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Identification of Furfural in the Coconut Shell Extract and its Study as Green Corrosion Inhibitor in Acid Media

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ABSTRACT

The extraction from the agricultural waste like coconut shell using sulfuric acid and the identification of furfural in the extract treated as corrosion inhibitor. Extracted furfural was characterized by Fourier Transform Infra-Red (FTIR) spectroscopy and High Performance Liquid Chromatography (HPLC). The inhibition efficiency of extracted furfural was studied on the corrosion of mild steel in 0.5M H_2SO_4 with different concentrations by weight loss method and Tafel plot techniques. The inhibition efficiency increases with also increase in the extract concentration. The effect of temperature on corrosion behavior was also studied over the temperature range of 30–45°C. The results indicate that the inhibition efficiency increases with increase in concentration of inhibitor and decreases with rise in temperature. Electrochemical polarization (Tafel plot) data explores the mixed mode of inhibition. The extracted Furfural follow Langmuir adsorption isotherm. Adsorption mechanism involves both physisorption and chemisorptions. The surface study of inhibited mild steel was performed by scanning electron microscopy (SEM).

Introduction

Corrosion is defined as the destruction or deterioration of a material due to a chemical or an electrochemical reaction with its environment. Corrosion behaviour of a material is mainly determined by its structure and composition. Corrosion rate may increases in acid medium. But in many industries for the removal of undesired scale, acid solutions are used. Sulfuric acid is being used for some important fields of application like acid pickling, industrial cleaning, acid descaling, oil-well acidizing and petrochemical processes [1–3]. Therefore, the protection of mild steel from sulfuric acid solutions is most importance for the growing industries. Various methods like cathodic protection, anodic protection, coating method, lubrication, painting, electroplating and inhibitors are used to protect the mild steel corrosion. Among these methods for the protection of mild steel in acid media using inhibitors are the most effective and viable method. [4–9].

Inhibitors are the chemical compounds that react with an exposed metallic surface giving them certain level of protection. Inhibitors often adsorb themselves on the metallic surface, protecting the metallic surface by forming a thin film. The majority of the inhibitors are synthesized organic compounds which are more expensive and toxic in behavior. To control the use of synthetic organic compounds, an investigation of the non toxic, biodegradable, renewable and inexpensive materials has been carried out from plant products. Extracted plant products are incredibly rich sources of naturally synthesized chemical compounds that can be extracted by simple procedures with low cost [10].

Furfural is an organic compound (Fig.1) can be extracted by distilling the agricultural byproducts like Corncobs, oat hulls, cottonseed hulls, rice hulls, sawdust and bagasse with aqueous sulfuric acid [11,12]. Which is renewable chemical used in fuel dyes, coatings and adhesives. Coconut plant is the rich source of medicinal properties due to presence of biologically and chemically active components. The nut contains white meat and sweet water have anti-bacterial and antifungal, anti-viral anti-inflammatory [13, 14], antioxidant [15] activity. Water extracted coconut husk have antimicrobial activity [16] and coconut fruit wall are rich polyphenol content [17]. Literature review reveals that different parts of the coconut plants are used for biological study. But no much work has been focused on coconut shell. These factors motivated to determine the inhibitory action of coconut shell extract on mild steel. In the present study to identification of furfural from acid extracted coconut shell and its application as green corrosion inhibitor in 0.5M H_2SO_4 solution using gravimetric and electrochemical study has been carried out.

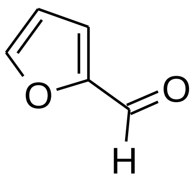

Figure 1. Molecular structure of Furfural.

Experimental

Mild Steel preparation

Mild steel coupon having the chemical composition (wt %): 0.16% C, 0.032% Mn, 0.18% Si, 0.026% S, 0.03% P and remaining Fe with the dimension of 2.6 cm x 2 cm x 0.025 cm were used for weight loss, electrochemical and SEM studies. Prior to each experiment, the mild steel coupons were well cleaned; the exposed coupon area was mechanically abraded with different grades of emery paper and washed

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with doubly distilled water and acetone, dried and stored in desiccators.

Preparation of Coconut Shell Extract

Dried waste coconut shells were collected and ground into fine powder in a mechanical grinder. 10 g of dried powder with 100 mL of 50% H_2SO_4 solution was irradiated in a micro-wave oven for about 20 minutes. Filtered the hot solution, collected filtrate containing aqueous and non-aqueous layers and was subjected to partitioning using chloroform as a solvent in a separating funnel. Furfural was isolated from the non-aqueous solution using rotary evaporator with temperature not exceeding 40^o C. Brown coloured oily liquid is obtained and refrigerated. The test solutions were prepared in 0.5 M H_2SO_4 solution and used for corrosion inhibition study.

Extracted Coconut Shell Characterization

FTIR Study

Fourier-transform infrared (FT-IR) spectra were recorded for the extracted furfural by Prestige-21 FT-IR spectrophotometer with the wave number range from 4000 to 400 cm^{-1}.

HPLC Study

Identification and purity of extracted furfural was performed by using SYS LC-138 instrument. Separations were carried out on 4.6 x 250 mm i.d. column packed with 5 µm C18 Kromstar^{TM}. The mobile phase was Methanol: Water (50:50 v/v) at 1 mL/min by injecting 20 µL of the sample or standard into the column with UV detection wavelength of 280 nm at room temperature.

The standard solution of Furfural (Sigma Aldrich) and extracted furfural were prepared by HPLC grade methanol.

Gravimetric Studies

Gravimetric studies were performed on precleaned and dried mild steel specimen, which were suspended in beaker containing 100 mL of test solutions over a period of 1hour with the help of glass hook. The weight loss was calculated by change in weight between the initial and final weights of the specimens in the absence and presence of different concentrations of extracted furfural at 303, 308, 313 and 318 K. The experiments were carried in triplicate and mean values are taken for further calculations.

From the weight loss measurements the rate of corrosion was calculated by the equation [18]

$$C_R = \frac{87.6 \times W}{Atd} \qquad (1)$$

Where C_R is the corrosion rate in mili miles penetration per year (mmpy), W is the weight loss of mild steel (mg), A is the area of specimen (cm^2), t is immersion time in hours, and d is the density of mild steel (7.85 g cm $^{-3}$).

The percentage inhibition efficiency η (%) was calculated by the formula

$$\eta\,(\%) = \frac{(W_1 - W_2)}{W_1} \times 100 \qquad (2)$$

Where, W_2 and W_1 are the weight loss of the mild steel in presence and absence of the coconut shell extract respectively.

Electrochemical Studies

The electrochemical studies were performed by using CHI6O4E Electrochemical Analyzer (CH instruments). Experiment were carried out in a conventional three electrode cell assembly with mild steel specimen used as working electrode, Platinum electrode as the auxiliary electrode, and a saturated calomel electrode as standard reference electrode. In the Tafel plot studies and electrochemical impedance spectroscopic measurements (EIS), mild steel was suspended in the test solution over a period of 40 min to reach steady-state open-circuit potential (OCP). In the Tafel plot studies, the applied electrode potential from +200mV (anodic) to -200mV (cathodic) with respect to open circuit potential (OCP) at 0.01V/s scan rate. The EIS measurements were performed by using an AC signal at OCP in the frequencies of 100,000 to 1 Hz with the amplitude of 0.01 V.

Scanning Electron Microscopy (SEM) Studies

For surface morphology experiments mild steel were immersed in the solution of 0.5 M H_2SO_4 in the absence and presence of 0.4 g/L furfural extract for 5 hours at room temperature. The SEM images of dried specimens were taken using CARL ZEISS FESEM instrument at high vacuum and the accelerating voltage of 5 kV.

Results and Discussion

Characterization of Extracted Furfural

FTIR Analysis

FTIR is an important technique to identify the functional group present in the molecule. The recorded FTIR spectrum (Fig. 2) of extracted furfural showed very strong absorption band at 1666 cm^{-1} indicates the presence of conjugated carbonyl (C=O) group, the absorption band observed at 1567 – 1463 cm^{-1} due to aromatic C=C group. The presence of aldehyde (CHO) group was confirmed by existence of two peaks at 3132 and 2853 cm^{-1}. The C-O stretching vibration was observed at 1158 – 1018 cm^{-1}. The presence of aromatic =CH bending at 927 and 881 cm^{-1} and aldehydic CH bending at 1392 cm^{-1}. These IR regions confirm the structure of furfural present in coconut shell extract.

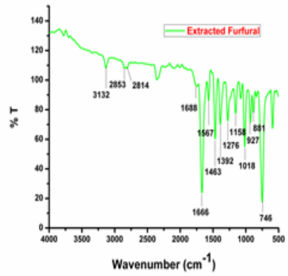
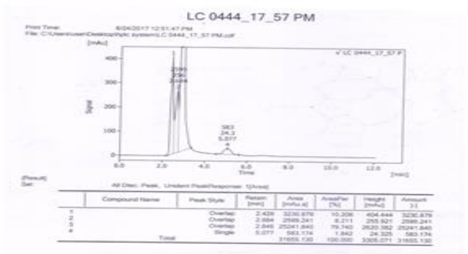


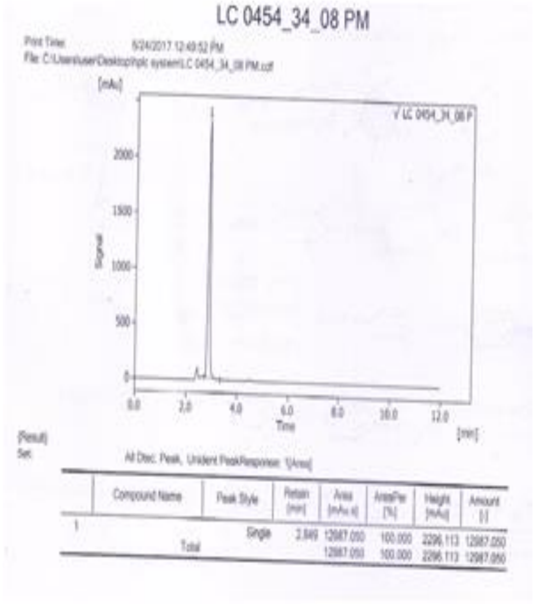
Figure 2. FTIR spectrum of extracted furfural.

HPLC Analysis

The presence of furfural in coconut shell extract was further assertained with the help of High performance Liquid Chromatography. The methanol, standard furfural and coconut shell extract were injected separately as blank, standard and sample respectively to the LC-138 instrument. The 20 µl of extract in methanol was injected and recorded the chromatogram in Fig. 3A; from the chromatogram the retention time at 2.8 min shows the major peak of the purity in area percentage 79.5 %. It clearly shows the presence of furfural which was confirmed by injecting the standard furfural in methanol eluted at 2.8 min RT (Fig. 3B). The retention time of standard furfural and sample were matching with one another thereby confirming the presence of furfural in coconut shell extract.



(a)



(b)

Figure 3. LC chromatogram (a) coconut shell extract and (b) standard furfural.

Gravimetric Results

The weight loss due to corrosion of mild steel in 0.5 M H_2SO_4 solution was determined by Gravimetric measurements. The investigation of the corrosion inhibition efficiency of coconut shell extract and the rate of corrosion on mild steel at different temperature like 303, 308, 313 and 318 K. Table 1 show the calculated values of corrosion rate, inhibition efficiency and surface coverage. From the table it is clear that the percentage inhibition efficiency increases with increase in concentration of coconut shell extract and inhibition efficiency decreases with increase in temperature [19] as shown in Fig 4. The rate of corrosion decreases with increasing extract concentration. Due to adsorption of inhibitor molecule on mild steel, the surface coverage of the metal increases and due to higher temperature desorption of inhibitor molecule on mild steel, the surface coverage of the metal decreases [20, 21].

Table 1. Effect of temperature on corrosion rate, Inhibition efficiency and Surface coverage

Temp K	Conc g/L	C_R mmpy	IE %	θ
303	Blank	170.065	-	-
	0.1	73.485	56.790	0.568
	0.2	64.037	62.346	0.623
	0.3	54.589	67.901	0.679
	0.4	47.240	72.222	0.722
308	Blank	213.107	-	-
	0.1	96.580	54.680	0.547
	0.2	86.083	59.606	0.596
	0.3	75.585	64.532	0.645
	0.4	66.137	68.966	0.690
313	Blank	280.293	-	-
	0.1	135.423	51.685	0.517
	0.2	122.825	56.180	0.562
	0.3	110.228	60.674	0.607
	0.4	95.531	65.918	0.659
318	Blank	309.687	-	-
	0.1	158.518	48.814	0.488
	0.2	144.871	53.220	0.532
	0.3	128.074	58.644	0.586
	0.4	112.327	63.729	0.637

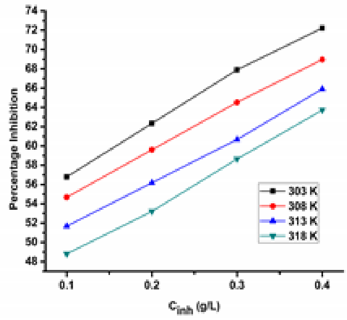


Figure 4. Effect of concentration of inhibitor on percentage inhibition at different temperature.

Temperature Effect

The rate of corrosion on mild steel is mainly influenced by change of temperature. As temperature increases corrosion rate also increases as shown in Fig. 5. In kinetic study activation energies were calculated by using Arrhenius equation [21],

$$\ln C_R = \ln A - \frac{E_a}{RT} \qquad (3)$$

Where C_R is the corrosion rate, E_a is the apparent activation energy, R is the universal gas constant, A is the Arrhenius pre-exponential factor and T is the temperature.

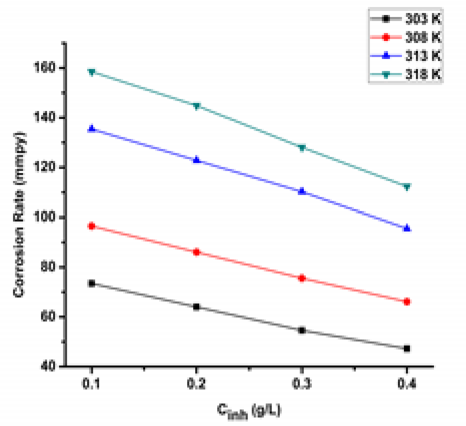


Figure 5. Temperature effect on corrosion rate.

Using an Arrhenius plot of log C_R against 1/T (Fig. 6) shows straight lines with slope $-E_a/R$ and intercept of ln A to calculate activation energy listed in Table 2. The E_a values are higher in presence of inhibitor than uninhibited solution and also Ea values increase from 0.1g/L to 0.4 g/L of extracted solution due to their adsorption onto the metal surface. It indicates the addition of coconut shell extract decreases metal dissolution in 0.5 M H_2SO_4 solution [22].

Kinetic parameters such as enthalpy and entropy of corrosion process were calculated by transition-state equation [23],

$$\ln \frac{C_R}{T} = \left[\ln \frac{R}{Nh} + \frac{\Delta S^*}{R}\right] - \frac{\Delta H^*}{RT} \qquad (4)$$

Where ΔS^* is the entropy of activation, ΔH^* is the enthalpy of activation, N is Avogadro's number, and h is Planck's constant.

Fig. 7 shows the plot of log(C_R/T) against 1/T. Straight lines were obtained with a slope of (ΔH^*/2.303R) and an intercept of [log(R/N$\hbar$)) + (ΔS^*/2.303R)]. The calculated ΔH^* and ΔS^* values are complied in Table 2. The positive or negative signs of ΔH^* indicate the reaction is endothermic or exothermic respectively. In the present study ΔH^* values are positive (Table 2) shows the dissolution process of mild steel is endothermic in nature. From Table 2, it has been noticed that ΔS^* values are negative and larger in presence of inhibitors. The results show the activated complex in the ratedetermining step represents an association rather than dissociation. This shows the decrease in disorder on moving from reactants to the activated complex [24-27].

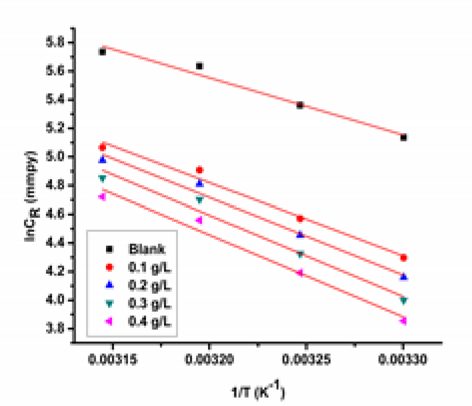


Figure 6. Arrhenius Plot.

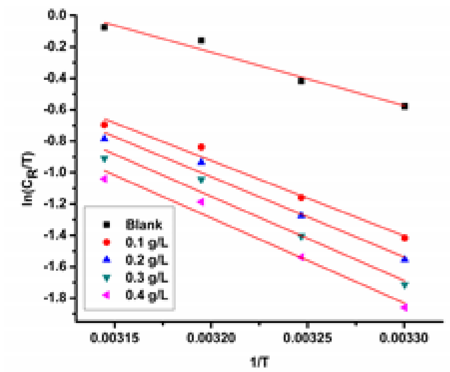


Figure 7. Transition Plot.

Table 2. Activation parameters.

Concentration g/L	E_a^* KJ/mol	ΔH* KJ/mol	ΔS* J/mol/K
Blank	33.26	28.32	-108.86
0.1	42.42	39.84	-77.73
0.2	44.99	42.41	-70.35
0.3	47.12	44.54	-64.61
0.4	47.60	45.02	-64.17

Adsorption Isotherm

The adsorption isotherm explains the mechanism of organo-electrochemical reaction and shows the interaction of inhibitor and metal surface. Mode of adsorption on mild steel surface was evaluated in the temperature range 303-318 K. From the experimental data several adsorption isotherms were verified but Langmuir adsorption isotherm showed to good agreement with the equation [28]

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (5)$$

Where *Θ* is the degree of surface coverage, *C* is the inhibitor concentration, and K_{ads} is the equilibrium constant of the adsorption process. The plot of C_{inh} v/s C_{inh}/Θ shown in Fig. 8 gives straight line with almost unit slope and from the intercept ($1/K_{ads}$) equilibrium constant was calculated. The equilibrium constant and the free energy of adsorption (ΔG^0_{ads}) are related by [29]

$$\Delta G^0_{ads} = -2.303RT\log(K_{ads}\ C_{H_2O}) \quad (6)$$

Where *T* is the absolute temperature, C_{H_2O} is the concentration of water in solution equals 1000 g/L and *R* is the gas constant. The adsorption enthalpy (ΔH^0_{ads}) was calculated by the slope obtained from the plot of $\Delta G^0_{ads}/T$ v/s 1000/T as shown in Fig. 9. From the Gibbs–Helmholtz equation [30] the entropy of adsorption (ΔS^0_{ads}) was calculated

$$\Delta S^0_{ads} = \frac{\Delta H^0_{ads} - \Delta G^0_{ads}}{T} \quad (7)$$

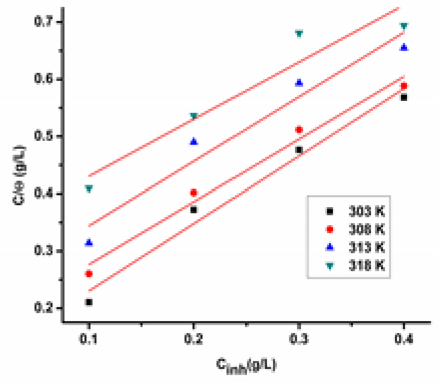


Figure 8. Langmuir adsorption isotherms.

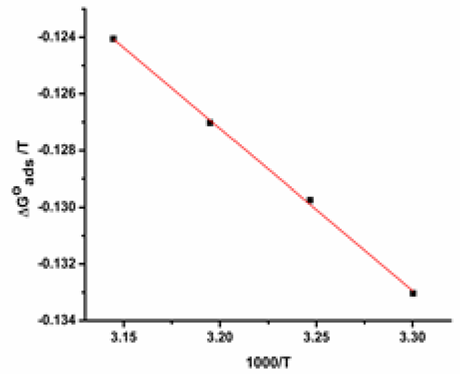


Figure 9. Plot of $\Delta G^0_{ads}/T$ v/s 1000/T.

Table 3. Thermodynamic parameter for adsorption on mild steel.

Temp. (K)	K_{ads} L/mol	ΔG^0_{ads} KJ/mol	ΔH^0_{ads} KJ/mol	ΔS^0_{ads} J/mol/K
303	8897.59	-40.31	-57.13	-55.51
308	5986.59	-39.96	-57.13	-55.75
313	4320.40	-39.76	-57.13	-55.50
318	3022.52	-39.45	-57.13	-55.60

Table 3 lists the calculated values of K_{ads}, ΔG^0_{ads}, ΔH^0_{ads} and ΔS^0_{ads}. The temperature effect on equilibrium constant of adsorption has been shown in Table 3. It shows adsorption on mild steel decreases as temperature of the solution increases. The temperature effect on adsorption explains thermal agitation of inhibitory molecules and replaceable of inhibitor molecule on the mild steel surface by the solvent molecules [31]. In the present study average ΔG^0_{ads} value is -39.87 KJ mol^{-1}, the negative value shows spontaneous adsorption on mild steel surface and ΔG^0_{ads} value more than -20 KJ mol^{-1} and less than -40 KJ mol^{-1} indicates the adsorption mechanism of coconut shell extract on mild steel in 0.5M H_2SO_4 solution was both physical and chemical adsorptions (physisorption and chemisorption). It means adsorption process between inhibitor molecule and metal surface involves both electrostatic interaction and electron transfer processes [32-37].

The adsorption processes are either exothermic or endothermic phenomena depend on negative or positive values of ΔH^0_{ads}. The exothermic process shows either physical or chemical adsorption or mixture of both but the chemisorption process is due to endothermic nature of enthalpy of adsorption [38]. It is clear from Table 3, that ΔH^0_{ads} value is -57.13 KJ mol^{-1}, indicates adsorption process is an exothermic phenomenon and involves both physical and chemical adsorption processes. From the calculations ΔS^0_{ads}

Table 4. Tafel results in 0.5 M H_2SO_4 solution at 303 K.

Concentration (g/L)	E_{corr} (mV)	I_{corr} x 10^{-3} (A)	β_c (V/dec)	β_a (V/dec)	R_p (Ω)	η_p (%)
Blank	-332.7	11.7	5.621	6.137	109	-
0.1	-481.8	7.13	5.492	6.971	187	38.86
0.2	-464.7	4.51	5.36	7.599	302	61.30
0.3	-454.5	3.11	4.818	7.266	404	73.30
0.4	-467.5	2.98	5.937	8.669	514	74.49

shows negative values, suggests the decrease in entropy means the molecules of the adsorbate are held on surface of the solid adsorbent during adsorption process [39].

Electrochemical Studies

Potentiodynamic Polarization Studies

The polarization resistance is the slope of the applied potential and the recorded current. The polarization experiments help to study the corrosion inhibition effect of the inhibitor and kinetics of anodic and cathodic reactions. In order to find the corrosion potential E_{corr}, Corrosion current I_{corr}, cathodic (β_c), and anodic (β_a) Tafel slope, Potentiodynamic Polarization Studies were performed in the absence and presence of 0.1 g/L to 0.4 g/L of coconut shell extract in 0.5 M H_2SO_4 solution. The Tafel plot obtained from plot of Potential, *E* Vs *log I*, were recorded in Fig. 10. Polarization resistance (R_p) values were calculated using the Stern–Geary equation [40] with the help of I_{corr}, β_c and β_a generated from software installed in the instrument.

$$R_p = \frac{\beta_a \times \beta_c}{2.303 \times (\beta_a + \beta_c) \times I_{corr}} \quad (8)$$

The percentage inhibition efficiency (η_p) was calculated from the I_{corr} values using the formula

$$\eta_p = \frac{I^0_{corr} - I_{corr}}{I^0_{corr}} \times 100 \quad (9)$$

Where I^0_{corr} and I_{corr} are the corrosion current densities in absence and presence of inhibitor, respectively.

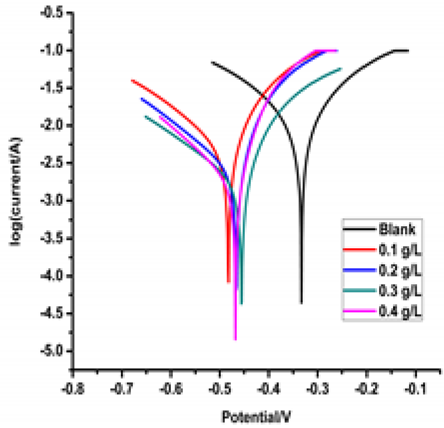


Figure 10. Tafel Plot.

The Tafel results are presented in Table 4. It shows that as the coconut shell extract concentration increases I_{corr} values decreases, which indicates that the inhibitor molecules adsorbed on the mild steel surface and formation of surface film control the diffusion of ions from the metal surface. Percentage inhibition efficiency increases (η_p) with increasing extract concentration and these results agree with gravimetric results. The gradual increase in polarization resistance (R_p) with increase in extract concentration shows that mild steel corrosion is reduced. In the present study the change E_{corr} value is less than 85 mV indicates the mixed type inhibitor [41, 42]. The addition of inhibitor changes the cathodic Tafel line (β_c) and anodic Tafel lines (β_a).

Scanning Electron Microscopy Results

The SEM analysis shows the damage on metal surface due to corrosion. From Fig. 11A it is observed that the surface is rough and highly porous in the absence of inhibitor and from Fig. 11B, the surface of the metal is in better condition and shows smooth surface in presence of 0.4 g/L concentration of the coconut shell extract. From SEM analysis we can understand that surface of the mild steel is protected by the extract solution in acid media.

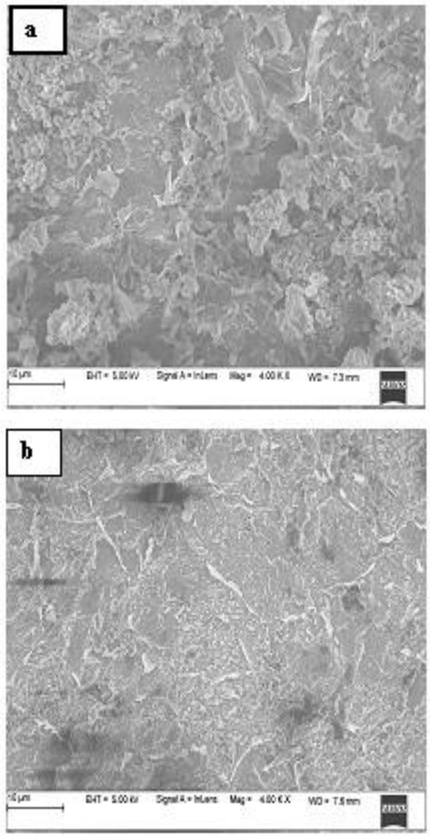


Figure 11. SEM images of mild steel in the absence (a) and presence (b) of 0.4 g/L Coconut shell extract.

Conclusion

The present study shows the identification of furfural from coconut shell extract and the extract acts as a green corrosion inhibitor in acid media. The data obtained from FTIR and HPLC confirm the presence of furfural in extract. Gravimetric studies show that the extracted compound is an excellent corrosion inhibitor for mild steel. Inhibition efficiency increases with increase in extract concentration and decreases with increase in temperature. The protection of metal by adsorption process in acid media obeys Langmuir adsorption isotherm. From the free energy value the adsorption mechanism of the inhibitor involves both physisorption and chemisorptions. Tafel plot studies show that the coconut shell extract acts as a mixed type inhibitor. The results obtained from gravimetric and electrochemical studies reveal that there is a good agreement values in inhibition efficiency.

The SEM image also confirms the protection of mild steel surface in presence of coconut shell extract. From these results we concluded that the agricultural waste product like coconut shell extract can be used as corrosion inhibitor in acidic media.

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