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The Relation Between Adsorption of Metronidazole as Green Inhibitor and the Microstructure of Carbon Steel (CS) in 1M HCl Medium

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Abstract

In this paper using the Metronidazole pharmaceutical drug compound as a green corrosion inhibitor that can decreasing the rate of corrosion on metallic surface, as a result of the adsorption of metronidazole on the metal surface. In this regard, we simultaneously present an overview of metronidazole compound performance, as a corrosion inhibitor in 1 M HCl, and with presence different concentrations of the drug. By using potentiodynamic polarization, technique that illustrate the nature of adsorption, the effect of medium on the corrosion processes, and the effect of polarization for the orientation of the inhibitor molecule to the CS surface. The surface examination by Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX), Atomic Force Microscopy (AFM), and Fourier Transforms Infrared (FT-IR) are confirmed the formation thin film that adsorbed on the metal surface according to the mechanism of the adsorption processes on the polarized metal surface.

Keywords: Corrosion Inhibition; Potentiodynamic Polarization Technique; Metronidazole Inhibitor; SEM; EDX; AFM; FT-IR.

Introduction

Most organic compounds containing nitrogen (N-heterocyclic), sulfur, long carbon chain, or aromatic, and oxygen atoms are used as a corrosion inhibitor. Among them, organic compounds have many advantages such as large molecular size, soluble in water, availability, cheap, low toxicity, easy for using, and easy production [1]. Natural heterocyclic mixes have been utilized for the corrosion inhibitor on the C-steel copper aluminum and various metals in various aqueous medium [1-5]. Adsorption of the drug molecules on the metal surface facilitates its inhibition [6]. Heterocyclic mixes have demonstrated more hindrance effectiveness, for C-steel in both HCl and H_2SO_4 arrangements such as the medications are used inhibitors, that can compete favorably with green inhibition of corrosion, and the most medications can be synthesized from natural products [7,8]. Selection of some medication as corrosion inhibitors due to the followings: drug molecules contain oxygen, sulfur, and nitrogen as active sites, it is environmentally friendly furthermore vital in organic responses, drugs can be easily produced, and purified, nontoxic compering organic inhibitors. Some medications have been investigated to be great corrosion inhibitors for metals such as Biopolymer gave 86% inhibition efficiency (IE) for Cu in NaCl pyromellitic di-amide linked to oxadiazole cycle gave 84.6% IE for mild steel (CS) in HCl 2-mercaptobenzimidazole gave 82% IE for CS in HCl Antidiabetic Drug Janumet gave 88.7% IE for CS in HCl [9-11]. Januvia gave 79.5 % IE for Zn in HCl Cefuroxime Axetil gave 89.9% IE for Al in HCl Phenytoin sodium gave 79% for CS in HCl Aspirin gave71% IE for CS in H_2SO_4 Septazole gave 84.8% IE for Cu in HCl and Chloroquine diphosphate gave 80% IE for CS in HCl [12-17]. Study on Structural, Corrosion, and Sensitization Behavior of Ultrafine and Coarse Grain 316 Stainless Steel Processed by Multiaxial Forging and Heat Treatment [18]. Investigating the corrosion of the Heat-Affected Zones (HAZs) of API-X70 pipeline steels in aerated carbonate solution by electrochemical methods [19]. Predictions of corrosion current density and potential by using chemical composition, and corrosion cell characteristics in microalloyed pipeline steels [20]. Predictions of toughness, and hardness by using chemical composition, and tensile properties in microalloyed line pipe steels [21].

The scope of this article is used metronidazole drug as save corrosion inhibitor for CS in the acid medium by electrochemical method, and to elucidate the mechanism of corrosion inhibition.\

Experimental

Metal samples

The sample of CS was used in this study that have the chemical composition of the metal sample was determined by using an emission spectrometer, with the aid of ARL quant meter (model 3100-292 IC) and listed in the Table 1.

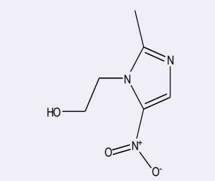
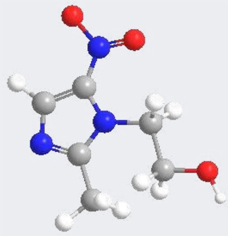
Table 1: Chemical Compositions of Carbon Steel Sample

Sample	C%	Mn%	V%	Fe%	Si%
CS	0.26	0.77	0.11	98.51	0.35

Metronidazole drug as an inhibitor

Metronidazole drug information's is describing in Table 2.

Table 2: The Components and Molecular Structure of Investigated Inhibitor

Inhibitor Structure		IUPAC Name	Molecular Weight	ActiveCenters	Chemical Formula
 2-(2-Methyl-5-nitro-1H-imidazol-1-yl) ethanol		2-(2-Methyl-5-nitro-1H-imidazol-1-yl) ethanol	171.156 g.mol^{-1}	3N, 3O, 3π	$C_6H_9N_3O_3$

Solution

The aggressive solution, 1M HCl was prepared by dilution of analytical grade 36% of HCl with bi-distill water. The concentrations range of the inhibitor were used between 50 ppm to 250 ppm.

Potential-Dynamic Polarization Technique

Electrochemical polarization experiments using three electrodes in electrochemical cell such as saturated calomel electrode (SCE) that couple to a fine Luggin capillary act a reference electrode, platinum is counter electrode and working electrode that made up from square cut of metal (CS) sheet fixed in epoxy resin so that the surface area that exposed to the electrolyte 1.0 cm2 only. The working electrode prepared by polisher paper (SiC) with deferent sizes (800, 1000 and 1200) and immersed in corrosive medium at natural potential for 10 min until reach the steady state.

The potential was started from - 600 to + 600 mV vs. open circuit potential (E_{ocp}). Calculation of inhibition efficiency (% IE) and the degree of surface coverage (θ) as follows [22].

$$IE\ \% = \theta \times 100 = [1 - (i_{corr\ (inh)} / i_{corr\ (free)})] \times 100$$

Where, icorr (free) and icorr (inh) are the corrosion current densities in the absence and presence of metronidazole, respectively.

Surface Examinations

The morphology of the CS surface used for analysis and examination nature of the surface and study the changing that appeared on the metal surface. The specimens were prepared by abraded mechanically by using different emery papers up to 1200 grit size and immersed in 1M HCl acid (blank) and with 300 ppm of metronidazole at room temperature for one day (24 h). Then, after this immersion time, the specimens were washed gently with distilled water, carefully dried and take carefully to the system of surface examinations such as using scanning electron microscope (SEM), energy dispersive x-ray (EDX), FT- IR spectroscopy and atomic force microscope (AFM).

Result and Discussion

Electrochemical Techniques

Open circuit potential (E_{OCP})

From the Fig. 1 and Table 3 are shown several interesting points:

The EOCP in the blank solution started at -562.1 mV then shifted anodically and the steady state is occurred after 300 S. This indicate that the initial dissolution process (the attack on the surface of metal) and then the formed oxide film.

In the presence of metronidazole, the EOCP started at relatively positive potential compared with that in the absence of the inhibitor and then shifted anodically that starting from 483.3 to 475.1 mV according to the increasing the concentration 50 to 300 ppm respectively. The steady state is attained rapidly, compared with the blank. With increasing the concentration of the metronidazole, make shift in the open circuit potential that increases in the active direction pointing, this means the inhibitor might act mainly as mixed type inhibitor [23]. The classification of a compound as an anodic or cathodic type inhibitor, based on the E_{OCP} displacement; if the shift in E_{OCP} is at least ±43 mV compared to the one measured in the blank solution it can be classified as an anodic or cathodic inhibitor. However, from Fig. 1, the shift in E_{OCP} on adding metronidazole is about 15 mV revealing that the present inhibitor acts slightly more as anodically inhibitor.

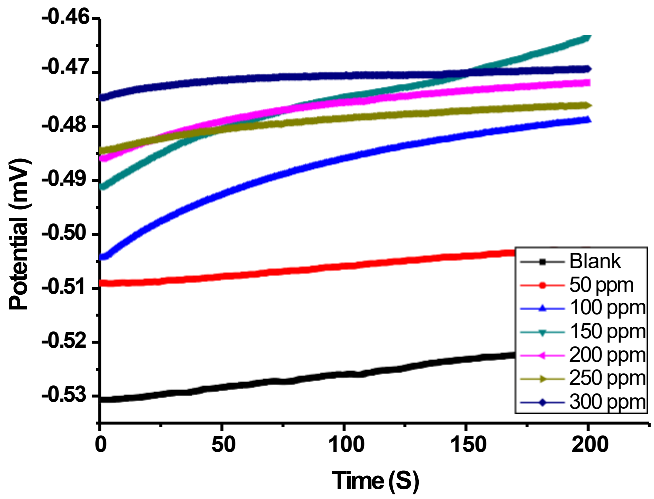


Figure 1: Open Circuit Potential, each vs. Time Relations for cs Submersed in 1m hcl in the Nonexistence and with Existence of Metronidazole Drug at 25oc.

Table 3: E_{co} of The Cs in The Nonexistence and in Existence of Metronidazole Drug At 25° C.

Conc.(ppm)	$-E_{Min}$ (mV)	$-E_{Max}$ (mV)
Blank	526.1	507.1
50	483.6	480.2
100	479.3	471.5
150	478.9	475.2
200	476.9	469.3
250	475.1	469.3
300	475.1	458.7

Potentiodynamic polarization (PP)

The results are drowning in nonexistence and with existence deferent doses of metronidazole drug in Fig. 2. The obtained potential-dynamic polarization parameters are given in Table 4. These results indicating that the cathodic and anodic curves obtained according to Tafel-type behavior. The form of the curves is slightly similar either in the cathodic or in the anodic side, which indicates that the mechanisms of CS dissolution and hydrogen reduction apparently remain in the presence of the inhibitor. Addition of metronidazole decreased both the cathodic and anodic current densities and caused mainly parallel displacement to the more negative and positive values respectively, i.e., the presence of metronidazole in solution inhibit both the hydrogen evolution and the anodic dissolution processes with overall shift of Ector to slightly fewer negative values.

The graphical also show that the anodic and the cathodic Tafel slopes (□a and □c) were slightly changed on increment of the doses of the metronidazole. It obvious that no change of the mechanism of inhibition in existence and nonexistence of metronidazole drug. The fact that the values of □c are slightly higher than the values of □a refer to the cathodic action of the metronidazole inhibitor. It obvious that the action of inhibitor control over the electrochemical reaction slightly cathodic control. This means that the metronidazole is mixed type inhibitor, but the cathodic is more specially polarized than the anodic. The values of Tafel slope are higher refer to the surface kinetic process instead of the diffusion-controlled process [24]. Ether the cathodic slope that obtained from the electrochemical measurements confirm the hydrogen evolution reaction was activation or cathodic controlled [25].

The addition of the inhibitor did not modify the mechanism of this process but appears that the inhibition mode of the metronidazole was used by simple adheres of the surface by adsorption process.

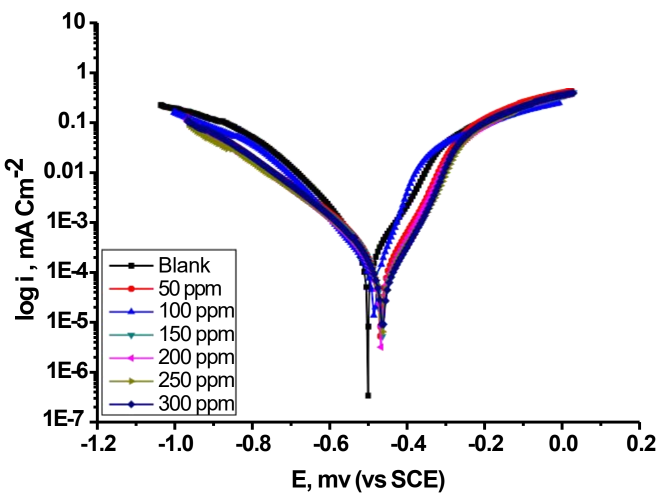


Figure 2: Pp Curves for The Corrosion of Cs In 1 M Hcl in The Nonexistence and Existence of Various Doses of Metronidazole At 25°C

Table 4: Pp Parameters (Ector, Iscor, Ba and Bc), Θ and % Ie in Nonexistence and with Existence Various Doses of Metronidazole In 1 M Hcl Medium At 25°C

Conc. ppm	I_{corr} mA/cm^{-2}	$-E_{corr.}$ mV (SCE)	β_a mV dec^{-1}	β_c mV dec^{-1}	C. R. Mpy	Θ	% IE
0.0	147.0	498	153	344	96.5	----	----
50	61.1	465	77.1	94.4	27.9	0.71	71
100	78.6	464	97.9	99.1	35.9	0.628	62.8
150	92.7	465	109.9	110.2	42.4	0.561	56.1
200	125	466	95.9	106.2	57.3	0.406	40.6
250	129	467	152.2	129.9	58.8	0.391	39.1
300	136	469	105.7	136	62.2	0.355	35.5

The positive potential is increased by anodic polarization, i.e., increase the dissolved component while that the potential decreased by cathodic polarization, i.e., increase the undissolved components. The dissolved component is formed as the following chemical equations [26].

$Fe \rightarrow Fe2+ \quad + 2e$

$Fe2^{+} + 2H2O \rightarrow HFeO^{-}_{2} \quad + 3H^{+}$

Where $HFeO^{-}_{2}$ Di-hypo-ferrite, green.

In the same time occurs as

$HFeO_{2}^{-} + H+ \rightarrow Fe\ (OH)_{2}$

Where the undissolved hydrated, and the (FeO) can be considered. So that at anodic polarization in the presence of HCl, the iron is dissolved, and formed ferrous chloride as:

$Fe^{2+} + 2HCl_{(aq)} \rightarrow Fe\ Cl_{2(aq)} + 2H^{+}$

And The Cathodic Processes in The Presence Of 1m Hcl Occurred As

i) $2H^{+} + 2e \rightarrow H2$ hydrogen evolution

ii) $O2(g) + 4H+_{(aq)} + 4e \rightarrow 2H_{2}O_{(l)}$ reduced of oxygen

The Hydrogen Ions Adsorbed on The Metal Surface Where an Electrochemical Reaction Takes Place in The Presence of O_2 As:

$M + H3O^{+} + e \rightarrow M{-}H + H_{2}O$

Where three steps can be done as:

a) $2M{-}H \rightarrow 2M + H_2(g)\uparrow$

b) $M{-}H + H_{3}O^{+} + e \rightarrow M + H_{2(g)}\uparrow + H_{2}O$

or

c) $4M{-}H^{+}$ + dissolved $O_{2} + 4e \rightarrow 4M + 2H_{2}O_{(l)}$

The positive potential is increased by anodic polarization, i.e., increase the dissolved component while that the potential decreased by cathodic polarization, i.e., increase the undissolved components. According to the following equation:

$Fe \rightarrow Fe^{2+} + 2e$ anodic reaction

$2H_2O + 2e \rightarrow H2\ (g)\uparrow + 2OH-$ cathodic reaction

In total process:

$Fe + H2O \rightarrow Fe^{2+} + 2OH^{-} \rightarrow Fe\ (OH)_{2}$

In the bulk the ferrous hydroxide dissolved as:

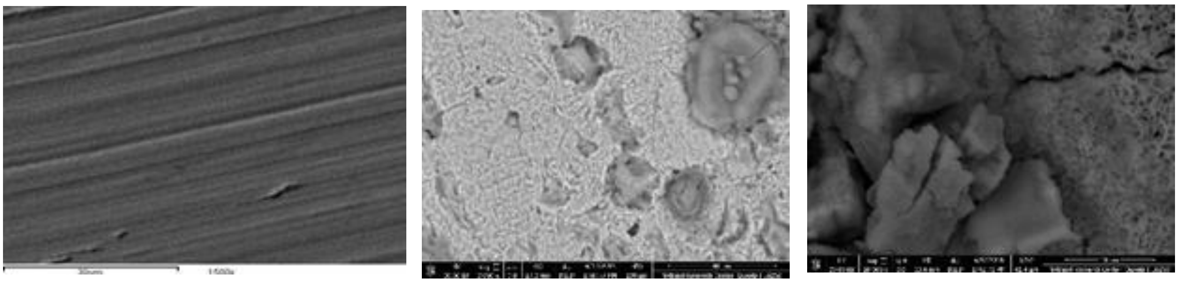
$2Fe\ (OH)_{2} \rightarrow 2FeOH^{++} + 2OH^{-}$

And,

$2Fe\ OH^{++} + 2e \rightarrow 2Fe\ O + H_{2}\uparrow$

Scanning Electron Microscopy (SEM)

The micrographs are obtained for CS specimens in the nonexistence, and in the existence of 300 ppm of metronidazole drug after exposure for immersion one day in corrosive medium 1M HCl. It is clear that CS has suitable surfaces for corrosion attack in the blank or corrosive medium only Figs. 3 a, b and c. When the metronidazole is existence in the corrosive medium, the morphology of CS surfaces is quite different from the previous one, and the specimen surface was smoother. It is clear that the formation of a thin film layer adsorbed on the metal surface, which distributed in a disorder way overall surface of the CS [27]. This may be due to the adsorption of the metronidazole on the CS surface, and made up the passive film in order to block the active site present on the CS surface. The metronidazole molecule is interacted with active sites of CS surface, resulting the decreasing contact between CS, and the corrosive medium. From the above sequentially metronidazole is exhibited excellent inhibition effect.



A-Free sample **B- Blank in 1M HCl** **B- Blank in 1M HCl**

300 ppm of Metronidazole

Figure 3: a, b and c SEM micrographs for CS in the nonexistence, and the existence of 300 ppm of the metronidazole after submersion for 1 day

Energy Dispersion Spectroscopy (EDX)

To determination the elements, and molecules that existence are adsorbed on the surface of CS after one day that immersion in acid with optimum doses of metronidazole by using the EDX spectra [28]. The EDX analysis of CS in 1M HCl with in the presence of 300 ppm of the metronidazole is given by Fig. 4. The spectra show additional lines, demonstrating the existence of C (owing to the carbon atoms of some metronidazole). These data show that the carbon, nitrogen, and oxygen atoms are covered the specimen surface. The EDX analysis is indicated that only carbon, nitrogen, and oxygen are detected, and show that the passivation film is contained the chemical formula of the metronidazole drag that adsorbed on the CS surface. It is clear that, the percent weight of adsorbed elements C, N, and O were presented in the spectra, and recorded in Table 5.

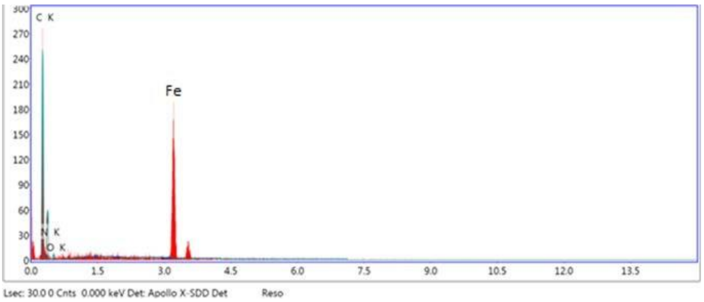


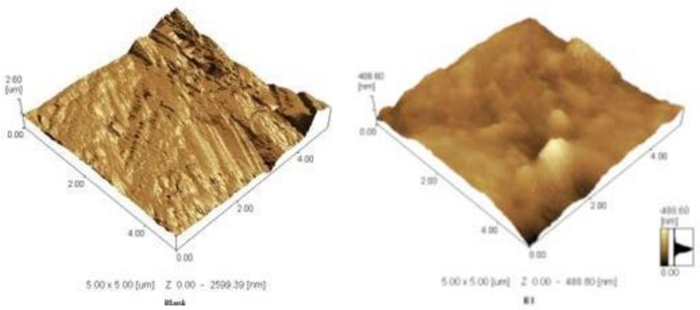
Figure 4: Eds Analysis on the Cs in the Existence 300 Ppm of the Metronidazole Drug for 1 Day that Immersion in 1m Hcl.

Table 5: Surface Composition (Wt. %) Of Cs After One Day that Immersion In 1m Hcl with 300 Ppm of the Metronidazole

Wt %	Fe	C	N	O
Metronidazole	75.97	43	50	5.21

Atomic Force Microscopy (AFM)

AFM is a powerful tool to investigate the surface morphology of various samples at nano- micro scale that is currently used to study the influence of corrosion inhibitor on the metal solution interface. From the analysis, it can be gained regarding the roughness on the surface. The roughness profile values are played an important role to identifying, and report the efficiency of the inhibitor under study. Among the roughness is tacked a role for the explanation of adsorption, and illustrated the nature of the adsorbed film on the metal surface [29-31]. Fig. 5 a, shows the 3D images as well as elevation profiles of polished of the CS in the absence, and the presence the metronidazole an inhibitor. Fig. 5 b, the surface of CS specimen (a) exposed to corroded solution affected vales structure with large, and deep crack but the surface (b) reveal that is covering film adsorbed on the metal surface. Conclusion, the adsorption film is protected the surface of the metal from corrosion process. From analysis the values, indicating that the higher value of Z parameter reached, which found (2.60 µm) for the blank solution which placed in 1M HCl one day and analyzed. The observation of the metal surface which immersed in 1M HCl in the presence of 300 ppm of the metronidazole as an inhibitor possess small roughness (488.6 nm) compared with the blank solution. It can be noted that the value is lower than that of the blank value. The decrease in the roughness value reflected to the adsorption of inhibitor molecule on metal surface thereby reducing the rate of corrosion.



a-Blank **b-In 300 ppm of Egy- dronate drug**

Figure 5: A And B The 3d of Optical Images of Afm in the Non-existence, and the Existence of the Metronidazole Drug.

Fourier Transforms Infrared Spectra (FT – IR)

The (FT – IR) spectrophotometer is a powerful instrument that can be used to identify the function group that presence in metronidazole and the type of interaction that occur between function group with metal surface. Since, pharmaceutical drug compound contain variety of organic compound, and these organic compounds (inhibitor) are adsorbed on the metal surface providing thin film that protection them against corrosion, they can be analyzed by using (FT – IR). To confirm the nature of the chemical constituent is adsorbed on the metal surface, by the Fourier transform infrared (FT – IR) spectra [32].

The pharmaceutical drug compounds are certain have function group according to the chemical formula like OH, C=C and C=O. In order to find the nature of constituents involved in the adsorption using (FT – IR) spectrum of material that are coated the metal surface gives in Fig. 6. The spectrum of metronidazole before, and after adsorption that seen the wave number of the function groups OH abroad peak at 3400 cm-1 starching, C=C is sharp peak at 1630 cm-1 starching, and C=O a sharp peak between 1140 – 1000 cm-1 starching. It is clear that the function groups of metronidazole inhibitor appear on the metal surface that confirm to the adsorption process [33].

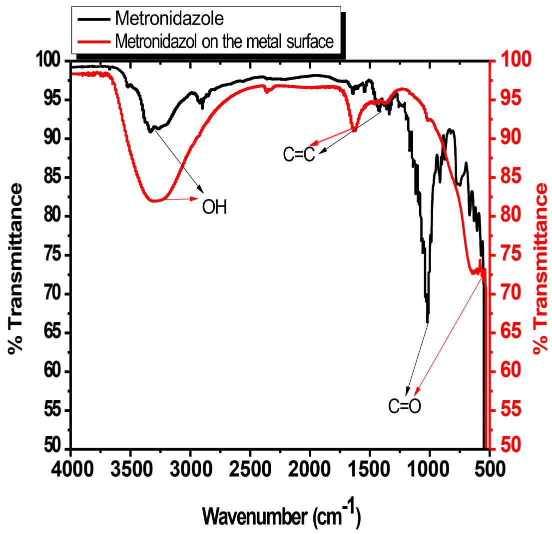


Figure 6: Ft – Ir Spectrum of Metronidazole Before and After Adsorption on the Cs Surface.

Mechanism of Inhibition

To illustrate the mechanism of inhibition of corrosion on the CS surface in acid medium by using pharmaceutical drug compound as an inhibitor, it is must be knowing the nature of metal surface, and the nature of the component of inhibitor structure. The CS is regarded the metal α-phase It is obvious that α-phase state consists of grains, and grain boundaries in the surface of the metal, Fig. 7. A cross-section of a piece or specimen of the metal that is a corroding to clarify that there are both anodic, and cathodic sites in the metal surface structure [34].

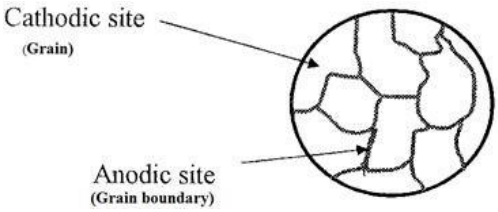


Figure 7: Schema Models of Metalα- Phase

The surface of iron is usually, coated with a thin film of iron oxide. However, if this iron oxide film develops some cracks, anodic area is created on the surface, while other metal parts acts as cathodic sets. It follows that the anodic areas are small surface, while nearly the rest of the surface of the metal large cathodes. Electrochemical corrosion involves flow of electric current between the anodic, and cathodic areas called inter-granular corrosion. Fig. 8, SEM image is shown the corrosion of the CS in 1M HCl in one day immersion that illustrated inter-granular corrosion.

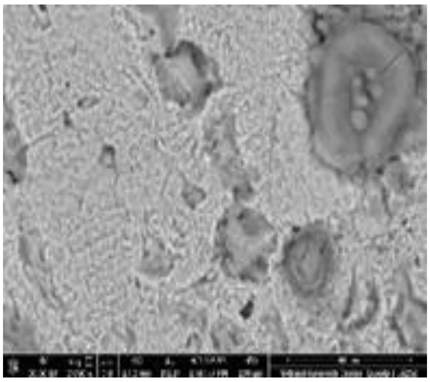


Figure 8: Sem Image Illustrated Inter-Granular Corrosion After Immersion the Specimen in 1m Hcl One Day

All previous results prove that the pharmaceutical drug compound under study were actually inhibit the corrosion of the CS in 1M HCl solution as a corrosive medium. The corrosion inhibition is due to their physical, and chemical adsorption for formation of protection thin film adsorbed on the metal surface. The effect of metronidazole as inhibitor may be corresponding to the accumulation of the inhibitor molecules on the metal surface, which prevent the direction contact of the metal surface with corrosive environment. The surface of the CS sample has positively charge in aqueous acid solution, and the adsorption occur according to [35].

- The unshared electrons of nitrogen, oxygen atoms, and electron density of □ bonding donate to the vacant orbital on the metal surface make chemisorption.
- The partial negative charge that presents in function group containing Oxygen, nitrogen, and electron density of □-bond in metronidazole may be adsorbed on the positively charge of the metal surface like electrostatic attraction between the opposite charge, in the form of neutral molecules, that involving displacement of water molecules from the metal surface.

The inhibition action of the metronidazole can be accounted by the interaction between the lone pair of electrons in the nitrogen, oxygen, and electron density of □-bond with positively charged (anodic sites) on the metal surface, and the skeleton of inhibitor compound cover the cathodic sites this action form thin layer adsorbed on the metal surface and prevent corrosion processes Fig. 9.

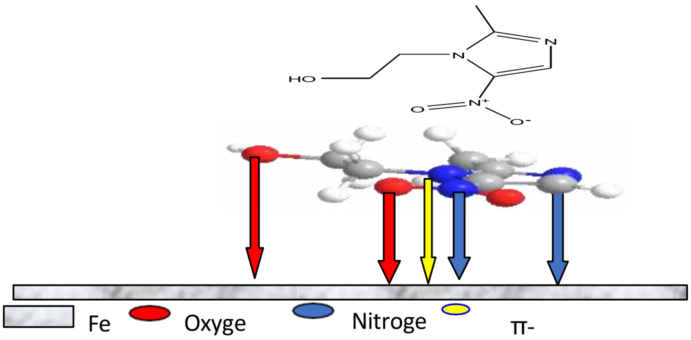


Figure 9: Schema Model Illustrated the Adsorption of The Metronidazole Structure on the Cs Surface.

This meaning, the metronidazole molecule attached with anodic site, and covered somewhat of cathodic area, so that the corrosion rate in presence of metronidazole is anodic-cathodic control.

Conclusion

Inhibition of the corrosion of the CS in 1M HCl solution by metronidazole is determine by potential-dynamic polarization, technique, and surface examination by Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX), Atomic Force Microscopy (AFM), and Fourier Transforms Infrared (FT-IR). It was found that the inhibition efficiency depends on concentration, nature of metal surface, and the type of adsorption of the inhibitor [36].

The observed corrosion data in the presence the metronidazole as an inhibitor

- The tested metronidazole inhibitor establishes a very good an inhibition efficiency for the CS corrosion in 1M HCl solution.
- Metronidazole inhibits the CS for the corrosion by the adsorption on its surface, and make thin film layer protective them from corrosion process.
- The inhibition efficiencies of the metronidazole increase with the increasing of their concentrations.
- The values of inhibition efficiencies obtained from all techniques that using are seen the validity of the obtained results.
- The metronidazole molecule attached with anodic site, and covered somewhat of cathodic area, so that the corrosion rate in the presence of the metronidazole is anodic-cathodic control.

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