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## Analytical and electroanalytical techniques in the determination of (Fe<sup>+2</sup>) results from iron corrosion: effect of omeprazole drugs as green inhibitors

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### ABSTRACT

Seven analytical and electroanalytical techniques, namely gravimetric, thermometric, gasometrical, acidimetric, atomic absorption spectroscopy (AAS), potentiodynamic polarization, and electrochemical impedance spectroscopy, EIS, were applied in the determination of ferrous (Fe<sup>+2</sup>) ions that result from corrosion of steel in aqueous lower pH medium, in the presence and absence of omeprazole drugs as corrosion inhibitors. The effects of pH, concentration and temperature were studied; the ferrous ion (Fe<sup>+2</sup>) concentration and consequently the corrosion rate decreased by increasing drug concentration, while the concentration of ferrous ions (Fe<sup>+2</sup>) and corrosion rate increased by lowering pH and with rising temperature. Potentiodynamic polarization techniques show that the used drugs act as mixed type inhibitors for steel in all studied pH values. The maximum inhibition efficiency, obtained using 300 ppm of omeprazole drugs, is 96% within the range when it is compared with the obtained efficiency in the cited references. All the used techniques are in good agreement with each other ( $\pm 2\%$ ) and show that the used drugs act as green corrosion inhibitors for the steel at lower pH values.

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### KEYWORDS

Gravimetric; thermometric; gasometrical; atomic absorption spectroscopy; green inhibitors; drugs; potentiodynamic

## 1. Introduction

The analytical and electroanalytical techniques are widely used in the determination of ferrous ion and the rate of corrosion of iron in different corrosive medium and at different pH (1–9). Ferrous ions in aqueous medium result from the iron corrosion, when the iron metal is subjected to corrosive environment, it is directly changed into ferrous ion in the solution and consequently the metal is damaged; the rate of corrosion and metal damaged is determined gravimetrically by the ferrous ion concentration passing into the solution, as the rate of corrosion increases, the concentrations of ferrous ions in the corrosive medium increase and vice versa. Quantification of iron ion contents in corrosive solutions was done by Barbouchi et al (10). The concentrations of the ions passed into the solution were measured by using Varian Spectra AA 220 atomic absorption spectroscopy (AAS) and in order to determine the concentrations of iron ions within corrosive solution when the green inhibitors were absent and present. Potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) were also used (10). Iron is usually found in the industry in the form of steel, steel is composed of about 99% iron and traces of alloying elements, such as manganese,

carbon, sulfur, phosphorus, and silicon; steel is a boon of heavy industries, corrosion is the process of oxidation of metals by its environment, leading to corrosion products and the destruction of the metal lattice; this phenomenon represents a terrible waste of both natural resources and money. So, the corrosion control of steel is of technical, economical, and environmental importance, when the steel was corroded it changed from hard metal into corrosive product which is hazard to the environment so the corrosion control of steel is considered a green process, also. The damage by corrosion incurs high cost for the maintenance and protection of materials used. Corrosion inhibitors are commonly used to reduce the corrosion attack on the steel surface (11,12). The presence of the inhibitors leads to decline in the rate of corrosion of steel and hence the lifetime increases (12). Most of the effective acid inhibitors are organic compounds, containing nitrogen, oxygen, and/or sulfur, which are adsorbed at the steel surface and hence slow down the rate of corrosion (11–15). The strength of the adsorption depends on several factors, such as the chemical structure of the inhibitors, the presence of electro donating or electro repelling group, molecular weight of the inhibitor, temperature and electrochemical potential at the metal/solution interface (15–19). Surfactant inhibitor has many

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advantages, such as high inhibition efficiency, low price, low toxicity and easy production (19–23). A large number of organic compounds were studied as corrosion inhibitor; unfortunately, most of the organic inhibitors used are very expensive and cause health hazards. Their toxic properties limit their field of application. Thus, it remains an important object to find cost-effective and non-hazardous inhibitors for the protection of metals against corrosion. In this connection, the influences of nontoxic organic compounds and drugs on the corrosion of metals in acid media were investigated by several authors (24–28). Obot, I.B. et al (29) investigated the effect of benzimidazoles as corrosion inhibitors for industrial metals in different aqueous systems, and suggested the mechanisms of corrosion protection of benzimidazoles. Umoren, S.A. et al (30) reviewed the effect of halide ions on the corrosion inhibition efficiency of various organic species, including polymers, organic dyes, surfactants, and drugs. They concluded that the inhibition depends on various factors, including nature and concentration of the additives, temperature and nature of the metal and aggressive environments. N. Palaniappan et al, (31) investigated the corrosion inhibition performance of expiry drugs as green inhibitors for steel in NaCl medium. He concluded that the expiry drug molecules are multifunctional groups and lone pair electron species, it could easily interact with metal surface and reduce major corrosion problem of the industry. R.S. Abdel Hameed et al (32) studied the effect of sulfanyl pyridazine derivatives as efficient corrosion inhibitors for carbon steel in the 1.0 M HCl using analytical techniques. He concluded that the analytical techniques were effective for the evaluation of the corrosion inhibition performance; different analytical and electroanalytical techniques were used to evaluate the corrosion inhibition efficiency of inhibitors (32–34), AAS was made available to the routine laboratory as an analytical technique that initially was intended to produce considerable simplification of procedures for the analysis of aqueous, acidic, or basic solutions, and thereby contribute to a reduction in costs. The industrial application of atomic absorption for routine analysis is divided into four areas: (1) the incoming inspection of all raw materials, (2) production testing, (3) final inspection of all products, and (4) environmental analysis. For rapid analysis during the production process, atomic absorption is mainly of an indirect value because, as the sequential character of the technique, it cannot be used for complete steel or slag analysis in a two- to three-minute period (35). AAS was used for corrosion rate prediction in different media, acidic, basic, and neutral based on fundamental chemistry of the solubility

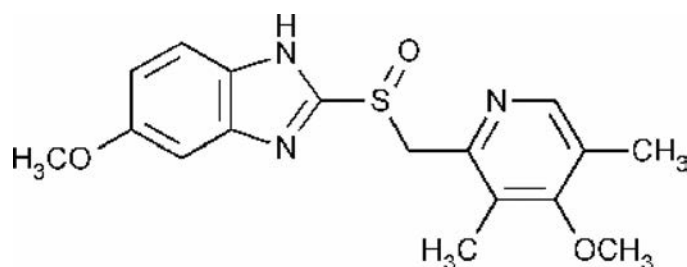
of the corrosion products. The AAS method was applied for the determination of Fe ions in mineral waters and natural waters (32,36). From the green chemistry point of view, the use of the drugs materials as inhibitors helps in (1) avoiding the toxic effect of some toxic inhibitors to human beings, (2) using drugs to save the energy and organic solvent used in the synthesis of the corrosion inhibitors in addition (3) there is no waste material during the use of the drugs as inhibitors, so the drugs are considered green inhibitors, because there is no waste for the process of using drugs as inhibitors as they are taken directly from the drug market to the laboratory, where they are used in their pharmacological form which is safe for human beings and the environment in very few doses. The aim of the present research is to study the concentration of ferrous ions and rate of iron corrosion in the absence and presence of Omeprazole drugs as green corrosion inhibitors using analytical and electroanalytical techniques, namely, gravimetric, thermometric, gasometrical, acidimetric, AAS, potentiodynamic polarization, and EIS techniques. The effect of green inhibitor concentrations and reaction temperatures were also studied.

The survey of literature reveals that omeprazole drug belongs to a group of medicines called proton-pump inhibitor (PPI); it is a medication used in the treatment of gastroesophageal reflux disease, peptic ulcer disease, and Zollinger–Ellison syndrome. It is also used to prevent upper gastrointestinal bleeding in people who are at high risk. It can be taken orally or injected into a vein (37). Omeprazole contains oxygen, nitrogen and sulfur heteroatoms in addition to active substituted aromatic groups and a tri-coordinated sulfinyl sulfur in a pyramidal structure, as shown in Figure 1. So the use of Omeprazole ampules as green corrosion inhibitors gives promise.

## 2. Experimental

### 2.1. Materials and test solution

The aggressive solution prepared in this study is 1.0 N hydrochloric acid of 0.0 pH, which was prepared from analytical grade 37% HCl (Sigma Aldrich) after diluted with double-distilled water and titrated against 1.0 N  $\text{Na}_2\text{CO}_3$ , then diluted to an appropriate required pH. Each experiment was performed in aerated stagnant solutions and was repeated at least three times under the same conditions to check the reproducibility and the average of the three replicated values was used for further processing of the data. The gravimetric composition of iron materials employed in this study is given in Table 1.



**Figure 1.** Chemical structure of the used inhibitor, Omeprazole.

Omeprazole drugs, with the molecular formula:  $C_{17}H_{19}N_3O_3S$  Molecular weight: 345.42 from Tabuk pharmaceutical Mfg. Company, Tabuk, Saudi Arabia, were used as green corrosion inhibitors for iron in the present study and its chemical structure as given in Figure 1 in the introduction section.

## 2.2. Gravimetric studies

The gravimetric method is widely used because it has the advantage of being simple and does not require complex equipment or procedures. All the experiments were done three times and the recorded data were taken to the medium value with ( $\pm 0.1$ ) error. In this study, two types of gravimetric tests were carried out, namely the weight loss measurement and the dosing of the metal passed in the solution by atomic absorption spectroscopy.

### 2.2.1. Weight loss measurement

The technique is for determining the weight loss (WL) of a sample (coupon) of the surface (S) immersed for a time (t) in the aggressive solution; the tests are performed in 100 ml glass vials in a non-aerated medium, at room temperature. The immersion time is 8 h. The iron samples are being cleaned with distilled water, degreased with acetone and dried before and after the immersion. The corrosion rate W was determined from the following relation (1):

$$W = \frac{m_i - m_f}{St} \quad (1)$$

where W ( $\text{mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ ) is the corrosion rate,  $m_i$  (mg) and  $m_f$  (mg) are the mass before and after exposure to test solution, respectively, S ( $\text{cm}^2$ ) is the surface of area of specimen, t (h) is the immersion time. The inhibitory efficiency I.E.% and the surface coverage ( $\theta$ ), which

represent the part of the metal surface covered by the inhibitors molecules, were calculated according to the following equations:

$$\%IE = ([W^0 - W]/W^0) \times 100 \quad (2)$$

$$\theta = ([W^0 - W]/W^0) \quad (3)$$

where  $W^0$  and W represent the corrosion rates in the absence and presence of the inhibitors, respectively.

### 2.2.2. Atomic absorption spectroscopy

Quantification of iron ion contents in corrosive solutions in the absence and presence of the used drugs as corrosion inhibitor was determined by AAS. Concentration of the ferric ions passed into solution has been determined by using Varian Spectra AA 220 atomic absorption spectroscopy. In order to determine the concentrations of iron ions within the corrosive solution when the inhibitor was absent and present, we dissolved the corrosive medium by aqua regia (10).

## 2.3. Gasometrical measurements (hydrogen evolution measurements)

The progress of the corrosion reaction was determined by volumetric measurement of the evolved hydrogen. The metal sample was put in a Büchner flask containing the test solution. The flask is sealed with a rubber bung, and from its hose barb protruding from its neck, rubber tubing is connected to the bottom of an inverted measuring cylinder which is fitted above a basin. The cylinder and the basin are filled with distilled water. The evolved hydrogen gradually displaces the distilled water and is collected at the top inside the cylinder, and its volume is measured directly with time. The experiment is done in the absence and presence of different concentrations of the tested inhibitor.

## 2.4. Thermometric measurements

The carbon steel sheet was press cut  $2 \times 2 \times 0.1$  cm with a chemical composition, as mentioned in Table 1. The measurements were carried out in a Dolvacpyrex flask covered with a sheet of aluminum foil. The reaction

**Table 1.** Gravimetric composition of the used iron materials.

| Element                | Mn    | Si    | S     | P     | C     | Fe        |
|------------------------|-------|-------|-------|-------|-------|-----------|
| Composition weight (%) | 0.517 | 0.201 | 0.009 | 0.007 | 0.157 | About 99% |

vessel consists of 50 ml of the acid test solution put into the flask covered with sheets of aluminum foil, corked with a check temp digital thermometer in place. The metal coupon was introduced into the corrosive solution and quickly covered. Thermometric measurements depend on measuring the temperature variation during the reaction of a metal with test piece with a definite volume of a corroding solution (38). The variation of temperature of the system was monitored with time and the reaction number (RN) is defined as (38)

$$RN(^{\circ}C/min.) = (T_m - T_i)/t_m \quad (4)$$

where  $T_m$  and  $T_i$  are the maximum and initial temperature, respectively,  $t_m$  is the time in minutes taken to attain the maximum temperature.

### 2.5. Acidification method

Each experiment was preceded by evaluation for the pH of the prepared solution by a standard BT-500 model pH meter (Germany). The pH was checked for the five concentrations (50, 100, 150, 200, 250, and 300 ppm) pre- and post-dipping in the checked solution for 5 hrs. The %IE computed by implemented in the following equation.

$$\%IE = 1([\Delta H^+]_{inh}/[\Delta H^+]_{uninh}) \times 100 \quad (5)$$

where  $\Delta H^+$  inh. and  $\Delta H^+$  uninhi. are changes in  $H^+$  concentration with and without the inhibitor, respectively (39).

### 2.6. Electroanalytical techniques

#### 2.6.1. The potentiodynamic polarization (PDP)

All the electrochemical experiments are performed using radiometer analytical, Volta master (PGZ301, DYNAMIC ELS VOLTAMMETRY). A platinum wire was used as a counter electrode and a saturated calomel electrode (SCE) as the reference electrode to which all potentials are referred. The iron-working electrode is of cylindrical shape (1 cm<sup>2</sup>). Before each test, the working electrode surface was successively abraded with silicon carbide abrasive papers of grade 600, 1000, 1200, 1500, and 2000, followed by washing with distilled water, degreasing in ethanol and cleaning with distilled water. The working electrode is maintained prior to immersion in free corrosion potential for 60 min. The scanning speed is 1 mV/s. The corrosion inhibitory efficiency % IE was obtained by using Equation (6):

$$\%IE = ([i - i^{\circ}]/i) \times 100 \quad (6)$$

where  $i$  and  $i^{\circ}$  are the corrosion current densities values

in the absence and presence of the inhibitors, respectively.

#### 2.6.2. Electrochemical impedance spectroscopy (EIS)

Electrochemical impedance was obtained using a Voltalab 40 for all EIS measurements with a frequency range of 100 kHz to 50 mHz with a 4 mV sine wave as the excitation signal at open circuit potential. If the real part is plotted on the X-axis and the imaginary part is plotted on the Y-axis of a chart, we get a Nyquist Plot. The charge transfer resistance value ( $R_{ct}$ ) was calculated from the difference in impedance at lower and higher frequencies.

### 3. Results and discussion

Analytical and electroanalytical techniques were used in estimation of the corrosion rate and ferrous ion determination in the presence and absence of omeprazole drugs as green corrosion inhibitors in acidic environment of the 1.0 N HCl solution. In the present research, the effects of inhibitor concentration and temperatures were studied, different techniques, such as gravimetric, thermometric, gasometrical, acidimetric, and AAS as analytical techniques soon after electroanalytical techniques which are potentiodynamic polarization and EIS. The used omeprazole drugs as green inhibitors are symbolized here as OMP. The studied concentrations are 50, 100, 150, 200, 250, and 300 ppm. As mentioned in the introduction part, the drugs as inhibitors considered as green corrosion inhibitors for many reasons mentioned and from the green chemistry point of view. In addition, the corrosion control of steel is of environmental importance, when the steel was corroded it changed from a hard to a useful metal into corrosive product which is hazardous to the environment, so the corrosion control of steel is considered the green process. The damage by corrosion results in high cost for maintenance and protection of materials used. Finally, the omeprazole drugs considered as green inhibitors as they are nontoxic inhibitors to human beings specially at very low concentrations, in addition, the prevention of the steel from corrosion is of environmental importance, as corrosion is a terrible waste of both natural resources and money.

#### 3.1. Gravimetric measurements

##### 3.1.1. Effect of concentrations

Gravimetric techniques were used in the determination of the corrosion rate and corrosion inhibition efficiency %IE of OMP drugs for the various concentrations of

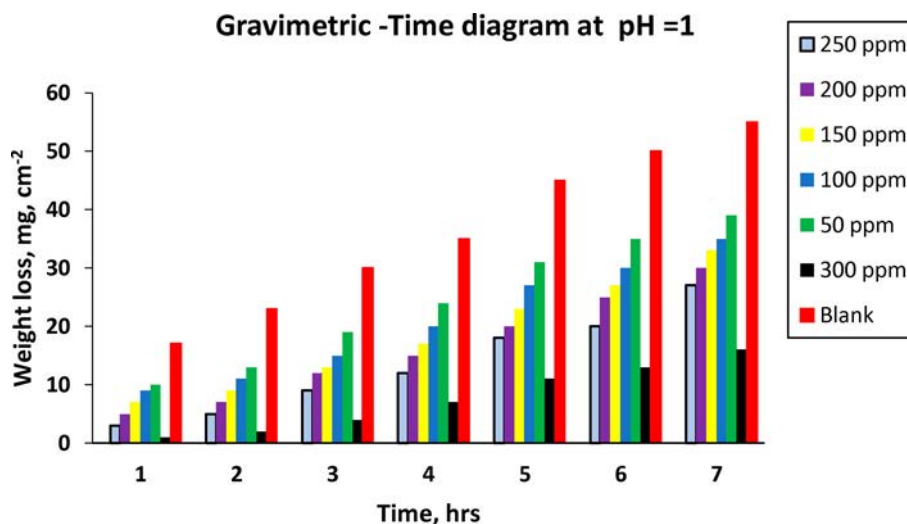
**Table 2.** Effect of increasing concentration of the OMP drugs on the corrosion parameters of iron in the HCl solution of different pH values obtained from the weight loss measurements at 303 K.

| Compound                                | pH | Concentrations ppm | W mg cm <sup>-2</sup> hr <sup>-1</sup> | I.E. % | $\theta$ |
|---|----|--------------------|--|--------|----------|
| Blank HCl of pH = 1 without inhibitor   | 1  |                    | 9.00                                   | –      | –        |
| OMP                                     | 1  | 50                 | 1.10                                   | 88     | 0.88     |
| HCl of pH = 1 with omeprazole inhibitor |    | 100                | 0.88                                   | 90     | 0.90     |
|   |    | 150                | 0.89                                   | 90.2   | 0.902    |
|   |    | 200                | 0.75                                   | 91.7   | 0.917    |
|   |    | 250                | 0.6                                    | 93.3   | 0.93     |
|   |    | 300                | 0.5                                    | 94.4   | 0.94     |
| Blank                                   | 3  |                    | 7.8                                    | –      | –        |
| HCl of pH = 3 without inhibitor         | 3  |                    |  |        |          |
| OMP                                     | 3  | 50                 | 0.84                                   | 89.7   | 0.897    |
| HCl of pH = 3 with omeprazole inhibitor |    | 100                | 0.71                                   | 91     | 0.91     |
|   |    | 150                | 0.56                                   | 93.5   | 0.935    |
|   |    | 200                | 0.47                                   | 94.5   | 0.945    |
|   |    | 250                | 0.39                                   | 95     | 0.95     |
|   |    | 300                | 0.27                                   | 96.5   | 0.965    |

OMP inhibitors after 7 h of immersion of iron coupon in hydrochloric acid corrosive environment of different pH values (pH 1 and 3). The values of corrosion rate (W) and corrosion inhibition efficiency %IE are given in Table 2 and Figure 2. According to gravimetric results, the weight loss and consequently the corrosion rate decrease and inhibition efficiency increases by adding the omeprazole drugs (OMP) green inhibitors and this behavior was increased by increasing the OMP inhibitor concentrations. It's clear that the inhibition efficiency increases as the inhibitor concentration increases to attain 94% at 300 ppm. The inhibitory efficiency % IE and the surface coverage ( $\theta$ ), which represents the part of the metal surface covered by the inhibitor molecules, were calculated according to Equations (2) and (3) mentioned in experimental part. Inspection of Table 2 reveals that the values of inhibition efficiency increase with

increasing concentration of drug and decrease by lowering pH values of the medium.

Figure 2 reveals that by increasing the concentrations of OMP drugs, the weight loss of carbon steel samples is reduced. This clarified that the occurrence of these compounds lowered the dissolution of iron in 1.0 N HCl solution indicating that these drugs act as inhibitors. The relation between weight loss with time in uninhibited and inhibited 1.0 N HCl is linear. This illustrates the absence of insoluble surface films during corrosion. In this case, the inhibitors are first adsorbed onto the metal surface and thereafter impede corrosion either by merely blocking the reaction sites (anodic and cathodic) or by altering the mechanism of the anodic and cathodic partial processes (40). The maximum inhibition efficiency, obtained using 300 ppm of omeprazole drugs, is 96% when it is compared by the



**Figure 2.** Weight loss of iron as a function time in the 1.0 N HCl in the absence and presence of OMP drugs as green inhibitor for steel corrosion.

obtained efficiency in the cited references (previous works) (24–27,31).

### 3.1.2. Effect of temperature

To elucidate the mechanism of inhibition and to determine the kinetic parameters of the corrosion process, Gravimetric (weight loss) measurements were done at different temperatures e.g. 303, 313, 323, 333 K. The effect of temperature on the percentage inhibition efficiency of steel in the presence of the inhibitor is graphically represented in Figure 3. It is obvious that the values of %IE decreased with the increase in temperature, leading to the conclusion that the protective film of these compounds formed on the steel surface is less stable at higher temperature, which may be due to the desorption of some adsorbed molecules from the surface of the steel at higher temperature due to which a greater area of the metal is exposed to the acidic environment. All the experiments were done three times and the recorded data were taken to the medium value with ( $\pm 0.1$ ) error.

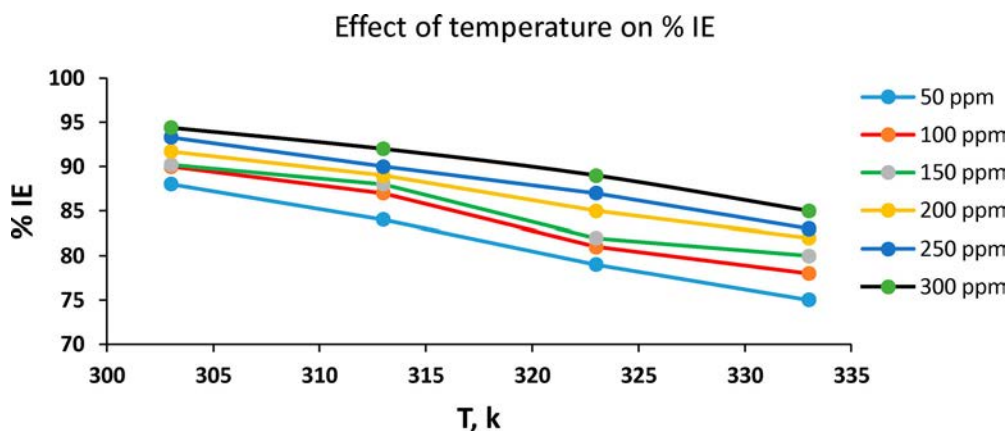
### 3.2. Atomic absorption spectroscopy

Iron corrosion is a complex process that occurs when iron is exposed to oxygen and humidity and is exacerbated by the presence of chloride ions. The deterioration of iron structures or other components can be costly to society and can be evaluated by following the properties of the corroding material (41). The iron ions released into the solution due to corrosion were detected directly by AAS and their concentration was determined using a calibration curve. The corrosion of the iron samples in solution was accelerated by high salinity, lowering pH, the presence of chloride ions and temperatures (41).

In the present study, the ferrous ion  $\text{Fe}^{+2}$  concentrations due to the corrosion of iron by acidic HCl of different pH were determined by AAS. The concentrations of the ions passed into the solution were measured by using AAS and in order to determine the concentrations of ferrous ions within corrosive solution when the green inhibitor was absent and present. The data of AAS are listed in Table 3 which shows that the ferrous ion  $\text{Fe}^{+2}$  concentrations in the corrosive medium were decreased by increasing inhibitor concentration and increased by lowering pH of the solution. Here ferrous ion concentration is taken as a function of corrosion rate consequently as the ferrous ions in the solution increase, the corrosion rate increases and vice versa. All processes are influenced by adding an inhibitor. The data in Table 3 agree well with the results obtained by the gravimetric (weight loss) method, so the tow gravimetric techniques are in good agreement with each other to indicate that the addition of omeprazole (OMP) drugs inhibits the corrosion of iron in acidic environment and decreases the iron dissolution process (ferrous ion concentration  $\text{Fe}^{+2}$ ) in this environment at lower pH values.

### 3.3. Gasometrical technique (Hydrogen evolution method)

Gasometrical technique (hydrogen evolution method) was used for the evaluation of omeprazole, OMP as green corrosion inhibitors for iron in the acidified hydrochloric acid solution of different lower pH values (pH 1 and 3). Iron metal is an active metal of higher oxidation potential, so it replaces the hydrogen of acid easily giving metal salt and the hydrogen evolved in the form of gas. Gasometrical techniques were used for measuring the evolved hydrogen gas with time in the absence and presence of inhibitors in acidic



**Figure 3.** Effect of temperature on % I.E for carbon steel in the 1.0 N HCl in the presence of different concentrations of the OMP drugs as green inhibitors.

**Table 3.** Effect of pH and inhibitor concentrations on ferrous ion concentrations as a result of the AAS technique.

| Sample     | Inhibitor concentration | Ferrous ion concentrations [Fe <sup>2+</sup> ], ppm |        |
|------------|-------------------------|---|--------|
|            |                         | pH = 1  | pH = 3 |
| Blank      | Free                    | 124   | 113    |
| Omeprazole | 50 ppm                  | 49  | 31     |
|            | 100 ppm                 | 38  | 22     |
|            | 150 ppm                 | 24  | 16     |
|            | 200 ppm                 | 17  | 12     |
|            | 250 ppm                 | 11  | 9      |
|            | 300 ppm                 | 9   | 6      |

environment. The volume of hydrogen evolved, during the corrosion reaction of iron acidic HCl medium in the absence and presence of different concentrations of the OMP green inhibitor, 50, 100, 150, 200, 250, and 300 ppm., was measured with time at room temperature (30°C). The inhibition efficiency was calculated using the following equation (29).

$$\% \text{IE} = [1 - (V_{inh.}/V_{free})] \times 100 \quad (7)$$

where  $V_{inh}$  is the volume of hydrogen gas evolved for the inhibited solution and  $V_{free}$  for the uninhibited solution. The values of evolved hydrogen volumes and inhibition efficiencies at different concentrations of the used green inhibitor, OMP, are shown in Table 4. The inhibition efficiency increases with increasing the inhibitor concentration, which indicates that the drug inhibitor compounds acts as a good inhibitor for carbon steel in HCl acidic environment.

### 3.4. Thermometric measurements

Reaction number (RN) values are known as a relative measure of retardation of the dissolution process (38).

The extent of corrosion inhibition can have expressed in terms of the percentage reduction in reaction number (% RR) given by the following equation (30).

$$\% \text{RR} = \frac{\text{RN uninhibited} - \text{RN inhibited}}{\text{RN uninhibited}} \times 100 \quad (8)$$

The thermometric parameters and inhibition efficiency are summarized in Table 5. The inhibition efficiency and  $t_m$  increase by increasing inhibitor concentrations. The inhibition efficiency and time delay ( $\Delta t_m$ ) of the used inhibitor decrease with increasing the pH value in the following order: pH 3 > pH 1. This order is similar to that obtained from both weight loss and gasometrical techniques.

### 3.5. Acidimetric measurements

The pH was checked for the five concentrations (50, 100, 150, 200, 250, and 300 ppm) pre- and post-dipping in the checked solution for 7 h. where  $[H^+]$  was computed in each case.

**Table 5.** Thermometric parameters for the iron corrosion in the acidic HCl solution of different pH values in the presence of 300 ppm of the omeprazole, OMP, green inhibitor.

| Thermometric parameters | OMP HCl of pH = 1 with omeprazole inhibitor | OMP HCl of pH = 3 with omeprazole inhibitor |
|-------------------------|---|---|
| $T_m$ , °C              | 37  | 36  |
| $t_m$ , min.            | 320   | 380   |
| RN, °C/min.             | 0.037                                       | 0.029                                       |
| % RR                    | 88  | 90  |
| $\Delta t_m$            | 275   | 335   |

**Table 4.** Hydrogen volumes and the percentage inhibition efficiency for the steel corrosion in the acidic HCl solution of different pH values with and without different concentrations of the OMP drug at room temperature after 7 h.

| Inhibitor Compound                          | pH | Concentrations | Volume of hydrogen (ml/cm <sup>2</sup> ) | IE % |
|---|----|----------------|--|------|
| Blank HCl of pH = 1 without inhibitor       | 1  | Free           | 56                                       | –    |
| OMP HCl of pH = 1 with omeprazole inhibitor | 1  | 50 ppm         | 15                                       | 73.2 |
|   |    | 100 ppm        | 12                                       | 78.5 |
|   |    | 150 ppm        | 10                                       | 82.1 |
|   |    | 200 ppm        | 7  | 87.5 |
|   |    | 250 ppm        | 5  | 91.0 |
|   |    | 300 ppm        | 4  | 92.8 |
| Blank HCl of pH = 3 without inhibitor       | 3  | Free           | 49                                       | –    |
| OMP HCl of pH = 3 with omeprazole inhibitor | 3  | 50 ppm         | 12                                       | 75.5 |
|   |    | 100 ppm        | 9  | 81.6 |
|   |    | 150 ppm        | 8  | 83.7 |
|   |    | 200 ppm        | 5  | 89.7 |
|   |    | 250 ppm        | 3  | 93.8 |
|   |    | 300 ppm        | 2  | 95.9 |



**Table 6.** Values of %IE for the acidimetric method for OMP inhibitor in the 1.0 N HCl at 303 K.

| Conc. (ppm) | $\Delta H^+ \times 10^4$ | $\theta$ | %IE  |
|-------------|--------------------------|----------|------|
| Blank       | 96                       | –        | –    |
| 50          | 19                       | 0.80     | 80   |
| 100         | 15                       | 0.84     | 84   |
| 150         | 13                       | 0.86     | 86   |
| 200         | 10                       | 0.895    | 89.5 |
| 250         | 7                        | 0.93     | 93   |
| 300         | 6                        | 0.938    | 93.8 |

The corrosion rate (W) of iron samples was computed utilizing the following equation:

$$W(\text{mole dm}^{-3}\text{cm}^{-2}\text{h}^{-1}) = \frac{\Delta H^+}{A t} \quad (9)$$

where  $[\Delta H^+]$  is the variation between the first and last concentration of  $H^+$ , A is the surface area of coupon in  $\text{cm}^2$  and t, the time in hrs. The %IE was computed utilizing the following equation:

$$\%IE = 1 - \left( \frac{[\Delta H^+]_{\text{inh}}}{[\Delta H^+]_{\text{uninh}}} \right) \times 100 \quad (10)$$

where  $\Delta H^+_{\text{inh}}$  and  $\Delta H^+$  are changes in  $H^+$  concentration with and without the presence of green inhibitor, OMP, respectively.

The values of surface coverage ( $\theta$ ) were computed utilizing the following equation: (39)

$$\theta = 1 - \left( \frac{[\Delta H^+]_{\text{inh}}}{[\Delta H^+]_{\text{uninh}}} \right) \quad (11)$$

As shown from Table 6 the values of corrosion inhibition efficiency and surface coverage were increased with increasing the concentration of green inhibitor OMP, due to the decrease of the hydrogen ion concentrations in the medium as inhibitor molecule acts as a ligand for protons in acidic medium.

### 3.6. Potentiodynamic polarization technique

Figure 4 illustrates the potentiodynamic polarization curves for carbon steel electrode in 1.0 N HCl (of pH = 1) solution devoid of and containing different concentrations of omeprazole drugs. Some electrochemical corrosion parameters, such as the values of cathodic ( $\beta_c$ ) and anodic ( $\beta_a$ ) Tafel slopes, were determined from the linear region of the polarization curves. The values of corrosion current density ( $I_{\text{corr}}$ ) were computed from the intersection of the anodic and cathodic lines with the corrosion potential ( $E_{\text{corr}}$ ).

The percentage inhibition efficiency percentage (%IE) was calculated from the corrosion current density values using the following equations (34, 42, 43).

$$\%IE = \left[ 1 - \frac{I_{\text{add}}}{I_{\text{free}}} \right] 100 \quad (12)$$

where  $I_{\text{free}}$  and  $I_{\text{add}}$  are the corrosion current densities in

the free and inhibited acid solutions, respectively. The values of  $\beta_a$ ,  $\beta_c$ ,  $E_{\text{corr}}$ ,  $I_{\text{corr}}$  and %IE are shown in Table 7.

Inspection of Table 7 concludes that the values  $\beta_a$  and  $\beta_c$  Tafel slopes that are approximately changed slightly proved that these compounds acted as mixed inhibitors. The surface area, available for anodic dissolution of iron and cathodic hydrogen evolution reaction, decreases without affecting the reaction mechanism. The values of  $E_{\text{corr}}$  change slowly to negative, indicating that these inhibitors are of mixed type inhibitors mainly cathodic. The values of  $I_{\text{corr}}$  decrease and hence the values of IE's increase, indicating that the inhibiting effect of omeprazole drugs toward the corrosion of iron in hydrochloric acid corrosive environment.

### 3.7. Electrochemical impedance spectroscopy

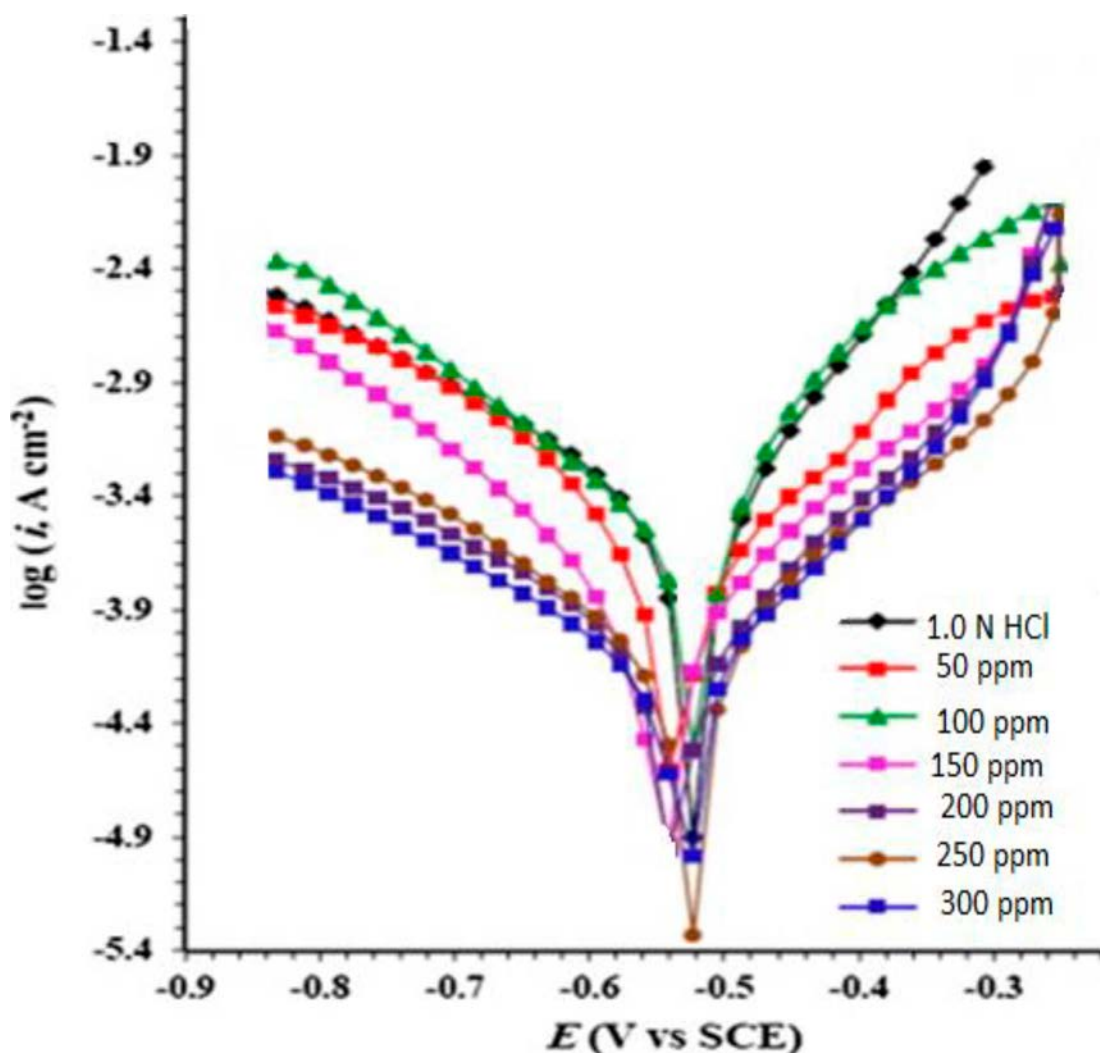
The impedance spectra (Nyquist plots) of iron in the 1.0 N HCl, containing various concentrations of the omeprazole drugs as green inhibitors at 30°C, are shown in Figure 5. Nyquist plots contain depressed semicircles with a center under real axis. The size of the semicircle increases with the inhibitor concentration, indicating the charge transfer process as the main controlling factor of the corrosion of iron. It is apparent from the plots that the impedance of the inhibited solution has increased with the increase in the concentration of the inhibitor. The experimental result of EIS measurements for the corrosion of iron in the 1.0 N HCl, in the absence and presence of OMP inhibitor, is given in Table 8.

It can be observed that polarization resistance ( $R_p$ ) value increased with an increase in the concentration of the inhibitor. Whereas values of the capacitance of the interface ( $C_{dl}$ ) start decreasing, with an increase in inhibitor concentration, which is most probably due to the decrease in local dielectric constant and/or increase in thickness of the electrical double layer. This suggests that the inhibitor acts via adsorption at the metal/solution interface (28) 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.

The inhibition efficiency is given by the following equation (28):

$$\%IE(R_p) = \frac{R_p(\text{inhi}) - R_p}{R_p} \times 100 \quad (13)$$

where  $R_p$  is the polarization resistance without inhibitor, and  $R_p(\text{inhi})$  is the polarization resistance with inhibitor.



**Figure 4.** Potentiodynamic polarization curves of iron in the 1.0 N HCl solution containing different concentrations of omeprazole drugs (OMP) at room temperature.

**Table 7.** Polarisation data for iron electrode in the 1.0 N HCl solution with various concentrations of omeprazole drugs (OMP) at room temperature.

| Inhibitors | Conc., ppm | $-E_{corr}$<br>mV (SCE) | $i_{corr}$<br>$\text{mA cm}^{-2}$ | $\beta_c$<br>$\text{mVdec}^{-1}$ | $\beta_a$<br>$\text{mVdec}^{-1}$ | % IE |
|------------|------------|-------------------------|-----------------------------------|----------------------------------|----------------------------------|------|
| Blank      | –          | 478                     | 1.2                               | 101                              | 97                               | –    |
| OMP        | 50         | 510                     | 0.21                              | 118                              | 103                              | 82.5 |
|            | 100        | 519                     | 0.19                              | 122                              | 112                              | 84.2 |
|            | 150        | 525                     | 0.12                              | 128                              | 118                              | 90.0 |
|            | 200        | 537                     | 0.10                              | 135                              | 126                              | 91.6 |
|            | 250        | 539                     | 0.098                             | 140                              | 132                              | 91.8 |
|            | 300        | 542                     | 0.096                             | 146                              | 139                              | 92.0 |

$C_{dl}$  value is obtained from the following equation:

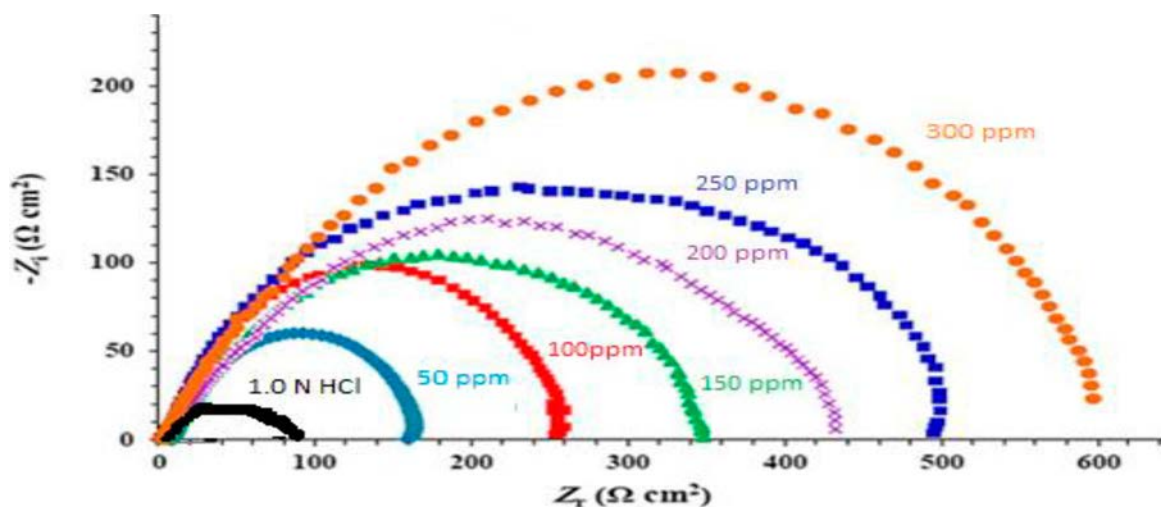
$$\omega_{max} = 2\pi f_{max} = 1/R_p C_{dl} \quad (14)$$

where  $f_{max}$  is the frequency at the top of the semicircle (where  $Z$  is maximum). The proposed equivalent circuit represents electrode impedance in Figure 6,  $Z$ , in this

case given by the mathematical equation (3, 5):

$$Z = R_s \left[ \frac{R_t}{1 + (2\pi f R_t C_{dl})^\alpha} \right] \quad (15)$$

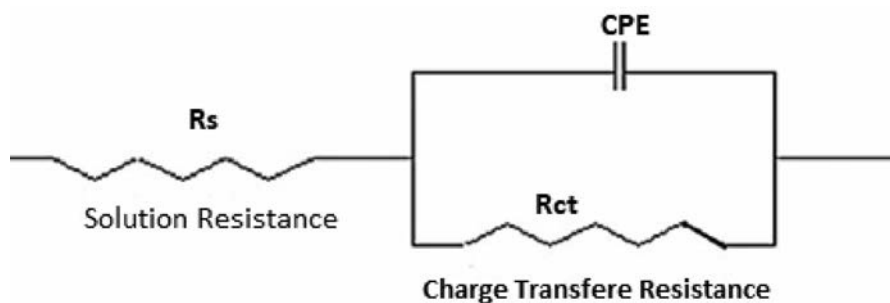
where  $\alpha$  denotes an empirical parameter ( $0 \leq \alpha \leq 1$ ) and  $f$  is the frequency in Hz. This equation takes into account the deviation from the ideal RC-behavior in terms of a distribution of time constant due to surface



**Figure 5.** Nyquist plots for iron in the 1.0 N HCl solution at 30°C containing various concentrations OMP drugs as green inhibitors.

**Table 8.** AC impedance data of iron electrode in the 1.0 N HCl solution at 30°C in the absence and presence of different concentrations of OMP drugs.

| Sample | R <sub>ct</sub><br>Ohm cm <sup>2</sup> | R <sub>s</sub><br>Ohm cm <sup>2</sup> | C <sub>dl</sub><br>μF cm <sup>-2</sup> | IE%  | θ     |
|--------|--|---------------------------------------|--|------|-------|
| Blank  | 93.84                                  | 2.39                                  | 38.9                                   | –    | –     |
| 50     | 397.41                                 | 1.66                                  | 8.56                                   | 76.4 | 0.764 |
| 100    | 450.65                                 | 1.59                                  | 4.19                                   | 79.2 | 0.792 |
| 150    | 540.43                                 | 2.25                                  | 3.13                                   | 82.6 | 0.826 |
| 200    | 650.23                                 | 1.88                                  | 2.25                                   | 85.5 | 0.855 |
| 250    | 798.45                                 | 1.75                                  | 1.42                                   | 88.2 | 0.882 |
| 300    | 890.34                                 | 1.63                                  | 1.21                                   | 89.5 | 0.895 |



**Figure 6.** The equivalent circuit model for the electrochemical impedance measurements.

heterogeneity, roughness effect, inhibitor adsorption, and variation of properties or composition of surface layers. The equivalent circuit, [Figure 6](#), 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 ( $R_s$ ).

## Conclusions

Analytical and electroanalytical techniques are effective in the determination of iron corrosion rate and ferrous

ion  $Fe^{+2}$  concentrations in the solution due to corrosion process. All the used techniques indicated that adding omeprazole drugs (OMP) into acidic environment decreases the ferrous  $Fe^{+2}$  ion concentrations so that OMP drugs are considered green corrosion inhibitors for iron in the acidic environment of lower pH values. Omeprazole drugs considered green inhibitors as they are nontoxic inhibitors to human being especially at very low concentrations. Also, prevention of the steel from corrosion is of environmental importance, as corrosion is a terrible waste of both natural resources and money. The corrosion inhibition efficiency increases by

increasing OMP inhibitor concentrations to reach to 96% at 300 ppm, as soon as the  $\text{Fe}^{+2}$  ion decreases. The data of AAS show that the ferrous ion  $\text{Fe}^{+2}$  concentrations were decreased by increasing inhibitor concentration and increased by lowering pH of the solution. The volume of hydrogen evolved during the corrosion reaction of iron in acidic HCl medium was decreased by increasing OMP inhibitor concentrations which may be because inhibitor compounds control the hydrogen evolution reaction. The reduction in the reaction number and time delay ( $\Delta t_m$ ) of the OMP inhibitor decrease by increasing the pH value in the following order:  $\text{pH } 3 > \text{pH } 1$ . The results of acidimetric method indicate that the hydrogen ion concentrations decreased in the inhibited medium as inhibitor molecule acts as a ligand for protons in acidic environment. Potentiodynamic measurement results indicate that the values of  $E_{\text{corr}}$  change slowly to negative, indicating that OMP inhibitors are of mixed type inhibitors. EIS shows that the size of the semicircle increases with the inhibitor concentration, indicating the charge transfer process as the main controlling factor of the iron dissolution process. The maximum inhibition efficiency obtained using 300 ppm of omeprazole drugs is 96% within the range when it is compared with the obtained efficiency in the cited references (previous works). The data obtained from the seven analytical and electroanalytical techniques are in good agreement with each other with ( $\pm 2$ ) to indicate that the addition of omeprazole (OMP) drugs inhibits the corrosion of iron in the acidic environment and decreases the iron dissolution process in this environment at lower pH values.

### Disclosure statement

No potential conflict of interest was reported by the author(s).

### Notes on contributor

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