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Short Communication

Electrochemical Techniques for Evaluation of Expired Megavit Drugs as Corrosion Inhibitor for Steel in Hydrochloric Acid

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Megavit zinc drugs is a physical and mental activity enhancer it is so dangerous to be left in the environment after its expired as it is harmful to the child's, so the present work introduces an idea for using it in the expired medicinal form as potential nontoxic inhibitor for steel corrosion against acidic 1.0 M HCl. The electrochemical techniques were applied in the evaluation of inhibition efficiency of megavit drugs in the expired form. Effect of expired drug concentration and reaction temperature were studied. The corrosion inhibition was found to increase by increasing concentration and decreased with rising the temperature. Potentiodynamic polarization data show that the expired drugs retard both anodic and cathodic potential meaning it is a mixed inhibitor. The electrochemical impedance spectroscopy EIS, techniques data shows that the expired drug help to increases the polarization resistance by adsorbing on metal/electrolyte interface. This kind of adsorption found to obeying Langmuir adsorption isotherm model. The data obtained from the used electrochemical techniques exhibit good agreement between them with (± 2) to prove that the used expired megavit zinc medicinal drugs act as green corrosion inhibitor for the c-steel in 1.0 M HCl acidic environment and in an industrial field**.**

Keywords: Electrochemical techniques; EIS; Potentiodynamic Polarization, Green inhibitors, Expired drugs; Megavit zinc drugs.

1. INTRODUCTION

Electrochemical techniques are highly effective and widely used for evaluation of large numbers of materials against corrosion protection of many metallic alloys in large types of the aqueous corrosive

environment, containing acids, alkaline, and aqueous salts [1-15]. Drugs is one of the most famous chemical materials of every daily and continuous use in our homes, the expired drugs considered as the dangerous materials in the environment cause the death of more than 2000 child's every year, in some countries the expired drugs were wasted in the holes in a deserts which leading to the pollution of the underground water by harmful materials. All this observations take the attentions of Reda Abdel Hameed to search about the new applications for the expired drugs, the using of the expired medicinal drugs as corrosion inhibitors for the metals and alloys was introduced at the first time by Abdelhameed 2009 and 2011, [16,17] when he applied the expire drugs ranitidine as potential nontoxic eco-friendly inhibitor for the corrosion of aluminum in hydrochloric acid solution, his work have been take attention of the another scientists and researchers to evaluate and study many of the expired drugs materials as corrosion inhibitors, Further, the research of unused drugs has been focused on corrosion inhibition of the steel in different corrosive environments [16-21]. In the last decade the scientific efforts in the field of corrosion inhibition were attended on the eco-friendly and potential nontoxic corrosion inhibitors known as green corrosion inhibitors. The most recent efforts are the use of expired drugs to solve not only the problem of soiled waste accumulations but also to introduce a potential nontoxic inhibitor also to save energy, money consumed in the preparing or sailing a chemical corrosion inhibitor as about of 7 % from the total income were consumed in the protection of metals from the corrosion in many industrial fields. Following the green chemistry concepts, the application of expired drugs in the metallic protections of metals helps in the important green condition namely, 1) avoid toxicity by of the toxic inhibitors to human, 2) save both solvent and energy consumed in preparation of others traditional corrosion inhibitors materials. 3)There is no waste while using drugs as inhibitors, in addition, 4) avoid the accumulation of drugs waste which is dangerous on the child's and ground water, so that the expired drugs considered as green corrosion inhibitors, because There is no any waste for the process of using drugs as inhibitors as it was taken from the drug market directly to the laboratory, where it was used in their pharmacological form in very few concentrations which is safe for humans and the environment. In addition, Corrosion is the oxidation process of the metals and alloys by effect of the environments and circumstances, which lead to the formation of the corrosion product in the medium results in the destruction of the metallic crystal, corrosion phenomena cause a terrible waste for both natural resource and money, consequently the control of steel corrosion is of technical, economic and environmental importance, when the corroded steel has been changed from a solid and useful metal to a corrosion product that considered a danger to environment, so controlling the corrosion of steel is a green process. The damage of metals and alloys by the corrosion increase the maintenance cost and need more metals production which is highly expensive process. Finally, expired drugs are a green inhibitor for corrosion as they are a nontoxic inhibitor to both human and environment especially at low concentrations of small concentrations, plus preventing steel from damage by corrosion has big environmental significance, as corrosion is a terrible waste for money and the natural resources together. In the previous works [18,19] expired paracetamol and expired Indomethacin were recycled and applied as inhibitors for the corrosion process of the steel in acidic and sodium chloride corrosive environment using electrochemical and chemical techniques. The aim of the present work is to recycling of expired megavit zinc drugs by reusing it as eco-friendly potential nontoxic green inhibitors for corrosion of the carbon steel alloys used in manufactural of petroleum pipe lines and petroleum industries in 1.0 M hydrochloric acid using

electrochemical techniques. The effect of expired drugs concentration and the reaction temperature were studied as soon as the adsorption isotherm mode to reach the corrosion inhibition mechanism of the studied expired megavit zinc drugs. The survey of the literature shows that the Megavit zinc drug is a physical and mental activity enhancer. Each capsule of the drug is a complex mixture containing, Ginseng extract (a mixture of Ginsenosides and Polysaccharides [22,23]) 50 mg, Essential phospholipids (EPL) 30 mg, Vitamin A 5000 IU, Vitamin D 400 IU, Vitamin E 20 mg, Vitamin B1 (25 mg), Vitamin B2 (5 mg), Nicotinamide 25 mg, Calcium Pantothenate 10 mg, Vitamin B6 (5 mg), Vitamin B12 (12 mg), Vitamin C 50 mg, Folic acid 0.4 mg, Biotin 300 mg, d-Inositol 25 mg, Lysine HCl 25 mg and other components. The drug is suitable herein to be experimented as a corrosion inhibitor for being rich with heteroatoms, and for being a medical waste [24,25]. From the literature we can concluded that the expired megavit zinc drugs is highly promise in the corrosion inhibition of steel alloys in acidic environment.

2. EXPERIMENTAL and Methods

2.1. Used Materials and Solutions

2.1.1. Materials

The used materials are analytical grade 37% HCl, Na_2CO_3, obtained from Sigma Aldrich chemical Co., Carbon steel samples employed in the study is similar to the carbon steel which used in manufacturer of petroleum pipe line with the gravimetric composition as given in table 1. Expired megavit zinc drugs used as corrosion inhibitor in this study was taken from home after expired date by 6 months, the chemical composition of megavit zinc drug is a complex mixture containing of: Ginseng extract (a mixture of Ginsenosides and Polysaccharides [22,23]) 50 mg, Essential phospholipids (EPL) 30 mg, multivitamins (vitamins A, D, E, B, C, and folic acid) as mentioned in introduction part with added minerals and other components.

Table 1. The gravimetric composition carbon Steel materials used in the study.

Elements	Manganese	Silicon	Sulfur	Phosphorus	Carbon	Iron
Gravimetric composition % Weight	0.5171	0.2013	0.0092	0.0073	0.1600	about 99.00

2.1.2. Solution Preparation

The corrosive hydrochloric acid of concentration 1.0 M HCl (pH = 0.0) which used as corrosive medium in the present research work was prepared using analytical grade (37% HCl) by dilution by distilled water then standardized by titration with 1.0 M Na_2CO_3, followed by dilution to required

appropriate pH.. All experiments were completed with aerated stagnant solution with three-time repetition at the same experimental conditions for reproducibility checking and the mean value of the three readings were taken for the data processing. The inhibitor solution is an expired megavit zinc drug, in the form of capsules produced by European Egyptian Pharmaceutical Industries (EEPI). The drug capsules were opened to obtain the powdered ingredients, and the powders were heated under reflux with alcohol for 5 h. Then the heterogeneous colloidal mixture was cooled to reach the room temperature then allowed to settle. The solution was filtered, and the filtrate was allowed to dry to yield soluble yellow extract.

2.2. The open circuit potential Measurements (OC)

Open circuit potential of the C- steel electrode was determined against the saturated calomel electrode (SCE) in 1.0 M HCl solution in the presence and absence of various concentrations of inhibitors at 30 ^{O}C. all the measurements were accomplished using Multi-tester until steady-state potential reached.

2.3. The Potentiodynamic Polarization (PDP)

The radiometer analytical was used to study the electrochemical experiments, type of Volta master (PGZ301, DYNAMIC ELS VOLTAMMETRY). A counter electrode is a platinum wire and the reference electrode to which all potentials are referred is a saturated calomel electrode (SCE). The acting (working) electrode is a steel cylindrical shape (1 cm^2). The surface of the c-steel working electrode was successively polished by silicon carbide abrasive sheets of grades, 800,1100, 1300, 1500, and 2000 before each experiment, followed by washing with bi distilled water, degreasing into ethanol and cleaning with bi distilled water. The used working steel electrode is maintained prior to immersion in free corrosion potential for 50 minutes. The scan rate is 1 mV/s. The % I.E percent of corrosion inhibitory efficiency. was calculated using the following equation **[17]:**

$$\% \boldsymbol{I.E.} \quad = \quad \left[\frac{\boldsymbol{i - io}}{\boldsymbol{i}}\right] \boldsymbol{x}\, \mathbf{100} \qquad (\mathbf{1})$$

Where as $\boldsymbol{i}$ and $\boldsymbol{io}$ are the values of the corrosion current densities in the absence and presence of the used inhibitors respectively. The cathodic and anodic Tafel slopes were obtained from the Tafel plot and the corrosion potential (Ecorr), corrosion current (Icorr) and Tafel constants (βa & βc) were obtained and tabulated.

2.4. The Electrochemical Impedance Spectroscopy (EIS)

Voltalab 40 was used for all electrochemical impedance spectroscopy, EIS, techniques, the used frequencies ranged from 100kHz to 50mHz with 4 Mv sine wave as the excitation signal at the open circuit potential. If the real part is plotted on the X-axis and imaginary part is plotted on Y-axis of the

chart we gain a Nyquist Plot shape. From the difference in the impedance at lower and at higher frequencies the charge transfer resistance values (Rct) was calculated. The polarization resistance Rp, was determined from the diameter of semicircle in the Nyquist plot representation.

3. RESULTS AND DISCUSSION

Following the green chemistry concepts by elimination of environmental pollution by accumulated waste like expired drugs and corrosion products which considered as environmental and ecological hazards materials, the application of expired materials in the corrosion inhibition process of metals and alloys is of economical, environmental, ethical impact, as the damage of metals by corrosion result in the high cost of maintenance and equipment's. In addition, the high cost and toxic effect of the traditional corrosion inhibitors, in this respect, megavit zinc drugs considered as potential nontoxic highly efficient corrosion inhibitor due to its promise chemical constituents. Megavit zinc drugs is a complex mixture contains multivitamins, plant extract, polysaccharides, nicotinamide, and minerals all of this constituents of rich functional groups cause the high corrosion inhibition efficiency of the used megavit zinc drugs. This constituent adsorbed on the steel surface and will affect both anodic and cathodic reaction. The corrosion inhibition efficiency was evaluated using high performances electrochemical techniques, namely open circuit potential, potentiodynamic polarization, and electrochemical impedance spectroscopy, EIS.

3.1. Open Circuit Potential Measurements

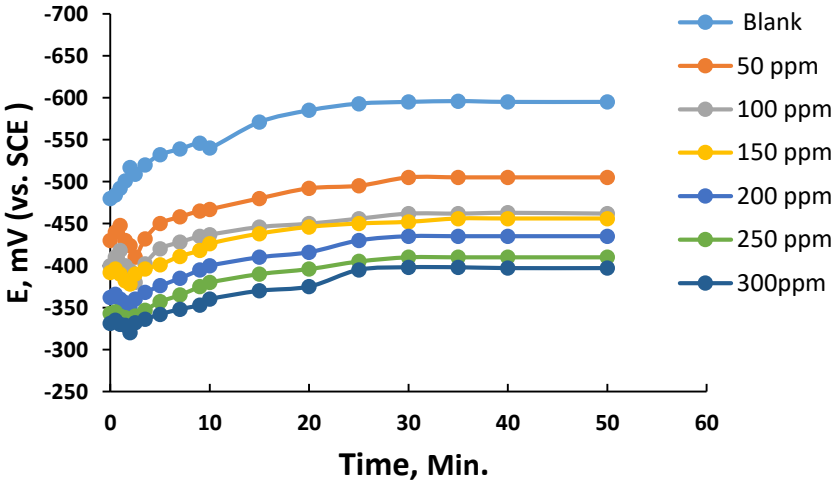

Figure 1. Variation of potential with time for steel immersed in 1.0 M HCl solution with and without expired Megavit zinc drugs as green inhibitor at 30 ^{0}C

Figure 1 represent the potential time curves of the carbon steel electrode immersed in 1.0 M HCl solution at 30 ^{0}C in the absence and presence of different concentrations of the expired megavit zinc

drugs. The open circuit potential was determined against SCE as a reference type electrode for about 50 min. as shown from figure 1 the potential E values of c-steel tends towards more negative potential then shifted slightly to the positive direction then reached to steady state. The same behavior was noted with addition the inhibitors bus the steady state potential shifted to more noble direction. The inhibitor molecules raise up the free corrosion potential of c-steel (shift it to the more noble direction) compared to the potential determined in the case of the blank solution. The apparent steady state values are observing always more negative compared to the immersion potential indicating the dissolution of the metal oxide film before the steady state is reached in all curves [8,29].

3.2. Potentiodynamic polarization (PDP) technique

Table 2. Polarization data of c-steel electrode in 1.0 M HCl solution containing different concentrations of expired megavit zinc drugs inhibitor at 30 ^{0}C.

Inhibitors	Conc., ppm	$-E_{corr}$ mV (SCE)	I_{corr} mA cm^{-2}	β_a mVdec^{-1}	β_c mVdec^{-1}	% I.E.	θ
Blank		478	1.2	97	118	-	-
Expired Megavit	50	517	0.29	106	123	75.8	0.758
	100	523	0.25	115	129	79.2	0.792
	150	535	0.2	119	137	83.3	0.833
	200	538	0.18	128	143	85.0	0.850
	250	542	0.13	131	149	89.1	0.891
	300	547	0.10	137	151	91.7	0.917

Figure 2 represent the potentiodynamic polarization (PDP)curves for the carbon-steel electrode immersed in 1.0 M HCl solution with and without different concentrations of expired megavit zinc drugs. The electrochemical parameters of corrosion like cathodic *(β_c)* and anodic*(β_a)* Tafel slopes were calculated and measured from the linear region of the polarization curves. The values of corrosion current density (I_{corr}) were calculated from the intersection of the anodic and cathodic lines with the corrosion potential ($E_{corr.}$). Percentage of inhibition efficiency (%I.E.) was calculated from corrosion current density values using equation 1(experimental section). Whereas the surface coverage θ values were obtained using the following equation [5-8].

$$\theta = \left[1 - \frac{I_{add}}{I_{free}}\right] \qquad (2)$$

where, I_{free} and I_{add} are the corrosion current densities in the free and inhibited acid solutions, respectively. The values of β_a, β_c , E_{corr} ,I_{corr} and %I.E. were listed in Table 2. It can concluded from the table 2 the followings: the Tafel slopes values β_a and β_c are nearly constant (slightly changed) indicatind that the inhibitor molecules affect both anodic and cathodic reactions(mixed inhibitor), this mean that the metal surface area available for both anodic dissolution of steel and the cathodic hydrogen evolution

process were decreased without any effect on the reaction mechanism. The slow change in the potential values $E_{corr.}$ to negative values prove that the inhibitor is mixed type mainly cathodic. The I_{corr}.values decreases consequently the I.E's increses proving the inhibition effect of expired Megavit zinc drugs toward the corrosion of steel in hydrochloric acid corrosive environment.

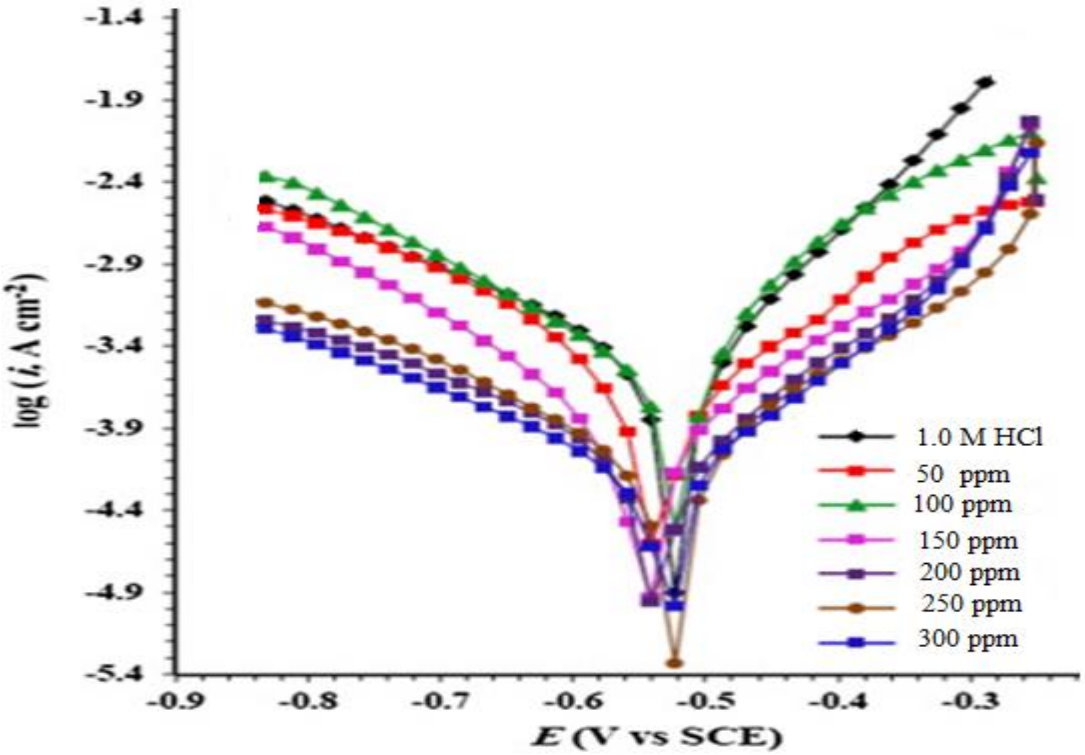

Figure 2. Potentidynamic polarization (PDP) curves for carbon steel in 1.0 M HCl containing different concentration of expired megavit zinc drug inhibitor at 30 ^{0}C.

3.3. Electrochemical Impedance Spectroscopy (EIS) Measurements

Electrochemical impedance spectra Nyquist plots obtained for corrosion inhibition of steel in 1.0 M HCl containing different concentration from the expired megavit drugs as green inhibitor at 30 ^{0}C is shown in Figure3. The Nyquist plots contain depressed semicircle with center under real axis. The semicircle size increases with the inhibitor concentration, suggesting the charge transfer process is the main controlling factor of the steel corrosion. It is apparent from the plots that; the impedance of the inhibited solution has increased with the increase in expired drug inhibitor concentrations. The EIS experimental results for corrosion of c-steel in 1.0 M HCl in presence and absence of expired megavit zinc inhibitor is given in Table3.

Table 3. AC Impedance data for carbon-steel electrode in 1.0 M HCl solution at 30ºC with and without various concentrations of the expired megavit zinc.

Expired Drug Concentrations	Rct Ohm cm^2	Rs Ohm cm^2	Cdl µF cm^{-2}	% I.E.	θ
(free)	93.84	2.4	39.0	-	-
50 ppm	540.4	1.7	8.6	82.6	0.826
100 ppm	650.3	1.6	4.2	85.5	0.855
150 ppm	798.5	2.3	3.1	88.2	0.882
200 ppm	890.3	1.9	2.3	89.5	0.895
250 ppm	960.5	1.8	1.4	90.2	0.902
300 ppm	998.6	1.6	1.2	90.6	0.906

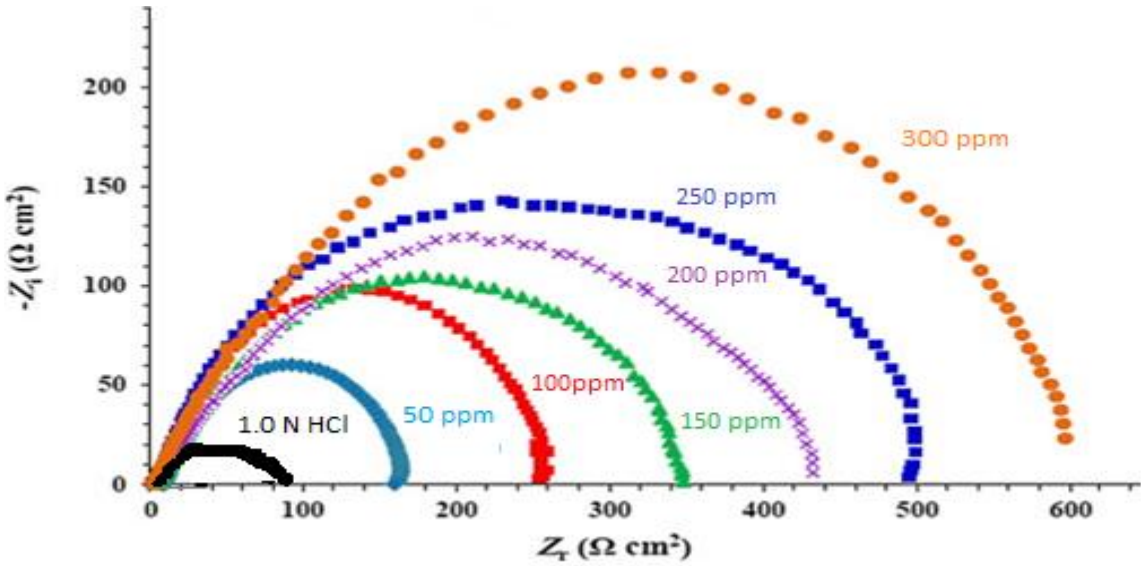

Figure 3. The Nyquist plots for carbon-steel electrode in 1.0 M HCl solution with and without different concentrations of expired megavit zinc as green inhibitor at 30ºC

As showed from the data the values of polarization resistance (R_p) increased with increasing the inhibitor concentration. Whereas of the capacitance of the interface (C_{dl}) values starts in decreasing, by increasing the inhibitor concentrations, this may be due to the decrease in the local dielectric constant and/or increase in thickness of the electrical double layer. This suggests that the drugs inhibitor molecules act via adsorption at the metal/solution interface **[5-8]** and the decrease in the C_{dl} values is caused by the gradual replacement of water molecules by the adsorption of the inhibitor molecules on the electrode surface, which decreases the extent of metal dissolution process.

The corrosion inhibition efficiency is given by the following equation **[25-29]**:

$$\%\, \boldsymbol{I.E.} = \frac{\boldsymbol{Rp(inhi) - Rp}}{\boldsymbol{Rp(inhi)}} \; \boldsymbol{x\,100} \qquad (\mathbf{3})$$

Where R_p is polarization resistance without inhibitor, and R_p(inhi) is polarization resistance with inhibitor. C_{dl} value is obtained from the following equation:

$$\boldsymbol{\omega\,(max) = 2\,\pi\,f\,(max)\; = \frac{1}{Rp\;Cdl}} \qquad \textbf{(4)}$$

Where, $f_{(max)}$ is the frequency at the top of the semicircle (where-Z" is maximum). The proposed equivalent circuit is represented in **figure 4** the electrode impedance, Z, in this case given by the mathematical equation **[25-29]:**

$$\boldsymbol{Z\; = Rs\;\; [\frac{Rt}{1+(2\,\pi\,f\,Rt\;Cdl)^{\alpha}}]} \qquad \textbf{(5)}$$

Where α represents the empirical parameter ($0 \leq\ \alpha \leq 1$) and f is equal to the frequency (in Hz). The equation number 5. Takes into account the normal deviation from the ideal RC behavior in view of the term of a time constant distribution process due to the surface heterogeneity, roughness effect, adsorption of the inhibitor, and the variation of properties or the composition of the surface layers. The suggested equivalent circuit **figure 4** consists of the double-layer capacitance (C_{dl}) in parallel to the charge transfer resistance (R_t) which is in series to the parallel inductive (Rs).

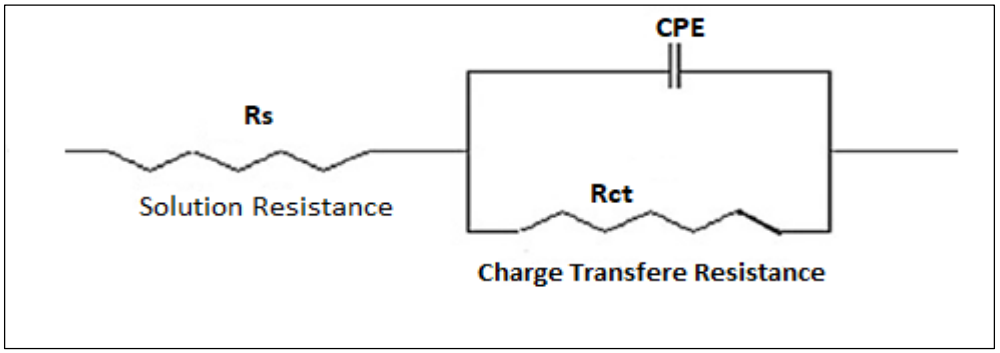

Figure 4. The suggested equivalent circuit model for electrochemical impedance(EIS) measurements.

3.4. Adsorption Isotherm

The process of adsorption affected mainly by the charge, metal surface nature, electronic characteristic of the metal surface, the temperature of the reaction, the presence of the electro repelling or electro donating groups in the derivatives, the solution-interfaces electrochemical potential, adsorption of the solvent and other ionic species.

Tafel polarization values are used to calculate the surface coverage (θ) values in the presence of different inhibitor concentrations and to reach the best fit isotherm, many adsorption isotherms were applied but the obtained results are completely agreed to the Langmuire adsorption isotherm as given from equation number (6) **[32]:**

$$\boldsymbol{\frac{Ci}{\theta}\;\; = \;\; \frac{1}{Kads.}\;\; + Ci} \qquad \textbf{(6)}$$

where, K_{ads} and Ci are the constant of the adsorption process and the concentration of the drugs, respectively [32]. Graphical representation of Ci/θ against Ci is a straight line shape as represented in

the figure 5. The slope of the obtained strait line nearly equal unity with intercept equal 1/K. The standard free energy for the adsorption process $\Delta G°_{ads}$ is given from the following mathematical equation:

$$Kads = \frac{1}{55.5} \exp\left(\frac{-\Delta G}{RT}\right) \quad (7)$$

Where K_{ads}. Is the equilibrium constant of the adsorption process, the value 55.5 equal the water concentration at the bulk of the solution(mole/liter), the absolute temperature is T, and R is the gas constant. The calculated value of ΔG_{ads} is(-34.25 kJ/mole). The value of the free energy of adsorption ($\Delta G°_{ads}$) is negative to indicate on the spontaneous adsorption process of the drugs molecules on the metal surface**[32].** On the other hand, the obtained value is less than the reported value (-40 Kj/mol) necessary for chemical adsorption this observation indicates that the adsorption mechanism is a physical adsorption type mechanism **[30-32]**. It can be concluded that the expired drug materials inhibito corrosion by the adsorption of its constituent molecules on the metal /electrolyte interface. Some researchers have been believed that the formation of passive film from the solid organic drug molecule on the atoms of the metal at the surface this suggestion is the most acceptable in the present case because the megavit zinc drugs is a high complex mixture from multivitamins, polysaccharides, plant extract, and minerals as mentioned in experimental part, all the constituent help in the formation of stronger adhesive film on the steel surface preventing steel from the corrosion by attached corrosive environment**[30-32]**

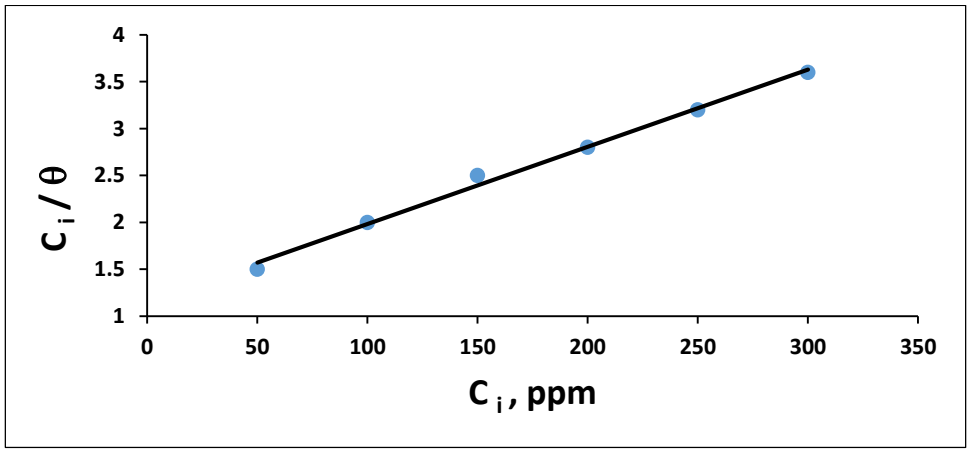

Figure 5. Langmuir isotherm for adsorption of C-steel immersed in 1.0M HCl solution at 30 °C.

4. CONCLUSIONS

Expired megavit zinc drugs considered as green corrosion inhibitor as it is a nontoxic inhibitor to the human being at the used low concentration, also the prevention of steel and metals from corrosion is of big impact from the environmental chemistry point of view.

Electrochemical measurements are of higher performance and accurate in evaluation of steel corrosion and in evaluation of the inhibition performance of the inhibitors.

The corrosion inhibition efficiency increase by increasing expired megavit zinc drugs inhibitor concentrations reach to 91.7 % at 300 ppm.

The high corrosion inhibition efficiency of the expired megavit zinc drugs due to its complex component mixture of multivitamins, plant extract, polysaccharides, nicotinamide, and minerals

Potentodynamic polarization measurements results indicated that the values of $E_{corr.}$ change slowly to more negative values to indicate that the expired megavit zinc inhibitors are of mixed type inhibitors.

Electrochemical impedance spectroscopy data indicating that the semicircle size increases by increasing inhibitor concentrations suggesting that the process of charge transfer is the controlling factoring steel dissolution process.

Corrosion inhibition process due to adsorption and adhesion of expired megavit zinc drugs on steel surface forming a passive layer on the steel surface which separate the steel from aggressive media the adsorption fit well with the Langmuir isotherm of adsorption.

The data obtained from the used electrochemical techniques exhibit good agreement between them with (± 2) to prove that the used expired megavit zinc medicinal drugs act as green corrosion inhibitor for the c-steel in 1.0 M HCl acidic environment. So expired megavit zinc drugs act as green corrosion inhibitor.

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