

Research Article

Study of Synergistic Effect of Iodide on the Corrosion Antagonistic Behaviour of a Heterocyclic Phenylhydrazone in Sulphuric Acid Medium on Carbon Steel

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Synergistic effect of KI on the corrosion inhibition efficiency of 3-acetylpyridine phenylhydrazone (3APPH) on carbon steel (CS) in 0.5 M sulphuric acid solution has been investigated using gravimetric studies, electrochemical impedance spectroscopy (EIS), and potentiodynamic polarization studies. Gravimetric corrosion studies revealed that 3APPH showed moderate corrosion inhibition efficiency up to 8 h and beyond this period it showed corrosion accelerating behavior. This antagonistic effect of 3APPH is due to the hydrolysis of the molecule in acidic medium. A very high percentage of inhibition efficiency at 24 h was obtained on the addition of KI due to the synergistic effect of iodide ions. The adsorption of 3APPH and 3APPH + KI on the surfaces of the corroding metal obey Langmuir isotherm as obtained by impedance measurements. Polarization studies revealed that 3APPH act as a mixed type inhibitor. Thermodynamic parameters (K_{ads} , ΔG_{ads}^0) were derived from the adsorption isotherms. Surface morphology of the corroding metal was investigated by SEM analysis.

1. Introduction

Industrial processes such as pickling and descaling will escalate the corrosion rate of metals enormously and the addition of certain organic compounds which contain π -bonds and heteroatoms like N, O, and S is considered as the most useful method for reducing the corrosion of carbon steel in acidic media [1–3]. Molecules equipped with the above mentioned active probes may not act as a good inhibitors against corrosion in severe aggressive solutions such as sulphuric acid. It has been found that the addition of halide ions to the sulphuric acid enhance the corrosion inhibition capacity of the organic molecules on carbon steel synergistically. The synergistic effect of the halides has increased [4, 5] in the order $Cl^- < Br^- < I^-$. Due to large size and ease of polarizability, iodide (I^-) shows the highest synergistic effect [6–10]. The present study was conducted to examine the corrosion response of carbon steel in sulphuric acid containing the phenylhydrazone 3APPH and the synergistic behavior of 3APPH with I^- on the corrosion of carbon steel [11–13].

2. Experimental

2.1. Synthesis. The heterocyclic phenylhydrazone (3APPH) was obtained by the condensation of equimolar mixture of 3-acetylpyridine and phenylhydrazine hydrochloride in ethanol. The reaction mixture was refluxed for 2 hours, cooled to obtain yellow precipitate, filtered, washed with water, and dried. Figure 1 shows the molecular structure of phenylhydrazone 3APPH. Anal. calcd for $C_{13}H_{13}N_3$: C, 73.93; H, 6.16; N, 19.9%. Found C, 73.47; H, 6.42; N, 19.44%; m.p. = 245°C; IR (KBr): $\nu_{C=N} = 1590\text{ cm}^{-1}$ Mass: M^+ peak m/z: 211.

2.2. Solutions. The aggressive solution of 0.5 M H_2SO_4 was prepared by the dilution of A.R grade 98% of H_2SO_4 (Merck) with deionized water. Solutions of 3APPH were prepared in the range of 0.2 mM–1.0 mM concentrations in 0.5 M H_2SO_4 .

2.3. Gravimetric Corrosion Studies. Carbon steel specimens of dimension $1 \times 1 \times 0.1\text{ cm}$ (composition: C, 0.58%; Mn, 0.07%; P, 0.02%; S, 0.015%; Si, 0.02%, and the rest Fe) were

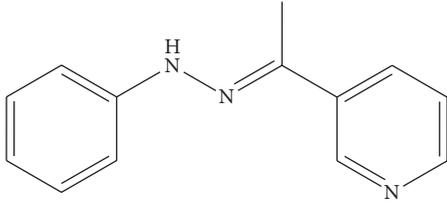


FIGURE 1: Molecular structure of 3APPH.

cut and abraded with various grades of silicon carbide papers (200, 400, 600, 800, 1000, and 1200). The exact area and thickness of each specimen were measured. Specimens were then degreased with acetone, washed with distilled water and dried. After weighing, specimens were immersed in 50 mL acid solutions at 30°C in the absence and presence of the 3APPH and 3APPH + KI (0.2 mM) at different concentrations using hooks and fishing lines. Weight loss of metal specimens was noted after 24 h. The experiments were carried out in duplicate and the average values were reported. The corrosion rate (ν) and the percentage of inhibition efficiency ($\eta_w\%$) were calculated by the following equations [14–16]:

$$\nu = \frac{W}{St}, \quad (1)$$

$$\eta_w\% = \frac{\nu_0 - \nu}{\nu_0} \times 100, \quad (2)$$

where W is the average weight loss of CS specimen, S is the total area of specimen, t is the time of treatment, and ν_0 and ν are the corrosion rates of uninhibited and inhibited specimens, respectively.

2.4. AC Impedance Studies. The impedance measurements were performed in a three electrode assembly. Saturated calomel electrode (SCE) was used as the reference electrode. Platinum electrode having 1 cm² area was taken as the counter electrode. Metal specimens with an exposed area of 1 cm² were used as the working electrode. The EIS experiments were carried out on an Ivium compactstat-e electrochemical system. 0.5 M H₂SO₄ acid was taken as the electrolyte and the working area of the metal specimens were exposed to the electrolyte for 30 minutes prior to the measurement. Impedance measurements were performed at constant potential (OCP) in the frequency range from 1 KHz to 100 mHz with an amplitude of 10 mV as excitation signal. The percentage of inhibition from impedance measurements were calculated using charge transfer resistance by the following expression [17, 18]:

$$\eta_{\text{EIS}}\% = \frac{R_{\text{ct}} - R'_{\text{ct}}}{R_{\text{ct}}} \times 100, \quad (3)$$

where R_{ct} and R'_{ct} are the charge transfer resistances of working electrode with and without inhibitors, respectively.

2.5. Potentiodynamic Polarization Measurements. Electrochemical polarization studies of CS specimens in 0.5 M

H₂SO₄ with and without 3APPH and 3APPH + KI were performed by recording anodic and cathodic potentiodynamic polarization curves. Polarization plots were obtained in the electrode potential range from -100 to +100 mV versus corrosion potential (E_{corr}) at a sweep rate of 1 mV/sec. To obtain corrosion current densities, Tafel polarization analyses were done by extrapolating anodic and cathodic curves. The percentage of inhibition efficiency ($\eta_{\text{pol}}\%$) was evaluated from the measured I_{corr} values using the following relation [19]:

$$\eta_{\text{pol}}\% = \frac{I_{\text{corr}} - I'_{\text{corr}}}{I_{\text{corr}}} \times 100, \quad (4)$$

where I_{corr} and I'_{corr} are the corrosion current densities of the exposed area of the working electrode in the absence and presence of inhibitor.

2.6. Scanning Electron Microscopy. Surface analyses of CS specimens were performed using scanning electron microscope (model Hitachi SU6600). SEM images of CS surface were taken by treating 0.5 M H₂SO₄ solutions in the absence and presence of the 3APPH and 3APPH (0.8 mM) + KI (0.2 mM) for 24 h at 30°C.

3. Results and Discussions

3.1. Gravimetric Studies. Weight loss of CS specimens in 0.5 M H₂SO₄ at 30°C was determined at 24 h in the presence of various concentrations of 3APPH and 3APPH + KI (0.2 mM). The corrosion rates and inhibition efficiencies are listed in Table 1. It is evident from the table that the rate of corrosion in the presence of 3APPH increased with concentration at 24 h (corrosion accelerating/antagonistic behavior). A marked rise in the inhibition efficiency was noted for CS specimens in the presence of 3APPH + KI. To investigate a detailed corrosion inhibition response of the 3APPH on CS, the gravimetric measurements were performed every 2 hours. The corrosion rate of CS was found to be decreased with the concentration of 3APPH up to 8 h or $\eta_w\%$ increased with the concentration. Beyond this period, 3APPH tend to behave as a corrosion accelerator. From the UV spectral studies it was confirmed that the 3APPH molecules undergo slow hydrolysis in sulphuric acid medium at room temperature into the parent ketone and phenylhydrazine. The hydrolyzed products were inhibited less on the CS surface when compared to the hydrazone (3APPH), and the same was confirmed by performing the gravimetric corrosion studies of CS in sulphuric acid by taking parent amine and ketone as the inhibitor (Figure 2). To check the synergistic effect of I⁻ with the parent amine, parent ketone and their mixture, gravimetric corrosion analysis of the systems like phenylhydrazine (PH) + KI, 3-acetylpyridine (3AP) + KI, PH + 3AP + KI was performed independently and compared with 3APPH + KI system (Figure 2). These studies clearly showed that the highest $\eta_w\%$ was obtained for 3APPH + KI for all the studied concentrations. The present investigation also emphasizes that 3APPH molecules

TABLE 1: Inhibition efficiency ($\eta_w\%$) from gravimetric measurements for different systems on CS corrosion at 24 h in 0.5 M H_2SO_4 at 30°C.

System/conc.	Corrosion rate (ν)	% of inhibition efficiency ($\eta_w\%$)
Blank	23.53	—
Blank + 0.2 mM KI	19.40	24.0
0.2 mM 3APPH	27.83	-18.28
0.4 mM 3APPH	32.67	-38.83
0.6 mM 3APPH	44.77	-90.26
0.8 mM 3APPH	45.31	-92.54
1.0 mM 3APPH	45.48	-93.27
0.2 mM 3APPH + 0.2 mM KI	2.84	87.90
0.4 mM 3APPH + 0.2 mM KI	2.50	89.38
0.6 mM 3APPH + 0.2 mM KI	1.06	95.47
0.8 mM 3APPH + 0.2 mM KI	0.99	95.80
1.0 mM 3APPH + 0.2 mM KI	0.82	96.50

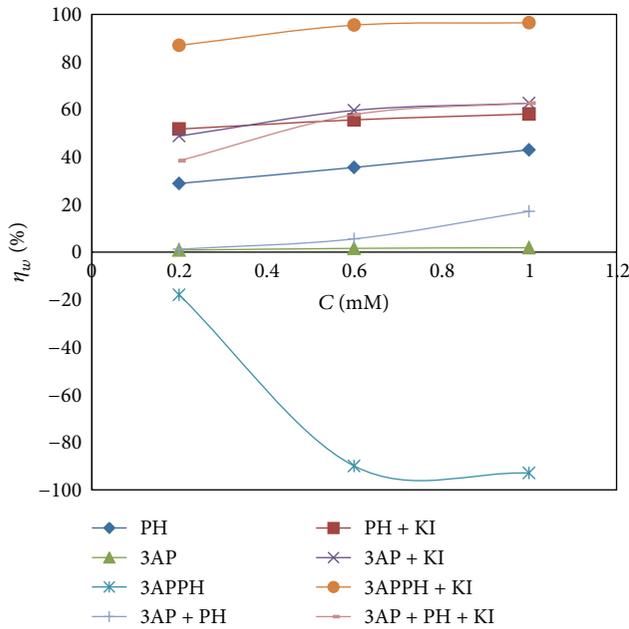


FIGURE 2: Comparison between the corrosion inhibition efficiencies of various systems.

attaching through the I^- ions on CS surface do not undergo hydrolysis by hydronium ions.

3.2. AC Impedance Studies. To emphasize the role of iodide ions on the corrosion inhibition of CS by 3APPH, impedance study was performed by exposing the corroding metal surface towards the 3APPH and 3APPH + KI solutions for 30 minutes. Figures 3 and 4 represent the Nyquist plots of CS specimens in 0.5 M H_2SO_4 in the presence of various concentrations of 3APPH and 3APPH + KI, respectively. It is evident from the plots that the impedance response of metal specimens has marked differences in the presence and absence of the KI with 3APPH. Equivalent circuit that fit into many electrochemical systems composed of a double layer capacitance, R_s and R_{ct} [20–22], can also be accepted in this

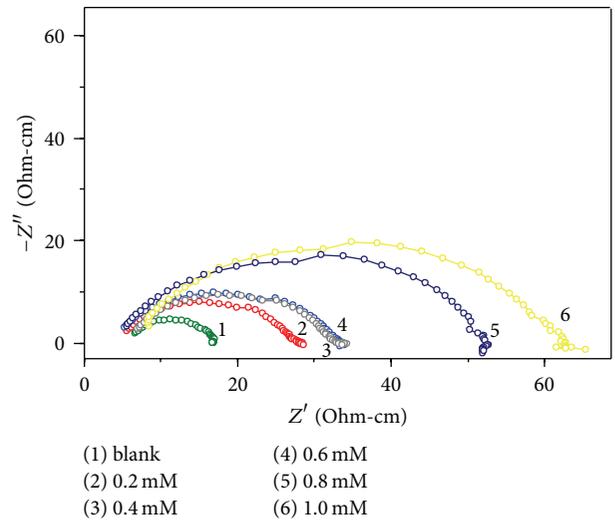


FIGURE 3: Nyquist plots for CS specimens in 0.5 M H_2SO_4 and 3APPH.

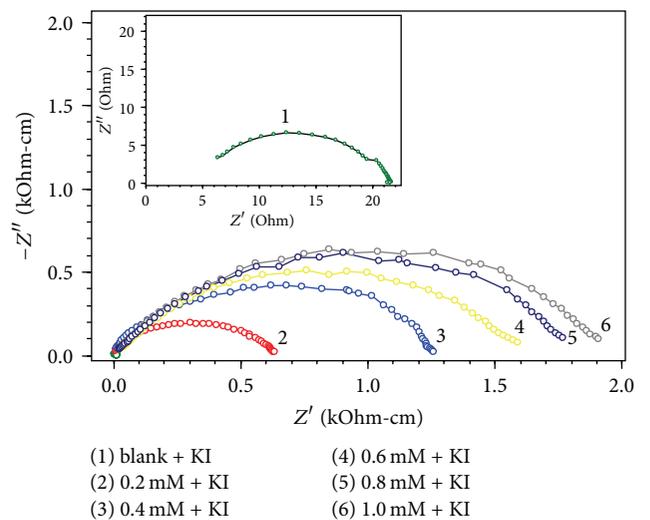


FIGURE 4: Nyquist plots for CS specimens in 0.5 M H_2SO_4 and 3APPH + KI.

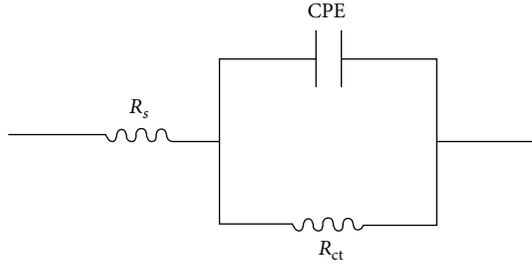
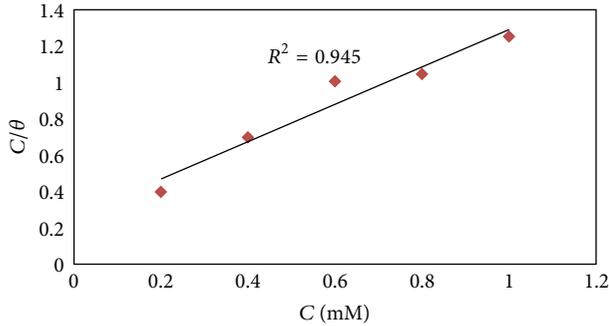


FIGURE 5: Equivalent circuit fitting for EIS.

FIGURE 6: Langmuir adsorption isotherm for CS specimens in 0.5 M H_2SO_4 and 3APPH.

study. To reduce the effects due to surface irregularities of metal, constant phase element (CPE) is introduced into the circuit instead of a pure double layer capacitance which gives more accurate fit as shown in the Figure 5 [23–25].

The impedance of CPE can be expressed as

$$Z_{CPE} = \frac{1}{Y_0(j\omega)^n}, \quad (5)$$

where Y_0 is the magnitude of CPE, n is the exponent (phase shift), ω is the angular frequency, and j is the imaginary unit. CPE may be resistance, capacitance, and inductance [26] depending upon the values of n . In all measurements the value of n ranges between 0.75 and 0.99, suggesting the capacitive response of CPE. The impedance parameters and the percentage of inhibition ($\eta_{EIS}\%$) are listed in Tables 2(a) and 2(b), respectively. From the data it is clear that R_{ct} values are increased with the 3APPH concentration. The capacitance values (C_{dl}) decreased with 3APPH concentration and this decrease in C_{dl} was enhanced upon addition of I^- ions to the corrosive environment. These results suggest that the 3APPH molecules function by adsorption at the metal/solution interface [27, 28] and this adsorption is reinforced by I^- ions. The addition of KI drastically increased the $\eta_w\%$ values. From Table 2 it is also evident that $\eta_w\%$ for KI in combination with 3APPH is higher than the sum of $\eta_w\%$ for single KI and single 3APPH, which is considered due to synergistic effect.

3.3. Adsorption Isotherm and Free Energy of Adsorption. In order to reveal the mechanism of inhibition, various adsorption isotherms such as Langmuir, Freundlich, Temkin, and

TABLE 2: Electrochemical impedance parameters of CS specimens in 0.5 M H_2SO_4 at 30°C in the absence and presence of 3APPH (a) and 3APPH + 0.2 mM KI (b).

(a)			
C (mM)	R_{ct} (Ωcm^2)	C_{dl} ($\mu F cm^{-2}$)	$\eta_{EIS}\%$
0	9.72	133	—
0.2	19.54	134	50.25
0.4	22.77	121	57.31
0.6	24.09	119	59.65
0.8	41.39	118	76.51
1.0	48.22	116	79.84
(b)			
C (mM)	R_{ct} (Ωcm^2)	C_{dl} ($\mu F cm^{-2}$)	$\eta_{EIS}\%$
Blank + KI	12.21	128	20.39
0.2 + KI	518.4	28.9	98.13
0.4 + KI	1071	26.2	99.09
0.6 + KI	1301	20.2	99.25
0.8 + KI	1499	21.5	99.35
1.0 + KI	1607	14.3	99.39

TABLE 3: Thermodynamic parameters from adsorption isotherms.

System	Adsorption isotherm	K_{ads}	ΔG_{ads}^0 (kJ)
3APPH	Langmuir	3.8×10^3	-30.9
3APPH + KI	Langmuir	5.0×10^5	-43.17

Frumkin were plotted and the most suitable one was selected with the aid of correlation coefficient. Among the isotherms considered above, the most adequate one for both experiments, that is, impedance measurements in the presence and absence of KI, was Langmuir adsorption isotherm. Figures 6 and 7, respectively, portray the adsorption isotherms for the inhibition of 3APPH in the absence and presence of KI. Langmuir adsorption isotherm can be expressed as [29]

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C, \quad (6)$$

where C is the concentration of the 3APPH, θ is the fractional surface coverage, and K_{ads} is the adsorption equilibrium constant. Table 3 explores the thermodynamic parameters such as adsorption equilibrium constants (K_{ads}) and free energies of adsorption ΔG_{ads}^0 .

It is evident from Table 3 that ΔG_{ads}^0 is negative for both investigations, which suggest the spontaneity of the processes. For the inhibition process by 3APPH on CS without KI, ΔG_{ads}^0 value implies that the mechanism of adsorption involve both physisorption and chemisorption. Since ΔG_{ads}^0 exhibited for the 3APPH solution containing KI was more than 40 kJ, the chemisorptions [30, 31] is the sole factor responsible for the inhibition process. This investigation clearly establishes the synergistic effect of I^- ions with 3APPH which cause adsorbing firmly the 3APPH molecules without hydrolysis on the metal surface by a strong chemical interaction.

TABLE 4: Potentiodynamic polarization parameters of CS specimens in 0.5 M H₂SO₄ at 30°C in the absence and presence of 3APPH.

C (mM)	E_{corr} (mV/SCE)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	b_a (mV/dec)	$-b_c$ (mV/dec)	$\eta_{pol}\%$
Blank	-468	1041	84	122	—
0.2	-461	591	67	131	43.1
0.4	-464	432	63	123	58.5
0.6	-455	395	68	124	62.0
0.8	-465	255	54	114	75.5
1.0	-462	175	46	106	83.2

TABLE 5: Potentiodynamic polarization parameters of CS specimens in 0.5 M H₂SO₄ at 30°C in the presence of KI and 3APPH + KI.

C (mM)	E_{corr} (mV/SCE)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	b_a (mV/dec)	$-b_c$ (mV/dec)	$\eta_{pol}\%$
Blank + KI	-475	799	80	123	23.3
0.2 + KI	-507	18.8	85	102	98.2
0.4 + KI	-540	12.6	118	121	98.8
0.6 + KI	-526	11.8	117	105	98.9
0.8 + KI	-515	8.9	102	119	99.1
1.0 + KI	-534	8.5	127	106	99.2

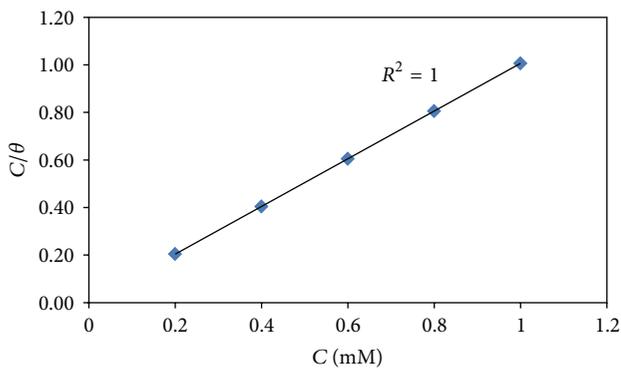
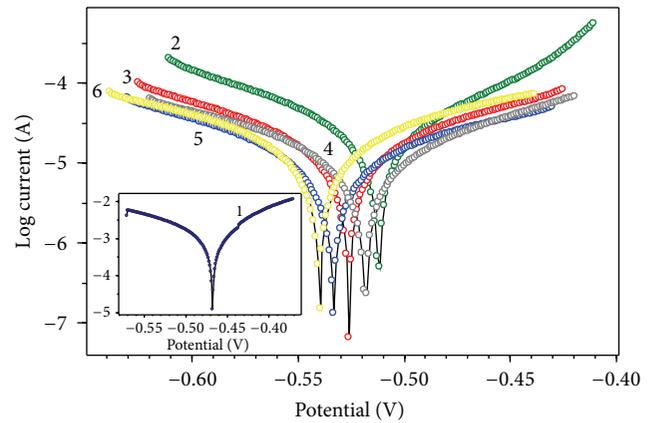
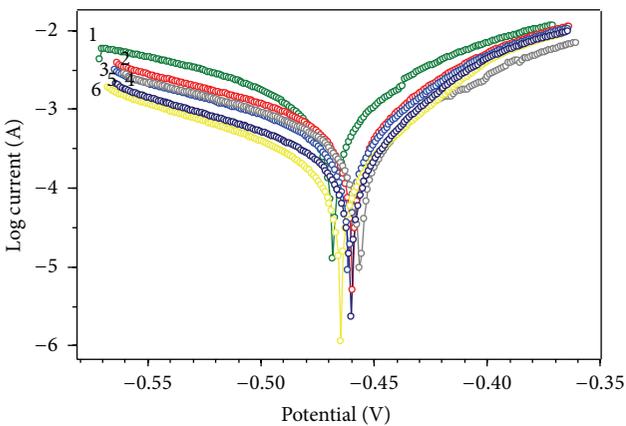


FIGURE 7: Langmuir adsorption isotherm for CS specimens in 0.5 M H₂SO₄ and 3APPH + KI.



- (1) blank + KI
- (2) 0.2 mM + KI
- (3) 0.4 mM + KI
- (4) 0.6 mM + KI
- (5) 0.8 mM + KI
- (6) 1.0 mM + KI

FIGURE 9: Tafel plots of CS specimens in 0.5 M H₂SO₄ at 30°C, with KI and 3APPH + KI.



- (1) blank
- (2) 0.2 mM
- (3) 0.4 mM
- (4) 0.6 mM
- (5) 0.8 mM
- (6) 1.0 mM

FIGURE 8: Tafel plots of CS specimens in 0.5 M H₂SO₄ at 30°C, with and without 3APPH.

3.4. Potentiodynamic Polarization Studies. Potentiodynamic polarization curves for 3APPH in 0.5 M H₂SO₄ at 30°C for CS specimens in the presence of various concentrations of 3APPH and 3APPH + KI are shown in Figures 8 and 9 respectively and the corresponding polarization parameters like corrosion current densities (I_{corr}), corrosion potential (E_{corr}), cathodic Tafel slope (b_c), anodic Tafel slope (b_a), and inhibition efficiency ($\eta_{pol}\%$) are listed in Tables 4 and 5 respectively. The data show that addition of 3APPH to acid medium affects both the cathodic and anodic parts of the curves. Addition of I^- ions to 3APPH/H₂SO₄ systems results in marked decrease in the corrosion current density (I_{corr}). In other words, both anodic and cathodic reactions are drastically inhibited and the 3APPH can be viewed as mixed type inhibitor for CS specimens in 0.5 M H₂SO₄. The values

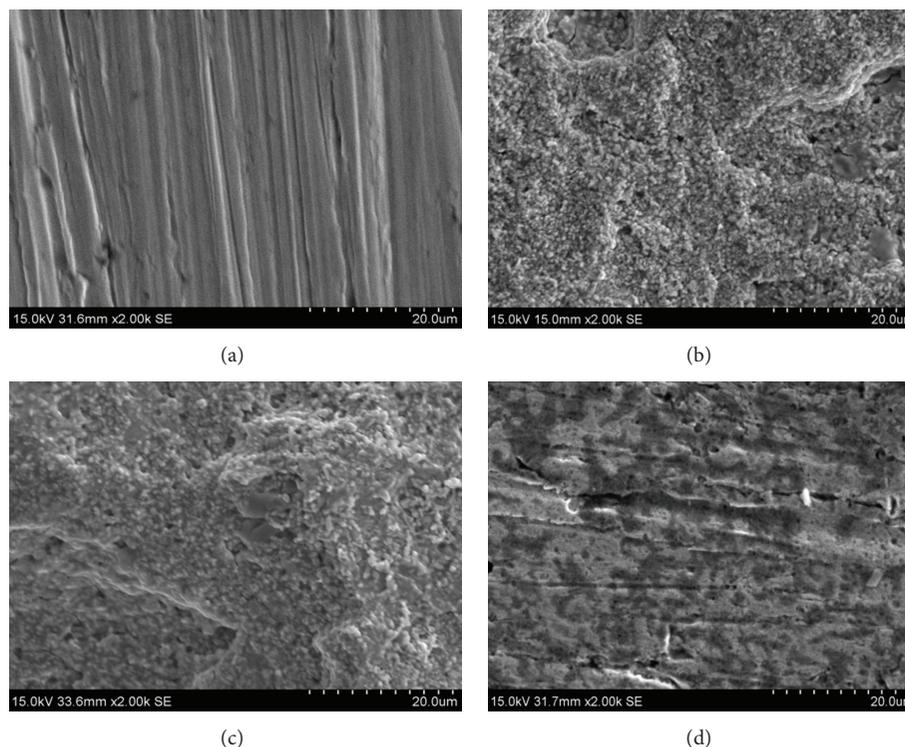


FIGURE 10: (a) SEM image of bare CS surface (b) SEM image of CS surface in 0.5 M H_2SO_4 , (c) SEM image of CS surface in 0.5 M H_2SO_4 and 3APPH (0.8 mM), and (d) SEM image of CS surface in 0.5 M H_2SO_4 and 3APPH (0.8 mM) + 0.2 mM KI.

establish that the $\eta_{\text{pol}}\%$ of 3APPH is increased in presence of KI and these results also assure the existence of strong synergism between 3APPH and KI in the corrosion inhibition process of CS.

3.5. SEM Analysis. To examine the effect of 3APPH molecules on CS surface, SEM analyses were performed [32, 33]. SEM images of CS surfaces are given in Figures 10(a)–10(d). On comparison of the Figures 10(a) and 10(b), it is evident that the surface of CS was seriously corroded in the acidic solution. Figure 10(c) represents the surface image of the metal in the presence of the 3APPH (0.8 mM, 24 h). Comparison of the textures of Figures 10(b) and 10(c) revealed that severe damage on the surface of CS happened in the presence of the 3APPH due to the antagonistic behaviour. Figure 10(d) is the image of CS surface treated with 3APPH and KI (0.8 mM + 0.2 mM KI for 24 h). Morphology of this image is entirely different from that of other images and least damage occurred for the surface. This suggests an increased inhibition of the 3APPH molecules on the CS surface through I^- ions.

4. Conclusions

3APPH undergo slow hydrolysis in sulphuric acid medium and showed moderate/poor corrosion inhibition efficiency up to 8 h. Beyond this period, 3APPH shows corrosion antagonistic character. In the presence of KI, the 3APPH molecules are firmly attached on the CS surface and do not

undergo hydrolysis and act as excellent corrosion inhibitor. Adsorption studies revealed that chemical interaction occurs between the 3APPH molecules and CS surface through I^- ions. This causes the enhancing of the inhibition efficiency greatly. Parent ketone and parent amine exhibit poorer corrosion inhibition efficiency than the 3APPH molecules. Polarization studies reveal that 3APPH act as a mixed type inhibitor.

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