RESEARCH PAPER

# Electrochemical Investigations on Corrosion Inhibition Performance of an Ecofriendly expired Drug on Corrosion of Mild Steel in Acid Media

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## ABSTRACT

The study investigated expired fenbendazole drug as a green corrosion inhibitor for mild steel in 1.0 M HCl and 0.5 M $H_2SO_4$. This was done using weight-loss, electrochemical impedance spectroscopy, polarization, adsorption isotherm, and potential of zero charge studies. The inhibitor showed efficient corrosion inhibition in acid media with maximum inhibition efficiency values of 96 and 94% in HCl and H2SO4 respectively at 5.0 mM. The electrochemical findings demonstrated that the charge transfer resistance increased, while the corrosion current density decreased as a result of the adsorption of the inhibitor molecules on the surface of the steel. The Langmuir isotherm model was followed for the adsorption. The adsorption was spontaneous with mixed adsorption behaviour from the results. The study results show that the expired Fenbendazole can prove to be an effective, economic as well as eco-friendly corrosion inhibitor for mild steel.

**Keywords**: *Expired Fenbendazole; Mild Steel Corrosion; Acid Corrosion Inhibition; Electrochemical Impedance Spectroscopy; Langmuir Adsorption Isotherm.*

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## Introduction

The low cost of mild steel, good mechanical strength, and adaptability contribute to its extensive use in industrial applications. Corrosion is highly likely in acidic conditions such as pickling, acid cleaning, and oil-well acidizing [1]. The material can become weaker and result in unsafe situations or monetary loss [2]. The efficiency of traditional corrosion inhibitors like chromates, heavy metal salts is high, but they are toxic and harmful to the environment [3]. Consequently, the focus turned towards designing inhibitors that are eco-friendly and are effective, cheap and less hazardous[4]. Among these materials, expired pharmaceutical drugs have been especially recognized as effective green corrosion inhibitors [5]. Numerous drug molecules possess heteroatoms including N, O and S besides the aromatic structures and polar functional groups which, can adsorb on mild steel surface, block the active sites of corrosion, inhibit the anodic and cathodic reactions, and reduce the corrosion rate [7]. Moreover, repurposing expired pharmaceuticals is seen as a value-added approach to waste management in terms of alleviating concern for disposal and lower inhibitor development costs. Techniques for electrochemical and surface characterization are quite useful for establishing adsorption behavior and for determining the nature of protective films formed on metals [9]. Furthermore, it is expected that the use of computational studies such as density functional theory (DFT) and molecular dynamics simulations will become increasingly common, alongside experimental studies, to evaluate the electronic factors of corrosion inhibitors [10]. Research has shown that expired drug substances inhibited high recovery efficiencies (80–95%) in acidic environments (HCl and $H_2SO_4$) as reported in many studies [5,11] Antibiotics and antifungal drugs after expiry, for example, can form stable adsorbed layers on mild steel in 1M HCl solutions [12].

Different mechanisms are proposed for physical and chemical adsorption. The adsorption behaviour often fits either Langmuir or Temkin isotherms, depending on the system [13]. The inhibition mechanism is from donation of lone pairs to iron atoms, $\pi$–electron interaction with the metal surface and protective film formation, which improves the charge transfer resistance [14]. The difference between physisorption and chemisorption can be established from the temperature studies and activation energy measurements [15]. Under certain circumstances, inhibition performance can be further enhanced by a synergistic effect of additives, halo ions or surfactants, and the like.

It is also important to look at what environmental and lifecycle issues might exist in this performance assessment. The reuse of plastic as active pharmaceutical ingredients can curtail the generation of pharmaceutical waste, although they must be evaluated before large-scale use [17]. Moreover, while implementing industrially, regulatory compliance and safety considerations such as handling procedures, leaching possibilities and degradation by-products formation are crucial [18]. While progress has been promising, a number of practical problems exist such as standardization of testing methods, transferring lab-scale findings to industrial conditions, and assessing long-term stability of inhibitors films [19]. Advances in the future could be in the area of integration of green formulation strategies with computational screening methods for an efficient and quicker way to identify in highly effective expired drug-based inhibitors [20].

Overall, expired pharmaceuticals could overcome mild steel corrosion in acidic medium sustainable and cost-effective way. Wider adoption will, however, require thorough experimental validation, understanding mechanisms of action and environmental risk assessment.

Given how expired Fenbendazole (methyl N-(6-phenylsulfanyl-1H-benzimidazol-2-yl) carbamate) is a broad-spectrum benzimidazole anthelmintic and a veterinary drug, this study focused on it. Notably, Fenbendazole is useful in treating parasitic worm infections. Additionally, it is reported to have antiproliferative property apart from its anti-parasitic action. The compound has been documented to have low toxicity and a high safety profile in animals [21]. Fenbendazole is effective against Giardia as well as roundworm, hookworm, whipworm, pinworm, Aelurostrongylus, Strongyloides, strongyles, Paragonimus and Taenia (tapeworm) except for Dipylidium caninum, the common dog tapeworm. Fenbendazole has a wide range of therapeutic uses and is often given to sheep, cattle, horses, dogs, cats and a range of rabbits and other experimental animals [21]. The molecular structure of the drug is shown in figure 1.

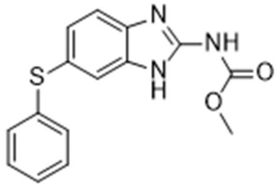

**Figure.1 Structure of Fenbendazole.**

## 2. EXPERIMENTS

### 2.1 Composition of the test material

The mild steel used in this investigation had the following elemental composition (wt.%): 0.086 C, 0.039 Mn, 0.027 P, 0.022 S, with the remaining balance consisting of Fe.

### 2.2 Preparation of the specimen

For gravimetric measurements, mild steel specimens of dimensions 2.5 × 1 × 0.1 cm were used. Prior to analysis, the specimens were mechanically polished using emery papers of different grades (220, 400, 600, 800, and 1200) to obtain a smooth surface, followed by rinsing with double-distilled water. The polished samples were then degreased using acetone, dried, and preserved in a desiccator until use. Electrochemical experiments were performed using a Teflon-coated cylindrical mild steel rod with an exposed surface area of 1 cm² as the working electrode. After each experimental run, the test electrode was cleaned following the same surface preparation procedure. All test solutions used throughout the study were prepared using double-distilled water.

### 2.3 Preparation of acid solutions

Solutions of HCl and $H_2SO_4$ were prepared by diluting analytically pure concentrated acids to 1000 mL using double-distilled water in a standard volumetric flask. The normality of the prepared acid solutions was determined by titration against a standard sodium carbonate solution. The inhibitor solutions were then prepared using these standardized acidic media.

### 2.4 Preparation of inhibitor solutions

Fenbendazole was prepared as a stock solution from commercially available tablets following the procedure reported [21]. To enhance solubility, 3% DMSO was added as a solvent. The stock solution was then diluted appropriately to obtain the desired inhibitor concentrations for the study.

### 2.5 Weight loss measurements

Weight loss measurements were carried out according to ASTM standard procedures to evaluate corrosion behavior and the performance of the corrosion inhibitor [22]. Pre-treated mild steel specimens, prepared in triplicate, were immersed in 100 mL of 1M HCl and 0.5M $H_2SO_4$ solutions containing different concentrations of the inhibitor at 30 ± 1°C. After the immersion period, the specimens were removed, cleaned, dried, and reweighed. The average weight loss values were used to calculate the corrosion rate, and the inhibition efficiency was determined using the following relationship [23].

$$\mathrm{IE}(\%) = \frac{\mathrm{W_{BS}} - \mathrm{W_{IS}}}{\mathrm{w_{BS}}} \times 100$$

Where, IE is the efficiency of the inhibitor, WBS is the weight loss in an inhibited solution and WIS is that of weight loss in an inhibited solution.

### 2.6 Electrochemical impedance spectroscopy

Electrochemical investigations were performed using a potentiostat/galvanostat (Zahner Zennium XC Superior Electrochemical Workstation). A conventional three-electrode cell system was employed, consisting of a Teflon-coated mild steel rod with an exposed area of 1 cm² as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and a platinum mesh as the counter electrode. Electrochemical impedance spectroscopy (EIS) measurements were carried out at open circuit potential after 30 minutes of immersion in the acidic medium, using an AC sinusoidal perturbation of 10 mV over a frequency range from $10^6$ to $10^{-2}$ Hz. The inhibition efficiency was calculated from the charge transfer resistance (Rct) values obtained through equivalent circuit fitting, as shown below [24].

$$\% \text{ IE} = \frac{R'_{ct} - R_{ct}}{R'_{ct}} \text{ x } 100$$

Where $R'_{ct}$ is the charge transfer resistance in the inhibited solution, Rct is the charge transfer resistance in the uninhibited solution.

## 2.7 Polarization measurements

Potentiodynamic polarization measurements were carried out by scanning the potential from values more negative than the open circuit potential (Eocp) to values more positive than Eocp at a scan rate of 1.67 mV $s^{-1}$. The Tafel slopes, βa and βc, were determined from plots of E versus log i. Anodic and cathodic polarization curves were constructed, and the corrosion current density (icorr) and corrosion potential (Ecorr) were obtained from the intersection of the extrapolated Tafel lines. The inhibition efficiency was then calculated using Equation (3) [25].

$$\% \text{ IE} = \frac{i_{corr} - i'_{corr}}{i_{corr}} \text{ x } 100$$

Where icorr is the corrosion current density in uninhibited solution and $i'_{corr}$is the corrosion current density of inhibited solution.

## 2.8 Measurement of the potential of zero charge (PZC)

Adsorption may arise from electrostatic interactions between charged adsorbed species, ionic species in solution, and the electrical charge present at the metal–solution interface. The surface charge of a metal in solution can be evaluated relative to its potential of zero charge (PZC), which provides insight into interfacial charge characteristics. The potential difference between the metal surface potential and the zero-charge potential, often referred to as the φ-potential, is particularly significant in understanding adsorption behavior and may differ from the potential measured on the hydrogen scale. To assess the influence of surface charge on organic adsorption, Antropov introduced the concept of the potential difference between the corrosion potential and the PZC of a corroding metal. A more positively charged metal surface tends to favor the adsorption of anions, whereas a more negative φ-potential enhances the adsorption of cationic species. The impedance response of the corroding electrode was recorded at 100 Hz with an AC perturbation amplitude of 10 mV. The double-layer capacitance (Cdl) at different applied DC potentials was evaluated, and the potential of zero charge (PZC) was determined from plots of differential capacitance versus applied DC potential.

The adsorption of the inhibitor on the metal surface is strongly influenced by the surface charge at the interface. To assess the effect of this charge on adsorption behavior, the sign of Er was determined using the relationship proposed by Popova [26], as expressed below.

$$E_r = E_{ocp} - E_{PZC}$$

Where,

$E_{PZC}$ = Potential of zero charge

Er = Antropov's rational corrosion potential

$E_{OCP}$ = Open circuit potential

## 3.RESULTS AND DISCUSSION

### 3.1 Weightless measurements

Table 1 presents the weight loss corrosion data for mild steel in 1.0 M HCl and 0.5 M $H_2SO_4$ solutions in the absence and presence of expired Fenbendazole drug at different concentrations. The results obtained show that corrosion rate of mild steel decreases with the increasing concentration of the inhibitor and the inhibition efficiency progresses in both acidic media. This behavior confirms that the expired Fenbendazole has an efficient surface adsorption corrosion inhibiting effect for mild steel.

**Table.1 Findings of weight loss corrosion of mild steel in 1.0M HCl and in 0.5M H2SO4 acid solutions.**

| Medium | Inhibitor Concentration (mM) | Corrosion Rate (mpy) | Inhibition Efficiency(IE%) |
|---|---|---|---|
| 1.0 M HCl | Blank | 1267 | - |
| | 0.1 | 710 | 44 |
| | 0.5 | 469 | 63 |
| | 1.0 | 203 | 84 |
| | 2.0 | 114 | 91 |
| | 5.0 | 51 | 96 |
| | 10.0 | 76 | 94 |
| 0.5 M H2SO4 | Blank | 1488 | - |
| | 0.1 | 877.92 | 41 |
| | 0.5 | 610.08 | 59 |
| | 1.0 | 401.76 | 73 |
| | 2.0 | 282.72 | 81 |
| | 5.0 | 89.28 | 94 |
| | 10.0 | 119.04 | 92 |

In a 1.0 M HCl medium, the mild steel in the uninhibited solution exhibited a corrosion rate of 1267 mpy. The continuous addition of expired Fenbendazole resulted in a reduction in the corrosion rate as well as consistent improvement in the efficiency of the inhibitor. This eventually led to the corrosion rate reducing to 710, 469, 203, 114 and 51 mpy which corresponds to inhibitor concentrations of 0.1, 0.5, 1.0, 2.0 and 5.0 mM. As a result, when as much inhibitor is mixed (0.10 mM) in the media, the iron inhibition is only 44%. The inhibition efficiency slightly decreased from 95% to 94% when the concentration was raised to 10.0 mM and thus, under the present experimental conditions, optimum inhibitor concentration was noted to be 5.0 mM. A comparable pattern was noted in the 0.5 M $H_2SO_4$ solution. The corrosion rate decreased from 1488 mpy (blank solution) to 877.92, 610.08, 401.76, 282.72, and 89.28 mpy for 0.1, 0.5, 1.0, 2.0, and 5.0 mM inhibitor concentrations. The inhibition efficiency steadily improved from 41% to 94% at the same time. Somewhat similar to that of hydrochloric acid medium, the inhibition efficiency was found to decrease slightly at 10.0 mM concentration. Here, the efficiency reduced to 92% and corrosion rate increased slightly to 119.04 mpy. This behavior may be due to partial desorption or reorientation of inhibitor molecules at too high concentrations, thus destabilizing the protective adsorbed film on the mild steel surface [27].

As the concentration increases, the efficiency of the inhibitor also increases; this can be explained on the basis of adsorption. With the increase in the concentration of expired Fenbendazole, more number of inhibitor molecules get adsorbed on to the metal surface increasing the surface coverage and blocking the active corrosion sites. Inhibitor molecules that are adsorbed to the surface of metals form a compact protective barrier which isolates the metal from the aggressive acidic medium, thus inhibiting corrosion.

The ability of expired Fenbendazole to inhibit corrosion can be ascribed to its molecular structure, which has heteroatoms (N, S, O) and aromatic rings with π-electrons. The active adsorption centers from these structural features enable the strong interaction of mild steel. The combined physical and chemical adsorption may be the inhibition mechanism.

The inhibitor molecules may attach themselves to the surface of mild steel through electrostatic attraction between protonated inhibitor species and charged mild steel surface in acid medium. Alternatively, chemisorption can also take place because of lone pair electrons present on the heteroatoms (especially, nitrogen and sulfur) that coordinate on vacant d-orbitals of iron. In addition, the π-electrons on the aromatic rings may interact with the metal surface via π-d interactions which might increase the stability and compactness of the protective film on the mild steel surface.

The inhibitors used showed a comparatively higher inhibition efficiency in 1.0 M HCl solution than in 0.5 M $H_2SO_4$ solution in all concentrations studied. This difference is likely due to the type of anions present in the acid medium [26,28]. Chloride ions facilitate adsorption of protonated organic inhibitor molecules by forming intermediate ion-pair interactions, enhancing their adsorption and improving surface coverage at the metal surface [26,29-31]. Conversely, sulfate ions have greater ionic size and more powerful hydration properties, reducing their capacity to aid adsorption of inhibitor species. As a result, there were found to be slightly lower inhibition efficiencies in sulfuric acid medium [26].

The adsorbed expired Fenbendazole molecules form a protective film which suppresses anodic iron dissolution and the cathodic hydrogen evolution reactions. The high inhibition efficiencies observed suggest that the inhibitor effectively reduces the active surface area. The observed marginal decrease in efficiency for concentrations beyond 5.0 mM indicates that the excessive build-up of inhibitor molecules may lead to repulsion between molecules, partial desorption, or obstruction in the orientation of the adsorbed species causing weakening of the protective layer.

The outcome of weight-loss experimentation proved that expired Fenbendazole drug acts as an efficient corrosion inhibitor for mild steel in HCl and H2SO4 media. The outstanding inhibition performance of the inhibitor at 5.0 mM indicates its strong tendency to adsorb strongly and protect the surface of mild steel.

### 3.2 Electrochemical Impedance Spectroscopy

The Nyquist impedance spectra of mild steel in 1.0 M HCl and 0.5 M H2SO4 are shown in Figures 2a and 2b, respectively, in the absence and presence of varying concentrations of expired Fenbendazole drug. The electrochemical impedance parameters acquired from the fit of the experimental data to an equivalent circuit model are collated in Table 2. Electrochemical impedance spectroscopy is a powerful technique for probing corrosion process and inhibition action at the metal/electrolyte interface because it allows the separation of electrochemical phenomena taking place at the wide frequency range [26].

The Nyquist plots in acidic media show depressed capacitive semicircles, indicating that the corrosion process is mainly controlled by charge transfer reactions at the surface of mild steel. The semicircles that are depressed in nature reveals that the electrode is non-ideal capacitor which could be due to surface roughness, heterogeneous nature of the electrode surface, non-uniform current distribution and adsorption of the inhibitor molecules [26]. To obtain a more precise fitting to experimental data, a constant phase element (CPE) was employed in the equivalent circuit analysis instead of an ideal capacitor[26, 32-34] .

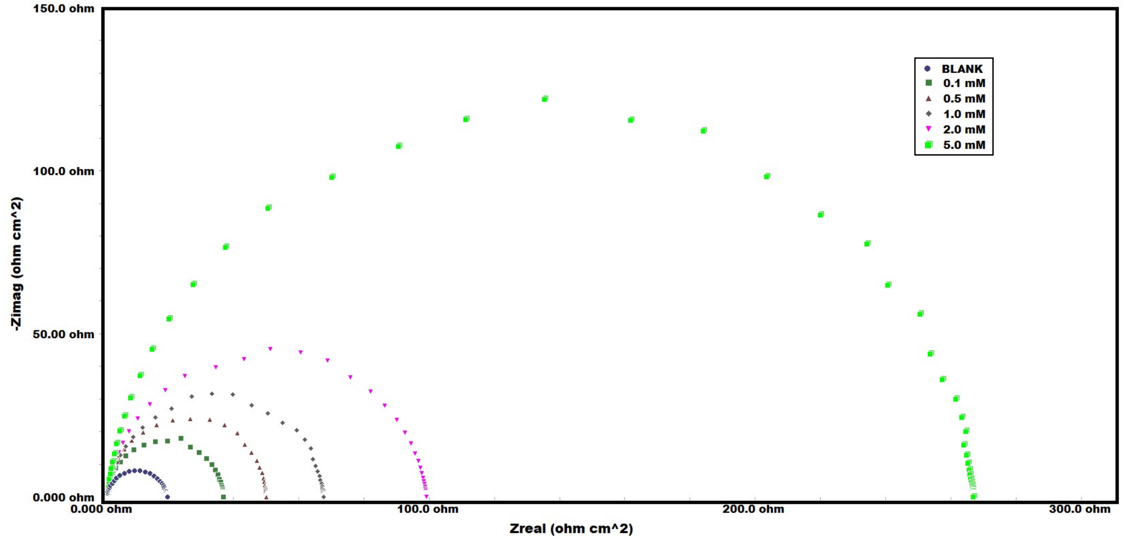

**Figure.2a Experimental Nyquist curves of corrosion of mild steel in the absence and the presence of Fenbendazole at various concentrations in 1.0M HCl solution.**

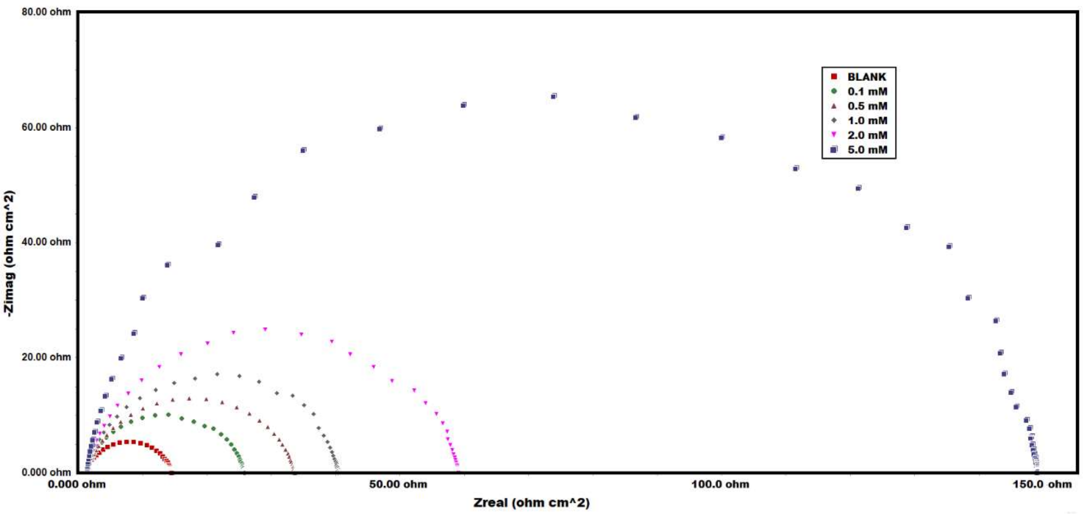

**Figure.2b Experimental Nyquist curves of corrosion of mild steel in the absence and the presence of Fenbendazole at various concentrations in 0.5M H2SO4 solution.**

The equivalent circuit employed for fitting the impedance spectra consists mainly of solution resistance (Rs), charge transfer resistance (Rct), and a constant phase element (CPE). The charge transfer resistance reflects the resistance offered to electron transfer across the metal/solution interface, whereas the CPE parameters provide information regarding surface roughness and adsorption behavior of inhibitor molecules [35].

**Table.2 Electrochemical impedance characteristics of corrosion of mild steel in 0.1M HCl and in 0.5 M H2SO4 acidic solution in the absence and the presence of Fenbendazole at various concentrations.**

| **S.NO** | **Medium** | **Concentration (ppm)** | **Rct (Ω cm-2)** | **Y0 (X10-6 Ω-1cm-2)** | **n** | **Rs** | **%IE** |
|---|---|---|---|---|---|---|---|
| 1 | 1.0M HCI | Blank | 18.6 | 88.37 | 0.911 | 1.112 | - |
| 3 | | 0.1 | 35.8 | 64.27 | 0.970 | 1.116 | 48 |
| 4 | | 0.5 | 48.9 | 53.66 | 0.985 | 1.225 | 62 |
| 5 | | 1.0 | 66.4 | 48.37 | 0.932 | 1.301 | 72 |
| 6 | | 2.0 | 97.9 | 39.24 | 0.928 | 1.314 | 81 |
| 7 | | 5.0 | 265.7 | 22.51 | 0.924 | 1.334 | 93 |
| 8 | 0.5M H2SO4 | Blank | 13.3 | 110.2 | 868.0 | - | - |
| 10 | | 0.1 | 24.6 | 90.64 | 878.0 | 1.227 | 46 |
| 11 | | 0.5 | 32.4 | 81.52 | 862.4 | 1.314 | 59 |
| 12 | | 1.0 | 39.1 | 76.65 | 894.9 | 1.311 | 66 |
| 13 | | 2.0 | 57.8 | 70.40 | 889.6 | 1.329 | 77 |
| 14 | | 5.0 | 147.8 | 64.8 | 923.1 | 1.338 | 91 |

As the concentration of expired Fenbendazole increased, the diameter of the capacitive semicircle in 1.0 M HCl medium increased which revealed an increase in corrosion resistance and adsorption of the inhibitor on mild steel surface. The charge transfer resistance increased significantly from 18.6 Ω cm$^2$ for the blank solution to 265.7 Ω cm$^2$ at the concentration of 5.0 mM. The efficiency of inhibition correspondingly increased from 48% at 0.1 mM to 93% at 5.0 mM. The significant rise in Rct values indicates that the inhibitor is effective as it reduces the electron transfer between metal and corrosive medium and hence the corrosion reaction [26].

Simultaneously, the admission parameter ($Y_0$) decreases from 88.37 × 10-6 Ω-1 cm-2 for the blank solution to 22.51 × 10-6 Ω-1 cm-2 at 5.0 mM concentration. The $Y_0$ value reduced because of the adsorption of inhibitor molecules at the metal/solution interface [26]. This indicates a lower double layer capacitance. Adsorption of the expired Fenbendazole displaces water molecules and aggressive chloride ions in the electrical double layer, thus decreasing the local dielectric constant, while conversely enhancing the thickness of the protective interfacial layer [26].

The values of n yielded from the fittings of CPE close to 1 (between 0.911 and 0.985) suggest that electrode surface is almost capacitive in nature. As the concentration of the inhibitor increases, slight enhancement in surface homogeneity of mild steel is noticed which is supported with the slight increase in the n values [26].

The Nyquist plots also show depressed capacitive semicircles in 0.5 M $H_2SO_4$ medium; indicating corrosion processes controlled by charge transfer [36]. The blank solution had a relatively lower charge transfer resistance of 13.3 O cm2 which indicates sulfuric acid provides highly aggressive corrosive medium for mild steel. The Rct values progressively increased to 24.6, 32.4, 39.1, 57.8 and 147.8 Ω cm², when 0.1, 0.5, 1.0, 2.0 and 5.0 mM expired Fenbendazole were added subsequently. Inhibition efficiency steadily increased from 46% to 91%.

The increase in charge transfer resistance reveals that inhibitor molecules get adsorbed strongly on the mild steel surface and block active corrosion sites effectively. The $Y_0$ values decreased continuously with increasing concentration of inhibitor like in HCl medium indicating the formation of a protective adsorbed film and decrease in interfacial capacitance [37]. The reduced capacitance may be the result of inhibitor molecule displacing water molecules and sulfate ions, thus leading to the formation of a thicker electrical double layer [26].

The efficiency of inhibition of expired fenbendazole was observed to be slightly better in HCl medium than $H_2SO_4$ medium. The distinction may be explained by the aggressive anions present in the acid solutions. According to previous studies, chloride ions can increase the adsorption of protonated organic inhibitor molecules through intermediate ion-pair formation between chloride ions and organic inhibitor molecules [26]. It also enhances the surface coverage on the mild steel surface. In contrast to chloride despite having a greater charge-to-radius ratio sulfate is larger and much more heavily hydrated and the presence of sulfate reduces adsorption of other molecules which results in a lesser corrosion inhibitor efficiency of sulfate than chloride [38].

The expired Fenbendazole effectively inhibits various metal ions due to the presence of nitrogen, sulfur, and oxygen heteroatoms with aromatic π-electron systems in the molecule [39]. The mild steel surface is able to interact strongly with the active adsorption centers that these structural features possess. Chemisorption involves the donation of lone pair electrons from the heteroatom to the vacant d-orbital of the iron atom. In addition, the protonated inhibitor molecules can adsorb onto the charged surface of the metal through electrostatic attraction [26,39]. Additionally, interactions between the π–d orbitals of the aromatic rings and the metal surface stabilize the protective layer. When an inhibitor is added to a corrosive environment, it forms a film on the surface of the metal which helps to block penetration of the corrosive into the metal [40]. It reduces the anodic dissolution of Fe2+ and subsequent cathodic evolution of H2 [26].

The Rct values are observed to continuously increase with decreasing Y0 confirming that corrosion inhibition takes place through adsorption of expired Fenbendazole on the mild steel surface. Overall, Fiducial studies reveal that the expired Fenbendazole is an effective corrosion inhibitor for mild steel in hydrochloric acid and sulfuric acid media. The inhibitor offers improved shielding ability in HCl solution because of stronger adsorption interactions along with the increased surface coverage. The presence of a dense and stable protective film considerably hampers charge transfer processes and improves the corrosion resistance of mild steel in aggressive acidic conditions.

### 3.3 Potentiodynamic Polarization Studies

The potentiodynamic polarization curves of mild steel in 1.0 M HCl (Fig. 3a) and 0.5 M $H_2SO_4$ solutions (Fig. 3b) in absence and presence of expired Fenbendazole drug at different concentrations. The electrochemical parameters including corrosion potential, Ecorr, corrosion current density, Icorr, anodic Tafel slope, βa, cathodic Tafel slope, βc, and Inhibition efficiency, %IE, are summarized in Table 3. The polarization studies provide valuable information regarding the kinetics of anodic metal dissolution and cathodic hydrogen evolution reactions taking place on the mild steel surface in acidic media.

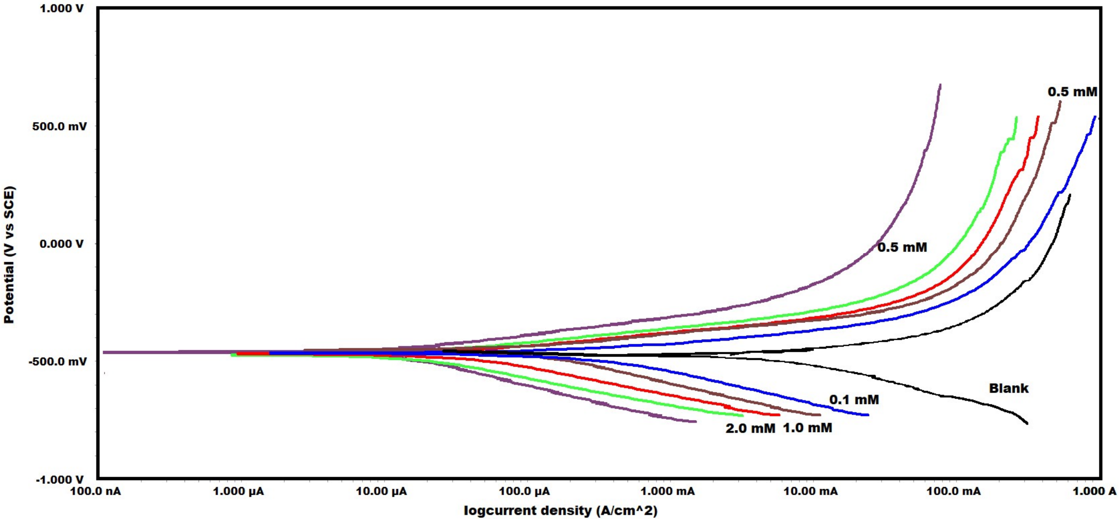

**Figure.3a Tafel plots of corrosion of mild steel during the absence and the presence of Fenbendazole in 1.0 M HCl solution.**

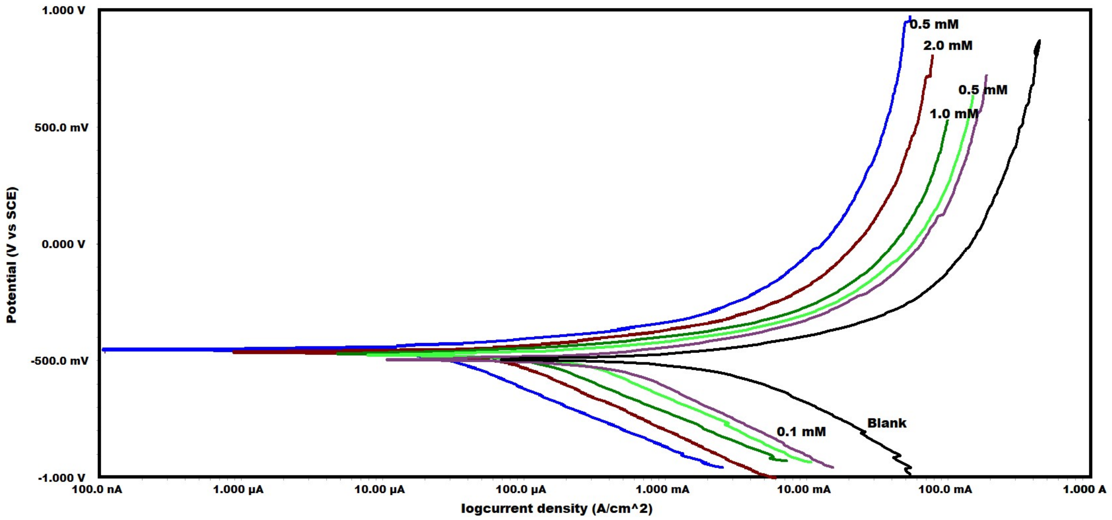

**Figure.3b Tafel plots of corrosion of mild steel during the absence and the presence of Fenbendazole in 0.5M H2SO4 solution.**

The polarization curves clearly indicate that the expired Fenbendazole addition significantly decreases the corrosion current density in the acidic solutions. The inhibition study confirms that the inhibitor inhibits the corrosion reaction at metal/electrolyte interface. In a 1.0 M HCl solution, the corrosion current density of uninhibited system was found to be 1712 µA cm$^{-2}$. After adding the inhibitor, the Icorr values decreased gradually to 1198, 924.5, 667.7, 530.7, 291.0, and 137.0 µA cm-2 at inhibitor concentrations of 10, 50, 100, 200, 300, and 400 ppm, respectively. With increasing inhibitor concentration, the inhibition efficiency steadily increased from 30% to 92%.

A similar trend was observed in 0.5 M $H_2SO_4$ medium. The blank solution exhibited a high corrosion current density of 2214 µA cm$^{-2}$, indicating severe corrosion of mild steel in sulfuric acid. In the presence of expired Fenbendazole, the corrosion current density decreased gradually to 1638, 1218, 952.0, 819.2, 531.4, and 243.5 µA cm$^{-2}$ as the inhibitor concentration increased from 10 to 400 ppm. At the same time, the inhibition efficiency increased from 26% to 89%. The reduction in Icorr values demonstrates that the inhibitor molecules adsorb effectively onto the mild steel surface and retard the corrosion process.

**Table.3 Potentiodynamic polarization parameters of corrosion of mild steel in 1.0 M HCl solution and 0.5 M H2SO4 acid solution in the presence and absence of Fenbendazole.**

| S. No | Medium | Concentration | βa (mV/dec) | βc (Mv/dec) | Icorr (mV/SCE) | Ecorr (mV/SCE) | %IE |
|---|---|---|---|---|---|---|---|
| 1 | 1.0M HCl | Blank | 179.2 | 188.6 | 1712 | -458.0 | - |
| 2 |  | 0.1 | 113.6 | 179.3 | 924.5 | -472.0 | 46 |
| 3 |  | 0.5 | 93.50 | 172.5 | 667.7 | -471.0 | 61 |
| 4 |  | 1.0 | 95.10 | 161.8 | 530.7 | -488.0 | 69 |
| 5 |  | 2.0 | 86.30 | 156.2 | 291.0 | -489.0 | 83 |
| 6 |  | 5.0 | 89.70 | 155.2 | 137.0 | -481.0 | 92 |
| 7 | 0.5M H2SO4 | Blank | 215.2 | 375.8 | 2214 | -480.0 | - |
| 8 |  | 0.1 | 155.3 | 331.9 | 1218 | -471.0 | 45 |
| 09 |  | 0.5 | 141.1 | 326.6 | 952.0 | -468.0 | 57 |
| 10 |  | 1.0 | 118.3 | 226.2 | 819.2 | -466.0 | 63 |
| 11 |  | 2.0 | 123.6 | 262.6 | 531.4 | -469.0 | 76 |
| 12 |  | 5.0 | 130.4 | 267.4 | 243.5 | -479.0 | 89 |

The inhibition efficiency in the medium of hydrochloric acid was slightly higher than in the medium of sulfuric acid for all concentrations studied. The reason for the difference could be the type of aggressive anions present in the acidic solution. The presence of chloride ions enhances the adsorption of protonated organic inhibitor molecules on the surface of mild steel through electrostatic interactions and ion-pair formation. This leads to better adsorption and surface coverage. Unlike sulphate ions, molecules with bigger ionic size and greater hydration reduce their capacity to promote the adsorption of inhibitor molecules leading to low inhibition efficiency.

The inclusion of the inhibitor in both acidic media causes only a minor shift in corrosion potential values (Ecorr). In the 1.0 M HCl solution, the Ecorr values which were / shifted / varied / modified / changed for the blank solution were −458 mV followed by −466 to −489 mV for the inhibited solution. Likewise, the Ecorr values in 0.5 M

$H_2SO_4$ solution varied only slightly from −480 mV to approximately −466 to −479 mV after addition of the inhibitor. Since the shift of corrosion potential is less than 85 mV in comparison to the blank solution, expired Fenbendazole can be classified as mixed type corrosion inhibitor. This indicates that the inhibitor inhibits the anodic iron dissolution reaction as well as the cathodic hydrogen evolution reaction.

The presence of inhibitor has changed anodic and cathodic Tafel slope values showing that the electrode kinetics got modified after the adsorption of the inhibitor molecule. As the concentration of inhibitor increased in hydrochloric acid medium, anodic Tafel slope decreased from 179.2 to 89.70 mV $dec^{-1}$. Also, cathodic slope decreased from 188.6 to 155.2 mV $dec^{-1}$. A similar change was noted when sulfuric acid was used. These variations demonstrate that the inhibitor molecules obstruct the metallic active sites involved in anodic as well as cathodic reactions.

The larger decrease in the cathodic current densities shows that the expired Fenbendazole has a relatively greater effect on the cathodic hydrogen evolution reaction. Nevertheless, the concurrent decrease in both the anodic and cathodic branches confirms the mixed-type inhibition of the inhibitor [26]. The mild steel surface eliminate exposure of metallic ion to acidic solution. While the complexation reaction with the metal ion may regenerate the inhibitor [26].

The expired Fenbendazole corrosion inhibition mechanism may be due to the presence of heteroatoms (N, S O) and the aromatic $\pi$- electron system on the Fenbendazole molecule. These structural characteristics here provide active centers of adsorption capable of exerting strong interactions with the mild steel surface [26,41]. Electrostatic attraction between protonated inhibitor molecules and charged metal surface in acidic medium and chemisorption due to the donation of lone pair electrons of heteroatoms to vacant d-orbitals of iron atoms may support adsorption. “Furthermore, the protective adsorbed film is stabilized by the $\pi$–d orbital interactions between the aromatic rings and the metal surface [26].”

As the concentration of inhibitor increases, the inhibition efficiency increases, suggesting that gradual surface coverage occurs. Inhibitors containing higher concentrations allow the formation of a more compact and adherent protective film on the metal surface to isolate the mild steel from acidic mediums. The presence of the adsorbed film slows down the corrosion rate, as it significantly restricts the diffusion of corrosive species towards the metal surface [26]. Totally, the potentiodynamic polarization result confirmed that expired Fenbendazole is an efficient corrosion inhibitor for mild steel in hydrochloric acid and sulfuric acid solution. The large drop in corrosion current density coupled with high inhibition efficiency values and slight shifts in corrosion potential indicates that the inhibitor works mainly through adsorption and acts generally as a mixed type corrosion inhibitor [26]. The better inhibition performance in HCl medium suggests stronger adsorption interactions, leading to more effective protective film formation in chloride-containing acidic environments.

### 3.4 Adsorption Isotherm

As depicted in Figures 4a and 4b, the adsorption isotherm graphs correspond to expired Fenbendazole on the surface of mild steel in 1.0 M HCl and 0.5 M $H_2SO_4$ respectively. Adsorption studies help in elucidating the mechanism of corrosion inhibition since the protective action of organic inhibitors mainly depends on their ability to adsorb on to the metal surface to form a barrier film that separates the metal from the corrosive acidic medium. The adsorption behavior gives information about the strength and nature of the interaction between the inhibitor molecules and mild steel.

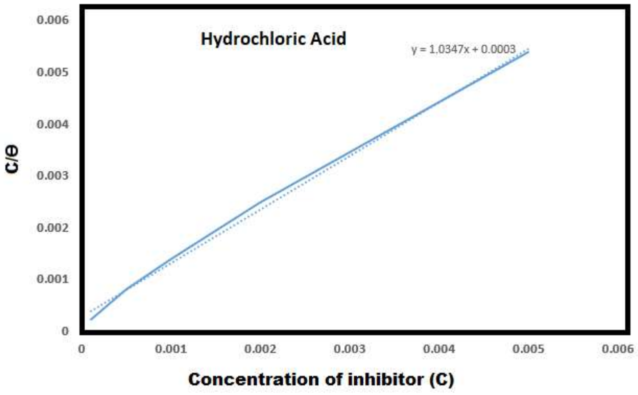

Figure.4a Adsorption isotherm for adsorption of Fenbendazole onto mild steel surface in 1.0 M HCl acid solutions.

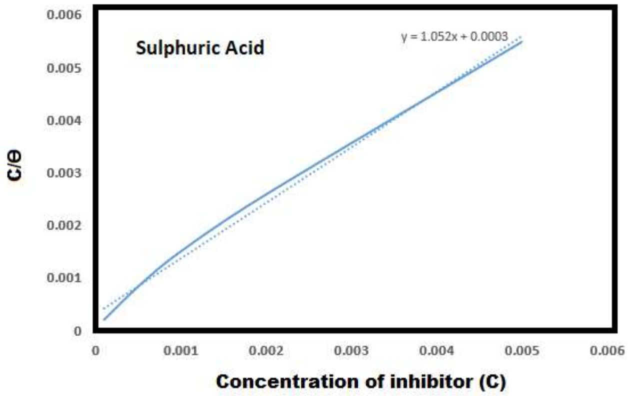

Figure.4b Adsorption isotherm for adsorption of Fenbendazole onto mild steel surface in 0.5 M H2SO4 acid solutions.

The Langmuir isotherm, which presumes a monolayer of inhibitor on a homogeneous surface of the metal, was used to analyze the variation of inhibitor concentration with surface coverage. Furthermore, the interaction between the adsorbed species is negligible. The Langmuir adsorption equation is given as.

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C$$

Where, C is the inhibitor concentration θ is the degree of surface coverage Kads is the adsorption equilibrium constant The isotherm plots of C/θ versus C obtained in both acid media were fairly linear with a slope close to unity confirming that the adsorption of expired Fenbendazole on mild steel surface obeys the Langmuir adsorption isotherm model [26]. The linear behavior suggests that the molecules of the inhibitor develop nearly a uniform monolayer on the mild steel surface and that the adsorption sites are practically of equal adsorption energy [26]. Absence of deviation from linearity indicates negligible interaction between adsorbed inhibitor molecules.

The relatively high values of adsorption equilibrium constant ($K_{ads}$) obtained from the Langmuir plots in both the acidic media indicate that expired Fenbendazole molecules have strong tendency to get adsorbed on the mild steel surface. The greater K value in hydrochloric acid medium as compared to sulfate acid medium indicates a greater interaction and adsorption of inhibitor molecules in HCl solution. The evidence from weight loss, impedance and polarization measurement which is in agreement.

The standard free energy of adsorption was determined by a relevant relation. Adsorption free energy can be derived from Langmuir adsorption isotherm using the equation:

$$\Delta G^{\circ}_{ads} = -RT\ln(55.5K_{ads})$$

R is the universal gas constant; T is the absolute temperature and 55.5 is the molar concentration of water in solution. The predicted negative values of $\Delta G^{\circ}_{ads}$show that the adsorption of expired Fenbendazole on the mild steel surface occurs spontaneously in both acidic environments. The free energy values indicate that the adsorption mechanism is a combination of physical and chemical adsorption. Typically, a value close to −20 kJ $mol^{-1}$ indicates an electrostatic interaction of the protonated molecule of the inhibitor and the charged metal surface, while a value close to −40 kJ $mol^{-1}$ indicates stronger chemical bonding involving electron sharing/transfer between the molecule of the inhibitor and iron.

Expired Fenbendazole is adsorbed on a mild steel by the presence of heteroatoms like N, S, O along with the presence of aromatic π-electron system in its molecular structure. The lone pair electrons present on these atoms can interact with the empty d-orbitals of iron atoms on the surface of the metal, promoting its chemisorption. In acidic medium, these inhibitor molecules can be protonated and lead to an electrostatic attraction with the negatively charged regions of metal sites to physisorption. Furthermore, the π-electrons located in the aromatic rings might be able to form π–d orbital interactions with the metal surface, leading to better stability of the adsorbed protective film.

The greater adsorption in hydrochloric acid medium due to contribution of chloride ions towards the adsorption of protonated inhibitor molecules. Chloride ions tend to first adsorb onto the mild steel surface, creating favorable electrostatic conditions for attraction of the positively charged inhibiting species. This causes higher surface coverage and improved inhibition efficiency due to greater adsorption. On the other hand, the sulfate ion is more vigorously hydrated and competes well with the inhibitor molecules for their place on the adsorbent; therefore, the inhibition efficiency is decreased in the sulfuric acid medium.

The adsorption process helps in the formation of a tight and adherent protective coating over the mild steel surface. When a film has been adsorbed, it will block some of the active corrosion sites. A film will reduce the direct contact of the metal with the acidic environment. A film will suppress decomposition of the metal on the anodic side, and reduce the evolution of an H2 molecule on the cathodic side. The adsorption studies and electrochemical results are in excellent agreement, confirming that effective corrosion inhibition of expired Fenbendazole is mainly controlled by adsorption to the mild steel surface.

The adsorption isotherm studies reveal that expired Fenbendazole is an effective corrosion inhibitor that spontaneously adsorbs to the surface following the

Langmuir adsorption isotherm model. Due to the combined varieties of physical and chemical adsorption, the high surface affinity of expired Fenbendazole along with the formation of a compact stable protective film at the surface is responsible for its high corrosion inhibition performance in hydrochloric acid and sulfuric acid media.

### 3.5 Potential of Zero Charge

Figures 5A–5D show the change in differential capacitance in mild steel in 1.0 M HCl and 0.5 M H2SO4 solutions in the absence and presence of expired Fenbendazole drug with applied potential. The electrochemical parameters determined from the PZC measurements (E_ocp, E_pzc and excess surface charge values) are shown in Table 4. The above parameters provide an insight into mild steel surface charge and adsorption of inhibitor molecules at the metal/solution interface.

The differential capacitance curves clearly indicated that the capacitance values decrease drastically in the presence expired Fenbendazole in both acidic media. The reduction of capacitance has been attributed to the adsorption of inhibitor molecules over the mild steel surface. Inhibitor molecules adsorbed onto the metal/electrolyte interface eliminate the influence of water molecules and aggressive ions. These changes raise the electrical double layer thickness and lower the local value of the dielectric constant. Thus, the interfacial capacitance lessens, confirming formation of an adsorption film on the metal surface.

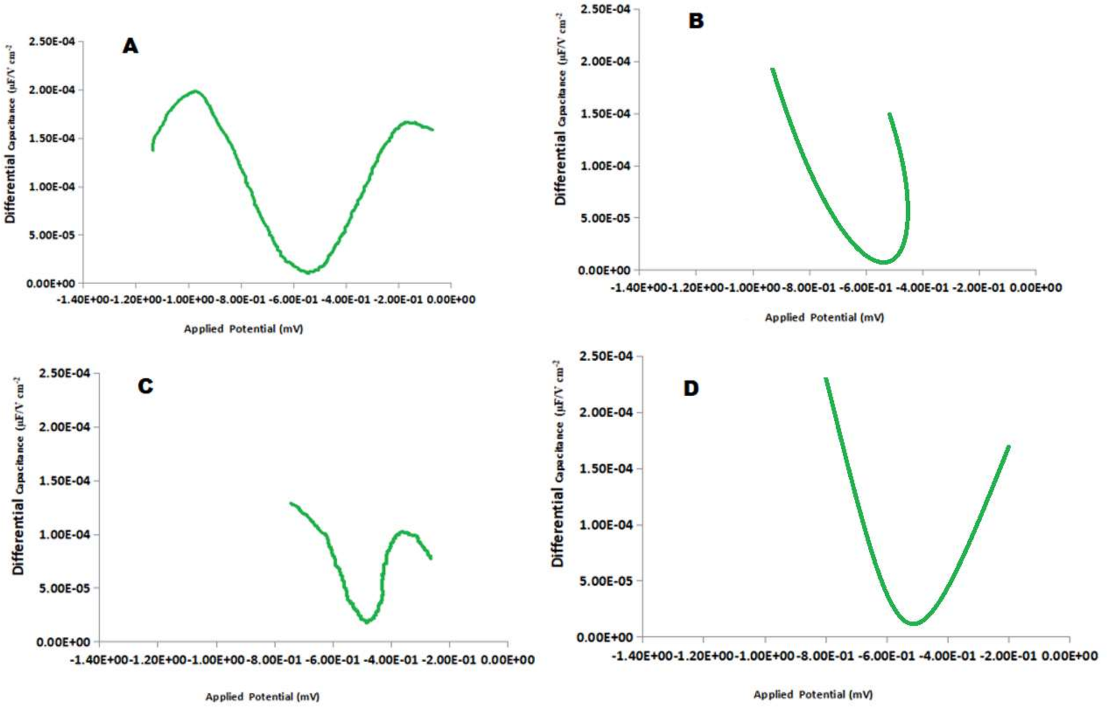

**Figure.5 A plot of Applied DC potential vs Differential Capacitance in A. 1.0 M HCl B. 1.0 M HCl + 5.0 mM Fenbendazole C. 0.5 M H2SO4 and D. 0.5 M H2SO4 + 5.0 mM Fenbendazole.**

In a solution of 1.0 M HCl, the uninhibited solution had an open circuit potential (Eocp) value of −490 mV/SCE and a potential of zero charge (Epzc) value of −532 mV/SCE. The excess surface charge was positive at +42 mV. The addition of expired Fenbendazole at 5.0 mM slightly shifted Eocp to −481 mV/SCE and did not affect Epzc to −532 mV/SCE, thereby reducing excess surface charge to +20 mV. The blank solution in the 0.5 M $H_2SO_4$ medium likewise exhibited an Eocp and Epzc value of −484 mV/SCE and −510 mV/SCE, respectively, producing a positive surface charge of +26 mV. With the introduction of 5.0 mM inhibitor, the Eocp value shifts to −479 mV/SCE the Epzc value shifted to −540 mV/SCE that gives the charge on the surface value +23 mV.

The positive excess surface charge values recorded in acidic media indicate that the mild steel surface has a net positive charge under open circuit potential conditions. Inhibitor molecules are attracted to positively charged surfaces due to electrostatic interaction. In acidic media, expired Fenbendazole molecules will likely be protonated owing to heteroatoms such as nitrogen and sulfur present in the molecular structure. The negatively charged adsorbed chloride or sulfate ions on the metal surface can effectively interact with these protonated species, thereby enhancing adsorption and the formation of a stable protective film.

In hydrochloric acid medium, surface charges decrease more than in sulfuric acid medium. This indicates stronger adsorption of expired Fenbendazole in HCl solution than in H2SO4 solution. The adsorption of mild steel surface will favours protonated organic inhibitor molecule adsorption due to the electrostatic condition created by chloride ion adsorption. In hydrochloric acid medium, the surface coverage and inhibition efficiency of chlorides are improved. On the contrary, the sulfate ion is bulky and more strongly hydrated, making them less effective in facilitating the adsorption of the inhibitor molecules. Consequently, this leads to relatively lesser inhibition performance in sulfuric acid solution.

The curves of differential capacitance further supports the adsorption mechanism of the inhibitor. When no inhibitor is present, the values of capacitance are relatively higher due to direct exposure of the metal surface to the acidic environment. As expired Fenbendazole is incorporated, the capacitive minima grow more significant and experience a slight shift, indicating that the adsorptive inhibitors modify the electrical double layer. A compact adsorbed layer formation results in a decrease in the active surface area available for corrosion reactions and inhibition of charge transfer processes at the metal surface.

The expired Fenbendazole may be adsorbed physically as well as chemically. The physisorption of the protonated inhibitor molecules on the charged metal surface is facilitated through electrostatic attraction while the coordination of lone pair electrons on nitrogen, sulphur and oxygen atom with empty d-orbitals of the iron atom may cause chemisorption [26]. Also, aromatic electrons present in the structure may be involved in π−d orbital interactions with the metal surface, thus enhancing the stability of the adsorbed protective film [26].

The reduction interfacial capacitance coupled with positive values of excess surface charge confirms the efficacy of expired Fenbendazole adsorption in both acidic media [42]. The film of the adsorbed inhibitor does not allow the corrosive species to reach the metal surface, inhibiting both anodic dissolution of iron and cathodic evolution of hydrogen [43].

On the whole, the findings of zero charge and differential capacitance studies strongly support the assertion that expired Fenbendazole acts as an efficient corrosion inhibitor by adsorbing on mild steel surface. The stronger adsorption and larger reduction of the surface charge taking place in the HCl medium explains the greater inhibition efficiency obtained in HCl than in H2SO4 solution. The development of a compact and stable protective coating greatly improves mild steel's resistance to corrosion in acid environments.

## 4. Conclusion

The study conducted in the present work reveals that expired Fenbendazole drug is an efficient corrosion inhibitor of mild steel in an acidic medium such as 1.0 M HCl and 0.5 M $H_2SO_4$. In weight-loss experiments, the rate of corrosion was found to decrease with increased concentration of the inhibitor while the efficiency of the inhibitor was found to increase with an increase in the concentration of the inhibitor. The maximum efficiency was found in the 5.0 mM concentration. The inhibitor performed much better in hydrochloric acid medium, compared to sulfuric acid medium that is probably due to the chloride ions being more effective in facilitating the adsorption interaction.

The results of the electrochemical impedance spectroscopy studies reflected an enormous rise in the charge transfer resistance and the decrease of the double-layer capacitance in the presence of the inhibitor. This confirms the formation of a stable protective adsorbed film on the surface of mild steel. The Potentiodynamic polarization results revealed drastic decreases in corrosion current density while only showing small shifts in corrosion potential. This proves that expired Fenbendazole functions as a mixed-type inhibitor. Therefore, it hinders anodic metal dissolution as well as cathodic hydrogen evolution reactions.

Results of the adsorption studies reveal that the inhibition process stalks the Langmuir adsorption isotherm, which indicates monolayer adsorption of the inhibitor molecules on the mild steel surface. The negative sign of free energy values indicated that adsorption took place spontaneously owing to both physical and chemical adsorption. Further substantiation of adsorption mechanism occurred with potential of zero charge and differential capacitance measurements, which exhibited electrode interface barrier film formation of compact configuration.

The expired Fenbendazole exhibiting excellent inhibition efficiency is due to the presence of heteroatoms such as nitrogen, sulfur and oxygen together with aromatic π-electronic systems which favours the interaction with the mild steel surface. On the whole, expired Fenbendazole is found effective, eco-friendly, economic, and sustainable corrosion inhibitor for mild steel in acidic medium. This is useful for protecting steel from corrosion and re-utilization of expired drug molecules.

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