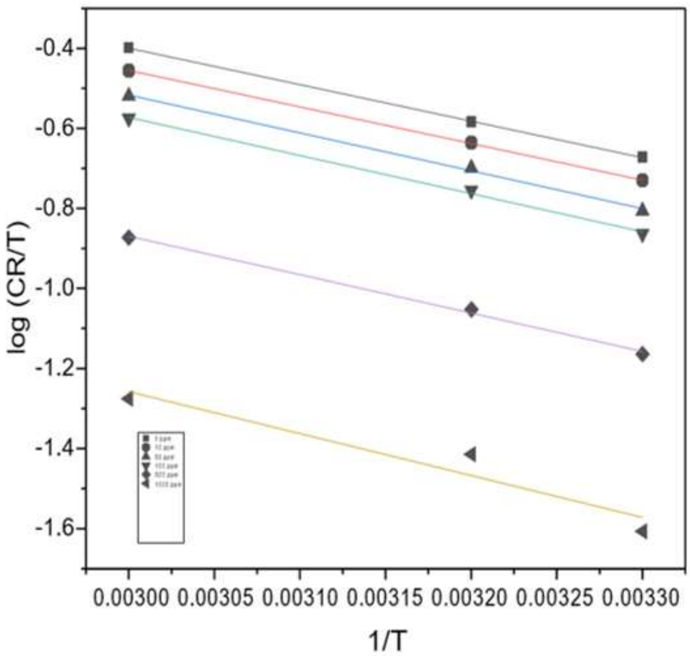


Fig-10. Transition plot of oil and gas pipeline steel in 1.0 N HCl comprising variousconcentration of CR extract.



Electrochemical Measurements

Polarization Studies

The potentiodynamic polarization curves for oil and gas pipeline steel in1.0N HCl in the presence and absence of different concentration of CR extract are shown in fig-11. The various electrochemical parameters such as corrosion current density (I_{corr}), corrosion potential (E_{corr}), and Tafel constants (b_a and b_c) are given in table-6. It is observed that the presence of CR extract lowers the corrosion current density (I_{corr}) from 3097 to 557.2 µA/cm^2.This significant reduction in corrosion current density clearly indicated that decrease in corrosion rate in the presence of inhibitor. The corrosion potential (E_{corr}) was shifted to noble direction -502.1 to -516mV.The percentage of inhibition efficiency is increased with increase of inhibitor concentration. This may be attributed to the formation of barrier film due to the adsorption of inhibitor molecules onto metal surface involving interaction between π- electrons of inhibitor molecules and vacant d-orbitals of metal ion. It is evident that the addition of an inhibitor shifts E_{corr} value exceeds ±85mv with respect to corrosion potential of solution in the absence of an inhibitor, the inhibitor acts as either anodic (or) cathodic type. In the present case the maximum displacement in E_{corr} is found to be within ±35mv, which indicated the CR extract acts as a mixed type of inhibitor by showing its inhibitory action on both hydrogen evolution and metal dissolution.

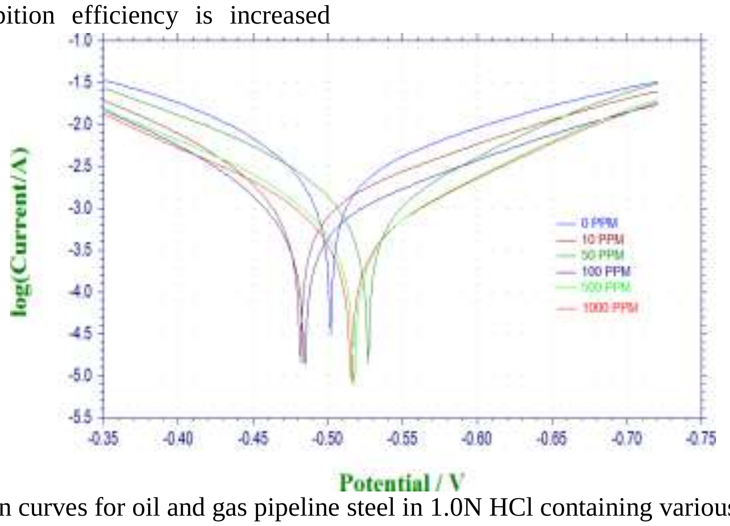


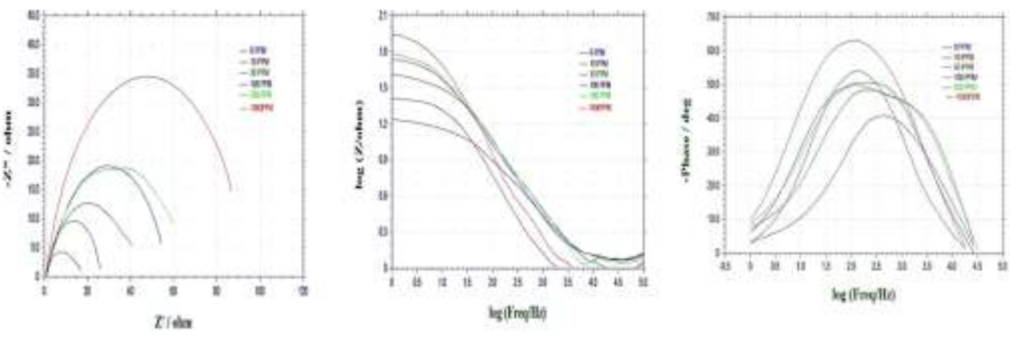
Fig-11.Polarisation curves for oil and gas pipeline steel in 1.0N HCl containing variousconcentration of CR extract

Electrochemical Impedance Studies (EIS)

Fig -12.shows that typical set of complex planes plot of oil and gas pipeline steel in 1.0N Hydrochloric acid in the absence and presence of various concentration of CR inhibitor at room temperature. It was obvious that the addition of inhibitor results in an increase of the diameter of the semicircle capacitive loop as shown in fig -12 (a), bode impedance plot fig-12(b) and the maximum phase angle fig -12 (c). Careful inspection of this data revealed that the value of charge transfer resistance (R_{ct}) increased from 16.54 to 91.63 Ωcm^2 of oil and gas pipeline steel in acid with increase of inhibitor concentration. The inhibition efficiency is increased from 34.13to 81.95% with increase of inhibitor concentration. It ensures that the formation of protective film on the metal surface. The double layer capacitance (C_{dl}) is decreased as the increase of inhibitor concentration may be due to the adsorption of the active compounds onto the metal surface leading to a film formation. It can be noticed that the charge transfer process may be controlling the dissolution of the metal.

Table –6. Parameters derived from electrochemical measurements of oil and gaspipeline steel in 1.0N HCl containing various concentration of CR extract

Con.of CR in ppm	Polarization measurements					Impedance measurements		
	-E_{corr} mV/decade	b_a (mV/decade	b_c (mV/decade	I_{corr} µA/cm^2.	%I.E	R_{ct} (Ωcm^2)	C_{dl} 10^{-4} Fcm^2	%I.E
0	502.1	134.92	163.99	3097	-	16.54	11.92	-
10	482.2	113.95	152.49	1323	57.28	25.11	5.005	34.13
50	527.0	124.58	113.29	1173	62.12	42.24	1.7917	60.84
100	485.0	107.38	147.38	804.6	74.02	55.35	1.0416	70.12
500	518.8	119.57	112.88	583.2	81.17	62.76	0.7929	73.65
1000	516.0	124.97	114.93	557.2	82.01	91.63	0.3848	81.95



(a) (b) (c)

Fig – 12. Electrochemical impedance plots (a-c) a. Nyquist b. Bode plot c. Phase anglefor oil and gas pipeline in 1.0N HCl containing various concentration of CR extract

Characterization of Corrosion Products

FTIR Spectra Analysis

FTIR spectra of this CR extract was obtained to verify the functional groups present in its chemical components in order to know the interaction mechanism between these components and Pipeline steel surface. In Fig. 13-a, the broad absorption peak centered at 3303 cm1 is ascribed to the O-H stretching vibration. The observed band at 1596 cm1 and 1396 cm1 are due to the carbonyl group. The transmission band at 1097 cm1 was observed due to C=O and C-O vibration. The appearance of CO stretching bands and OH is stretching suggested the successful functionalization of Extract with CR. The FTIR spectrum of the inhibitor film formed on the metal surface immersed in 1 M HCl solution containing CR extract on metal surface is shown in Fig. 13-b. A shift in the −OH group stretching from 3481 to 3234 cm^{-1} can be observed. The weak bands assigned for -CH asymmetric and symmetric stretching are shifted to 1743 and 1532 cm^{-1}, respectively. In addition, both -CO and C-C stretching vibrations were shifted to 1632 and 1487 cm^{-1}, respectively. These shifts indicate the adsorption of the extract into the active sites on the Pipeline steel surface

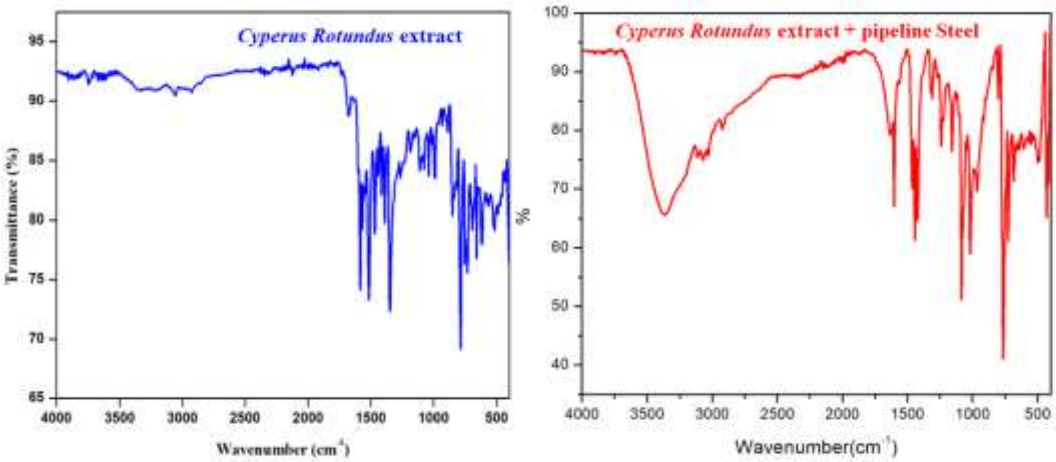


Fig-13. FTIR spectrum of CR extract and after contact with Pipeline steel Surface

EDX

Figs: 14(a-b) represents that the EDX spectra of the corrosion products on metal surface in the presence and absence of CR extract in 1.0N Hydrochloric acid. The elemental spectrum in 14b clearly indicates the presence of the hetero atoms like Oxygen and micro alloying, when compared to 14a where only major metal alloys are visible. These hetero atom peaks clearly reveals the attachment of inhibitor molecules on the metal surface

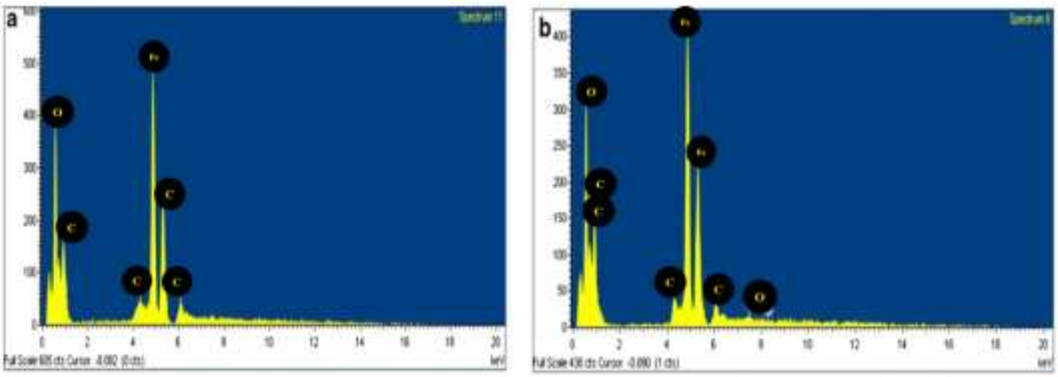


Fig-13. (a)EDX spectrum of the corrosion product on Pipeline steel surface in 1.0N HCl and (b) EDX spectrum of the corrosion product on Pipeline steel in the presence of CR extract in 1.0N HCl

SEM

Figure 15 presents image of the corrosion surface morphology of pipeline steel immersed in (a) Polished metal and (b) Metal in 1.0NHCl c) Metal in CR extracts. The acid aggressively corroded the sample in Figure 15(b) causing a rougher surface. The contrast was noticed in the sample in Figure 15(a) with smoother surface. The sample in Figure 15(c) surface is attributed to the ability of CR extract to form an adsorbed film on the surface of pipeline steel which was absent in Figure 14(b) sample. This is in agreement with the impedance results.

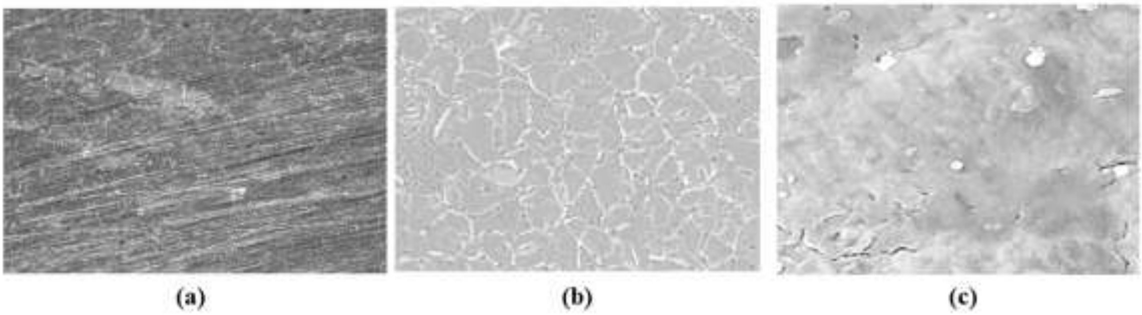


Fig-14. (a-c) SEM image of the Polished Pipeline Steel surface, Before and After immersed in 1.0N Hydrochloric acid with CR extract respectively

5. Conclusion

Cyperus Rotundushas shown good inhibition performance for oil and gas pipeline steel in 1.0N HCl environment. The inhibition efficiencyis increased with the increase of inhibitor concentration. The inhibition efficiency decreased with the rise in temperature to 88.40% for 303K respectively. It follows Physical adsorption mechanism. The value of activation energy (E_a), enthalpy of adsorption (ΔH_{ads}) and free energy changes (ΔG_{ads}) indicates that the adsorption of inhibitor on metal surface follows physisorption, exothermic and spontaneous process respectively.The close unity of R^2 values suggest that the inhibitor is found to obey Langmuir adsorption isotherms. The inhibition efficiency was found to be 81.95% by impedance studies and 82.01% of polarization studies. The study also showed that CR functioned as a mixed-type corrosion inhibitor in the acid environments studied and therefore presents it as a long –term inhibitor for the corrosion of oil and gas pipelinesteel.

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