

***Cocos nucifera* Shell as a Potential Inhibitor for Mild Steel Corrosion in Acidic Medium**

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The effect of coconut (*Cocos nucifera*) shell extract obtained by destructive distillation, on the corrosion of mild steel in H₂SO₄ and HCl in the temperature range of 30-60° C was studied by weight loss method and electrochemical measurements. Results obtained reveal that the extract reduces the corrosion rate. The inhibition efficiency increases with an increase in the concentration of the extract and appreciable increase with increase in temperature. Experimental results were fitted into various adsorption isotherms. Activation energy and thermodynamic parameters were evaluated from temperature studies. Electrochemical measurements reveal that the extract behaves as a mixed type inhibitor. A suitable mechanism is also proposed.

Key Words: Corrosion, Mild steel, Coconut shell, GC-MS, Activation energy, Adsorption isotherm, Thermodynamic parameters.

INTRODUCTION

Metals and their alloys are exposed to the action of acids in industries¹. Processes in which acids play important part are acid pickling, industrial acid cleaning, cleaning of oil refinery equipment, oil well acidizing and acid descaling^{1,2}. The exposures can be most severe but in many cases, corrosion inhibitors are widely used in industry to prevent or to reduce the corrosion rates of metallic materials in the acid media^{2,3}.

Because of the toxic nature and high cost of some chemicals currently in use, it is necessary to develop environmentally acceptable and less expensive inhibitors. Natural products can be considered as a good source for this purpose⁴. Several acid extracts of seeds, leaves and bark from the *Ficus virens* plant⁵, *Datura metel*⁶, *Allium sativum*⁷, *Olive*⁸, *Zenthoxylum alatum*⁹, juice extracts of *Magnifera indica* (mango)¹⁰, acidic extracts of *Ficus benghalensis*¹¹ and sprouted seeds of *Phaseolus aureus*¹² to mention a few. It was found that all the plant materials act as good corrosion inhibitor in acidic media. The corrosion inhibition of these plant materials have been attributed to the presence of active principles present in them. These active principles form protective films on metal surfaces by coordinating with metal ions through O, S or N atoms of the functional groups present in the active principles¹³.

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Coconut shell occurs naturally, is relatively cheap and is made up of hardened cellulose with chemical composition very similar to wood. In the present study the fruit shell of *Cocos nucifera* is used because of its easy availability, low cost nature and the presence of non-toxic chemicals.

The extract obtained from destructive distillation of coconut shell has been used as inhibitor on mild steel in 0.5 M H₂SO₄ and 1 M HCl medium. The aqueous distillation of *Cocos nucifera* shell extract obtained was characterized by GC-MS techniques for its detailed chemical composition.

EXPERIMENTAL

Preparation of shell extract: The *Cocos nucifera* fruit was collected from the tree and shell was removed, cleaned, chopped into small pieces, air dried and stored at room temperature prior to use. 500 g of air dried shell pieces was transferred to a 2000 mL round bottomed flask, directly heated on a heating mantle with water condenser at about 80 °C in the absence of air and without a carrier solvent (destructive distillation). The products obtained are (i) A gaseous mixture or wood gas. (ii) 150 mL of aqueous distillate obtained which was used as inhibitor. (iii) 50 mL of a thick black liquid or wood tar which was separated from the aqueous distillate using a separating funnel. (iv) 300 g of a solid residue or wood charcoal was left in the round bottom flask. It is popularly used as a domestic fuel.

GC-MS analysis: In order to identify the individual compounds in the aqueous distillate of *Cocos nucifera* shell extract gas chromatograph-mass spectrometry (GC-MS) technique was used¹⁴. The identification of the isolated volatile compounds was achieved by comparing obtained mass spectra of unknown peaks with those stored in the National Institute of Standards and Technology (NIST) and Wiley mass spectral electronic libraries. Identifications were confirmed by comparison with authentic substances used as references and by use of linear retention indices (LRI). Relative area values (as a percentage of total volatile composition) were directly obtained from total ion current (TIC). All analyses were carried out in duplicate. The chromatogram of aqueous distillate is shown in Fig. 1. The chemical compounds identified in the *Cocos nucifera* shell extract are phenol, 4-methyl-2-hydroxycyclopent-2-en-1-one, 2-methoxy phenol, 2-methoxy-4-methyl phenol, *p*-ethyl guaiacol, 1-(5-hydroxymethyl-2-furanyl)-1-ethanone, glycnamide hydrochloride, N-methyl-N-nitroso ethanamine, 1,2,3-trimethoxy-5-methyl benzene, 1-(4-hydroxy-3-methoxyphenyl)-2-propanone, 2-methoxy-4-(1'-methoxy propyl)phenol and 2-formyl-5-methyl pyrrole-1-acetic acid.

Mild steel specimen with the following chemical composition in wt % C, 0.11; Si, 0.061; Mn, 0.197; S, 0.023; P, 0.025; Ni, 0.010; Mo, 0.013; Cr, 0.037; Fe, 99.524; was used. Mild steel specimens were cut into 5 cm × 1 cm strips with 2 mm thickness, mechanically polished, their edges were abraded with fine grade of emery papers, degreased with acetone, washed in double distilled water, dried, stored in a dessicator and used for the entire immersion studies. 0.5 M H₂SO₄ and 1 M HCl solutions were prepared from analytical grade reagents (H₂SO₄ and HCl) and de-ionized water.

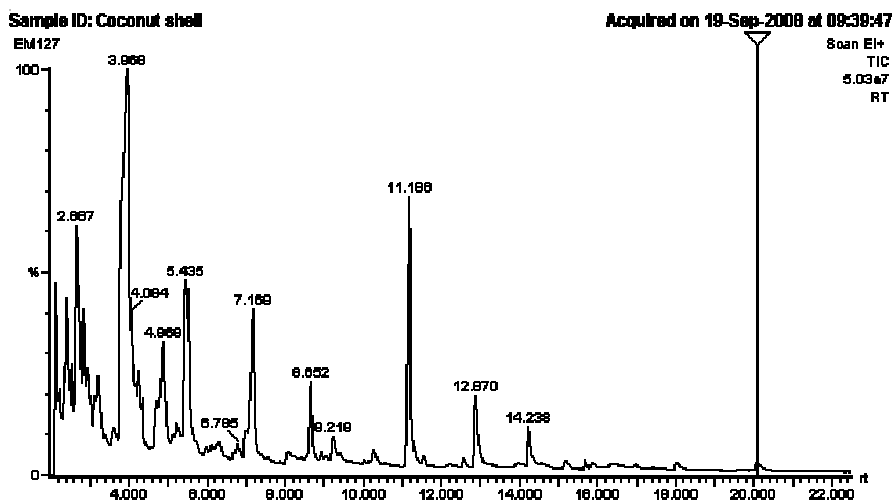


Fig. 1. Total ion chromatogram resulting from the aqueous distillate of coconut shell extract obtained by destructive distillation

Weight loss method: The weight loss measurements were carried out with mild steel specimens. The experiments were conducted at room temperature as well as higher temperatures. The concentration of the inhibitor was varied from 0.5 to 3.0 % v/v, the time of immersion was from 0.5 to 24.0 h at room temperature, temperature variation was from 303 to 343 K with a 10° difference.

Electrochemical measurements: For the electrochemical measurements, a conventional electrolytic cell was used with three electrode systems with platinum auxiliary electrode, saturated calomel as reference electrode and polished mild steel specimen as working electrode. All of the reported potential values were measured *versus* saturated calomel reference electrode. The working electrode was lacquered so as to expose 1 cm² area to contact with the solution. Electrochemical measurements were carried out using SOLARTRON electrochemical measurement unit (1284 B model) with a software package of Z plot and corware 2. The system includes a potentiostat, personal computer and frequency response analyzer (FRA). For potentiodynamic polarization studies the experiments were carried out over a potential range of -200 to +200 mV with respect to open circuit potential and its current response was measured at a scan rate of 1 mV/s.

Impedance measurements were carried out at corrosion potential. The AC amplitude of 10 mV was applied and the frequency was varied from 10 KHz to 10 MHz. The real and imaginary parts of the impedance were plotted in Nyquist plots. From the Nyquist and Bode plot, the charge transfer resistance and double layer capacitance values were calculated. Values of corrosion currents were obtained by Tafel extrapolation method, plot of η vs. log current were made and an extrapolation of linear portion to the corrosion potential gave the corrosion current and the slopes of the linear portion of the anodic and cathodic curves gave b_a and b_c , respectively.

RESULTS AND DISCUSSION

Weight loss method

Effect of concentration and period of immersion: Data on inhibition efficiency of shell extract of coconut in 0.5 M H₂SO₄ and 1 M HCl during immersion of mild steel with the current inhibitor are presented in the Table-1. Inspection of these data reveal that the inhibition efficiency increases with increasing the concentration of shell extract. Maximum inhibition efficiency in 0.5 M H₂SO₄ and 1 M HCl were found to be 98.57 and 98.06 % with 3 % v/v concentration of the inhibitor, respectively. Increase in inhibition efficiency with concentration of the extract may be due to the increase in the surface coverage of the adsorbed molecules on mild steel surface. In 0.5 M H₂SO₄ and 1 M HCl bath, as the immersion time increased from 0.5 to 24.0 h, the inhibition efficiency also increased from 92.68 % (0.5 h) to 98.57 % (24 h) and 89.13 % (0.5 h) to 98.06 % (24 h) with 3 % v/v concentration, respectively. The increase in inhibition efficiency with an increase in immersion time indicates the stability and persistence of the inhibitor layer on the mild steel surface¹⁵.

TABLE-1
INHIBITION EFFICIENCY AS A FUNCTION OF TIME OF IMMERSION
AND CONCENTRATION OF COCONUT SHELL EXTRACT

Acid medium	Inhibitor conc. (% v/v)	Inhibition efficiency (%)					
		0.5 h	1 h	3 h	6 h	12 h	24 h
0.5 M H ₂ SO ₄	0.5	77.98	90.13	90.52	92.30	89.37	91.9
	1.0	86.16	92.73	94.14	94.77	95.10	95.74
	1.5	88.72	93.95	96.17	96.27	96.94	97.08
	2.0	91.13	95.31	97.00	97.28	97.52	98.04
	2.5	91.66	95.35	97.10	97.88	97.85	98.42
	3.0	92.68	96.72	97.70	98.09	98.54	98.57
1 M HCl	0.5	71.39	76.35	89.07	91.01	90.50	93.25
	1.0	77.72	81.74	90.75	93.90	94.17	95.88
	1.5	82.58	87.64	93.24	95.21	95.67	96.58
	2.0	87.54	89.82	94.60	96.55	96.24	97.27
	2.5	88.60	92.00	95.02	97.17	96.70	97.73
	3.0	89.13	92.28	96.68	97.46	96.96	98.06

Effect of temperature: The effect of temperature on inhibition reaction is highly complex, because many changes may occur on the metal surface such as rapid etching, rupture, desorption of inhibitor and the decomposition and/or rearrangement of the inhibitor. The effect of temperature on the rate of corrosion process was studied in 0.5 M H₂SO₄ and 1 M HCl acid medium without and with various concentrations of coconut shell extract. By analyzing the results furnished in the Table-2, reveals that the extract works out very well at all temperatures. Maximum inhibition efficiency was found to be 96 % at 313 K and 323 K at 3 % v/v of the extract in H₂SO₄ bath. This investigation reveals that the extract is temperature resistant and there is a strong interaction between the adsorbed molecules and mild steel surface.

TABLE-2
EFFECT OF TEMPERATURE ON THE CORROSION OF MILD STEEL IN
0.5 M H₂SO₄ AND 1 M HCl IN THE PRESENCE OF VARIOUS
CONCENTRATION OF COCONUT SHELL EXTRACT

Acid medium	Inhibitor conc. (% v/v)	Inhibition efficiency (%)				
		303 K	313 K	323 K	333 K	343 K
0.5 M H ₂ SO ₄	0.5	77.98	85.21	83.04	81.36	77.95
	1.0	86.16	90.81	89.07	86.96	86.43
	1.5	88.72	92.45	92.73	91.35	88.53
	2.0	91.13	94.75	94.60	92.74	91.31
	2.5	91.66	95.97	95.86	93.99	92.99
	3.0	92.68	96.50	95.89	94.66	93.20
1 M HCl	0.5	71.39	73.93	72.04	60.82	54.27
	1.0	77.72	80.91	80.10	76.10	68.44
	1.5	82.58	85.07	85.67	79.57	72.65
	2.0	87.54	88.28	86.42	84.16	76.84
	2.5	89.13	89.71	89.64	86.69	81.90
	3.0	88.60	90.89	90.43	88.83	84.01

In 1 M HCl bath, the extract functions very well upto 333 K, furnishing around 90 % of inhibition efficiency at 3 % v/v concentration. There is slight decrease in inhibition efficiency at 343 K. This may be explained from the findings of previous researchers. Putilova¹⁶ explained the decrease in the inhibition efficiency of the inhibitor with increase in temperature might be due to adsorption and desorption of inhibitor. Adsorption and desorption of inhibitor molecules continuously occurs at the metal surface and an equilibrium exists between these two processes at a particular temperature. With the increase of temperature the equilibrium between adsorption and desorption processes is shifted leading to a higher desorption rate than adsorption until equilibrium is again established at a different value of equilibrium constant¹⁷.

Adsorption isotherm: It is generally accepted that organic molecules inhibit corrosion by adsorption at metal/solution interface and that the degree of adsorption depends on structure of the molecules, the chemical composition of the solution, the nature of the metal surface, the temperature and the electrochemical potential at the metal/solution interface¹⁸. Adsorption provides information about the interaction among the adsorption molecules themselves as well as their interaction with the electrode surface. A useful method that assists in the understanding of the mechanism of organo electrochemical reactions in the adsorption process is the adsorption isotherm¹⁹. An adsorption isotherm gives the relation between the coverage of an interface with the adsorbed species (the amount adsorbed and the concentration of species and the solution). Interpretation of the performance of the adsorbent type can be enhanced by fitting the data in one of the known isotherms. The adsorbed corrosion data in the presence of inhibitors namely: (a) The trend in variation of corrosion rate with inhibitor concentration. (b) Linear dependence of weight loss

on immersion time. (c) The increase in corrosion inhibition with temperature suggests that the corrosion inhibition of mild steel occurs through surface adsorption.

The degree of surface coverage ' θ ' for different concentration of the inhibitor has been evaluated from weight loss values. Data were tested graphically by fitting to various isotherms. A plot of $\log(\theta/1-\theta)$ vs. $\log C$ and θ vs. $\log C$ were linear for both the acids, suggesting that the adsorption of the compounds on the mild steel surface follows Langmuir and Temkin adsorption isotherms. This indicates that the behaviour of adsorption is affected by the heterogeneity of the electrode surface.

Activation energy (E_a): The values of corrosion rate at different temperature make it possible to calculate the activation energy (E_a) using Arrhenius equation. The estimated values of E_a for mild steel corrosion in the presence of coconut shell extract in 0.5 M H_2SO_4 and 1 M HCl are listed in the Table-3. Abiola *et al.*²⁰, Ebenso *et al.*²¹ and Ebenso²² reported that the values of $E_a > 80$ kJ/mol indicate chemical adsorption whereas $E_a < 80$ kJ/mol infer physical adsorption. The E_a values indicate that the process is activation controlled. In the present study, the activation energy value of 48.28 kJ/mol and 56.22 kJ/mol for both the systems support the fact that the inhibitors are physically adsorbed on mild steel surface. The estimated E_a in the presence of inhibitor infer that the interaction between the metal surface and the inhibitor was found to be strong enough to reduce corrosion.

TABLE-3
VARIATION OF THERMODYNAMICS AND KINETIC PARAMETERS
WITH CONCENTRATION AND TEMPERATURE (COCONUT
SHELL EXTRACT IN 0.5 M H_2SO_4 AND 1 M HCl)

Acid medium	Inhibitor conc. (% v/v)	E_a (kJ/mol)	- ΔG (kJ/mol)					ΔS (kJ/mol)	ΔH (kJ/mol)
			301 K	313 K	323 K	333 K	343 K		
0.5 M H_2SO_4	Blank	46.32	-	-	-	-	-	-	-
	0.5	47.61	15.02	16.78	16.88	17.09	17.00	+0.008	14.14
	1.0	48.28	14.70	16.38	16.39	16.34	16.70	+0.009	13.48
	1.5	46.96	14.26	15.89	16.50	16.49	16.09	+0.006	14.25
	2.0	47.91	14.21	16.15	16.58	16.22	16.15	-0.003	17.41
	2.5	45.8	13.82	16.29	16.73	16.17	16.18	-0.008	19.26
	3.0	47.74	13.72	16.20	16.27	16.01	15.76	-0.015	21.24
1 M HCl	Blank	43.78	-	-	-	-	-	-	-
	0.5	54.55	14.14	14.94	15.16	14.23	13.89	-0.040	27.97
	1.0	50.82	13.24	14.18	14.50	14.30	13.63	-0.018	20.24
	1.5	53.31	12.99	13.90	14.47	13.73	13.05	-0.032	24.53
	2.0	56.22	13.26	13.87	13.87	13.79	12.87	-0.030	23.76
	2.5	53.92	12.95	13.67	14.09	13.74	13.11	-0.020	20.31
	3.0	50.42	12.62	13.55	13.84	13.79	13.02	-0.016	18.92

Thermodynamic parameters

Free energy of adsorption, change in entropy and enthalpy: The values of free energy of adsorption can be calculated by using the standard equation:

$$\log C = \log \left(\frac{\theta}{1-\theta} \right) - \log B \quad (1)$$

where C is the inhibitor concentration and log B is $-1.74 - (-\Delta G/2.303 RT)$. Based on Table-3, it was shown that the free energy of adsorption ΔG is negative, which indicated the spontaneity of adsorption process on mild steel surface. The more the negative nature of ΔG value the more spontaneous would be its adsorption process on metal surface, thereby enhancing the corrosion inhibition activity. The value of ΔG upto -20 kJ/mol is consistent with the physical adsorption (Physisorption), whereas the value of ΔG , above -40 kJ/mol, is consistent with the chemical adsorption (chemisorption)^{23,24}. Therefore, coconut shell extract have the potent to interact with mild steel through physisorption because their ΔG values are within -20 kJ/mol for both acids.

The enthalpy of adsorption ΔH and entropy of adsorption ΔS are related to free energy of adsorption by Gibbs Helmholtz equation. A plot of $-\Delta G$ vs. T is a straight line. The values of ΔH are taken from intercept and that of ΔS from slopes. In the present case, the positive sign of ΔH indicates that the adsorption of inhibitor molecules is an endothermic process for the both acids²⁵. The positive and negative values of ΔS for 0.5 M H_2SO_4 indicate that the activation complex represents both association and dissociation step and negative values of ΔS for 1 M HCl indicates that the activated complex represents association rather than dissociation step²⁶.

Electrochemical measurements

Potentiodynamic studies: Potentiodynamic studies were conducted by using Solartron 1284 B model in the absence and presence of various concentrations of coconut shell extract. Electrochemical parameters such as current density (I_{corr}), Tafel slope constants (b_a and b_c), linear polarization resistance (R_p) are recorded in the Tables 4 and 5 for both the acids. From the Table-4 it is inferred that the values of current density I_{corr} decreases with increasing concentration of the inhibitor. No significant change was observed in E_{corr} values. The values of Tafel slopes (b_a and b_c), change with increase in concentration of the coconut shell. Potentiodynamic curve of mild steel in 0.5 M H_2SO_4 and 1 M HCl in the presence of coconut shell extract are recorded in Fig. 2. The values of R_p increases with increased in concentration of the coconut shell extract. From the above experimental results, the following conclusion can be drawn: (a) Values of E_{corr} indicate that the coconut shell extract behaves as mixed type inhibitor in both the acids. (b) Values of b_a and b_c indicate that the extract is acting as mixed type by controlling the cathodic hydrogen evolution as well as anodic metallic dissolution in both the acids. (c) Decrease in I_{corr} values and increase in R_p values in both the acids with increase in concentration of coconut shell extract revealed that the inhibition process takes place by adsorption of the extract on mild steel surface. Successive coverage of mild steel surface and growth of layer would have provided an effective corrosion. Inhibition efficiency calculated using I_{corr} and R_p indicate the inhibition efficiency increased with increasing concentration of the extract and maximum inhibition efficiency was found to be 97.45% in 0.5 M H_2SO_4 and 91.70% in 1 M HCl.

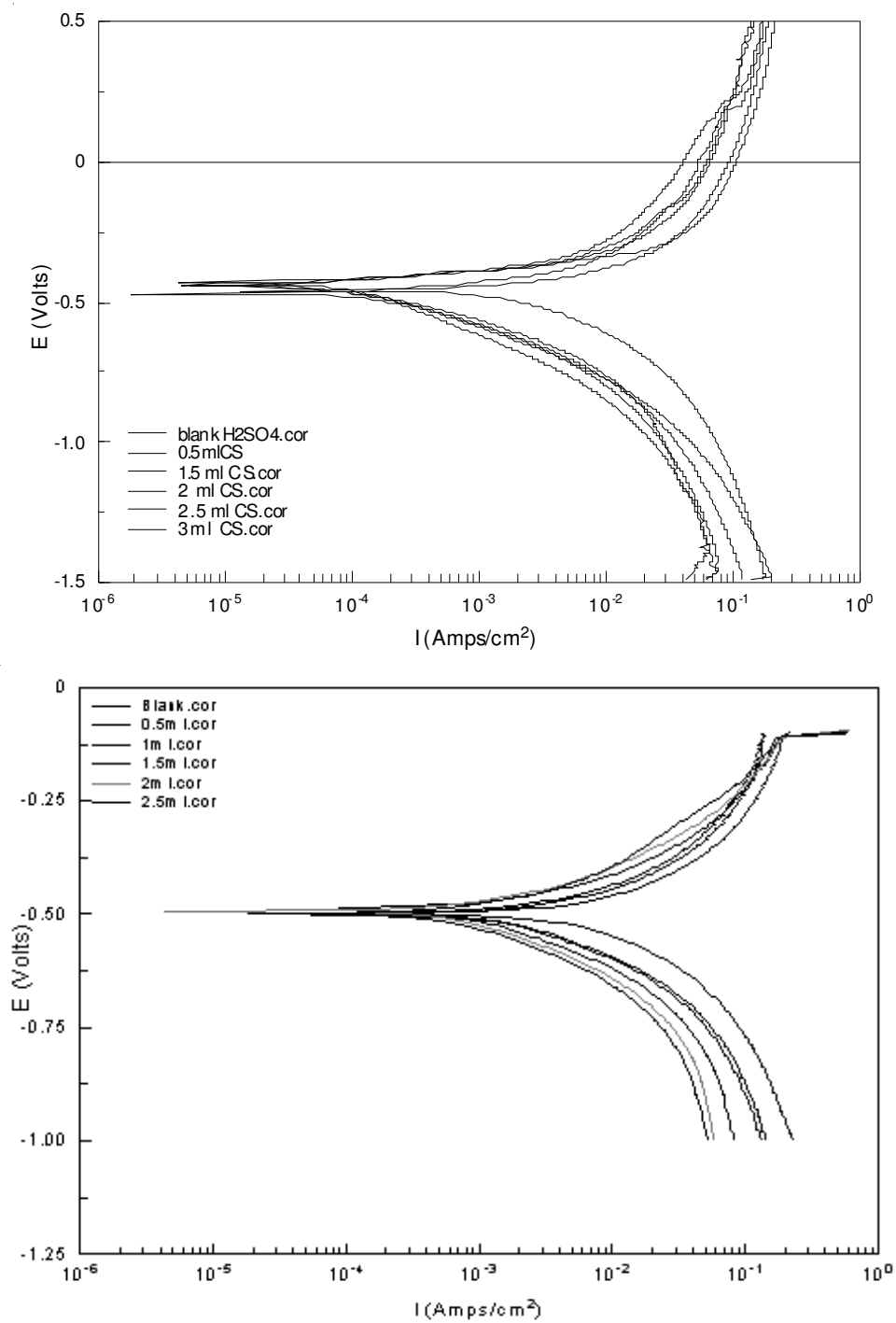


Fig. 2. Polarization curve for mild steel in 0.5 M H₂SO₄ and 1 M HCl with coconut shell extract

TABLE-4
TAFEL POLARIZATION MEASUREMENTS OF MILD STEEL IN
COCONUT SHELL EXTRACT IN 0.5 M H₂SO₄ AND 1 M HCl

Acid medium	Inhibitor conc. (% v/v)	I _{corr} (mA cm ⁻²)	-E _{corr} (V/dec)	b _a (mV/dec)	b _c (mV/dec)	IE (%)
0.5 M H ₂ SO ₄	Blank	2202	443	127.03	234.02	-
	0.5	301	440	84.29	348.07	86.33
	1.0	114	449	80.47	155.32	94.82
	1.5	95	442	69.3	139.53	95.68
	2.0	88	441	57.87	107.19	96.00
	2.5	73	443	65.52	121.86	96.68
	3.0	56	432	51.92	136	97.45
1 M HCl	Blank	8921	498	214.31	170.1	-
	0.5	3329	499	191.76	121.8	62.68
	1.0	2723	499	176.32	120.76	69.47
	1.5	1341	489	158.94	100.53	84.96
	2.0	806	491	134.22	99.55	90.96
	2.5	740	499	131.6	93.81	91.70

TABLE-5
IMPEDANCE MEASUREMENTS OF MILD STEEL IN COCONUT SHELL
EXTRACT IN 0.5 M H₂SO₄ AND 1 M HCl MEDIUM

Acid medium	Inhibitor conc. (% v/v)	R _{ct} (ohm cm ²)	IE (%)	C _{dl} × 10 ⁻⁴ Fcm ²	θ	R _p (ohm cm ²)	IE (%)
0.5 M H ₂ SO ₄	Blank	10.32	-	2.714	-	15.12	-
	0.5	58.44	82.33	2.676	0.014	103.48	85.38
	1.0	85.42	87.91	1.985	0.268	180.52	91.62
	1.5	172.37	94.00	1.958	0.278	217.65	93.05
	2.0	175.33	94.11	1.695	0.375	237.79	93.64
	2.5	184.47	94.40	1.221	0.550	240.74	93.71
	3.0	190.47	94.56	0.904	0.666	245.76	93.84
1 M HCl	Blank	17.98	-	2.415	-	4.720	-
	0.5	81.01	77.80	1.656	0.314	9.278	49.12
	1.0	99.35	81.90	1.060	0.561	10.977	57.00
	1.5	102.25	82.41	0.910	0.623	17.307	74.21
	2.0	186.44	90.35	0.687	0.715	23.556	76.96
	2.5	244.05	92.63	0.603	0.750	26.006	81.85

Electrochemical impedance spectroscopy: Electrochemical impedance spectroscopy is a sophisticated technique and it is an important tool to study the mechanism of inhibition process. Electrochemical impedance spectroscopic measurements are done in the presence and absence of coconut shell extract and the values of charge transfer resistance R_{ct} and double layer capacitance C_{dl} are tabulated in the Table-5. The Nyquist plots for both the acid system are represented in the Fig. 3. The depressed semicircles were obtained in the presence of the inhibitor. The lower frequencies in the Bode plots indicate that no diffusion control occurs under this condition for

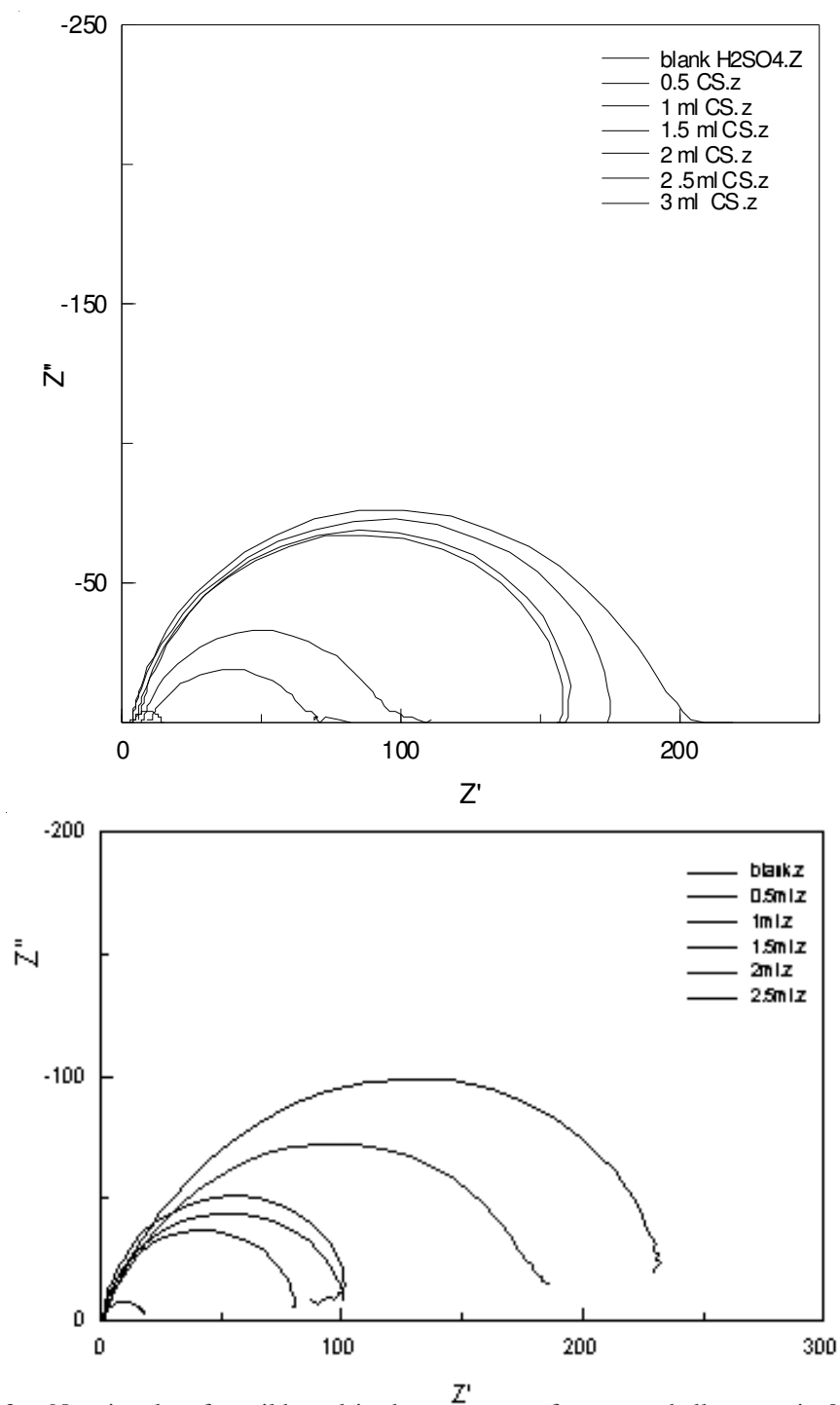


Fig. 3. Nyquist plots for mild steel in the presences of coconut shell extract in 0.5 M H₂SO₄ and 1 M HCl

both the acids. From the Table-5, it is clear that R_{ct} increased with increasing concentration of inhibitor. The depressed semicircles were obtained for the present system indicate that the adsorption of inhibitor molecule is charge transfer controlled. Maximum IE using R_{ct} values was found to be 93.84 % for 0.5 M H_2SO_4 and 81.85 % for 1 M HCl at 3 % v/v and 2.5 % v/v concentration. Values of C_{dl} decrease with increase in concentration of the extract, which indicate that the inhibition takes place through adsorption process.

Mechanism of corrosion inhibition: The coconut shell extract inhibits the corrosion by controlling both the anodic and cathodic reactions. In acidic solutions the chemical compounds present in the extract exist as protonated species. These protonated species adsorb on the cathodic sites of the mild steel and decrease the evolution of hydrogen. The adsorption on anodic sites occurs through the lone pair of electrons of nitrogen and oxygen atoms²⁷.

The chemical compounds present in the extract possess the hetero oxygen atom in the structure makes possible its adsorption by coordinate type linkage through the transfer of lone pairs of electron of oxygen atoms to the vacant *d*-orbitals of metal surface atoms, giving a stable chelate five or six membered ring with ferrous ions. The simultaneous adsorption of oxygen atoms forces the coconut shell extract molecule to be horizontally oriented at the metal surface, which led to increase the surface coverage and consequently protect efficiency even in the case of low inhibitor concentration.

Conclusion

(a) The efficiency of the coconut shell extract as inhibitor of corrosion of mild steel in 0.5 M H_2SO_4 and 1 M HCl increased with an increase in their concentration and decreased with an increase in temperature. Maximum IE was found to be 98 % in both acids. (b) Adsorption of the inhibitors in both acids were found to obey the Langmuir and Temkin adsorption isotherm, which represent the monolayer formation with heterogeneity on the mild steel surface. (c) The free Gibbs adsorption energy values, ΔG_{ads}° , is negative in both acids, which indicate the spontaneity of adsorption process on mild steel surface and also have the potential to interact with mild steel through physisorption. (d) From the potentiodynamic polarization curves it can be observed that the coconut shell extract acted primarily as mixed-type inhibitors in both acids. (e) Electrochemical impedance spectroscopy showed that the mild steel dissolution is controlled by charge-transfer mechanism in both acids. (f) The planarity of structure, the presence of electron donating groups, the steric hindrance and the electron density of molecular structure are some crucial factors of phenol and its derivatives present in coconut shell extract that acted as an excellent inhibitor in controlling corrosion of mild steel in both acid mediums and (g) Cost effective and eco-friendly inhibitor.

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