

## Corrosion Inhibition Performance of Cetrizine on Mild Steel in 1M H<sub>2</sub>SO<sub>4</sub>

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### Abstract

The inhibitive action of cetrizine on corrosion of mild steel in 1M H<sub>2</sub>SO<sub>4</sub> was investigated by using weight loss, corrosion potential, potentiodynamic polarization, SEM and EDX techniques. Results obtained from weight loss and polarization curves indicate that cetrizine act as an efficient corrosion inhibitor. The compound influences the anodic dissolution of mild steel as well as hydrogen evolution reaction in 1M H<sub>2</sub>SO<sub>4</sub>. The inhibition was due to adsorption of inhibitor molecule on metal surface and experimentally obtained adsorption isotherm follows Langmuir equation. An adherent layer of inhibitor on metal surface acted as barrier between metal and aggressive solution.

**Keywords:** Mild Steel; Inhibitors; Adsorption; Electrochemical techniques; Scanning Electron Microscope; Energy Dispersive X-ray Analysis.

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## 1. Introduction

Mild steel has found wide industrial application due to its excellent mechanical properties and low cost. The study of corrosion of iron and its alloys are of great importance due to economic, safety and conservation [1-3]. Sulphuric acid solutions are widely used for chemical cleaning, descaling, pickling and oil well acidizing industry which leads to electrochemical corrosion. Among the various methods available, use of inhibitor is one of the most economical and effective in protecting metal from corrosion attack in acidic media [4-8]. A corrosion inhibitor can function in two ways, as environment modifiers and by adsorption. In case of environment modifiers, the action and mechanism of inhibition is its simple interaction with the aggressive species of environment thus reducing attack on metal. Adsorption inhibition involves the interaction of inhibitor molecule with metal atoms present on metal surface [9]. Organic compounds containing nitrogen, sulfur, and oxygen atom in their structure are considered to be most effective and efficient corrosion inhibitors for steel in acidic media [10-13]. The inhibition property of compounds containing these hetero atoms is attributed to presence of lone pair of electrons which can be donated to the vacant d orbital of metal for adsorption. Nitrogen containing heterocyclic compound are found very effective and efficient corrosion inhibitor of mild steel in acidic media. There is a wide range of studies in the literature regarding corrosion inhibition by N-containing heterocyclic compound in acid media [14-17].

Effectiveness of corrosion inhibitors also depends upon their molecular structure, steric effect and aromaticity [18-19]. Organic inhibitors are generally adsorbed on metal surface and forms protective film which acts as barrier between the metal and aggressive solutions. The mechanism of inhibition is also affected by concentration, pH, nature of the anion of acid and nature of the metal [20].

Many organic compounds with pharmaceutical application have potential corrosion inhibition property. El Naggar have studied corrosion inhibition property of sulphaguanidine, sulphamethazine, sulphamethoxazole, sulphadiazine and reported them as potential corrosion inhibitor of mild steel in HCl and H<sub>2</sub>SO<sub>4</sub> [21]. Fluoroquinolones such as ciprofloxacin, norfloxacin and ofloxacin were studied for corrosion inhibition of mild steel in acidic media [22]. Corrosion inhibition of mild steel by tramadol in HCl and H<sub>2</sub>SO<sub>4</sub> has been studied by Prabhu et al [23]. The effect of pharmaceutically

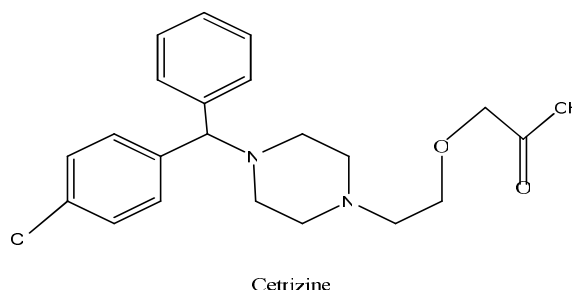
active compounds cefalexin, doxycycline and cefotaxime on the corrosion inhibition of mild steel in HCl was studied by Shukla et al [24-26].

Present study is aimed to investigate inhibitive performance of cetirizine on corrosion behavior of mild steel in 1M H<sub>2</sub>SO<sub>4</sub>. Inhibitive performance of cetirizine was evaluated using weight loss, potentiodynamic polarization and scanning electron microscopy.

## 2. Experimental

### 2.1 Chemicals and Materials

Mild Steel coupons having composition wt % (C- 0.16%, Si- 0.10%, Mn-0.40%, P- 0.013%, S- 0.02% and remaining as Iron) have been used as working electrode in the present investigation. For electrochemical polarization and weight loss studies, coupons of 1.0cm x 3.0cm x 0.025cm were sheared from the commercial grade sheet. The surface of mild steel coupons were abraded successively by different grades of metallographic emery papers 1/0, 2/0, 3/0, and 4/0 obtained from Sianor, Switzerland, so as to get the surface free from scratch and other apparent defects. The polished samples were washed with soap solution, rinsed with bi-distilled water, degreased with acetone and finally dried. The surface treatments were carried out immediately before each experiment of corrosion test. The aggressive solution was made of AR grade Sulphuric acid obtained from Merck Chemicals. One molar solution of sulphuric acid is prepared with double distilled water. The organic inhibitors cetirizine (C<sub>21</sub>H<sub>25</sub>ClN<sub>2</sub>O<sub>3</sub>) was used as received without further purification. The chemical structure of cetirizine is shown in fig.1. The measurements were carried out in aerated non stirred 1m sulphuric acid solution at concentration range of 10 ppm to 500 ppm as the corrosion inhibitor.



**Fig. 1:** Chemical structure of cetirizine.

## 2.2 Weight loss measurements

Weight loss measurements were carried out in a glass vessel with 100 ml of 1M H<sub>2</sub>SO<sub>4</sub> solution with and without concentration of inhibitors ranges from 10 ppm to 500 ppm. The immersion time for weight loss was 24 hrs. at  $30 \pm 1$  °C. After immersion the coupons were withdrawn, rinsed with double distilled water, washed with acetone, dried and weighed. The experiment was carried out in duplicate and the average value of weight loss noted.

## 2.3 Electrochemical measurements

Electrochemical measurements were carried out on the steady state open circuit potential (OCP). The variation of corrosion potential of mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> was measured against saturated calomel electrode in absence and presence of various concentrations of inhibitors. The time dependence of OCP for different experiments was recorded for 2 hours exposure period. Then same sample was used for potentiodynamic polarization (PD) experiments. Potential was swept between -0.5V to 0.5V at the scan rate of 5mV/second. Different electrochemical results obtained from potentiodynamic polarization are reported in Table No.1. The polarization studies were carried out in unstirred solutions. For electrochemical polarization studies (corrosion potential, and potentiodynamic polarization) flag shaped specimens with sufficiently long tail were cut from the mild steel sheet. These samples were polished as described earlier leaving a working area of 1cm<sup>2</sup> on both sides of the flag and a small portion at the tip for providing electrical contact. Rest of the surface was coated with enamel lacquer including side edges. The test specimen was connected to the working electrode holder through the tip of the tail. About 50ml of the corrosive medium was taken in a mini corrosion testing electrochemical cell. This volume was appropriate to permit desired immersion of electrodes.

Electrochemical Measurement System, DC 105, containing software of DC corrosion techniques from M/S Gamry Instruments Inc., (No. 23-25) 734, Louis Drive, Warminster, PA-18974, USA has been used for performing corrosion potential and polarization experiments. The electrochemical studies were performed in a three electrodes Pyrex glass vessel with mild steel coupon as working electrode, saturated calomel electrode as reference electrode and spectroscopic grade graphite rod as counter electrode.

## 2.4 SEM and EDX analysis

The composition and surface morphology of corrosion product on mild steel sample after 24 hours immersion in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of 250 ppm of cetirizine respectively, was studied by a scanning electron microscope and EDX examination using energy dispersive spectrometer Hitachi S-3400 N with thermo-noran EDS analyzer. The accelerating voltage for SEM picture was 15.0 KV.

## 3. Results and discussion

### 3.1 Weight loss study

The data of surface coverage ( $\theta$ ) and inhibition efficiency (% IE) obtained by weight loss method at different concentration of cetirizine in 1M H<sub>2</sub>SO<sub>4</sub> at 30 °C after 24 hours of immersion time are recorded in Table 1.

Inhibitor	Concentration (ppm)	Weight loss (mg.)	Surface Coverage ( $\theta$ )	Inhibition efficiency (%IE)
Blank	-	168	-	-
Cetirizine	500	13	0.9226	92.26
	250	24	0.8571	85.71
	100	28	0.8333	83.33
	50	31	0.8154	81.54
	30	41	0.7560	75.60
	10	51	0.6964	69.64

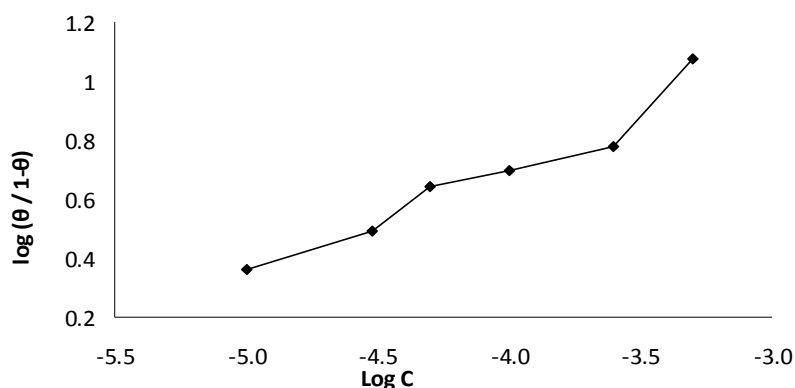
**Table 1:** Gravimetric data for inhibition of corrosion of mild steel exposed to 1M H<sub>2</sub>SO<sub>4</sub> with different concentrations of cetirizine.

Inhibition efficiency was calculated using the relation [26]:

$$(\% \text{ IE}) = \frac{(W_0 - W_{\text{corr}})}{W_0} \times 100 \quad \text{----- (1)}$$

$W_{\text{corr}}$  and  $W_0$  are the corrosion rates of steel with and without inhibitors, respectively.

Corrosion rate of mild steel decreases with increase in inhibitor concentration. Percentage inhibition efficiency (%IE) increases with increasing concentration. Fig. 2 shows the effect of inhibitor concentration on %IE for 24 hours immersion at 30 °C for inhibitor studied. Maximum inhibition efficiency was shown at 500 ppm concentration of cetirizine in 1M  $\text{H}_2\text{SO}_4$  at 303 K. The plot of  $\log c$  vs.  $\log (\theta/1-\theta)$  shows that adsorption phenomenon follows Langmuir adsorption isotherm which reveals chemisorptions of inhibitor molecules on mild steel surface can be seen. The results obtained by weight loss study were in good agreement with electrochemical data.



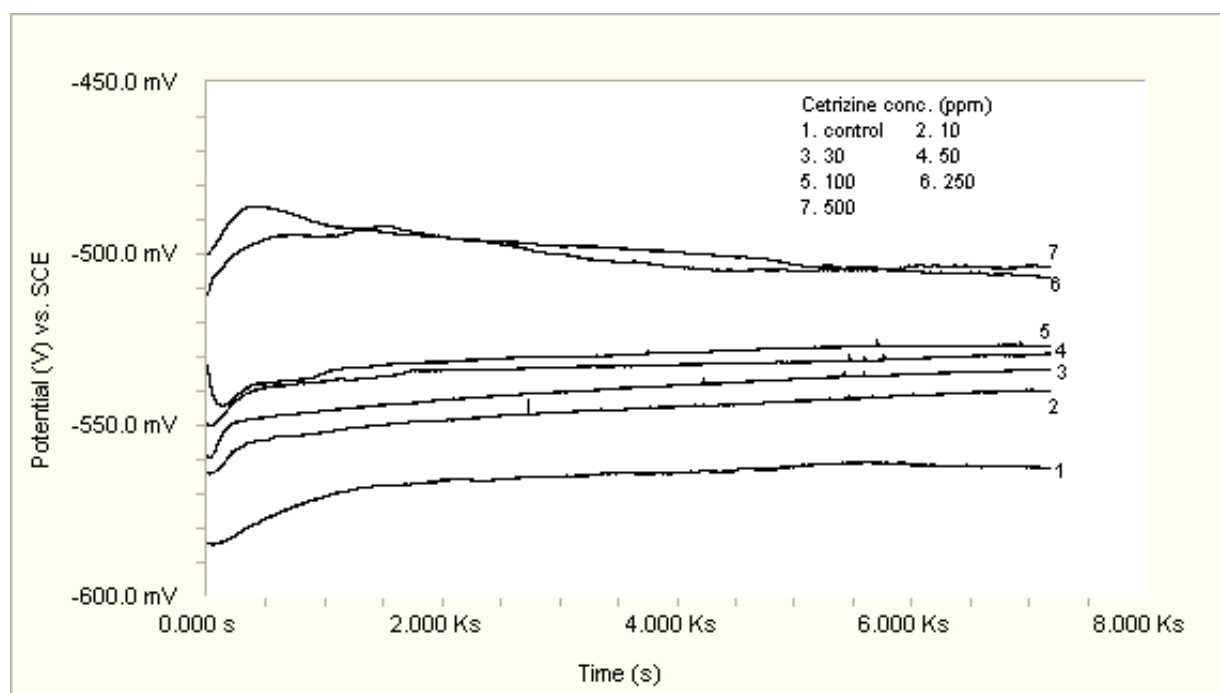
**Fig. 2:** Adsorption isotherm plot for cetirizine on mild steel in 1M  $\text{H}_2\text{SO}_4$ .

### 3.2 Open circuit potential measurement (OCP)

The electrochemical behavior of mild steel in 1M  $\text{H}_2\text{SO}_4$  was studied on the basis of change in corrosion potential ( $E_{\text{corr}}$ ) with time. The change in open circuit potential of mild steel in absence and presence of various concentration of cetirizine in 1 M  $\text{H}_2\text{SO}_4$  is shown in fig.3. The change in OCP of mild steel in absence and presence of inhibitors were measured for period of two hours with sample

period of one data per second. The potential attains steady state after exposure of approximately 30 minutes. The steady state potential is observed at an equilibrium state at which  $I_{ox}$  is equal to  $I_{red}$ . It has been observed that OCP of mild steel from moment of immersion in 1M  $H_2SO_4$  tends towards more negative value in absence of inhibitor. This shows corrosiveness of medium which is due to breakdown of pre-immersion, air formed oxide film on the metal surface. In the presence of various concentrations of inhibitors, the steady state potential of mild steel shifts more towards positive value. This is due to adsorption of inhibitors on metal surface resulting in passivation of metal.

The influence of various concentration (10, 30, 50, 100, 250, and 500 ppm) of cetirizine on OCP of mild steel in 1M  $H_2SO_4$  is given in fig.3. It is obvious from the figure that, it exhibit good inhibition performance at concentration 100 ppm and above. In this case inhibition efficiency increases with increase in concentration of inhibitor.



**Fig. 3:** Corrosion potential of mild steel exposed to 1M  $H_2SO_4$  solution with different concentrations of cetirizine.

### 3.3 Potentiodynamic polarization measurement

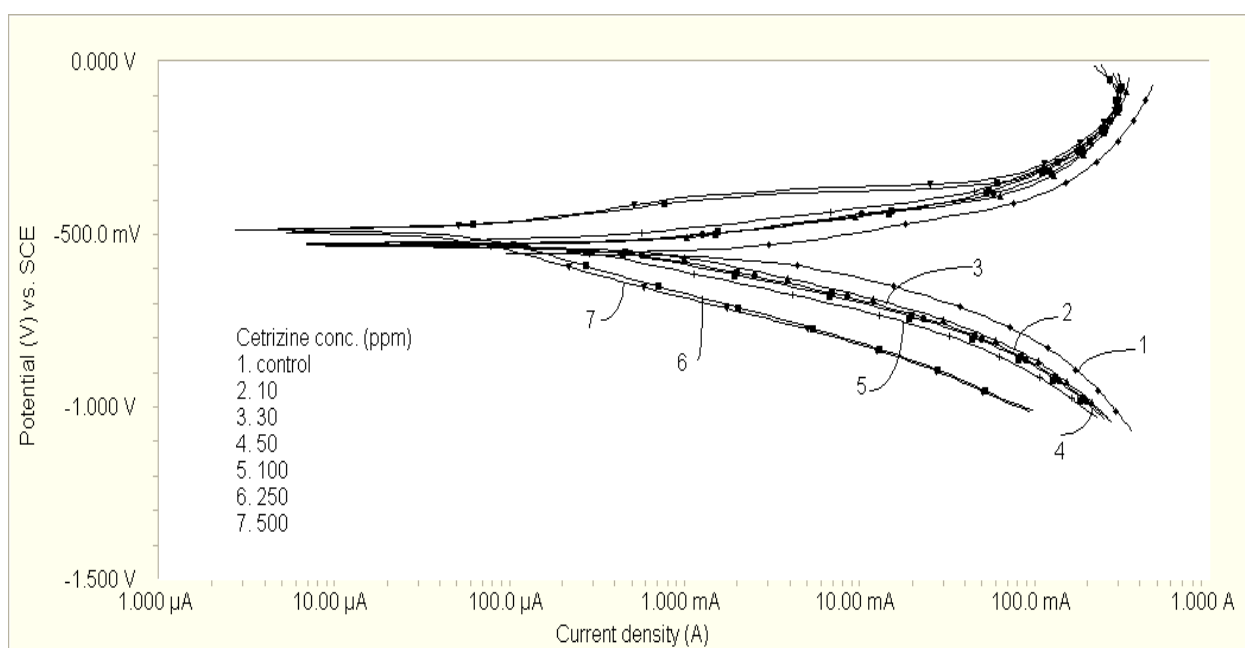


The corrosion potential ( $E_{\text{corr}}$ ), corrosion current density and anodic and cathodic slopes are obtained by the anodic and cathodic regions of the Tafel plots. The corrosion current density can be obtained by extrapolating the Tafel lines to the corrosion potential [27] and the corrosion inhibition efficiency was calculated from the electrochemical relation [28, 29]:

$$\text{Inhibition efficiency (IE \%)} = 100 (i_0 - i) / i_0 \quad \dots (2)$$

Where  $i_0$  and  $i$  are the corrosion current densities in the absence and presence of inhibitor in the solution respectively.

It is shown in fig. 4 that increasing cetrizine concentration in aggressive solution reduces both cathodic and anodic current densities. Cetrizine can be considered as mixed type of inhibitor. It means addition of cetrizine reduces anodic dissolution of mild steel and also retards cathodic hydrogen evolution.



**Fig. 4:** Potentiodynamic polarization curves of mild steel exposed to 1M  $\text{H}_2\text{SO}_4$  with different concentrations of cetrizine.

The values of electrochemical parameters and percentage of inhibition efficiency etc. determined from these experiments are summarized in Table 2.



Conc.	$\beta_a$	$\beta_c$	I <sub>corr</sub>	E <sub>corr</sub>	Corr. Rate	% of Inhibition
(ppm)	(V/dec.)	(V/dec.)	( $\mu\text{A. cm}^{-2}$ )	(mV)	(mpy)	Efficiency
Blank	123.2e-3	176.2e-3	4110.0	-555.0	1878.0	----
<b>Cetirizine</b>						
500	62.2e-3	120.1e-3	212.0	-524.0	96.83	94.84
250	79.4e-3	138.1e-3	539.0	-532.0	246.4	86.87
100	78.1e-3	36.9e-3	601.0	-535.0	274.5	85.38
50	76.8e-3	133.8e-3	695.0	-533.0	317.5	83.09
30	80.8e-3	145.6e-3	865.0	-522.0	395.3	78.95
10	76.7e-3	136.3e-3	1110.0	-512.0	506.3	72.99

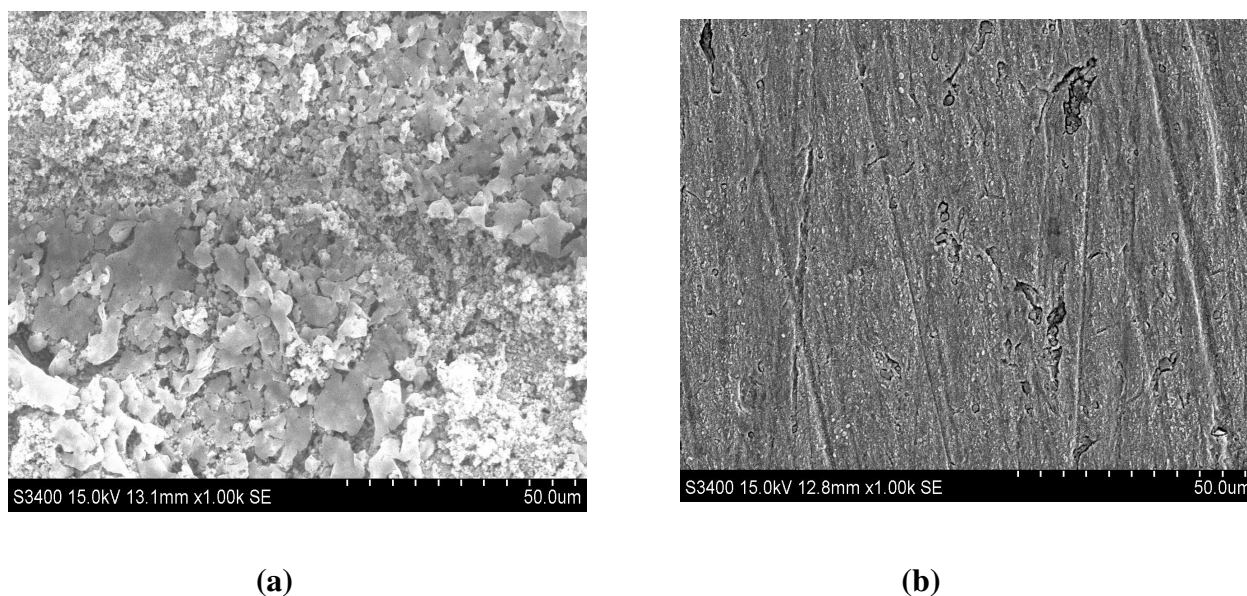
**Table 2:** Electrochemical parameters for inhibition of corrosion of mild steel exposed to 1M H<sub>2</sub>SO<sub>4</sub> with different concentration of cetirizine.

The corrosion rate decreases with increase in inhibitor concentration. The reduction of H<sup>+</sup> ions at the mild steel surface occurs through charge transfer mechanism [30]. It is evident from parallel cathodic Tafel line that hydrogen evolution mechanism is not modified by addition of inhibitor to 1M H<sub>2</sub>SO<sub>4</sub> solution but is activation controlled [31]. Effect of cetirizine on the kinetics of hydrogen evolution is indicated by the change in value of  $b_c$ . The shift of anodic tafel slope ( $b_c$ ) may be due to adsorption of inhibitor molecule or sulphate ion leading to formation of protective film on mild steel surface [32,33]. It seems from the anodic polarization curve (fig. 4) that at potential higher than -300 mV/SCE, inhibitor did not show corrosion inhibition effect. This potential can be defined as desorption potential [34-36]. This behavior could be significant dissolution of mild steel. The dissolution of mild steel leads to desorption of adsorbed film of inhibitor on the mild steel surface.

The increase in corrosion current with increase in potential shows that desorption rate of inhibitor film is higher than its adsorption rate.

### 3.4 SEM and EDX examination of mild steel surface

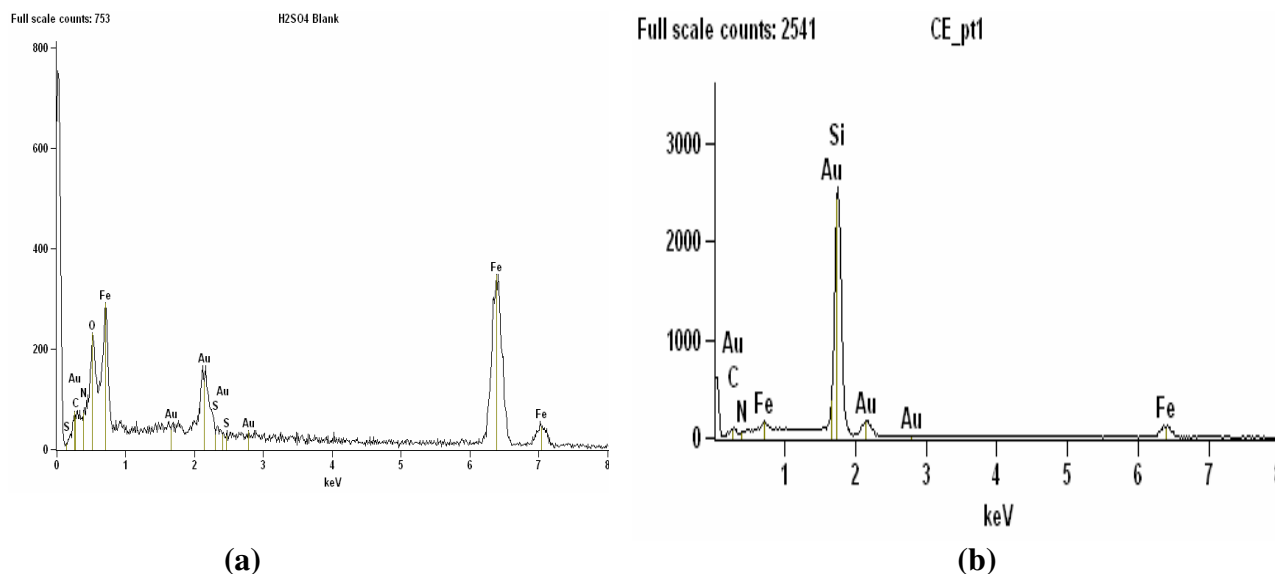
The results obtained from weight loss and electrochemical methods were further supported by SEM & EDX analysis. SEM micrographs obtained from mild steel surface after specimens immersed in 1.0 M  $\text{H}_2\text{SO}_4$  for 24 hrs. in the absence and presence of 250 ppm cetirizine are shown in Fig. 5a and b respectively. It could be visualized from Fig. 5a that the specimen surface was rougher and was damaged in the absence of the inhibitor. Fig. 5b shows SEM photographs of the mild steel surface after immersion in 1.0 M  $\text{H}_2\text{SO}_4$  containing 250 ppm cetirizine. It could be observed that extent of damage to mild steel surface is very less. The rate of corrosion was reduced considerably in the presence of inhibitors; it may be contributed to the presence of protective film adsorbed on metal surface, which act as a barrier and was responsible for the inhibition of corrosion.



**Fig. 5:** SEM micrographs of mild steel samples (a) after immersion in 1M  $\text{H}_2\text{SO}_4$  solution without inhibitor, (b) after immersion in 1M  $\text{H}_2\text{SO}_4$  solution in presence of 250 ppm cetirizine.

EDX spectra recorded for mild steel samples exposed to 1M  $\text{H}_2\text{SO}_4$  in the absence and presence of 250 ppm cetirizine is shown in Fig.6a-b. In inhibitor containing solution the EDX spectra

(fig. 6b) shows signal for existence of N and in addition intensity of C, enhanced appreciably. The appearance of N signal and enhancement of C signal are due to adsorption of N and C of cetrizine molecule. EDX data shows that carbonaceous organic molecule containing N-atom has formed a protective covering. The N signal and high intensity of C signal are not observed in EDX spectra (fig. 6a) of mild steel surface exposed to 1M H<sub>2</sub>SO<sub>4</sub> without inhibitor.



**Fig. 6:** EDX of mild steel samples (a) after immersion in 1M H<sub>2</sub>SO<sub>4</sub> solution without inhibitor, (b) after immersion in 1M H<sub>2</sub>SO<sub>4</sub> solution in presence of 250 ppm cetrizine.

It is also clear from Fig. 6a-b that Fe peaks are considerably suppressed in the samples containing cetrizine. This suppression of Fe line is due to presence of inhibitor film on the surface of mild steel. The result from EDX also confirms gravimetric and polarization measurement which suggests that corrosion inhibition is due to formation of protective surface film resulting in retardation of hydrogen evolution reaction.

#### 4. Conclusions

Corrosion inhibition studies of mild steel in 1M H<sub>2</sub>SO<sub>4</sub> solution using cetrizine as inhibitor studied by gravimetric, polarization, SEM and EDX techniques shows following conclusions:

1. Cetrizine is a good inhibitor for mild steel in 1M H<sub>2</sub>SO<sub>4</sub> solution.

2. The inhibitor efficiency increases with concentration of inhibitor to attain ~ 95 % inhibition at 500 ppm.
3. The adsorption of cetirizine on mild steel surface obeyed Langmuir adsorption isotherm.
4. All the cathodic curves of cetirizine appeared as Tafel lines indicate that hydrogen evolution reaction occurs through pure mechanism of activation.
5. SEM and EDX examination of mild steel surface showed that protective surface film is formed on metal surface which inhibits metal dissolution in  $H_2SO_4$  and retards hydrogen evolution. (Mixed-type inhibitor)
6. Cetirizine acts as mixed type inhibitors.

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