Expired concor drug as potential nontoxic corrosion inhibitor for 304 stainless steel in hydrochloric acid solution

ABSTRACT

The effect of the expired concor drug as the corrosion inhibitor for stainless steel type 304 (304SS) in 2M HCl solution has been investigated using electrochemical and non-electrochemical techniques. Some kinetic corrosion parameters were calculated. The inhibition efficiency (I%) increases with rising concentration of the drug and maximum efficiency (85.8 %) reached 300 ppm. The polarization measurements indicated that the expired concor drug was of the mixed type and inhibits corrosion by adsorption on the surface of 304SS due to the presence of more than one active center in its molecule. The adsorption was obeyed Langmuir adsorption isotherm. The activation energy and thermodynamic parameters were also calculated and discussed.

Keywords: 304 SS; expired concor drug, HCl, Langmuir isotherm, corrosion inhibition.

1. INTRODUCTION

Stainless steel type 304 has found wide application in a variety of industries. It is covered with a highly protective film of chromium oxyhydroxides; this makes it resistant to corrosion in many aggressive environments. The hydrochloric acid solution, however, readily attack 304SS. It is possible to reduce the corrosion rate to the safe level by adding inhibitors. Most of the well-known inhibitors investigated for corrosion of steel in acidic solution are heterocyclic compounds [1-7]. It is clear that the adsorption on the metal substrate is the initial step in inhibition. Generally, a stronger coordination bond causes higher inhibition efficiency (I%) [8]. Due to the toxicity of widely used corrosion inhibitors and the ever-tightening environmental regulations surrounding their use and disposal, there is great interest in replacing harmful inhibitors with effective nonhazardous alternatives. Over the past two decades, extensive research and development have led to the discovery of new classes of corrosion inhibitors, and the importance on the use of several drugs as corrosion inhibitors has grown [9].

Recently, the use of antibiotics and other drugs have been investigated and their inhibition efficiencies have been linked to their heterocyclic nature [10]. Most of the heterocyclic drugs are environmentally friendly and can favorably compete with the natural products. In recent years, the use of pharmaceutical compounds offer interesting possibilities for corrosion inhibition due to the presence of heteroatoms like nitrogen, sulfur, oxygen and π-bond in their structure and are of particular interest because of their safe use, high solubility in water and high molecular size. Some of the azosulpha and antimalarial drugs have been reported as good corrosion inhibitors [11,12]. There are great efforts to use drugs as corrosion inhibitors for steel [13-15], but the use of the expired drugs as corrosion inhibitors for steel is limited. Some antibacterial drugs have been used as corrosion inhibitors [16-18]. Cyclic voltammogram was drawn before by some authors [19] on platinum in order to assess the stability of pharmaceutically active substances from drugs at the metal-corrosive environment interface.

The present review reports the new trends aimed at the recycling of expired drugs and using it as corrosion inhibitors for metals in different media. The expired concor drug was used as the corrosion inhibitor for 304SS in 2M HCl using different electrochemical and non-electrochemical methods.

Figure 1 shows the structure of the expired concor drug. This drug obtained from Sandozinc and Pfizer Inc companies, Egypt.
Abd El-Aziz S. Fouda et al. Expired concor drug as potential nontoxic corrosion inhibitor ...

2. EXPERIMENTAL METHOD

The composition of the SS type 304 is as follows (weight %): C 0.08, Cr 18.0, Ni 8.0, S 0.03, Mn 2.0, Si 0.075, Fe balance. The 304SS strips of 2x2x0.1 cm size were used for WL measurements. In electrochemical studies, a cylindrical rod embedded in Araldite with an exposed bottom surface area of 1cm² was used. The electrode was abraded with different grades of emery papers, degreased with acetone and rinsed with bi-distilled water. WL measurements were carried out as described elsewhere [19]. The specimens are immersed in 100 ml test solution for the period of 3 hrs electrochemical measurements were carried out using the Gamry (PCI 300/4) Instrument Potentiostat / Galvanostat/ZRA. This includes a Gamry framework system based on the ESA400. Gamry applications include DC105 for corrosion measurements and EIS300 for electrochemical impedance spectroscopy along with a computer for collecting data. Echem Analyst (5.58) software was used for plotting, graphing, and fitting data. The electrode potential was measured against a saturated calomel electrode (SCE) and a platinum foil was used as an auxiliary electrode.

3. RESULTS AND DISCUSSION

3.1. Weight loss (WL) tests

304SS coupons were exposed for 3 hrs in naturally aerated 2M HCl with and without the addition of the drug [Fig.2]. From the data of WL, the % I at each drug concentration was calculated using the equation:

$$\% I = \frac{\theta_x 100}{CR_{inh}/CR_{free}} \times 100 \quad (1)$$

where CR_{inh} and CR_{free} are the corrosion rates in the absence and presence of the drug, respectively. It can be seen that the maximum of 75.1 % inhibition efficiency is achieved at 300 ppm of drug concentration and % I increases with increasing the drug concentrations [20] [Table 1]. This is mainly due to the active chemical constituent of viz, π bonds, heteroatoms (O and N). The almost greater than 75% of surface coverage (θ) is due to the coordination between the metal and the heteroatom present in the drug.

Table 1. (CR) and (% I) at different concentrations of the expired concor drug for the corrosion of 304 SS after 120 min immersion in 2M HCl at 30°C

<table>
<thead>
<tr>
<th>Conc. ppm</th>
<th>Conc.</th>
<th>CR x10^3 mg cm^{-2} min^{-1}</th>
<th>% I</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>4.2</td>
<td>45.88</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>3.5</td>
<td>54.90</td>
<td></td>
</tr>
<tr>
<td>150</td>
<td>3.1</td>
<td>60.18</td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>2.7</td>
<td>64.56</td>
<td></td>
</tr>
<tr>
<td>250</td>
<td>2.5</td>
<td>67.78</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>1.05</td>
<td>75.10</td>
<td></td>
</tr>
</tbody>
</table>

Figure 2. WL-time curves for the corrosion of 304 SS in 2M HCl without and with different concentrations of the expired concor drug at 30 °C

Slika 2. WL-vremenske krive za koroziju 304 SS u 2M HCl bez i sa različitim koncentracijama lekova koji je istekao vremenski rok na 30°C
3.2. Effect of temperature on % I

The % I for 304SS corrosion in the presence of various concentrations of the investigated expired drug and at different temperatures was calculated and were listed in Table 2. The results of Table 2 illustrate the variation of CR and % I with drug concentration at different temperatures. The obtained data revealed that the % I decreased with an increase in the drug concentration. This suggests that the drug species are adsorbed on the 304 SS/solution interface where the adsorbed species mechanically form a protecting film on the metal surface which retards the action of the corrosion. The value of % I was decreased with a suggested that physical adsorption mechanism [21]. These results indicate that the adsorption of investigating drug shields the metal surface at room temperature [22]. However, it may be shielded from the surface with a rise in temperature. It is also clear that the corrosion rate of 304SS in the absence and presence of drug obeyed Arrhenius type equation as with raising solution temperature. The dependence of corrosion rate ($i_{corr}$) on the temperature can be expressed by Arrhenius equation 2:

$$i_{corr} = A \exp\left(-\frac{E_a^*}{RT}\right)$$

(2)

where $A$ is the pre-exponential factor and $E_a^*$ is the apparent activation energy of the corrosion process. Arrhenius corrosion of 304SS in 2 M HCl solution temperature. The dependence of corrosion rate $i_{corr}$ on the temperature can be expressed by Arrhenius equation 2:

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(2)

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3.3. Adsorption Isotherms and Thermodynamic Parameters

Adsorption isotherm values are important to explain the mechanism of corrosion inhibition of organo-electrochemical reactions. The most frequently used isotherms are Langmuir isotherm (Fig. 5). Thermodynamic parameters for the adsorption of concor on a 304SS surface in 2M HCl at different temperatures were listed in Table 4.
The Langmuir adsorption isotherm may be formulated \((\theta/1-\theta) = 1/K_{ads} + C\). The plot \((1-\theta)/\theta\) vs \(C\) found to be linear for the additive (Fig. 5). The equilibrium constants \((K_{ads})\) for an adsorption-desorption process for the drug can be calculated from the reciprocal of the antilogarithm of the intercept. The \(K_{ads}\) values were listed in Table 4. The thermodynamic parameters free energy \((\Delta G^0_{ads})\) and entropy \((\Delta S^0_{ads})\) of the investigated drug can be calculated from the thermodynamic relations:

\[
\Delta G^0_{ads} = -RT \ln (55.5 K_{ads}) \tag{4}
\]
\[
\Delta G^0_{ads} = \Delta H^0_{ads} - T \Delta S^0_{ads} \tag{5}
\]

In the above expression, 55.5 is the concentration of water in solution in M\(^{-1}\)[25]. To calculate \(\Delta H^0_{ads}\), a plot of log \(K_{ads}\) vs. \(1/T\) was done and the slope of the line gave the \(\Delta H^0_{ads}\) (Fig. 6). The calculated values of \((\Delta G^0_{ads}),\ (\Delta H^0_{ads})\) and \((\Delta S^0_{ads})\) are recorded in Table 4. The negative values of \(\Delta G^0_{ads}\) indicate the spontaneous adsorption of the additives on the surface of 304SS and its values also, indicates that the inhibition process becomes less favorable as the temperature is increased from 30°C to 45°C. This conclusion agrees with the values of % I. These results prove that the best %I occurs at 30°C. This agrees with what expected since the adsorption is an exothermic process and is always accompanied by a decrease of entropy change. \(\Delta S^0_{ads}\) decreases with increasing temperature. The negative sign of \(\Delta H^0_{ads}\) indicates the exothermic process of adsorption. The low value of \(\Delta S^0_{ads}\) supports the higher absorbability of concor on the surface of SS.

Table 4. Thermodynamic adsorption parameters for the adsorption of the drug on SS304 in 2M HCl at different temperatures

<table>
<thead>
<tr>
<th>Inhibitor</th>
<th>Temp. °C</th>
<th>(K_{ads}\times 10^{-4}) M(^{-1})</th>
<th>(-\Delta G^0_{ads}) kJ mol(^{-1})</th>
<th>(-\Delta H^0_{ads}) kJ mol(^{-1})</th>
<th>(-\Delta S^0_{ads}) J mol(^{-1}) K(^{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>concor</td>
<td>30</td>
<td>68.2</td>
<td>20.8</td>
<td>68.50</td>
<td></td>
</tr>
<tr>
<td></td>
<td>35</td>
<td>61.7</td>
<td>20.8</td>
<td>67.67</td>
<td></td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>50.8</td>
<td>20.7</td>
<td>66.05</td>
<td></td>
</tr>
<tr>
<td></td>
<td>45</td>
<td>46.8</td>
<td>20.8</td>
<td>65.37</td>
<td></td>
</tr>
</tbody>
</table>

Figure 5. Langmuir isotherm of concor on 304SS surface in 2M HCl at different temperatures

Slika 5. Termodinamički parametri adsorpcije za adsorpciju istrošnog leka na SS304 u 2M HCl na različitim temperaturama
3.4. Polarization (PP) tests

Addition of different concentrations of this drug on the anodic and cathodic polarization of 304SS was studied in 2M HCl. The corrosion current \( (i_{\text{corr}}) \) density was determined from the intersection of linear parts of cathodic curves successively by the stationary corrosion potential \( (E_{\text{corr}}) \). The % I at each concentration was calculated using the equation:

\[
\% I = \theta = 100 \times \left( 1 - \frac{i_{\text{corr}}}{i_{\text{corr}}^0} \right)
\]

Where \( i_{\text{corr}} \) and \( i_{\text{corr}}^0 \) are the current densities in the presence and absence of inhibitor, respectively.

<table>
<thead>
<tr>
<th>[Inh.] ppm</th>
<th>(-E_{\text{corr}}) mV vs SCE</th>
<th>(i_{\text{corr}}) mA cm(^{-2})</th>
<th>(b_c) mV dec(^{-1})</th>
<th>(b_a) mV dec(^{-1})</th>
<th>(k_{\text{corr}}) mmy(^{-1})</th>
<th>% I</th>
<th>(\theta)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank</td>
<td>372</td>
<td>598</td>
<td>135</td>
<td>64</td>
<td>27.33</td>
<td>100</td>
<td>----</td>
</tr>
<tr>
<td>50</td>
<td>391</td>
<td>310</td>
<td>126</td>
<td>58</td>
<td>17.39</td>
<td>36.3</td>
<td>0.363</td>
</tr>
<tr>
<td>100</td>
<td>367</td>
<td>338</td>
<td>131</td>
<td>55</td>
<td>15.45</td>
<td>43.5</td>
<td>0.435</td>
</tr>
<tr>
<td>150</td>
<td>393</td>
<td>315</td>
<td>128</td>
<td>63</td>
<td>14.38</td>
<td>47.3</td>
<td>0.473</td>
</tr>
<tr>
<td>200</td>
<td>393</td>
<td>283</td>
<td>94</td>
<td>43</td>
<td>12.91</td>
<td>52.7</td>
<td>0.527</td>
</tr>
<tr>
<td>250</td>
<td>391</td>
<td>356</td>
<td>119</td>
<td>49</td>
<td>11.68</td>
<td>57.2</td>
<td>0.572</td>
</tr>
<tr>
<td>300</td>
<td>395</td>
<td>315</td>
<td>96</td>
<td>56</td>
<td>9.83</td>
<td>64.1</td>
<td>0.641</td>
</tr>
</tbody>
</table>

The parallel cathodic Tafel lines (Fig.7) suggested that the addition of the drug to 2M HCl solution do not modify the hydrogen evolution mechanism and the reduction of H\(^+\) ions at 304SS surface, which occurs mainly through a charge transfer mechanism [26]. The change in the values of \( b_c \) in the presence of the inhibitor clearly indicates the effect of the drug on the kinetics of hydrogen evolution. The shift in anodic Tafel slope \( b_a \) values may be due to the adsorption of inhibitor molecules onto the 304SS surface [27]. The studied drug acts as a corrosion inhibitor suppressing both anodic and cathodic reactions by getting adsorbed on the 304SS surface blocking the active sites and these results suggested that the addition of the drug reduces the anodic...
dissolution and also retards the cathodic hydrogen evolution reaction, indicating that concor drug exhibits cathodic and anodic inhibition effect [28]. The polarization curves exhibit no steep slope in the anodic range (Fig. 7), meaning that no passive films are formed on 304SS surface. Table 5 shows that there is no definite trend in the shift of $E_{corr}$ values, in the presence of various concentrations of concor in 2M HCl solution. This result indicated that the drug can be classified as a mixed type of inhibitor in 2M HCl solution. The I% calculated from WL and PP measurements was in good agreement.

Figure 7. PP curves for the dissolution of 304SS in 2M HCl without and with different concentrations of expired concor drug at 30°C

Slika 7. PP krive za rastvaranje 304SS u 2M HCl bez i sa različitim koncentracijama istrošenog leka na 30°C

3.5. EIS measurements

Impedance spectra (Nyquist plots) of SS in 2M HCl containing various concentrations of the expired concor at 30°C are shown in Fig. 8. Nyquist plots contain depressed semicircles with the center under the real axis, indicating the charge transfer process as the main controlling factor of the corrosion of 304SS. It is apparent from the plots that the impedance of the inhibited solution has increased with the increase in the concentration of the drug. The experimental results of EIS measurements for the corrosion of 304SS in 2M HCl with and without drug are given in Table 6. The equivalent circuit was used to fit the EIS data (Fig. 9).

Figure 8. The Nyquist plots for the corrosion of 304 SS in 2M HCl in the absence and presence of different concentrations of the concor at 30°C

Slika 8. Nyquist -ove krive za koroziju 304 SS u 2M HCl u odsustvu i prisustvu različitih koncentracija istrošenog leka na 30°C
Figure 9. Electrochemical equivalent circuits used to fit the impedance measurements that include a solution resistance \( R_s \), a constant phase element \( \text{CPE} \) and a polarization resistance or charge transfer \( R_{ct} \)

Slika 9. Elektrohemijska ekvivalentna kola koja se koriste za merenje impedancije koje uključuju otpornost na rastvor \( R_s \), konstantni fazni element \( \text{CPE} \) i otpornost na polarizaciju ili transfer punjenja \( R_{ct} \)

Table 6. EIS parameters for the corrosion of SS in 2M HCl without and with different concentrations of investigating drug at 30°C

Tabela 6. EIS parametri za koroziju SS u 2M HCl bez i sa različitim koncentracijama istraživanog leka na 30°C

<table>
<thead>
<tr>
<th>Conc. ppm</th>
<th>( C_{dl} \times 10^{-5} ) μF cm(^{-2} )</th>
<th>( R_{ct} ) Ω cm(^2 )</th>
<th>% I</th>
<th>θ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank</td>
<td>22.99</td>
<td>62.38</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>50</td>
<td>13.79</td>
<td>218.1</td>
<td>71.4</td>
<td>0.714</td>
</tr>
<tr>
<td>100</td>
<td>11.86</td>
<td>275.7</td>
<td>77.4</td>
<td>0.774</td>
</tr>
<tr>
<td>150</td>
<td>12.52</td>
<td>282.3</td>
<td>77.9</td>
<td>0.779</td>
</tr>
<tr>
<td>200</td>
<td>13.00</td>
<td>286.4</td>
<td>78.2</td>
<td>0.782</td>
</tr>
<tr>
<td>250</td>
<td>11.38</td>
<td>413.0</td>
<td>84.9</td>
<td>0.849</td>
</tr>
<tr>
<td>300</td>
<td>11.82</td>
<td>438.3</td>
<td>85.8</td>
<td>0.858</td>
</tr>
</tbody>
</table>

It is seen that the addition of the concor increases the values of charge transfer resistance \( R_{ct} \) and reduces the double layer capacitance \( C_{dl} \). The values of \( C_{dl} \) decreased with an increase in drug concentration. This is due to an increase in the surface coverage by the drug molecules, resulting in an increase in % and also due to the reduction in local dielectric constant and/or an increment in the thickness of the electrical double layer [29]. The phenomenon proposed that the inhibitor molecule function by adsorption at the metal/solution interface. The increase in \( R_{ct} \) values is due to the formation of the protective film on the metal/solution interface [30].

3.6. EFM tests

EFM is a nondestructive corrosion measurement like EIS; it is a small signal ac technique. Unlike EIS, however, two sine waves (at different frequencies) are applied to the cell simultaneously. The great strength of the EFM is the causality factors which serve as an internal check validity of the EFM measurement [31]. With the causality factors, the experimental EFM be verified. The results of EFM experiments are a spectrum of current response as a function of frequency. The spectrum is called the intermodulation spectrum. The spectra contain current responses assigned for harmonical and intermodulation current peaks. The larger peaks were used to calculate the corrosion current. The % \( I \) calculated from equation 1 increases with increasing the studied drug concentrations. Intermodulation spectra obtained from EFM measurements were constructed for 304SS in 2M HCl (Fig. 10) solution as a function of 300 ppm (Fig.11) of concor at 30°C. Each spectrum is a current response function of frequency; shown here. Corrosion kinetic parameters, current density \( i_{corr} \), Tafel constants \( b_m, b_c \) and causality factors \( (CF-2, CF-3) \) were listed Table 7 as a function of concentrations of investigated drug at 30°C. The causality factors in Table 7, values according to the EFM theory, should guarantee the validity of and corrosion current densities. The standard values for CF-2 and CF-3 are 2.0 and 3.0, respectively [32].

Figure 10. EFM spectra for SS304 in 2M HCl

Slika 10. EFM spektri za SS304 u 2M HCl
Figure 11. EFM spectra for SS304 in 2M HCl in the presence of 300 ppm concor drug

Table 7. Electrochemical kinetic parameters obtained from the EFM technique for SS in 2M HCl without and with different concentrations of expired drug

3.7. Mechanism of Corrosion Inhibition

It is generally accepted that the first step in the adsorption of an organic inhibitor on a metal surface usually involves the replacement of one or more water molecules adsorbed at the metal surface. The adsorption is influenced by the nature and charge of metal, the chemical structure of the drug and the type of the aggressive electrolyte. In acidic solutions, this compound could be protonated easily, because the molecule is made of the aromatic ring, which contains π electrons and with unshared electron pairs of N and O-atoms. So, it is easily protonated in HCl solution as:

\[ \text{Inh} + n\text{H}^+ = [\text{inh} - H_n]^n+ \]

Both molecular and protonated species can adsorb on the metal surface. Accordingly, Cl ions are first adsorbed on the metal surface in HCl medium and consequently, the metal surface becomes negatively charged and the protonated concor molecule is physically adsorbed on the metal surface [33]. Adsorption of the protonated concor molecules at the cathodic sites on the SS surface will retard the hydrogen evolution reaction. Adsorption on the anodic sites of SS surface can occur through O and N atoms in the molecule to retard SS dissolution reaction. Expired Carbamazepine and Paracetamol tablets were used to obtain corrosion inhibitors. For the former, the corrosion % I of carbon steel in 0.1 M sulfuric acid solution was about 90%, whereas for the latter, the corrosion % I of the same material in the 0.25 M acetic acid / 0.25 M sodium acetate buffer solution was about 85%. Dapsone gave corrosion %I 93% at 400 ppm and 90% in H2SO4 at 400 ppm in HCl. All tests confirmed that concor is an efficient inhibitor for SS in 2 M HCl solution. Generally, the use of expired drugs as corrosion inhibitors for steel is scanty, and show real promise.

4. CONCLUSIONS

The expired concor drug has proved to be an efficient inhibitor for corrosion of 304SS in 2M HCl solution. This inhibitor acts as mixed type inhibitor
and the I% was found to increase by increasing the drug concentration and decreasing of temperature. The I% obtained from the electrochemical measurements showed good agreement with those obtained from WL experiments. The inhibition of 304SS in 2M HCl solution at different temperatures was found to obey the Langmuir adsorption isotherm. The thermodynamic values obtained from this study $\Delta H^*$ and $\Delta S^*$ indicate that the presence of the expired drug increases the activation energy and the negative values of $\Delta G^o_{ads}$ indicate the spontaneous adsorption of the drug on the surface of SS.

5. REFERENCES

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IZVOD

VREMENSKI ISTEKAO LEK KAO POTENCIJALNI NETOKSIČNI INHIBITOR KOROZIJE ZA 304 NERĐAJUĆI ČELIK U RASTVORU HLOROVODONIČNE KISELINE

Efekat vremenski isteklog leka kao inhibitora korozije za nerđajući čelik tipa 304 (304SS) u 2M HCl rastvoru je ispitivan korišćenjem elektrohemijskih i ne-elektrohemijskih tehnika. Izračunati su neki parametri kinetičke korozije. Efikasnost inhibicije (% I) povećava se sa rastućom koncentracijom leka i maksimalna efikasnost (85,8%) je dostigla 300 ppm. Polarizacijska merenja pokazala su da je vremenski istekli lek bio inhibitor mešovitog tipa i inhibirao je koroziju adsorpcijom na površini 304SS zbog prisustva više od jednog aktivnog centra u njegovom molekulu. Adsorpcija je ponašala po izotermi adsorpcije Langmiura. Takođe, proračunati su i razmatrani energija aktivacije i termodynamicni parametri.

Ključne reči: 304 SS, vremenski istrošeni lek, HCl, Langmirova izoterm, inhibicija korozije.