IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

Investigation Of Corrosion Inhibition Properties Of 2-Acyl Pyridine Derivative On Mild Steel In 1 M Hcl: Surface Morphological Studies.

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Abstract

Inhibition properties of 2-Acyl pyridine derivative on mild steel in 1 M HCl was investigated using Field Emission Scanning Electron Microscope (FE-SEM) and Atomic Force Microscope (AFM). FE-SEM images of mild steel surface in the presence of inhibitors remains smooth with very less no. of pits as compared to blank acid solution. In the presence of inhibitors, AFM studied show that the variation in height sensor and the average surface roughness values obtained, are very less as compared to that obtained in blank solution. FE-SEM and AFM results showed that both the inhibitors 2-APN and ACN possess very good inhibition ability but 2-APN show comparatively better inhibition property than ACN. Both the results obtained from FE-SEM and AFM studied are in good agreement.

Keyword: Acid corrosion, corrosion inhibitor, mild steel, FE-SEM, AFM

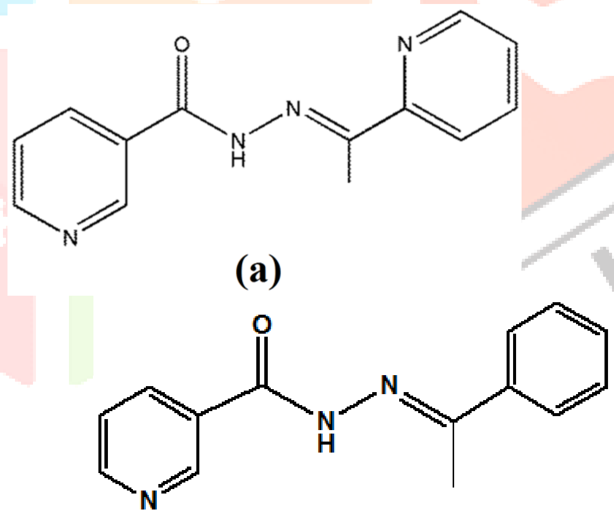
1. Introduction

Corrosion of metals, especially mild steel, in acidic media has long been a serious matter of concern as in modern era as corrosion leads to losses worth billions of dollars every year world-wide**.** There are various methods of protection of metal in acid media among which use of inhibitor is most popular and efficient method [1,2]. Literature review reveals that organic compounds containing heteroatoms like nitrogen, oxygen, sulphur, =C=N- group, aromatic ring etc. show good inhibition properties against mild steel [3-7]. In recent time, studies of the inhibition properties of Schiff's bases for metal in acidic solution have attracted the attention of scientist, researchers, academicians etc [8-9]. It is due to the fact that Schiff's bases are very easy to synthesize with very cheap materials and most of them show very good inhibition properties against metal corrosion in acidic solutions [10-11]. D.K. Singh. et al have investigated 2-Acetyl Pyridine nicotinic acid hydrazone (2-APN) and Acetophenone nicotinic acid hydrazone (ACN) as corrosion inhibitors for mild steel in 1 M HCl solution using electrochemical, gravimetric and quantum chemical calculation methods [12]. In these studies, authors have reported that 2-APN and ACN inhibitors show 96.5% and 87.7% inhibition efficiency respectively at 2 mM inhibitors concentrations. So far, the effect of these inhibitors on the morphology of mild steel surface have not been studied. Field Emission Scanning Electron Microscopy (FE-SEM) and Atomic Force Microscopy (AFM) techniques have also been used to studies the corrosion inhibition properties of the inhibitors for the metals in acid solutions like HCl etc. The aim of the present study was to investigate the effect of addition of these inhibitors at selected concentrations of 2 mM and 4 mM on the surface morphology of mild steel surface in 1 M HCl solution at 30 °C using FE-SEM and AFM techniques. The specific concentrations of 2 mM and 4 mM are selected, because at these concentrations, studied inhibitors has shown maximum inhibition efficiency as reported earlier [12].

2. Materials and Methods

2.1. Synthesis of 2-Acetyl Pyridine nicotinic acid hydrazone (2-APN) and Acetophenone nicotinic acid hydrazone (ACN)

2-Acetyl Pyridine nicotinic acid hydrazone (2-APN) and Acetophenone nicotinic acid hydrazone (ACN) were prepared as per reported method **[12]** by condensation of nicotinic acid hydrazide with selected aldehydes/ ketones (AR, supplied by Sigma Aldrich). The chemical structure and abbreviations for the prepared inhibitors are given in **Fig. 1**. The structures and purity of the compounds were confirmed by TLC, FT-IR and 1H NMR spectroscopic methods.



(a)

(b)

Fig. 1. Chemical structures of the synthesized inhibitors: **(a)** 2-Acetyl pyridine nicotinic acid hydrazone (2-APN) and **(b)** Acetophenone nicotinic acid hydrazone (ACN).

2.2. Material and sample preparation

In this study, mild steel having chemical composition (wt%): C- 0.07, Mn-0.42, P-0.029, S-0.014, Al-0.041 and rest Fe was used for corrosion inhibition properties test. The mild steel specimens of dimension (3 cm × 2 cm × 0.3 cm) were used for FE-SEM and AFM techniques. Prior to each experiment the exposed mild steel surface was abraded with emery papers (grade 320 to 1200), washed with double distilled water and dried with cool air. Analytical grade 37% HCl solution was used to prepare the test solution (1 M HCl) using double distilled water.

2.3 Field Emission Scanning Electron Microscope (FE-SEM)

To explore the effect of addition of inhibitor on the surface morphology of the abraded mild steel surface immersed in 1M HCl solution in the presence of inhibitors were studied using Field Emission scanning electron microscope (FE-SEM). It was performed by FE-SEM Supra 55 (Carl Zeiss, Germany). The mild steel samples for FE- SEM analysis were prepared as described above and then immersed in 1 M HCl in the presence of 2 mM and 4 mM of the inhibitors for 4 hrs. at 30 ˚C and then taken out from the test solution, cleaned with double-distilled water and dried with cool air.

2.4 Atomic Force microscope (AFM)

To get quantification of surface roughness of mild steel in the presence and absence of inhibitors owing to attack of acid solution or in other words, to enhance surface morphology investigation, the mild steel surface was also investigated using atomic force microscopy (AFM). In this study, three-dimensional topography and the average surface roughness of the mild steel surface over area scale of (10×10 μm^2) was calculated using Dimension Icon with one controller (Nanoscope V) from Bruker in tapping mode.

3. Results and Conclusions

3.1 *Field Emission Scanning Electron Microscopy (FE-SEM)*

The FE-SEM images of mild steel specimens after immersion of 4 hrs. in 1 M HCl in the presence of 2-APN and ACN at 2 mM and 4 mM concentrations at 30°C are represented in Fig. 2. On comparisons of these figures with that of Fe-SEM images of the abraded mild steel and mild steel in the 1 M HCl at 30 °C without inhibitors as reported earlier by D.K. et al **[13-14**]. it is very clear that mild steel surface in the absence of inhibitors becomes very rough with numerous pits and deposition of agglomerates as in the reported FE-SEM **[13-14].** It is due to aggressively attack of corrosive acid solution on the surface of mild steel. On addition of inhibitors 2-APN and ACN, the mild steel surface is found to be comparatively smoother with very less no. of pits and cracks with respect to that obtained in the absence of inhibitor. It suggests that the addition of inhibitors to acid solution forms protective layer which reduces the aggressive attack of corrosive acid solution. It can be explained as the formation of protective film due to adsorption of inhibitors on the mild steel surface. From Fig. 2, it is clear that surface morphology of mild steel surface 2-APN inhibitor at 2 mM concentration remains more smoother having less no. of pits as compared to mild steel surface morphology in the presence of 4 mM concentration of 2-APN. It may owing to aggregation or association at higher concentration.

FE-SEM images reveal that although both the inhibitors 2-APN and ACN can exhibit good corrosion inhibition properties but 2-APN has more inhibitive potential than ACN.

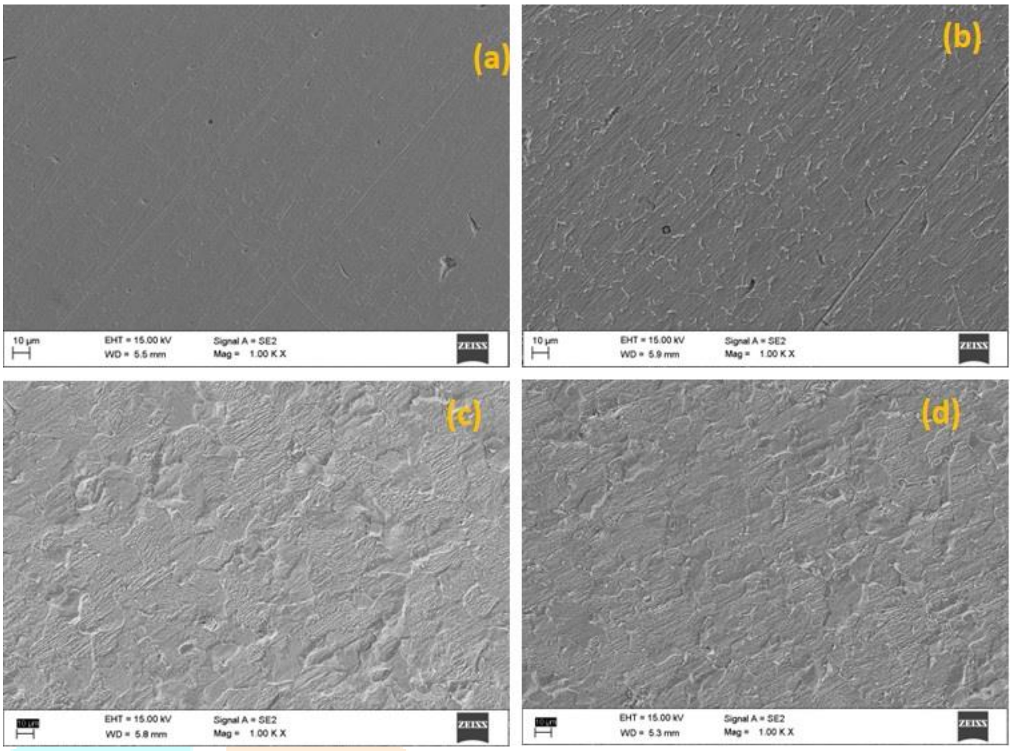


Fig.2 FE-SEM images of mild steel after 4 hrs. immersion in 1 M HCl in the presence of inhibitors: (a) 2 mM 2-APN (b) 4 mM 2-APN (c) 2 mM ACN (d) 4 mM ACN at 30 ˚C.

3.2 Atomic Force Microscopy (AFM)

AFM has became a powerful tool to investigate the surface morphology of the metal surface qualitatively as well as quantitatively at micro to nano level. Now a days, it has also been applied in investigating various organic compounds as corrosion inhibitors for metals in acidic media **[15-17]. Fig.3** represents the three-dimensional AFM images of mild steel after immersion for 4 hrs. in 1 M HCl in the presence of different concentrations (2 mM and 4 mM) of inhibitors at 30 ˚C. From these figures, it can be seen that the surface morphology of mild steel in the presence of 2-APN and ACN inhibitors remains very smooth as compared to that in blank 1 M HCl [13,14]. It may be owing to protective film of inhibitors molecules on the surface of mild steel which protect mild steel surface against corrosive attack of acid solution.

From height sensor, surprisingly, it is seen that the variation of height sensor in the presence of 2 mM concentration of 2-APN is lesser than that in the presence of 4 mM concentration of 2-APN. It may be owing to association or aggregation of 2-APN molecules at higher concentrations due to which surface coverage of metal surface by the inhibitor molecules decreases.

It is also studied surface morphology of mild steel surface by calculating average surface roughness after 4 hrs. immersion it in the presence of inhibitor and then compared the same with fresh abraded mild steel surface and mild steel surface in blank 1 M HCl solution that is without adding inhibitor. **Table 1** represents the average surface roughness (*R*a) for mild steel in 1 M HCl in the presence and absence of different concentration (2 mM and 4 mM) of inhibitors. The data of *R*a in the absence of inhibitors and fresh abraded mild steel surface were taken from earlier reported [13-14]. It is obvious that the average surface roughness decreases significantly with increasing concentration of the inhibitors, and the decrease in a value of average surface roughness is more prominent in the presence of 2-APN. These results indicates that both show good inhibition properties for mild steel in 1 M HCl solution. These results also support good inhibition ability of 2-APN as compared to ACN in 1 M HCl for mild steel and in good agreement with that obtained from FE-SEM.

Table 1
Average surface roughness of the mild steel surface in 1 M HCl in the presence and absence of inhibitors after immersion for 4 hrs at 30˚C

Inhibitors	C (mM)	Average surface roughness of mild steel, Ra (nm)
2-APN	0.00	235.00
	2.00	23.30
	4.00	76.04
ACN	2.00	129.00
	4.00	51.01
Polished MS		8.63

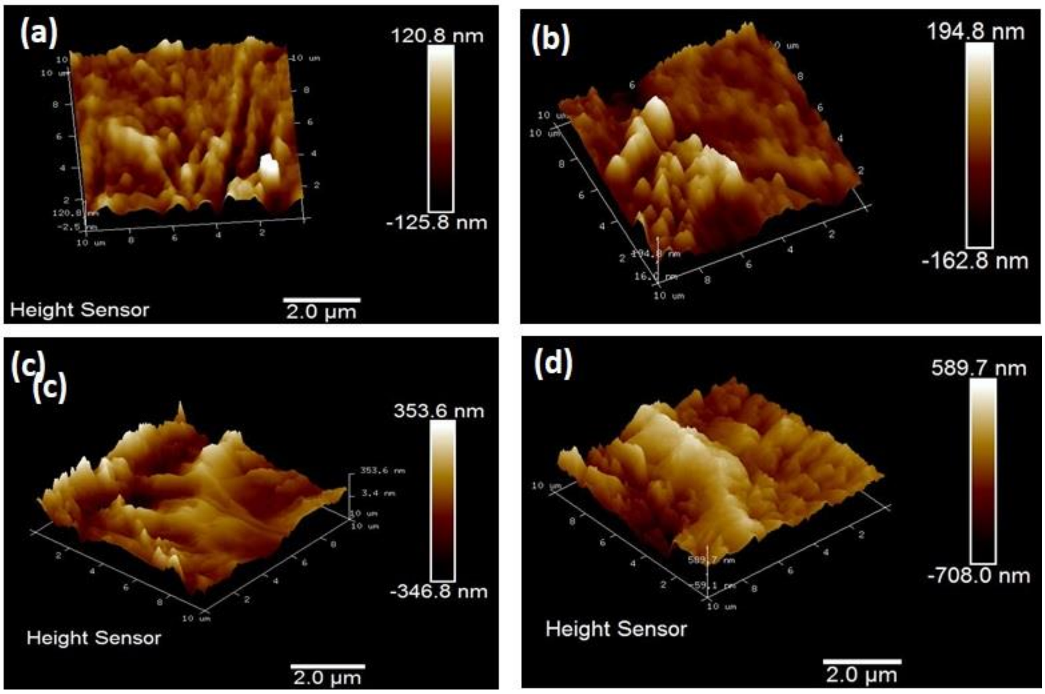


Fig.3 AFM images of mild steel after 4 hrs. immersion in 1 M HCl in the presence of inhibitors: (a) 2 mM 2-APN (b) 4 mM 2-APN (c) 2 mM ACN (d) 4 mM ACN at 30 ˚C.

4. Conclusions

(i) 2-APN and ACN both show good inhibition properties for mild steel in 1 M HCl solution.

Order of inhibition ability: 2-APN < CAN

(ii) 2-APN shows higher inhibition ability at 2 mM as compared to 4 mM concentration.

(iii) The surface roughness of mild steel surface decrease on addition of inhibitors again the surface roughness value decreases on increasing inhibitors concentration.

(iv) Both FE-SEM and AFM results are in good agreement.

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