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Expired Colxacillin, Amoxicillin, and Ceflaxin Drugs as Inhibitors for Low Carbon Steel Corrosion in Sodium Chloride

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Abstract

The ability to inhibit corrosion of low carbon steel in a salt solution (3.5%NaCl) has been checked with three real expired drugs (Cloxacillin, Amoxicillin, Ceflaxin) with variable concentrations (0, 250, 500, 750) mg/L were examined in the weight loss. The inhibition efficiency of the Cloxacillin 750 mg/L showed the highest value (82.8125 %) and the best inhibitor of the rest of the antibiotics. The different concentrations of Cloxacillin drug (0, 250, 500, 750) mg/L and temperature (25, 35, 45, 55) °C were studied as variables with potentiodynamic polarization, Scanning Electron Microscopy (SEM) for surface morphology and electrochemical impedance spectroscopy (EIS) depending on current values and the resistance of charge to calculate the inhibition efficiency. The main observations of these tests were that polarization curves showed a mixed-type inhibition of expired Cloxacillin. The inhibition efficiency increased with increasing Cloxacillin concentration but not with increasing temperature.

***Keywords**: Polarization, Inhibitor, Expired Drugs, Cloxacillin, Amoxicillin, Ceflaxin, Salt corrosion.*

1. Introduction

Due to the good mechanical properties of low Carbon steel alloy, this material is the generally used in processing plants and household applications, availability, and comparatively low cost[1, 2]. Chloride-containing solutions, like seawater, are in most cases, corrosive because the depolarization impacts carbon ions, the main case is the low resistance of low carbon steel to corrosion [3, 4]. It has been found that one of the preferable ways of keeping metals from corrosion comprises the use of expired antibiotics as corrosion inhibitors. The prevention or decreasing of corrosion impacts on metals like low carbon steel by using expired antibiotics is a method created because of the toxicity of many corrosion inhibitors, therefore; the researchers in the corrosion field selected compounds that are friendly to the environment, safe, and non-toxic such as pharmaceutical drugs [5]. Their capability to make a complex with metal ions on the metal surfaces through their functional groups. Perfectly the drugs soluble in the water besides their high purity and inexpensively as expired antibiotics are important factors in choosing expired drugs as corrosion inhibitors [6]. These characteristics would support the employment of expired drugs as corrosion inhibitors in various aqueous solutions.

Generally, expired antibiotics are large surface area materials, including functional groups (N) with an effective cover above the surface of low-carbon steel for the adsorption process[7, 8, 9].

The following table gives a comparison of (Inhibition Efficiency) IE percentage between various antibiotics (Table 1.).

Table 1,
Examples of pharmaceutical inhibitors with their IE percentage[1].

Inhibitor	Sample	Medium	IE %
Carvedilol	Carbon steel	HCl	98.94
Amifloxacin	Mild steel		17.1
Enrofloxacin	Mild steel		18.3
Cefotaxime	Mild steel		90.0

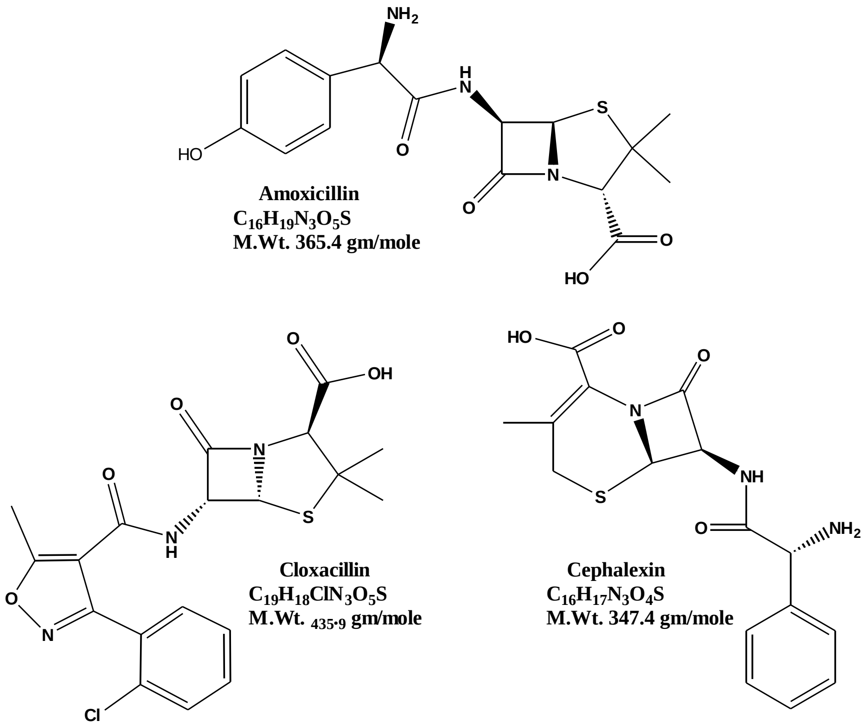


Fig.1. Chemical structure of the expired drugs used in the test with molecular weights for each one.

Here, the main goal is to test the corrosion–inhibition relationship as its rate and its effects on the low carbon steel in a medium of (NaCl) with the absence and presence of an inhibitor and on another occasion when the inhibitor is used in different concentrations with different temperatures. The inhibitors, as shown in the experimental section, were real expired antibiotics because of their cost and economical serving forwarding limitation of environmental pollution with pharmaceutically active compounds.

2. Experimental Methods
2.1 Materials

The sample is low carbon steel with a chemical composition of “weight %” (0.18 Carbon, 0.9 Manganese, 0.05 Sulphur and the rest is Iron). The expired drugs "Cloxacillin, Amoxicillin, and Ceflaxin” were tested as corrosion inhibitors. The antibiotics were collected from (Samara`a drugs industry, Iraq, Samara) as a pure expired antibiotics for two years. Sodium Chloride solution NaCl as a corrosive medium from CDH company, India.

2.2 Weight loss method

The experiments of this method were done by immersion of low carbon steel specimens with size (3x3x1) cm^3 in 3.5% NaCl (2 g of salt in 60 ml of water) solution for five days. Also, low carbon steel was immersed in the same corrosive media with the presence of one of the three bioactive inhibitors (250, 500, 750) mg/L for a period of 5 days. The obtained results were tabulated in Table 2. using the equations listed below [10]. The specimens are cleaned before and after the test using a different aluminum paper of various degrees (p40, p50, p100, p140, p220).

$$IE\% = \theta * 100 \quad \dots (1)$$

$$IE\% = 1 - \frac{W_{inh}}{W_{free}} \quad \dots (2)$$

$$corrosion\ rate = \frac{weight\ loss\ (grams)*k}{alloy\ density\ \frac{g}{cm^3}*exposed\ area*exposure\ time(hr)} \quad \dots (3)$$

Where IE% is the inhibition efficiency, θ is the surface coverage while W_{inh} and W_{free} are the weight loss in the presence and absence of inhibitor respectively.

2.3 Electrochemical tests

From weight loss results, the inhibitor Cloxacillin shows the best results in protecting metal surfaces due to the high molecular weight of the inhibitor [11]. Cloxacillin was chosen to complete the other tests as SEM and electrochemical tests. The electrochemical tests were done in a three-electrode cell with platinum as the counter electrode, saturated calomel as the reference electrode (SCE), and low carbon steel as the working electrode. Open circuit potentials OCP were carried out in the electrolyte before running the electrochemical measurements [12]. The specimens were immersed in the electrolyte for 30 min, followed by OCP measurement for 60 min. When calculating the corrosion current, the method can be used is the Stern-Geary by extrapolation of anodic and cathodic Tafel lines to the point that gives $\log i_{corr}$ and the conformable corrosion potential (E_{corr}) for inhibitor-free salt and different concentration of the antibiotics used. All practical experiments were done at 25°C [13].

2.3.1 Tafel polarization

A Potentiostat was used in this test, and kinetics parameters were found from Tafel curves such as corrosion current (I_{corr}), cathodic Tafel slope (β_c), anodic Tafel slope (β_a), corrosion potential E_{corr} and IE%. The experiment was done by using three concentrations of Cloxacillin as corrosion inhibitor (0, 250, 500, and 750) mg/L, with four degrees of temperature (25, 35, 45, and 55) °C for each concentration of inhibitor.

2.3.2 Electrochemical Impedance Spectroscopy (EIS)

Electrochemical Impedance Spectroscopy (EIS) was carried out at open circuit potential in the frequency range of (10^{-1} -10^{5}) Hz. EIS plots are found in the Nyquist and bode diagrams. The electrochemical studies were carried out with an electrochemical CS 310 CorrTest workstation, Iraq, Baghdad, University of Baghdad (Al Khwarizmi Engineering College).

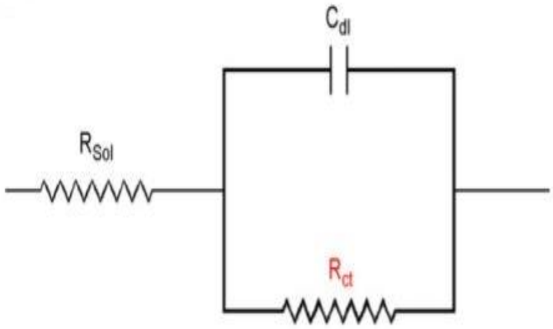


Fig. 2. Randle circuit in Impedance spectroscopy.

2.4 Surface study

Surface morphology as an indicator of how much salts affect the pieces of low carbon steel and how the corrosion inhibitors resist the corrosion and save the surface is tested by a Scanning Electron Microscope (SEM). The specimens with Cloxacillin (inhibitor) were chosen for SEM by the Tescan mirah3 instrument (Dansh Bryan Company, Tehran, Iran) where the scanned area was (1-6) µm.

3. Result and Discussion

3.1 Weight loss test

Weight loss measurements of low carbon steel samples were reported after 5 days for three real expired drugs (Cloxacillin, Amoxicillin, Ceflaxin) as shown in Table 2. The maximum IE, % at Cloxacillin 750 mg/L showed the highest value (82.8125 %) and the best inhibitor of the rest of the antibiotics. Table 3 showed the weight loss in the absence of inhibitors.

Table 2,
Weight loss results of the three bioactive inhibitors. [W $_{free}$ = 0.064 gm]

Inhibitor name	Antibiotic conc., mg/L	W $_{inh}$, gm	Surface coverage (θ)	IE, %
Cloxacillin	250	0.023	0.640625	64.0625
	500	0.021	0.671875	67.1875
	750	0.011	0.828125	82.8125
Amoxicillin	250	0.033	0.484375	48.4375
	500	0.030	0.53125	53.1250
	750	0.021	0.671875	67.1875
Ceflaxin	250	0.034	0.46875	46.8750
	500	0.031	0.515625	51.5625
	750	0.028	0.562500	56.2500

Table 3,
Weight loss results in the absence of inhibitor.

Solution kind	Original weight (g)	Final weight (g)	The difference in weight(g)	Corrosion rate (mm/a)
NaCl	7.03	6.9834	0.064	0.182

3.2 Electrochemical tests

Potentiodynamic polarization measurements have been carried out. Potentiodynamic polarization curves for low carbon steel metal in 3.5% NaCl solution with different concentrations of Cloxacillin expire drug are displayed in Figures (3-5) with different temperature (25, 35, 45, 55)^{o}C. Cloxacillin was chosen for the test of Tafel curves because it gave the best result in the weight loss experiment as 82% IE%.

May be noticed Table 4, the anodic and cathodic Tafel slopes (βa, βc) curves with the presence of inhibitors and which turns lower current density (I_{corr}). It can be observed in the presence of inhibitors both the anodic and cathodic curves are shifting towards lower current density. Which reveals that inhibitor molecules are adsorbed on the metal surface. Although the corrosion potential of the metal nearly remain constant, there is a slight shift towards more positive potential at higher concentrations with respect to the corrosion potential observed in the absence of inhibitor. Figures (3-6) show the effect of inhibitor concentration and temperature on current density and voltage with four temperature degrees (25, 35, 45, 55)◦C respectively.

It is noticed that the same concentration of the inhibitor with different temperature sometimes increases and other decreases, but at 750 mg/l of Cloxacillin at 35 ^{o}C gives minimum corrosion rate.

Table 4,
Electrochemical study of Cloxacillin

Conc., mg/L	Temp.^{o}C	E_{corr} (V/SCE)	I $_{corr}$*10^{-6}(A/cm^{2})	Corrosion rate (mm/a)	β_a(V/dec)	β_c(V/dec)	IE%
Blank	25	-0.95073	15.5	0.18200	267.47	112.79	-
	35	-0.72475	21.4	0.25097	86.564	456.7	-
	45	-0.86188	69.2	0.81135	252.57	156.08	-
	55	-0.77695	15.1	0.17756	77.437	231.38	-
250	25	-0.78254	6.91	0.081114	71.752	200.77	55.3
	35	-0.84599	4.60	0.053967	82.62	147.35	78.5
	45	-0.84487	6.07	0.071241	88.361	150.83	91.2
	55	-0.83344	4.46	0.05821	77.581	140.9	70.5
500	25	-0.77939	6.50	0.076252	71.197	212.99	58.0
	35	-0.80915	7.07	0.082971	73.877	137.09	66.9
	45	-0.79384	11.7	0.13686	70.256	211.44	83.1
	55	-0.82055	8.67	0.10175	74.232	173.69	42.7
750	25	-0.75942	1.95	0.22825	67.527	67.527	87.4
	35	-0.85093	3.51	0.041119	86.244	86.244	83.6
	45	-0.84281	5.58	0.065418	79.704	79.704	91.9
	55	-0.84777	5.70	0.066828	88.548	88.548	62.4

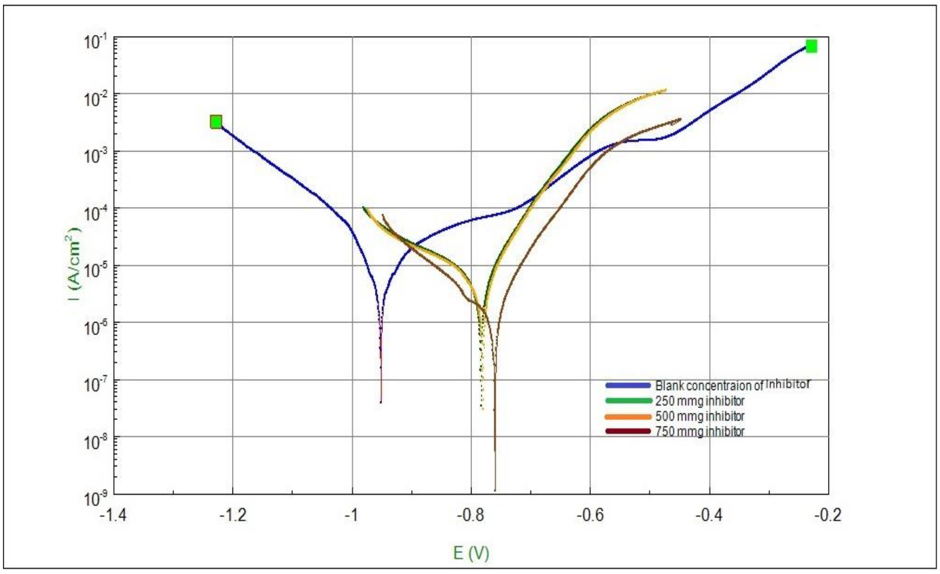


Fig. 3. Variation of inhibitor concentration on current density and voltage at 25°C.

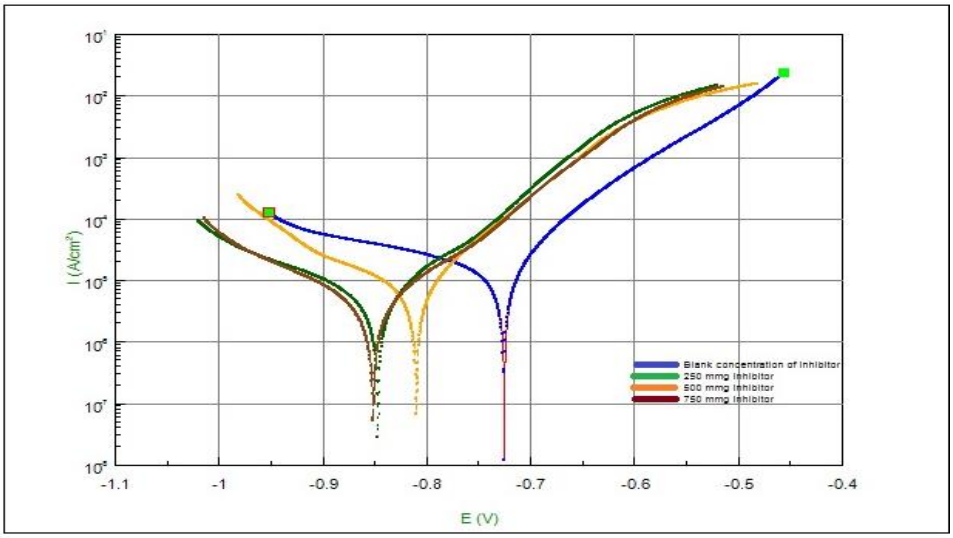


Fig. 4. Variation of inhibitor concentration on current density and voltage at 35oC

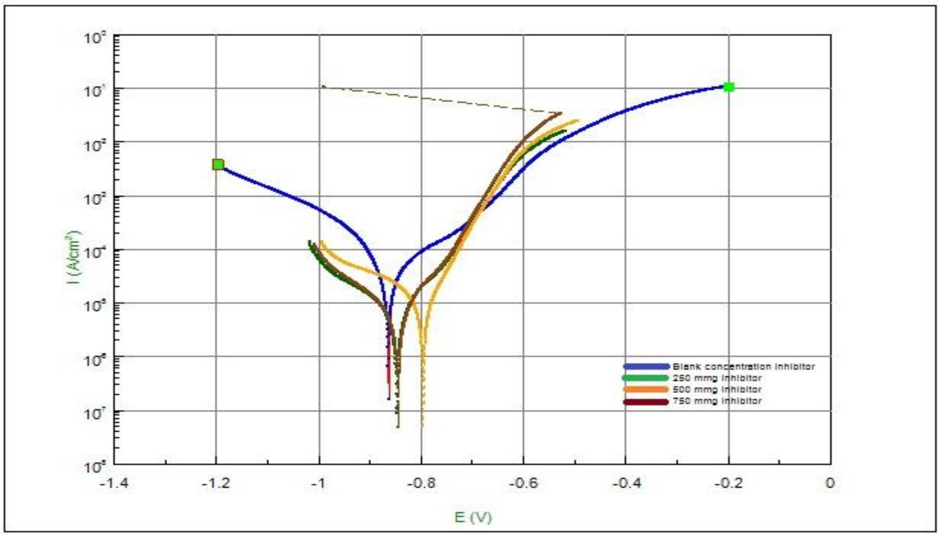


Fig. 5. Variation of inhibitor concentration on current density and voltage at 45oC

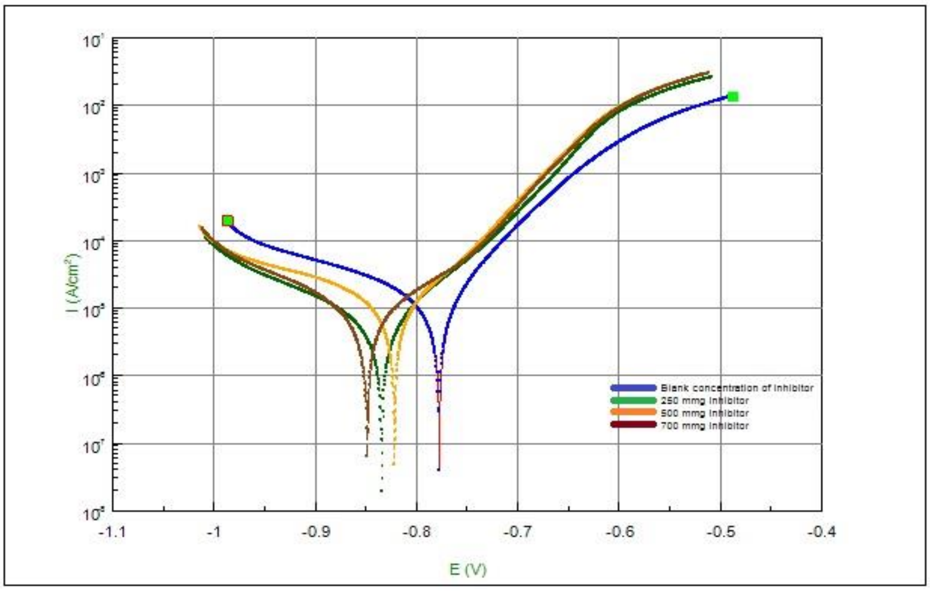
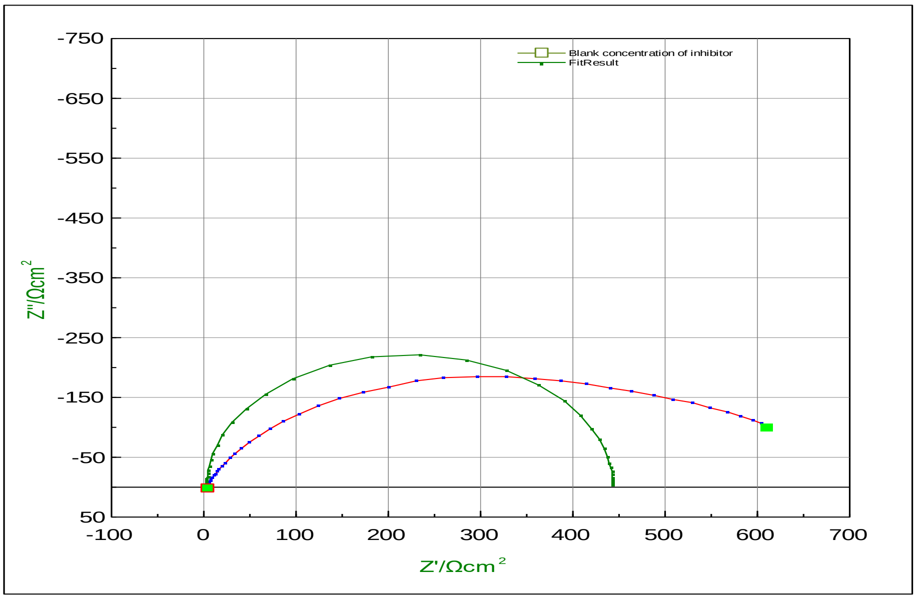


Fig. 6. Variation of inhibitor concentration on current density and voltage at 55 oC.

3.3 Electrochemical Impedance Spectroscopy (EIS)

In these experiments, Cloxacillin was used with 3.5% NaCl solution at different temperature degrees by using an electrical circuit called Randle circuit where C_{dl} is called double-layer capacitor, R_{ct} is the charge transfer resistance and R_{sol} is the solution resistance as shown in Table 4 [14]. Experimental results of this method are shown in Figures (7-14) Nyquist, Bode and (log(f)-log(z)) and (f)-phase) diagrams of low-carbon steel, which were obtained in 3% of NaCl solution in the absence and presence of various concentrations of Cloxacillin are shown. It can be seen from Figures (7a-14a) the Nyquist plot of the metal shows a depressed semi-circular shape. The obtained graphs (7b,c-14b,c) showed how bio-inhibitor affected the real and imaginary resistance introduced where Z'' is the imaginary resistance, Z' is the real resistance, f is the frequency and θ° is the phase values.



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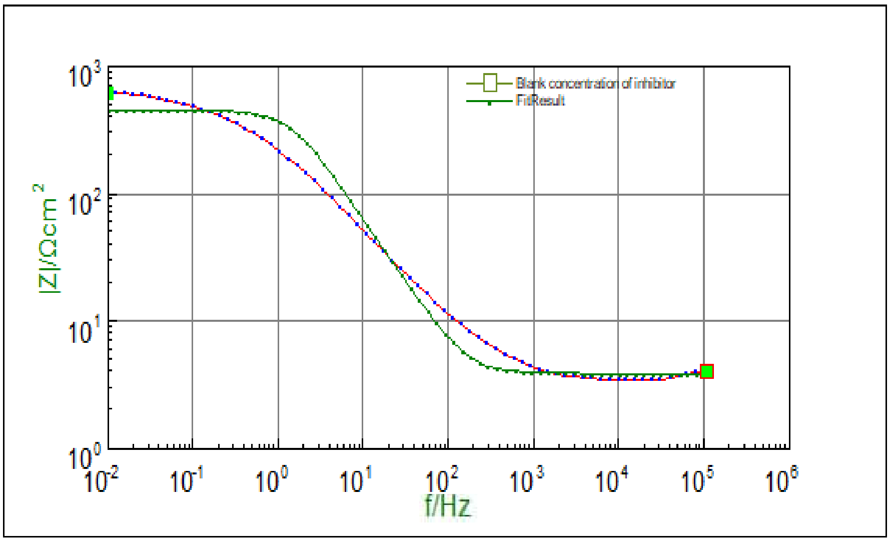

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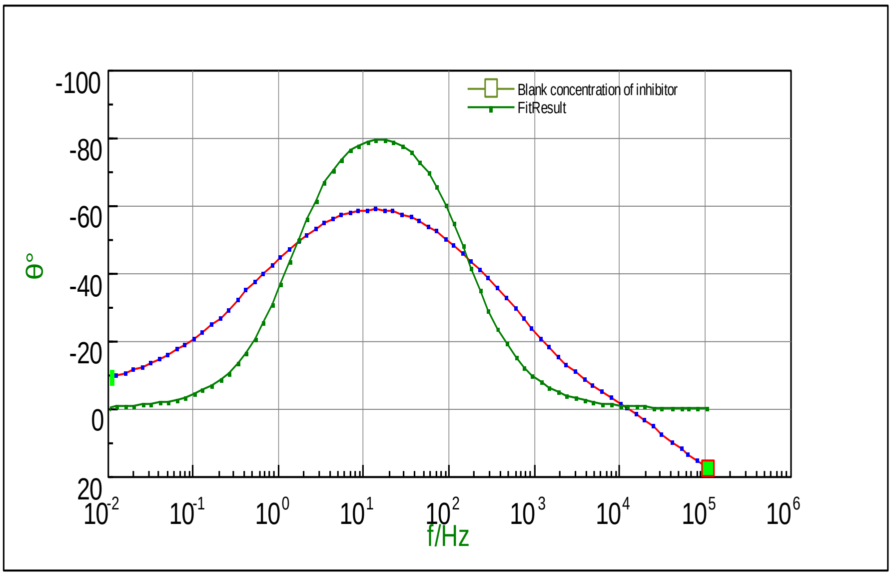
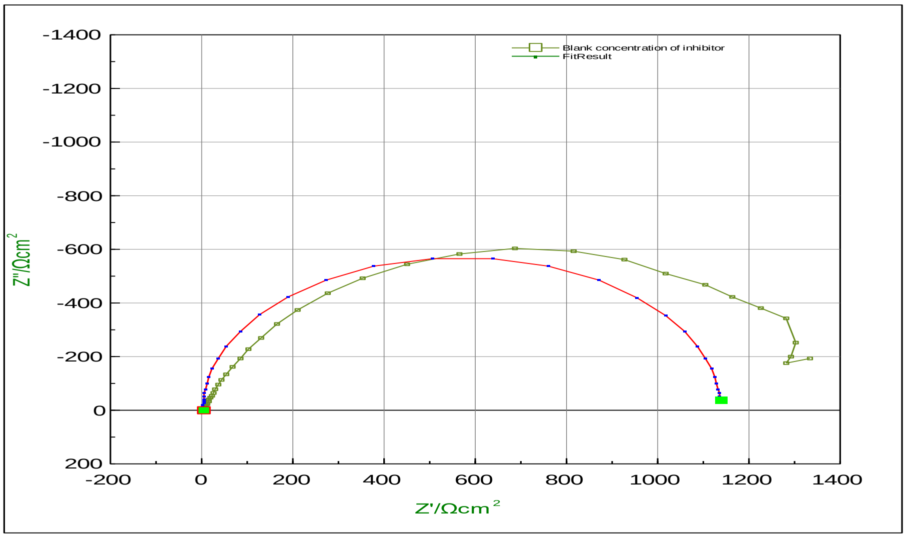


(c)

Fig. 7. The EIS test for blank concentration of inhibitor at 25◦C with the fitting result. . (a) Nyquist plot, (b, c) Bod (log versus |z|), and phase angle (log f versus $\propto^{0}$) plots.



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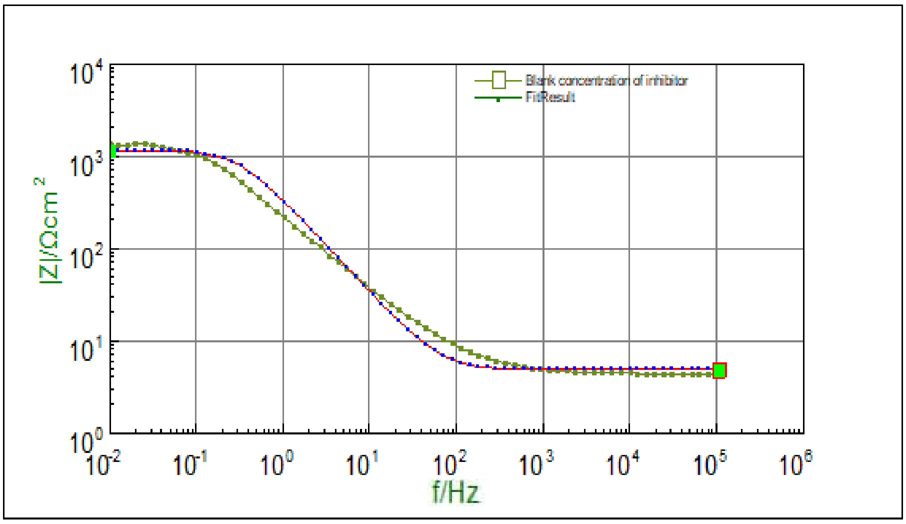

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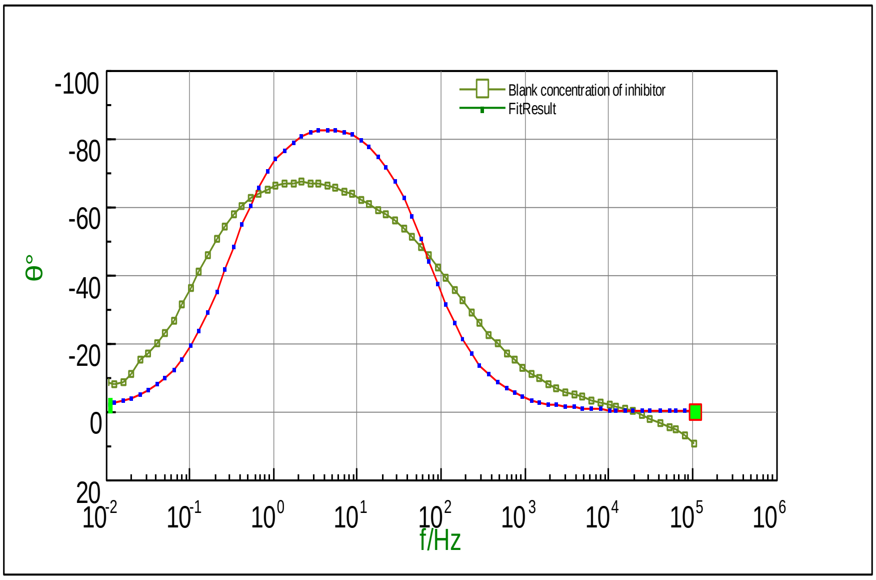

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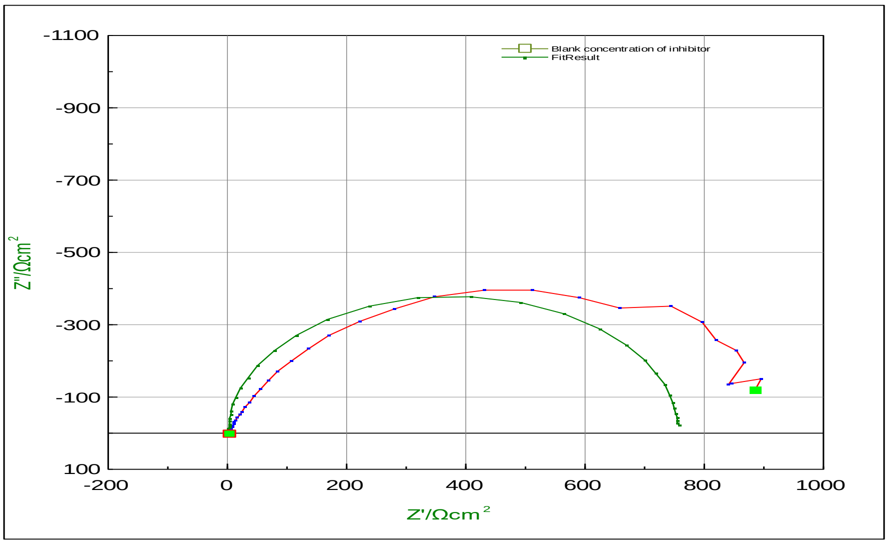
Fig. 8. The EIS test for blank concentration of inhibitor at 35◦C with the fitting result. . (a) Nyquist plot, (b, c) Bod (log versus |z|), and phase angle (log f versus $\propto^{0}$) plots.



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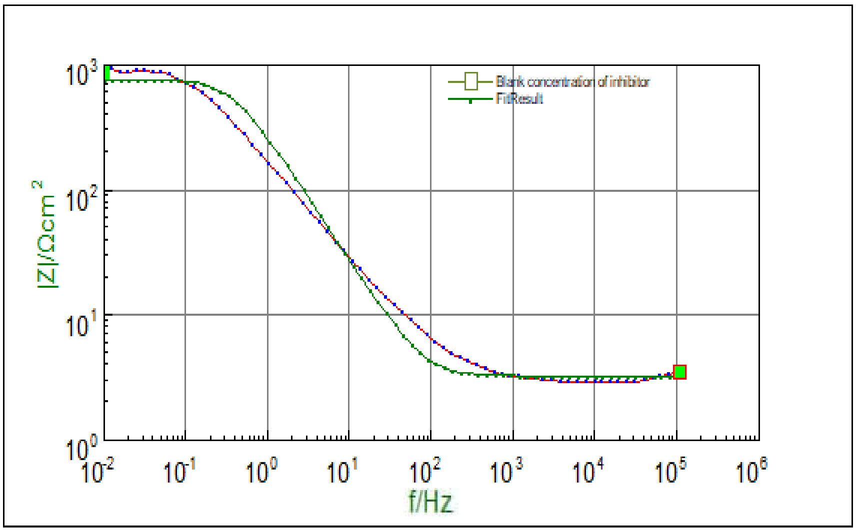

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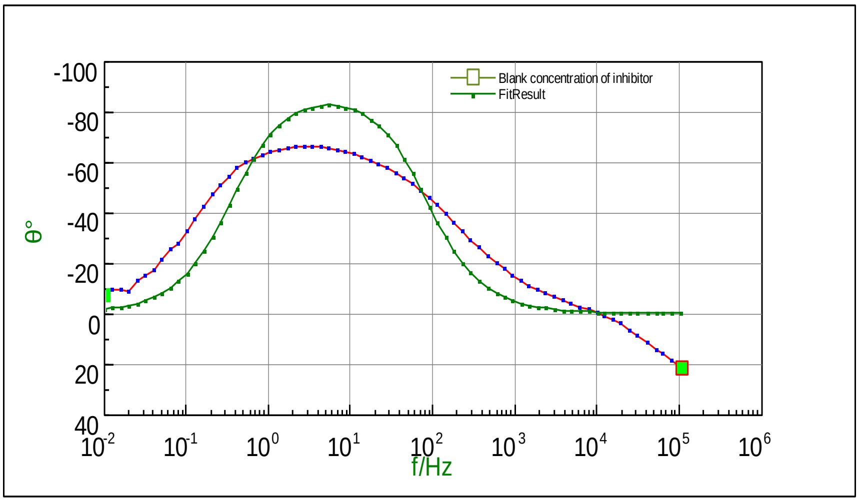

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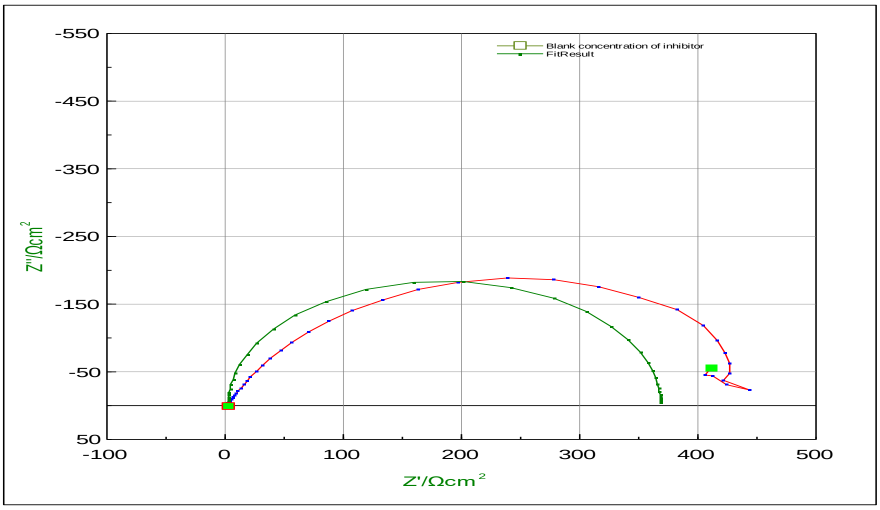
Fig. 9. The EIS test for blank concentration of inhibitor at 45◦C with the fitting result.. (a) Nyquist plot, (b, c) Bod (log versus |z|), and phase angle (log f versus $\propto^0$) plots.



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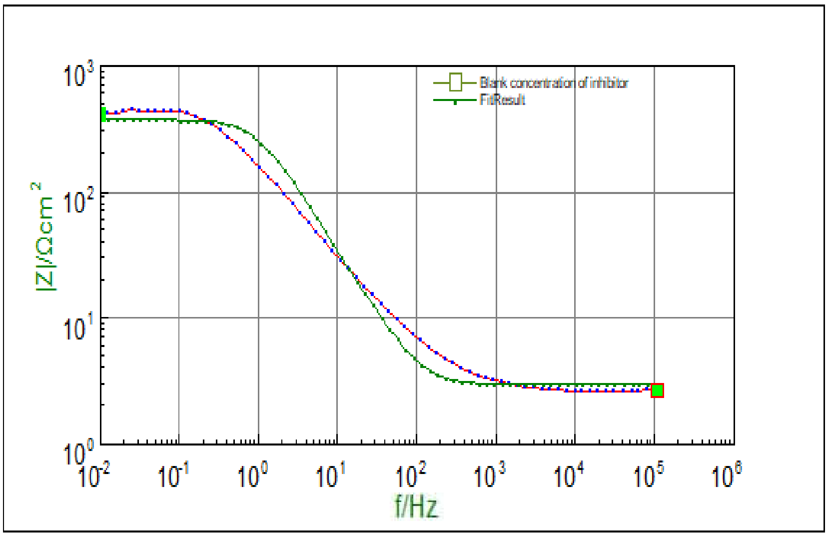

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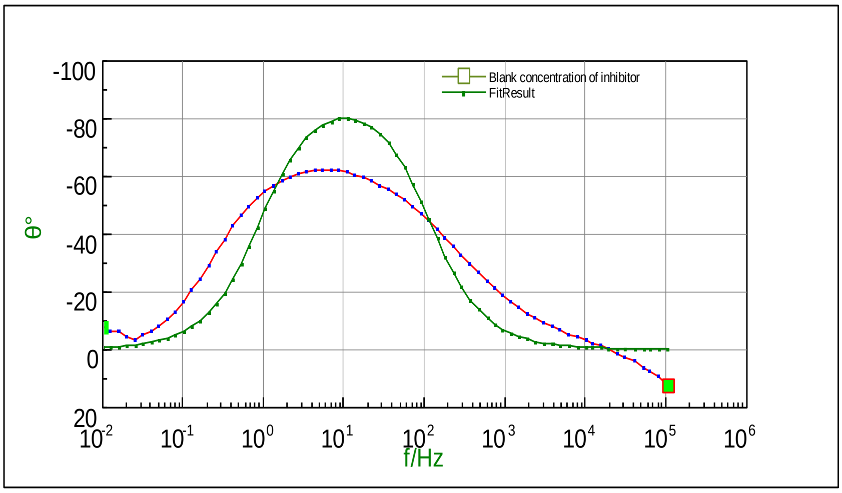

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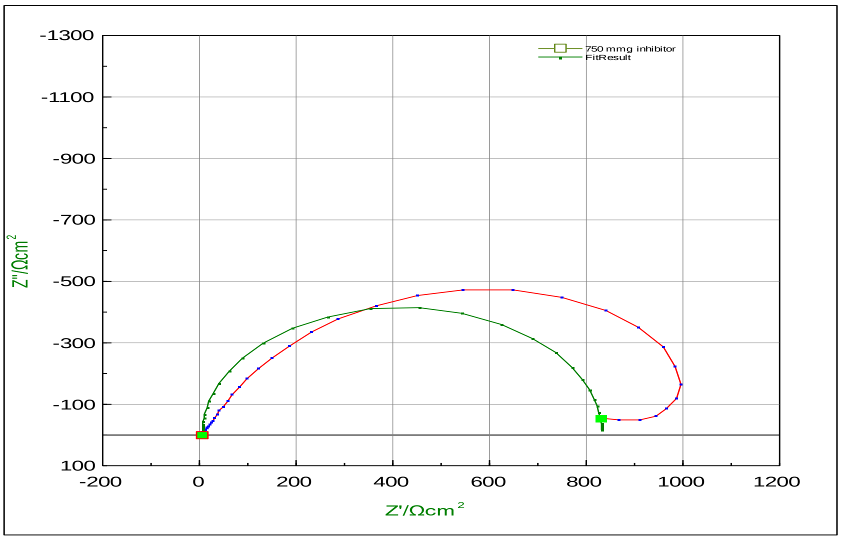
Fig. 10. The EIS test for blank concentration of inhibitor at 55◦C with the fitting result. (a) Nyquist plot, (b, c) Bod (log versus |z|), and phase angle (log f versus $\propto^0$) plots.



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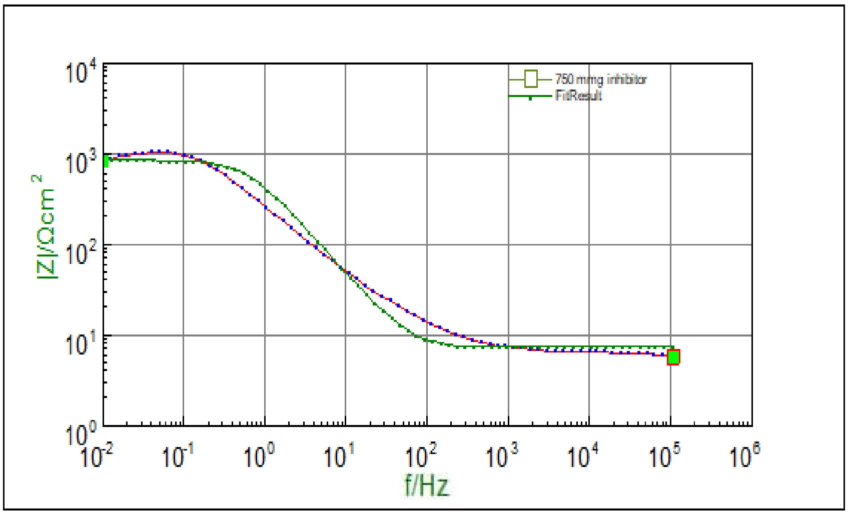

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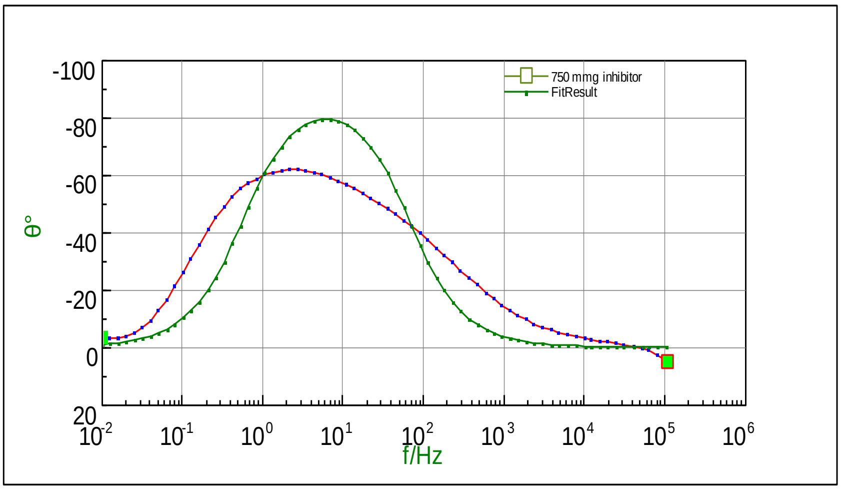

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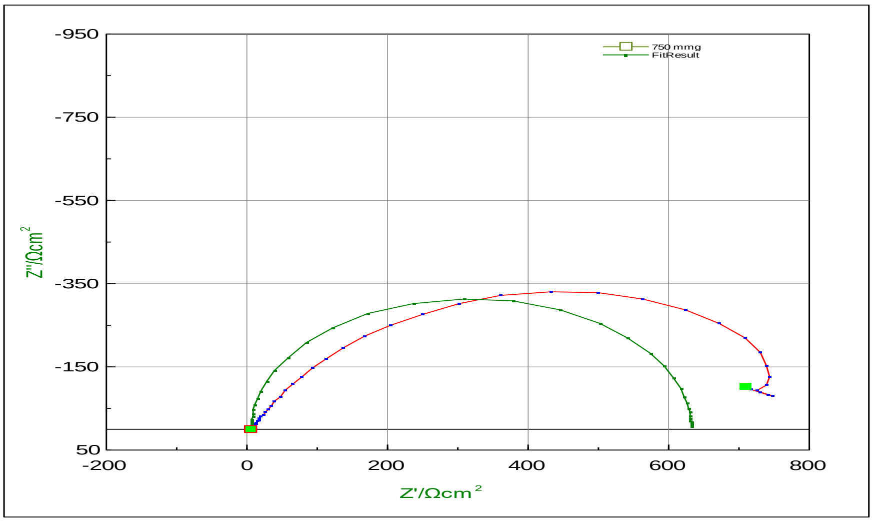
Fig. 11. The EIS test for 750 mg/l concentration of inhibitor at 25◦C with the fitting result. (a) Nyquist plot, (b, c) Bod (log versus |z|), and phase angle (log f versus $\propto^{0}$) plots.



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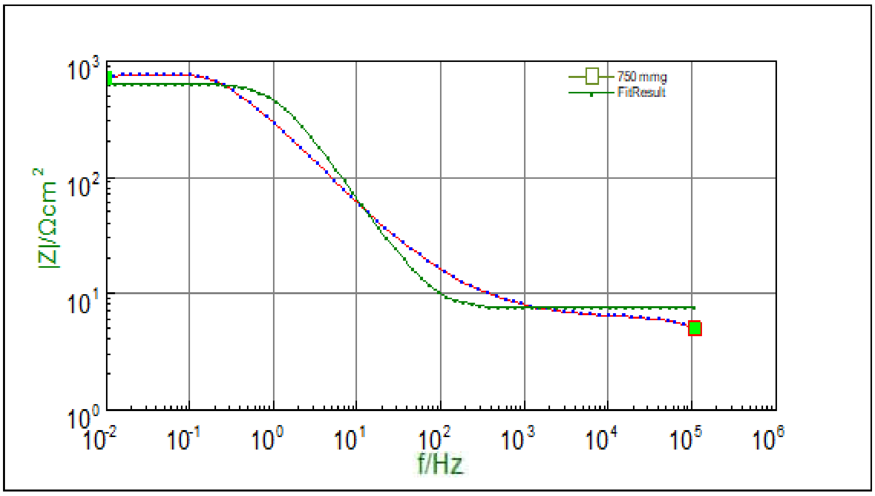

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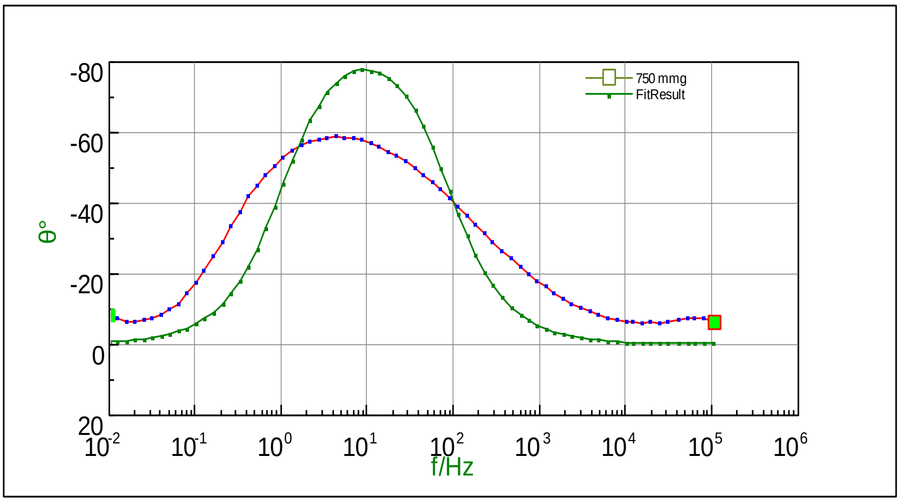

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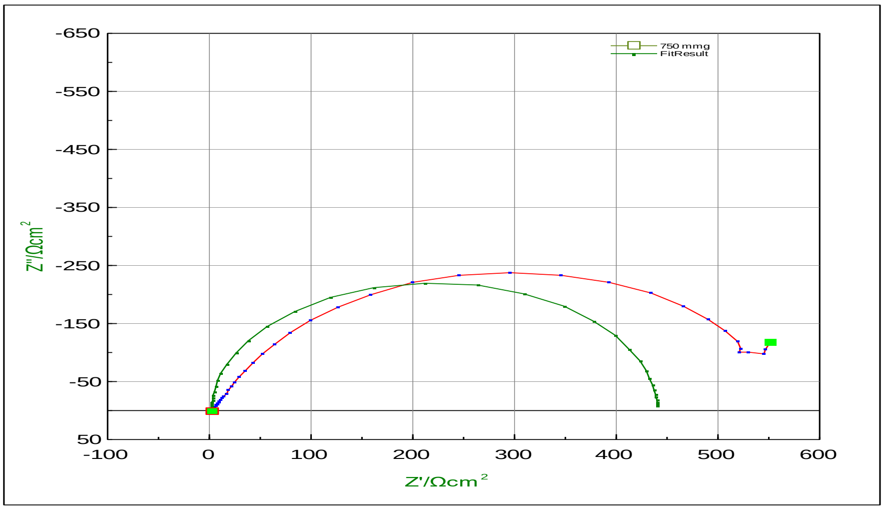
Fig. 12. The EIS test for 750 mg/l concentration of inhibitor at 35◦C with the fitting result. (a) Nyquist plot, (b, c) Bod (log versus |z|), and phase angle (log f versus $\propto^{0}$) plots.



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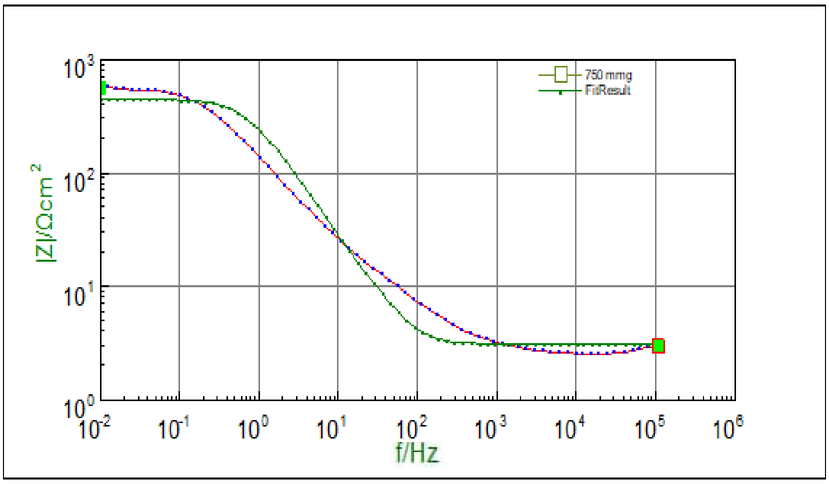

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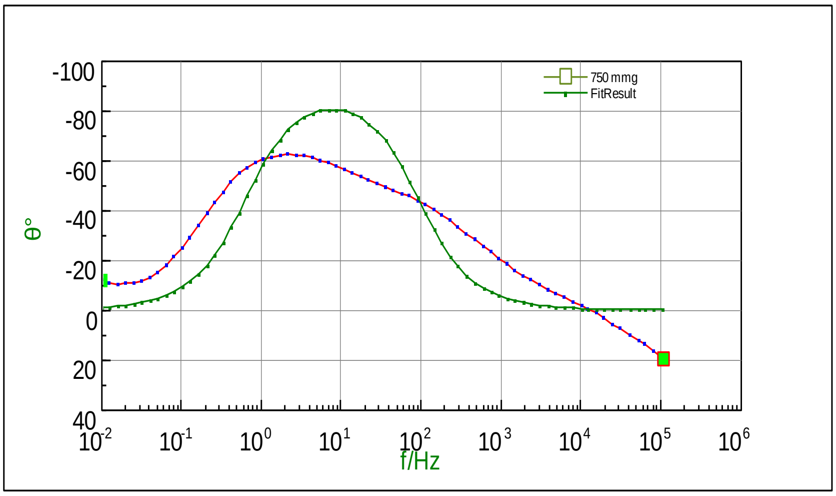

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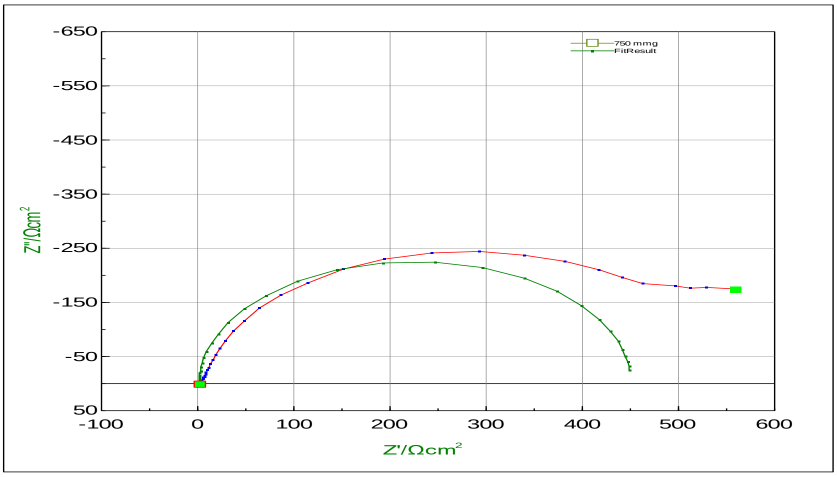
Fig. 13. The EIS test for 750 mg/l concentration of inhibitor at 45◦C with the fitting result. (a) Nyquist plot, (b, c) Bod (log versus |z|), and phase angle (log f versus $\propto^0$) plots.



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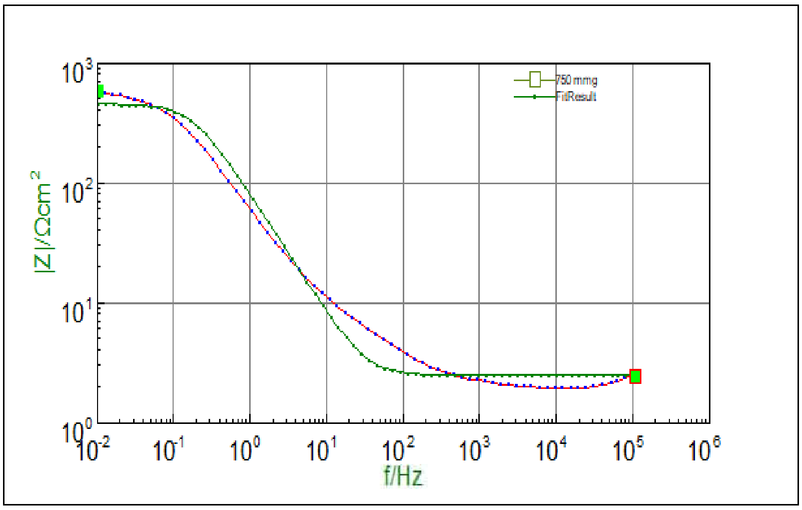

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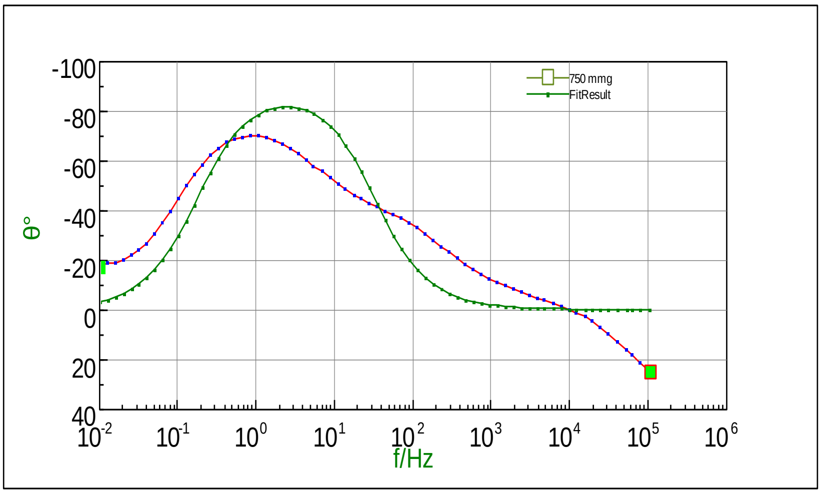

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Fig. 14. The EIS test for 750 mg/l concentration of inhibitor at 55◦C with the fitting result. (a) Nyquist plot, (b, c) Bod (log versus |z|), and phase angle (log f versus $\propto^{0}$) plots.

It was noticed that the impedance spectra exhibit a semi-circle caused by the frequency dispersion effect which means that the corrosion of low carbon steel in NaCl solution was controlled by a charge transfer process[15]. The diameter of the capacitive loop in the presence of an inhibitor was larger than in the absence of the inhibitor (blank concentration) and increased with inhibitor concentration. This observation conflicts with the increasing impedance of inhibited metal with increasing concentration.

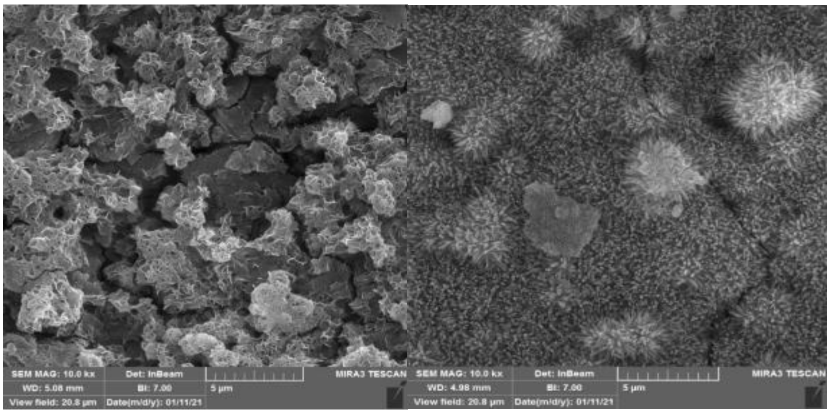
This irregular behavior is generally caused by the roughness and inhomogeneity of the alloy surface [5]. The charge transfer resistance R_{ct} value increased with increasing of concentration. But it turns out that the capacitance values (C_{dl}) of the double-layer decrease, and this decrease is expected in the presence of the local dielectric constant. This causes an increase in the thickness of the electrical double layer [16]. It is observed from Table 5 the 750 mmg concentration of Cloxacillin at 45°C give the maximum IE% and maximum R_{ct} .

Table 5,
Values of the EIS test for absence and presence of Cloxacillin.

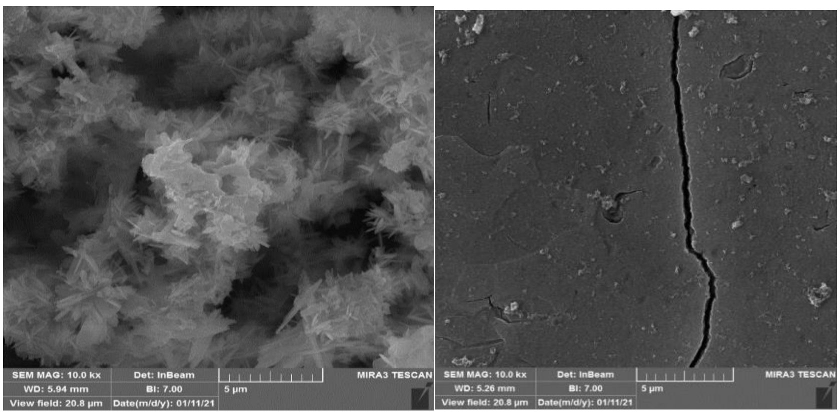
Conc.mg/l	Temp.C	R_{Ct}	IE%	$C_{dl}\ \mu f$
Blank	25	174.65	-	0.543
Blank	35	600.01	-	0.345
Blank	45	347.95	-	0.231
Blank	55	135.86	-	0.124
750	25	835	88.04	0.431
750	35	955	83.78	0.335
750	45	1135	90.46	0.211
750	55	795	60.76	0.105

3.4 Surface Analysis

The SEM micrographs for low carbon steel in presence and absence of 750 mg/L of Cloxacillin are shown in Figure 15(a-d). It is obviously seen from Figure 15a that the metal surface is strongly damaged in the absence of the inhibitor. In Figure 15d showed that the film is stable and protective which support the results of electrochemical measurements which are discussed above.



(a) (b)



(c) (d)

Fig. 15. SEM of (a) blank (b) 250 mg/L (c) 500 mg/L (d) 750 mg/L of Cloxacillin.

3.5 The mechanism of inhibition

The adsorption of expired antibiotics are to be the first step at the alloy/solution interface done in the mechanism of corrosion inhibition in the salt solution medium[17]. This becomes acceptable for the first step during the adsorption of an antibiotics drug as organic materials on the surface of the alloy, and this involves by substituting one or more water molecules adsorbed on the surface of the alloy[18], as in equation below[19] :

Organic (inhibitor) + $xH_2O \rightarrow$ organic (adsorbed) + x H_2O … (3)

4. Conclusion

The studied of expired antibiotics drugs, (cloxacillin, amoxicillin, cephalexin) can be used as good organic inhibitors for corrosion in salt solution on the low carbon steel metal in 3% NaCl as a salt solution. Cloxacillin has better inhibition, because of its high molecular weight, molecular surface, and functional groups as compared to the two others expired antibiotic drugs. Increasing the concentration of antibiotics increased the inhibition efficiency and decreased corrosion rate.

The minimum corrosion rate appears at temperature 35°C with 750 mg/l concentration of Cloxacillin.

EIS plots indicated that R_{ct} values increase whereas C_{dl} values decrease in presence of Cloxacillin. The changes in the impedance parameters confirmed the strong adsorption of the inhibitor on the steel surface.

SEM studies revealed that Cloxacillin form protective surface film.

5. References

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المضادات الحيوية المنتهية الصلاحية (الكولكساسيلين و الاموكسيسيلين والسيفلاكسين) كمثبطات للصدأ الحاصل للحديد القليل الكاربون في محلول ملح كلوريد الصوديوم

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الخلاصة

تم التحقق من القدرة على منع التآكل في الفولاذ منخفض الكربون في محلول ملح (3,5 % كلوريد الصوديوم) بثلاثة أدوية منتهية الصلاحية. تم اختبار التثبيط باستخدام إنقاص الوزن، واستقطاب الجهد الكهربي، والتحليل الطيفي للمقاومة الكهروكيميائية (EIS) والفحص المجهري الإلكتروني (SEM) لتشكل السطح. في إنقاص الوزن، تم فحص ثلاثة عقاقير منتهية الصلاحية (كلوكساسيلين، أموكسيسيلين، سيفلاكسين) بتركيزات متغيرة (0، 250، 500، 750) ملجم / لتر بهذه الطريقة تبين أن كفاءة التثبيط تزداد مع زيادة التركيز لكل المضادات الحيوية. حيث أظهر 750 ملجم / لتر من الكلوكساسيلين أعلى قيمة لكفاءة التثبيط (82.8125٪)، وهذا جعل الكلوكساسيلين أفضل مثبط لبقية المضادات الحيوية.

تمت دراسة تأثير التراكيز المختلفة لعقار كلوكساسيللين (0، 250، 500، 750) ملجم / لتر ودرجة الحرارة (25، 35، 45، 55) درجة مئوية كمتغيرات مع الاستقطاب الديناميكي الفعال والتحليل الطيفي للمقاومة الكهروكيميائية (EIS) اعتمادًا على القيم الحالية ومقاومة الشحنة لحساب كفاءة التثبيط. كانت الملاحظات الرئيسية لهذه الاختبارات هي أن منحنيات الاستقطاب أظهرت تثبيطًا مختلطًا من نوع الكلوكساسيللين منتهي الصلاحية. زادت كفاءة التثبيط مع زيادة تركيز الكلوكساسيلين ولكن ليس مع زيادة درجة الحرارة.