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Antibacterial Inhibitor as an Expired Metoclopramide in 0.5M Phosphoric Acid

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Abstract

Expired drug Metoclopramide was investigated as an antibacterial corrosion inhibitor for carbon steel in 0.5M H_3PO_4 solution using the electrochemical method at 30ºC and 60ºC. The results showed that this drug is an efficient inhibitor for carbon steel and the efficiency reached to 82.244 % for 175 ppm at 30ºC and 76.146% for 225 ppm at 60ºC. The adsorption of drug on carbon steel surface follows Langmuir adsorption isotherm with small values of adsorption-desorption constant. The polarization plots revealed that Metoclopramide acts as mixed-type inhibitor. Some parameters of inhibition process were calculated and discussed. The surface morphology of the carbon steel specimens was analyzed using scanning electron microscope and energy dispersive X-ray analysis. Antibacterial activity of inhibitor was also tested. Fourier-transform infrared (FTIR) spectroscopy confirmed the inhibitive role of Metoclopramide drug.

Keywords: *Antibacterial test; Carbon steel; Expired Metoclopramide; Phosphoric acid.*

1. Introduction

Metoclopramide is a drug used mostly for esophageal, stomach diseases and for treat and prevent vomiting and nausea. It is also used to treat migraine headaches. Using antibacterial materials for corrosion inhibition leads to reduce biocorrosion which is caused by the presence of microorganisms in corrosive mediums through prevent the biofilm to be formed [1-9], these drugs have organic compounds containing hetero-atoms such as nitrogen, sulfur and oxygen in their structures as active centres which allow the adsorption of component on the steel surface. This component interact with the chathodic and anodic reaction, blocking the active sites and create protective layer on the steel surface resulting a decrease in the corrosion rate. Drugs are nontoxic, cheap, negligible negative effects on environment, so it suggested to replace the traditional toxic corrosion inhibitors. Many researches highlighted on the use of many types of drugs as corrosion

inhibitors for carbon steel and aluminum in acidic and alkaline [10]. The objective of the present work is preventing corrosion of carbon steel in 0.5M H_3PO_4 solution at 30 and 60°C by adding five concentration of expired Metoclopramide drug.

2. Experimental Part

2.1. Materials and Chemicals

Square shaped samples were cut of low carbon steel with chemical composition (C 0.161, Mn 0.42, P 0.0073, S 0.0049 and Fe rem. wt%) grinded and polished with emery papers (600 to 1200) to get flat surface, degreased with acetone and washed with distilled water. The test solution of 0.5M H_3PO_4 was prepared by the dilution of analytical grade from (Merck) with water. Metoclopramide was purchased as expired drug from a medicine shop. Five concentrations were added to corrosive medium involve (75, 125, 175, 225 and 275 ppm) that prepared by dissolving specified amount of inhibitors in acidic solution.

2.2. Tests for Active Material

The active material in drug was investigated by high performance liquid chromatography HPLC to identify the recovery material, this instrument obtained from Japan, model RESERVOR TRAY, column dimension 25x4.6 cm, 75methanol/25water as solvent, wavelength (λ) =280 nm and flow rate = 1.8 m/min. The extracted active material in drug was studied for its antibacterial efficiency by agar well diffusion method. The gram negative and gram positive bacteria including Escherichia coli (E. coli) and Staph. were used. Each bacterial isolates were suspended in Muller Hinton agar and diluted to 105 colony forming unit (CFU) per ml. 5 ml diameter wells were cut from the agar using a sterile cork-borer and 20 µL in dimethyl sulphoxide and poured into the wells. The petri plates were incubated for 24 h at 37°C for bacterial growth. The diameter of the zone of inhibition in mm measured as antibacterial activity. These results of HPLC agreement with Moxifloxacin drug as green corrosion inhibitor for carbon steel in 1 M HCl [10].

2.3. Electrochemical Measurements

Potentiostat/Galvanostat M Lab 200 (Germany) was used to measure electrochemical parameters with three electrode cell; Pt as an auxiliary electrode, SCE as a reference electrode and Steel specimens as working electrode. All experiments were achieved at two temperatures 30 and 60 °C using water bath to temperature control. Tafel extrapolation method was used to measure the date of corrosion potential (E_{corr}), corrosion current density (i_{corr}) and Tafel slopes (b_c & b_a).

2.4. Inspections

Some inspections were achieved for inhibited surfaces include scanning electron microscopy (SEM) is an electron microscope that appearance the images of a sample by scanning the surface with a focused beam of electrons. The electrons react with atoms exist in the sample, producing different signals that involve information about the surface topography and composition of the sample. The electron beam is scanned in a raster scan pattern, and the location of the beam is joint with the detected signal to produce an image (FEI company – nether lands, inspect S50 – model), energy-dispersive x-ray spectroscopy (EDX) is an analytical technique utilized for the chemical analysis or elemental characterization of a specimen. It depended on an interaction of several source of X-ray excitation and a specimen. Its characterization abilities are due in big part to the fundamental principle that each element has a unique atomic structure letting a unique group of peaks on its electromagnetic emission spectrum (bruker company – Germany, X flash 6L10 – model) and Fourier-transform infrared spectroscopy (FTIR) is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high-spectral resolution data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer, which measures intensity through over a narrow range of wavelengths at one time (FTIR – 8400S, class1 – laser product) for film formed of adsorbed drug using KBr disc.

3. Results and Discussion

3.1. Tests for Active Material in Drug

The recovery of active material from expired drug was investigated by HPLC technique as illustrated in Fig. (1). This figure indicates the standard material in a small profile, while the recovery material shows in a big figure, this test confirms the obtaining of active material in drug as it is. The antibacterial of expired drug by the disk diffusion method was tested as shown in Fig. (2).

The inhibition zone against bacteria was measured in mm diameter as shown in Table (1). The antibacterial property of expired drug has proved to have cell death. The Metoclopramide has a good antibacterial activity against both gram positive and gram negative bacterial culture.

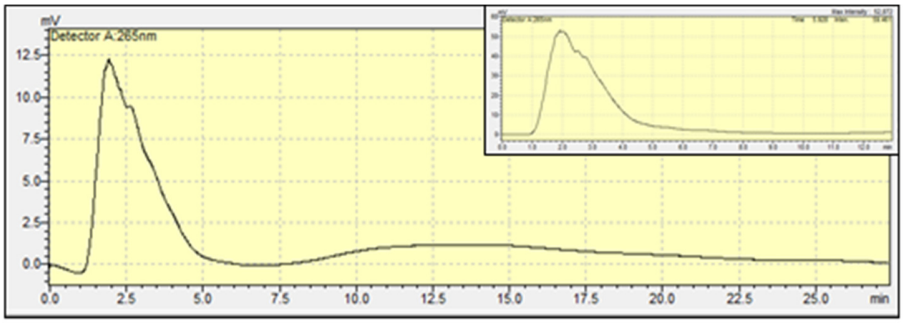
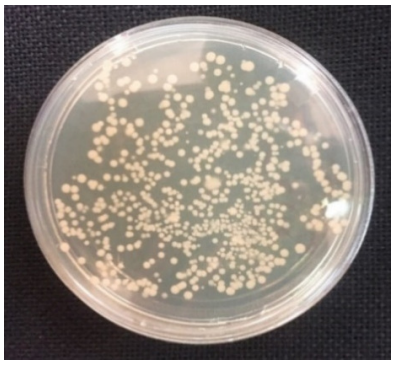
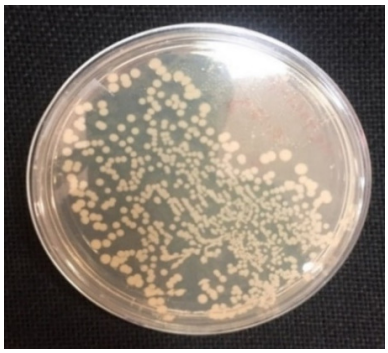


Fig. 1. HPLC analysis of extracted active material from expired drug compared with standard in small profile.



E. coli



Staph.

Fig. 2. Antibacterial activity of expired drug against E. coli and Staph. by the well diffusion method.

Table 1,
Data of antibacterial test.

Type of bacteria	Survival bacteria	Antibacterial percentage
Ecoli (Negative)	22.4%	73.4%
Staph. (Positive)	19.6%	80.4%

3.2. Polarization Behavior

Polarization behavior of low carbon steel corrodes in phosphoric acid in the presence of expired Metoclopramide drug as inhibitor as shown in Fig. (3) at 303 and 333 K. The presence of drug as organic molecules woks as an inhibitor by film forming on steel surface. Tables (2) and (3) indicate the corrosion inhibition data at 303 and 333 K respectively. These tables show that the corrosion potential shifted to either noble or active direction indicating that the selected drug is a mixed-type inhibitor. Also, cathodic and anodic Tafel slopes were decreased confirming the role of drug as inhibitor by decreasing polarization getting closed values for cathodic and anodic slope at constant concentration. Corrosion current density was decreased after adding expired drug; this means that the corrosion rate (C_R) which is calculated by the following equation also decreased [11]:

$$C_{R\,(mpy)} = 0.13 \times i_{corr} \left(\frac{e}{\rho}\right) \qquad \ldots(1)$$

Corrosion current densities before and after inhibition are used to calculate inhibition efficiency percentage (IE%) as follow [12, 13]:

$$IE\% = \left[1 - \frac{i_{corr,inhibited}}{i_{corr,uninhibited}}\right] \times 100 \qquad \ldots(2)$$

where $i_{corr,inhibited}$ and $i_{corr,uninhibited}$ are corrosion current density in inhibited and uninhibited medium respectively. The data of IE% that listed in Table (4) indicates that 175 ppm of expired drug is the best concentration at 303 K, while 225 ppm is the best concentration at 333 K due to the effect of temperature which leads to decreasing the adsorption of inhibitor on metallic surface. The same results were obtained by Fouda when he used septazole drug as green corrosion inhibitors for copper in hydrochloric acid solutions [14].

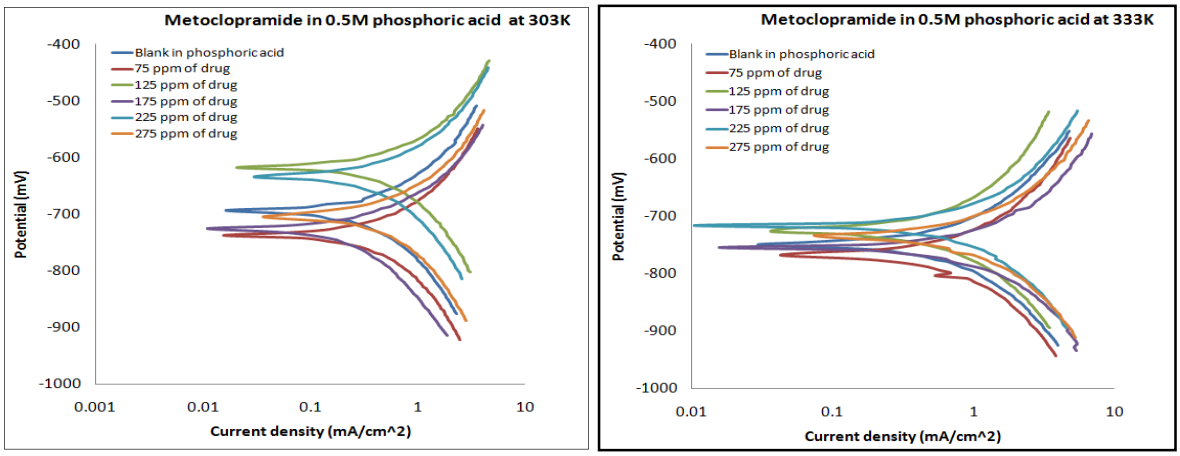


Fig. 3. Tafel plots in phosphoric acid at 303 and 33 K.

Table 2,
Measured data of polarization behavior for inhibition at 30°C in 0.5M H_3PO_4 solution.

Conc. ppm	E_{corr} mV	i_{corr} µA.cm^{-2}	$-b_c$ mV.decade^{-1}	$+b_a$	C_R mpy
0	-692	416.2	227.3	158	194.23
75	-737	143.93	66.1	63.3	67.16
125	-618	129.28	47.0	49.0	60.33
175	-726	73.90	54.0	41.2	34.48
225	-634	137.16	60.6	52.2	64.01
275	-705	155.74	61.2	62.9	72.67

Table 3,
Measured data of polarization behavior for inhibition at 60°C in 0.5M H_3PO_4 solution.

Conc. ppm	E_{corr} mV	i_{corr} µA.cm^{-2}	$-b_c$ mV.decade^{-1}	$+b_a$	C_R Mpy
0	-750	619.1	185.7	184.3	288.913
75	-767	190.33	55.9	53.8	88.820
125	-726	151.74	49.4	58.0	70.812
175	-754	200.94	53.2	40.9	93.772
225	-716	147.68	35.7	38.4	68.917
275	-733	220.31	61.3	47.1	102.811

3.3. Adsorption Isotherm

There are many factors affect adsorption process. The adsorption either is physical process with weak interaction or chemical process with directed interaction between the adsorbate and adsorbent.

The degree of surface coverage (θ) for various concentrations is used to estimate the adsorption isotherm according to following equation [15, 16]:

$$\theta = \left[1 - \frac{i_{inh.}}{i_{unin.}}\right] \quad ...(3)$$

The plots of C_{inh}/θ against C_{inh} for the expired drug at 303 and 333 K were straight lines as shown in (Fig. 4), indicating that the drug obey Langmuir adsorption isotherm by the following formula [16]:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad ...(4)$$

where, K_{ads} is the equilibrium constant of the process for the adsorption/desorption and C_{inh} is concentration of inhibitor in the bulk solution. The linear regression coefficient close to unity; where R^2 values were 0.9586 and 0.9703 at 303 and 333 K respectively.

The apparent free energy of adsorption (ΔG^o_{ads}) is calculated from the relation [16]:

$$\Delta G^o_{ads} = -2.303RT\ log 55.5\ K_{ads},$$
$$where\ K_{ads} = \frac{\theta}{C(1-\theta)} \quad ...(5)$$

The values of θ, K_{ads} and ΔG^o_{ads} are shown in Table (4).

Table 4,
Calculated data of polarization behavior for inhibition.

Conc. ppm	IE (%) 303K	333K	K_{ads} 303K	333K	-ΔG (kJ.mol^{-1}) 303K	333K
75	65.418	69.256	0.02522	0.030036	0.2552	0.3340
125	68.938	75.490	0.01775	0.024640	0.1796	0.2740
175	82.244	67.543	0.02646	0.011891	0.2678	0.1322
225	67.044	76.146	0.00904	0.014187	0.0915	0.1577
275	62.580	64.414	0.00608	0.006582	0.0615	0.0732

The values of ΔG^o_{ads} were negative indicating the spontaneous process of drug adsorption. The small values of K_{ads} and ΔG^o_{ads} indicate the physically adsorption of drug, where the free energy value up to –20 kJ/mol refer to electrostatic interaction between the charges of molecules and the metal surface (Physical adsorption).

From the corrosion rates at different temperatures, the values of the activation energy were calculated following the equation [17]:

$$log\frac{C_{R2}}{C_{R1}} = \frac{E^*_{app}}{2.303R}\left[\frac{1}{T_1} - \frac{1}{T_2}\right] \quad ...(6)$$

where CR1 and CR2 are the corrosion rates at the temperature T1 and T2 (K) respectively and E^*_{app} is the activation energy.

The data are given in Table (5) may be generalized that the E^*_{app} values are in general higher in presence of the drug as inhibitor than the value in the absence of it. This means that the adsorption process is decreases with increasing temperature and the adsorption of the drug molecules occurs rather than the attraction H_2O molecule to the metal surface. Therefore, the best concentration (175 ppm) has the highest activation energy equal to (27.976 kcal.mol^{-1}) compared with the activation energy without inhibitor which was (2.654 kcal.mol^{-1}).

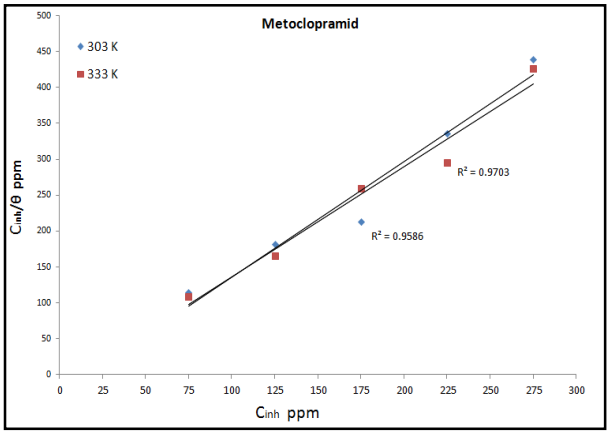


Fig. 4. Langmuir adsorption isotherm of expired drug on steel at two temperatures.

Table 5,

The energy of activation for adsorption drug on steel surface.

Conc. (ppm)	0	75	125	175	225	275
E^*_{app}**(kcal.mol^{-1})**	2.654	7.815	4.480	27.976	2.067	9.700

3.4. Examination of Inhibited Surface

Fig. (5) indicates the images of polished surface with little atmospheric corrosion and some oxide layer of FeO. The image of corroded carbon steel in phosphoric acid shows the product of corrosion with deposit P_2O_5 from environment, while the images of inhibited carbon steel in presence of 175 and 225 ppm at 303 and 333 K respectively show the adsorption of expired drug with other oxides (FeO and P_2O_5).

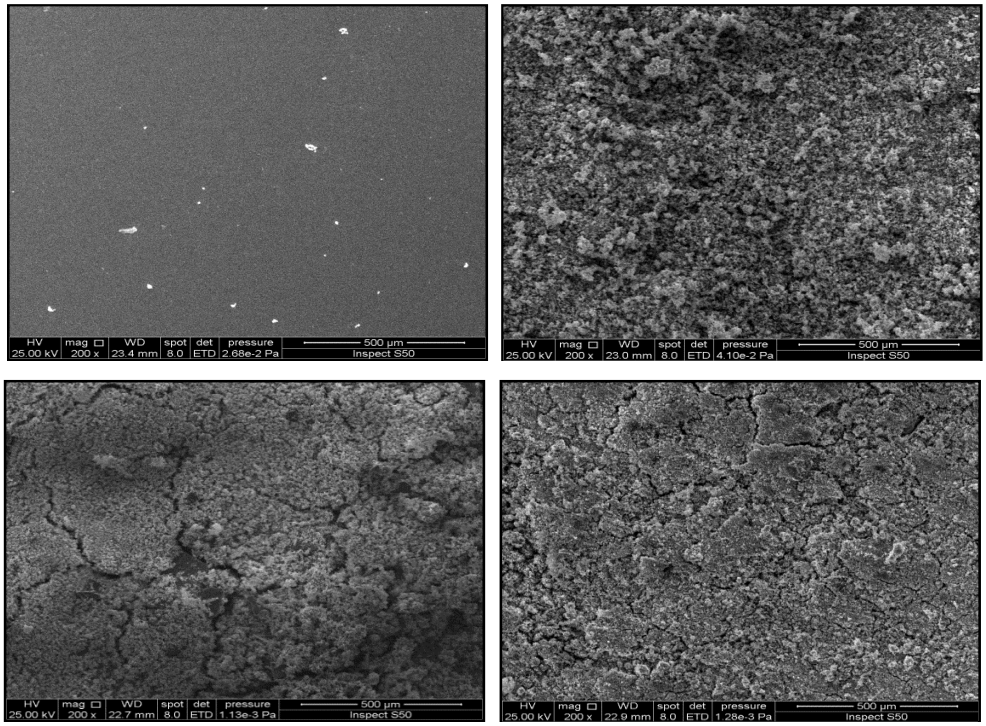
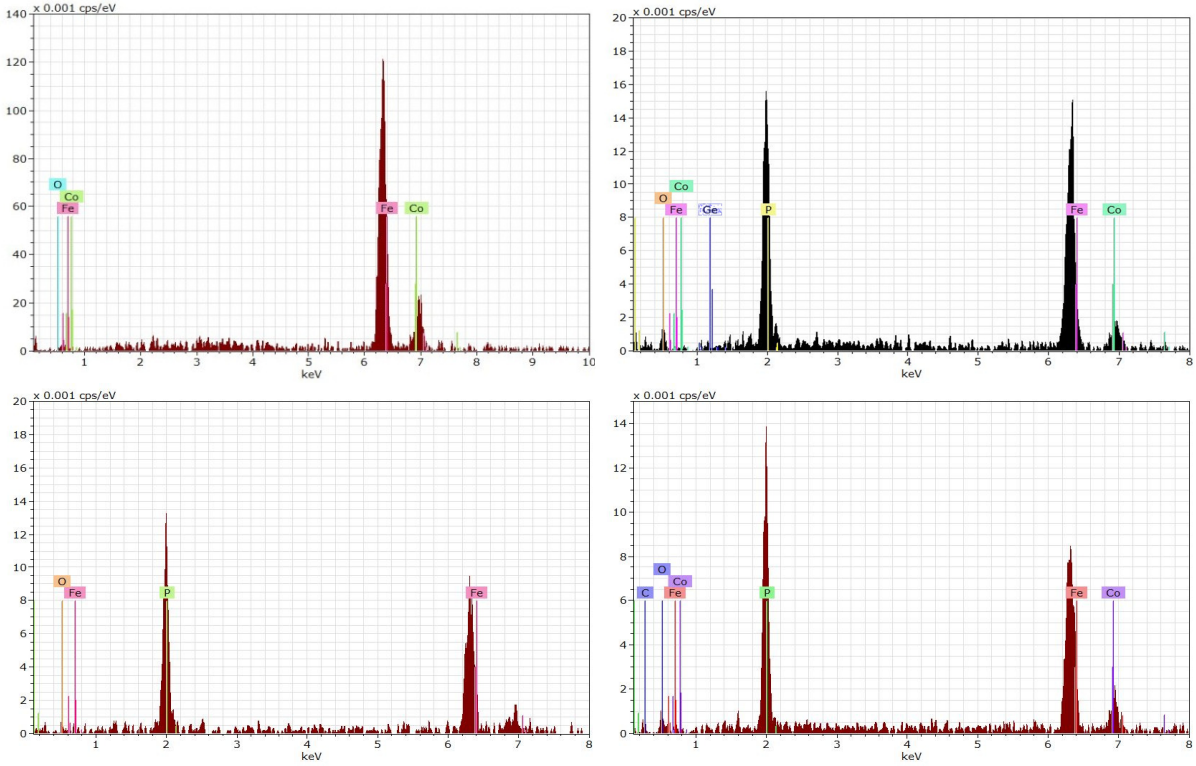


Fig. 5. SEM micrographs of carbon steel surface (a) before immersion in 0.5M H_3PO_4, (b) after corrosion in 0.5M H_3PO_4, (c) after inhibition 0.5M H_3PO_4 + 175 ppm of drug at 30^{o}C, and (d) after inhibition 0.5M H_3PO_4 + 225 ppm of drug at 60^{o}C.

The energy-dispersive x-ray spectroscopy (EDX) is an analytical technique utilized for the chemical analysis or elemental characterization of a specimen Fig. (6) indicates that the corroded and inhibited samples gave lower percents of Fe due to coverage the surface by passive layer and adsorbed drug. While the percent of oxygen was increased because of forming passive layers of FeO and P2O5 in addition to presence it in adsorbed drug. Carbon peak was appeared in the presence of the inhibitor. These results of SEM and EDX agreement with Moxifloxacin drug as green corrosion inhibitor for carbon steel in 1 M HCl [10].



Mass %	Fe	Co	O	P	C	FeO	P_2O_5
Fee	66.99	13.82	19.19	-	-	86.18	-
In H_3PO_4	28.92	3.38	39.34	24.36	-	35.42	55.81
175 ppm of drug at 303K	25.01	1.02	44.48	28.45	1.04	34.77	65.23
225 ppm of drug at 333K	24.98	7.05	40.99	25.96	1.02	33.43	59.52

Fig. 6. EDX analysis of (a) polished, (b) corroded, (c) inhibited sample at 303K and (d) inhibited sample at 333K.

The Fourier-transform infrared spectroscopy (FTIR) is spectrometer simultaneously collects high-spectral resolution data over a wide spectral range. Metoclopramide shows characteristic peaks as shown in Fig. (7); the characteristics peaks of Metoclopramide are the stretching of C-H aliphatic and aromatic at 3020.41-3377.47 cm-1 in addition to appear N-H stretching of amine at the same range. C=O stretching of amide occurs at 1599.04-1651.12 cm-1 and C=C stretching aromatic around 1500 cm-1, while C-N stretching appears at 1259.56 cm-1. N-H bending out of plane sometimes observes near 800 cm-1. C–O stretching in ether appears in the range of 1000-1300cm-1, and the peaks that appear between ≈600 to 800 cm-1 are indicated to C–Cl stretching vibration. The obtained same results with pantoprazole sodium expired drug as a corrosion inhibitor for high carbon steel in hydrochloric acid solution [18].

The FTIR spectrum of film formed on carbon steel after inhibition with two concentrations of drug indicates that there are more than one functionally groups to be attractive to the iron ions such as amine, amide and ester group as shown Fig. (7); in by the shifting in the vibration of these groups in FTIR spectra.

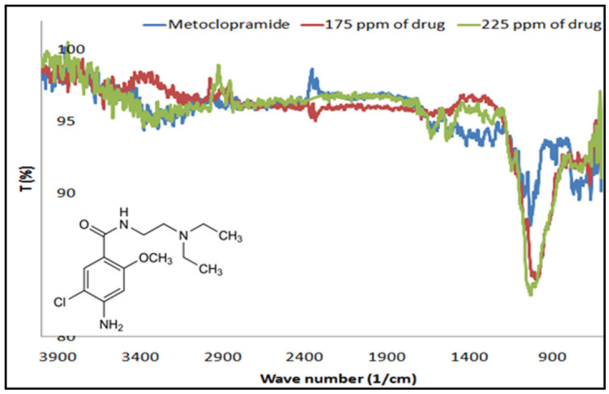
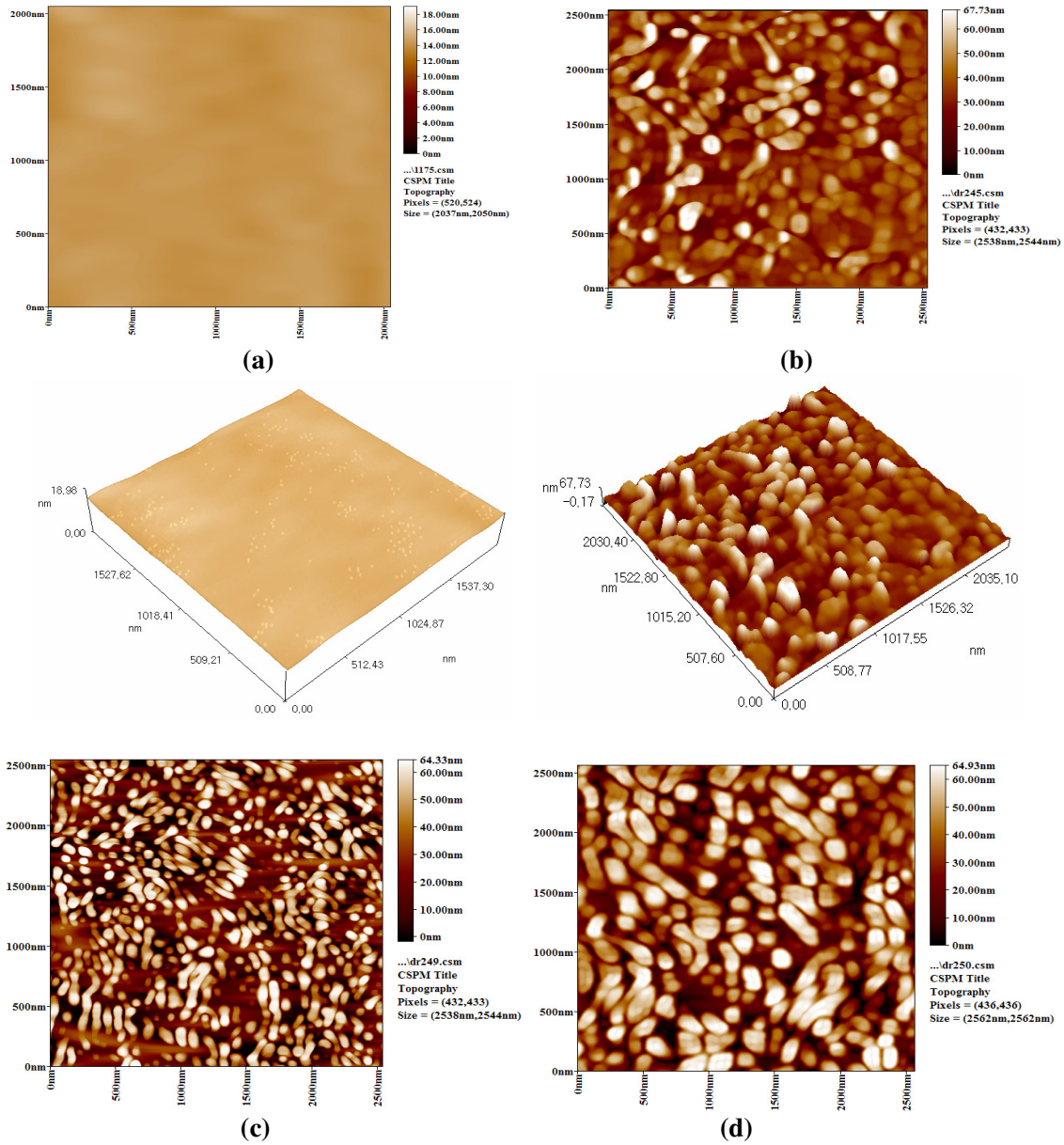


Fig. 7. FTIR spectra of drug (a) and film formed after adsorption on steel surface (b).

Fig. (8) shows the 2D and 3D images of polished, corroded and inhibited samples in presence of 175 and 225 ppm of drug in medium. These images describe the distribution of inhibitor molecules (drug) on steel surface to achieve the inhibition, the results were obtained with electrochemical and AFM study of corrosion inhibition of carbon steel by octanesulphonic acid-zinc ion system [19].

The average roughness of surface was increased after adsorption the drug on the surface due to the big size of drug molecules. The average diameter was decreased for inhibited surface especially in presence of 175 ppm of drug as shown in Fig. (8) and Table (6). This decreasing in diameter is attributed to the compact and perfect adsorbed layer of molecules on steel surface.



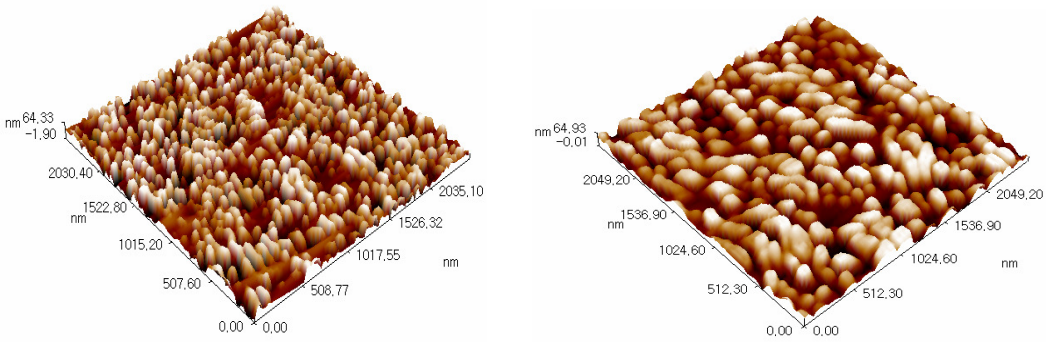


Fig. 8. 2D and 3D images of (a) polished, (b)corroded, (c) inhibited sample with 175ppm of drug and (d) inhibited sample with 225ppm of drug.

Fig. (9) shows granularity accumulation distribution charts for different samples, these charts indicate the different distribution of particles on the steel surface.

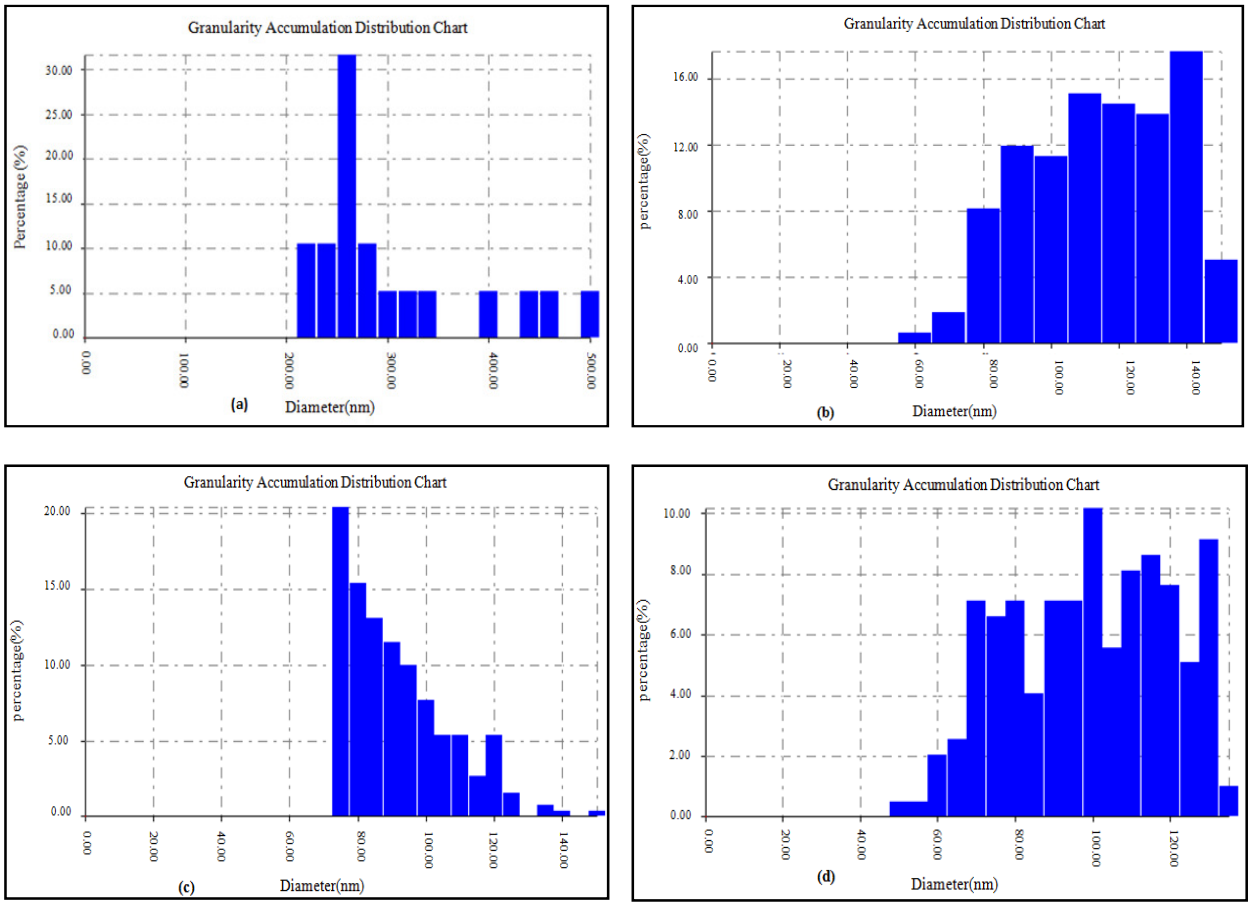


Fig. 9. Granularity accumulation distribution of particles for (a) polished, (b) corroded, (c) inhibited sample with 175ppm of drug and (d) inhibited sample with 225ppm of drug.

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المثبط المضاد للبكتريا كميتوكلوبراميد منتهي الصلا□يه في ٠,٥ مولاري من□امض الفسفوريك

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الخلاصة

الدواء المنتهي الصلاحيه ميتوكلوبراميد تم اختباره كمثبط التأكل المضاد للبكتريا للفولاذ كاربوني في محلول ٠,٥ مولاري من حامض فسفوريك بأستخدام الطريقة الكهروكيميائية في ٣٠ و ٦٠ درجة مئوية. وأظهرت النتائج أن هذا الدواء هو مثبط فعال للفولاذ كاربوني والكفاءة وصلت إلى ٨٢,٢٤٤ % لل ١٧٥ جزء من مليون عند ٣٠ درجه مئويه و٧٦,١٤٦ % لل ٢٢٥ جزء من مليون عند ٦٠ درجه مئويه. امتزاز الدواء على السطح فولاذ كاربوني يتبع متساوي الحراره الامتزاز مع القيم الصغيره لثابت امتصاص الامتزاز. كشفت سير الاحداث في الاستقطاب ان ميتوكلوبراميد يعمل كمثبط من نوع المختلط . بعض معامل عمليه التثبط تم حسابها ومناقشتها. التشكل السطحي للعينات فولاذ كاربوني تم تحليلها بأستخدام المجهر المسح الألكتروني و تحليل الأشعة السينية المشتتة للطاقة. تم اختبار المثبط الفعال المضاد للبكتريا. أكد التحليل الطيفي دور المثبط للدواء ميتوكلوبراميد.