



King Saud University
Arabian Journal of Chemistry

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ORIGINAL ARTICLE

Effect of Tryptophan on the corrosion behavior of low alloy steel in sulfamic acid



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Received 10 June 2011; accepted 22 November 2011

Available online 17 December 2011

KEYWORDS

Corrosion inhibition;
Acid cleaning;
Sulfamic acid;
Amino acids;
EIS;
EFM

Abstract Sulfamic acid is widely used in various industrial acid cleaning applications. In the present work, the inhibition effect of Tryptophan (Tryp) on the corrosion of low alloy steel in sulfamic acid solutions at four different temperatures was studied. The investigations involved electrochemical methods (electrochemical impedance spectroscopy; EIS and the new technique electrochemical frequency modulation; EFM) as well as gravimetric measurements. The inhibition efficiency and the apparent activation energy have been calculated in the presence and in the absence of Tryp. It is most probable that the inhibition property of Tryp was due to the electrostatic adsorption of the protonated form of Tryp on the steel surface. Adsorption of the inhibitor molecule, onto the steel surface followed the Temkin adsorption isotherm. The thermodynamic parameters of adsorption were determined and discussed. All of the obtained data from the three techniques were in close agreement, which confirmed that EFM technique can be used efficiently for monitoring the corrosion inhibition under the studied conditions.

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1. Introduction

The scaling or deposition on the boiler and heat exchange tubes is a phenomenon of common occurring. Eventually, removal of scales from the boiler tubes becomes essential if dam-

age to the boiler is to be prevented. The common way of removing scales is to chemically clean the boiler. The important step in the chemical cleaning process involves the use of acid to dissolve the scales. If proper cleaning or descaling was not carried out then it would greatly affect the efficiency of the plant. Therefore, industrial acid cleaning is a very important procedure applied chiefly to remove scales and other unwanted deposits from steam generating equipment and from chemical and petrochemical reaction vessels (Majnouni and Jaffer, 2003; Natarajan and Sivan, 2003).

Several acid solutions will effectively remove waterside deposits. Hydrochloric, sulfuric, sulfamic and citric acids are employed for such purpose (Sathiyarayanan et al., 2006).

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Peer review under responsibility of King Saud University.



Although, sulfamic acid is widely used in many diversified industrial acid cleaning applications, for unknown reasons researchers have not been interested in studying the corrosion characteristics of different metal materials in sulfamic acid media. Sulfamic acid is a crystalline solid, and is highly stable. In addition to its strength as an effective solvent for iron oxides and variety of water-formed scales it has also many other advantages such as that it is suitable for use with alloy steels and austenitic stainless steels. The usual concentration of sulfamic acid is 5–10 wt.% by weight at a temperature range of 55–65 °C (Majnoui and Jaffer, 2003; McCoy Jemes, 1984; Morad, 2008).

Because of the general aggressivity of acid solutions, corrosion inhibitors are commonly used to reduce corrosive attack on metallic materials (Ashassi-Sorkhabi et al., 2005; Abdel-Rehim et al., 2006). The uses of corrosion inhibitors in specific applications such as the acid cleaning of steam generator components have been reviewed briefly (Bhrara et al., 2008).

Amino acids are from a class of organic compounds that are completely soluble in aqueous media, relatively cheap, easy to produce with high purity; nontoxic and considered as environmentally friendly compounds. These properties enhance their use as corrosion inhibitors for iron, steel and stainless steel (Oguzie et al., 2007; Ashassi-Sorkhabi et al., 2005; Kalota and Silverman, 1994; Madkour and Ghoneim, 1997; Morad et al., 2002). In the present work, Tryptophan (Tryp) is used as environmental safe corrosion inhibitor.

The purpose of this study is to determine the inhibition efficiency of Tryp on the corrosion of low alloy steel (LAS) in sulfamic acid solutions and to study the reliability of the EFM technique for monitoring the corrosion inhibition under the studied conditions.

2. Experimental

Low alloy steel (ASTM A213 grade T12) strips composed of 0.35 wt.% Si, 0.64 wt.% Mn, 2.30 wt.% Cr, 0.86 wt.% Mo and the remaining 95.85 wt.% Fe were used. Generally, low alloy steels are widely employed for manufacturing the steam generating sections (Dobrzanski, 2004; Ghanem et al., 1996).

All solutions were freshly prepared from analytical grade chemical reagents using ultrapure water. The experiments were conducted in stagnant aerated solutions at different temperatures 25, 40, 50 and 60 °C ± 1 °C.

Measurements of weight changes were performed on rectangular coupons of size 1.5 cm × 1 cm × 0.2 cm with total exposed area of (4 cm²). The weight loss was determined by weighing the cleaned samples before and after 24 h immersion in tested solutions at different temperatures.

The electrochemical experiments were carried out using a three-electrode glass cell assembly of 150 cm³ volume capacity. The cell consists of a low alloy steel electrode embedded in epoxy resin with an exposed area of (1 cm²) as working electrode, a saturated calomel electrode as reference electrode, and a platinum foil (1 cm²) as counter electrode. The working electrodes were mechanically abraded with different grades (240, 400, 600 and 1200) of abrasive papers, degreased with acetone in an ultrasonic bath, then washed with ultrapure water and finally dried before use.

The electrochemical impedance spectroscopy (EIS) measurements were carried out using AC signals of amplitude

5 mV peak to peak at the open circuit potential in the frequency range between 15 kHz and 0.3 Hz.

The electrochemical frequency modulation (EFM) is a new technique that provides a new tool for electrochemical corrosion monitoring. The great strength of the EFM is the causality factor, which serves as an internal check on the validity of the EFM measurement. The theory and features of EFM technique was reported previously (Bosch et al., 2001; Abdel-Rehim et al., 2006; Amin et al., 2009).

All electrochemical experiments were carried out using Gamry PCI300/4 Potentiostat, EIS300 software, EFM140 software and Echem Analyst 5.21 for results plotting, graphing, data fitting and calculating.

The corrosion behavior was further confirmed by optical microscope observations.

3. Results and discussion

3.1. Effect of inhibitor concentration and solution temperature

The inhibition property of Tryp on the corrosion of LAS in sulfamic acid solutions at different temperatures 25, 50, and 60 °C ± 1 was examined using the following techniques.

3.1.1. Electrochemical frequency modulation studies

Fig. 1a and b represents the EFM intermodulation spectra of LAS in stagnant 0.6 M sulfamic acid devoid of and containing 0.04 M Tryp at 60 °C as an example. Similar results were collected for the other concentrations at different temperatures.

The EFM results; corrosion current density (I_{corr}), Tafel constants (β_c and β_a) and the causality factors (CF2 and CF3) are given in Table 1.

The inhibition efficiency (IE%) of Tryp was calculated using the following equation:

$$IE\% = \left[1 - \left(\frac{I_{corr}}{I_{corr}^0} \right) \right] \times 100 \quad (1)$$

where I_{corr}^0 and I_{corr} are the corrosion current densities for uninhibited and inhibited solutions, respectively.

The calculated values of the inhibition efficiency (IE%) at different concentrations of Tryp and at different temperatures (25–60 °C) are also included in Table 1. Analysis of the collected data listed in this table indicates the following:

- Tryp has good inhibitory property in corrosion of LAS in 0.6 M sulfamic acid solution. The presence of different concentrations of Tryp reduces the corrosion current density (I_{corr}) values and this suppression in I_{corr} increases as the inhibitor concentration increases, indicating that Tryp inhibits the corrosion of LAS in sulfamic acid solution, through adsorption on steel surface.
- The value of I_{corr} was directly proportional to temperature as a result of partial de-sorption of inhibitor species from the metal surface.
- The values of the anodic and cathodic Tafel slopes were almost unchanged, indicating that the presence of Tryp in sulfamic acid solution has no effect on the mechanism of the dissolution process of the metal and the adsorbed mol-

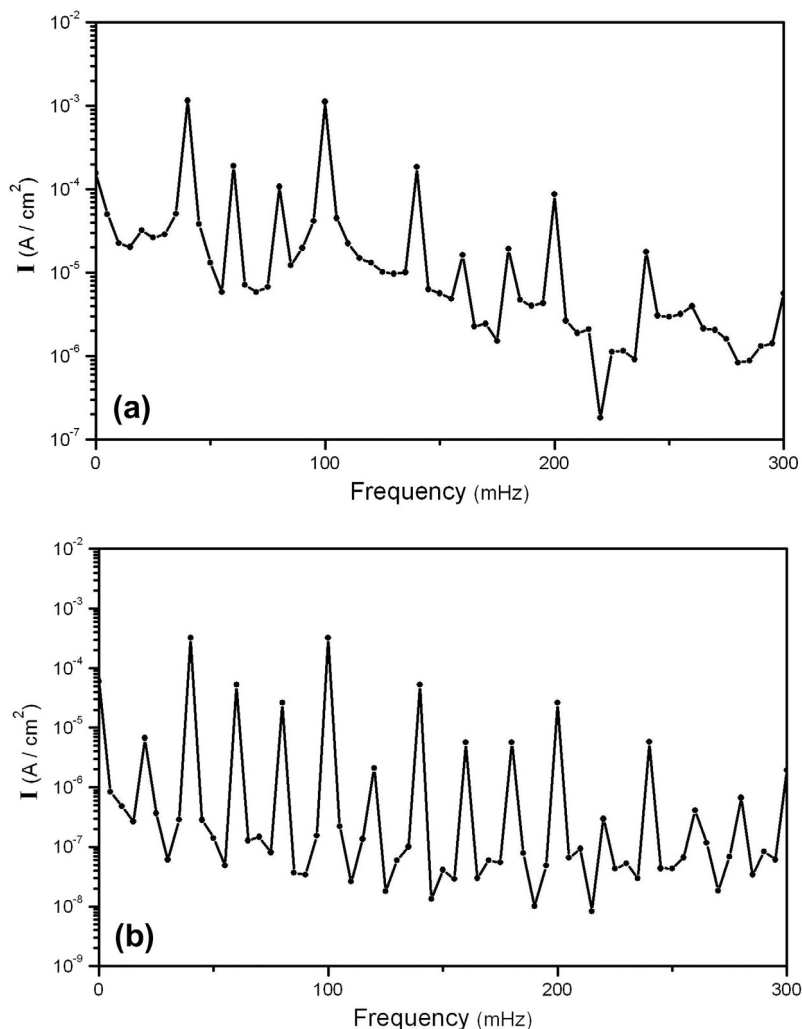


Figure 1 Intermodulation spectrum for LAS in 0.6 M sulfamic at 55 °C (a) in absence and (b) in presence of 0.004 M Tryp.

ecules screen the covered part of the electrode and therefore protects it from the action of the corrosion medium (Abd El-Maksoud and Fouda, 2005).

- (d) The values of causality factors (CF2 and CF3) obtained under different experimental conditions were approximately equal to the theoretical values 2 and 3 indicating that the measured data are reliable (Bosch et al., 2001).
- (e) The values of inhibition efficiency (IE%) show that the corrosion process depends on the two factors, the inhibitor concentration and the solution temperature. The IE% increases by increasing of concentration of Tryp but decreases with the increase of solution temperature.

In order to evaluate the results of EFM technique as an effective corrosion monitoring technique, weight loss and EIS measurements were performed.

3.1.2. Weight loss studies

The weight losses of LAS in static 0.6 M sulfamic acid solutions devoid of and containing 0.04 M of Tryp at different

temperatures were determined for 24 h. The weight losses ($\text{mg cm}^{-2} \text{min}^{-1}$) were listed in Table 2.

The weight loss and therefore the rate of corrosion were enhanced with increasing the solution temperature but decreases with the increase of inhibitor concentration.

The inhibition efficiency (IE%) of Tryp was calculated under different experimental conditions by using the following equation:

$$\text{IE}\% = \left[1 - \left(\frac{\text{CR}}{\text{CR}^0} \right) \right] \times 100 \quad (2)$$

where CR^0 and CR are the corrosion rates obtained from weight loss measurements in the absence and presence of inhibitor, respectively. The calculated values of inhibition efficiency (IE%) were also listed in Table 2.

The results reveal that the inhibition efficiency (IE%) of Tryp increases with increasing their concentration as a result of increasing the surface coverage by inhibitor species. However, at a given inhibitor concentration, the inhibition efficiency of Tryp decreases with rising the temperature. This behavior is due to the decrease in the strength of adsorption process by increasing temperature, suggesting that physical

Table 1 Electrochemical kinetic parameters obtained from EFM technique for LAS in 0.6 M sulfamic acid with various concentrations of Tryp at different temperatures.

Temp (°C)	Tryp conc (M)	β_a (mV dec ⁻¹)	β_c (mV dec ⁻¹)	CF2	CF3	I_{corr} ($\mu\text{A cm}^{-2}$)	IE%	θ
25	0.00	90.9	195.5	1.825	2.889	652.5	0.00	0.000
	0.005	88.3	192.7	1.973	3.140	380.1	41.75	0.417
	0.01	84.6	191.2	1.735	2.856	280.4	57.03	0.570
	0.02	93.3	199.4	1.953	2.875	140.0	78.54	0.785
	0.04	75.7	190.7	1.851	2.851	73.12	88.79	0.888
40	0.00	73.4	122.5	1.958	2.840	902.7	0.00	0.000
	0.005	72.5	118.5	2.156	3.281	589.6	34.68	0.347
	0.01	79.8	125.4	1.846	2.856	419.2	53.56	0.536
	0.02	80.6	123.5	2.264	3.211	261.4	71.04	0.710
	0.04	78.9	118.4	1.895	2.886	145.3	83.90	0.839
50	0.00	78.3	132.6	2.125	3.074	1273	0.00	0.000
	0.005	85.5	148.3	1.898	2.812	931.5	26.83	0.268
	0.01	81.6	136.2	1.962	2.820	675.1	46.97	0.470
	0.02	82.7	154.2	2.211	3.259	452.5	64.45	0.645
	0.04	80.6	126.3	2.021	3.127	265.3	79.16	0.792
60	0.00	96.3	121.1	1.984	2.790	2171	0.00	0.000
	0.005	91.5	115.3	1.894	3.101	1705	21.46	0.215
	0.01	86.2	110.2	2.354	2.910	1248	42.51	0.425
	0.02	98.4	125.5	2.089	3.128	904.8	58.32	0.583
	0.04	111.5	132.2	1.991	2.901	621.2	71.39	0.714

Table 2 Weight loss results for LAS in 0.6 M sulfamic acid at different temperatures.

Temp (°C)	Tryp conc. (M)	Weight loss (mg)	CR ($\text{mg cm}^{-2} \text{min}^{-1}$)	IE%
25	0.00	62	0.0108	0.00
	0.04	5	0.0009	91.94
40	0.00	76	0.0132	0.00
	0.04	15	0.0026	80.26
50	0.00	109	0.0189	0.00
	0.04	24	0.0042	77.98
60	0.00	206	0.0358	0.00
	0.04	67	0.0116	67.48

adsorption may be the type of adsorption of the inhibitor on the sample surfaces (Abd El Rehim et al., 2002).

3.1.3. Electrochemical Impedance studies

Impedance diagrams of LAS in stagnant 0.6 M sulfamic acid solution free, and containing different concentrations of Tryp at 50 °C (as an example), are shown in Fig. 2a and b in the Nyquist and Bode representation, respectively.

All the plots have the same shape where a single depressed capacitive semicircle was obtained. This frequency dispersion can be attributed to inhomogeneities and roughness of the electrode surface (Paskossy, 1994; Growcock and Jasinski, 1989).

The electrochemical response to the impedance measurements was best simulated with the equivalent circuits as shown in Fig. 3. According to Fig. 3, the polarization resistance (R_p), which corresponds to the diameter of Nyquist plot, includes charge transfer resistance (R_{ct}), diffuse layer resistance (R_d), film resistance (R_f) and all accumulated species at metal/solution interface (R_a). Therefore, in the present study, the difference in real impedance at lower and higher frequencies is considered as the polarization resistance ($R_p = R_{ct} + R_d +$

$R_f + R_a$) (Lorenz and Mansfeld, 1986; Solmaz et al., 2008; Sam et al., 2010).

Table 3 shows the impedance parameters, the polarization resistance (R_p) and the double layer capacitance (C_{dl}). Inspections of these data indicate the following:

- The polarization resistance (R_p) increased, while the double layer capacitance (C_{dl}) decreased with the increase of inhibitor concentration. This may be the result of decreasing film capacitance due to increase in the surface coverage by the inhibitor molecules.
- The values of R_p decreased, while the C_{dl} values increased with increase in the solution temperature.

The above results can be explained on the basis that the electrostatic adsorption of inhibitor species at the metal surface leads to the formation of a physical protective film that retards the charge transfer process and therefore inhibits the corrosion reactions, leading to the increase of R_p value. Moreover, the adsorbed inhibitor species decreases the electrical

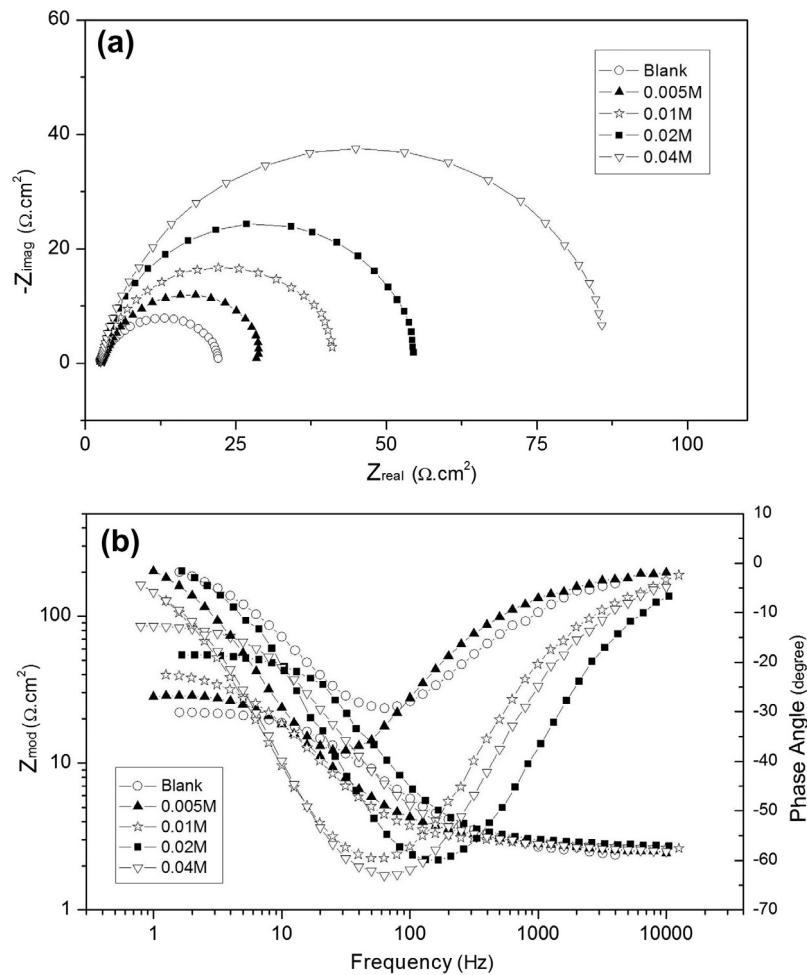


Figure 2 Nyquist (a) and Bode (b) plots for LAS in 0.6 M sulfamic without and with various concentrations of Tryp at 45 °C.

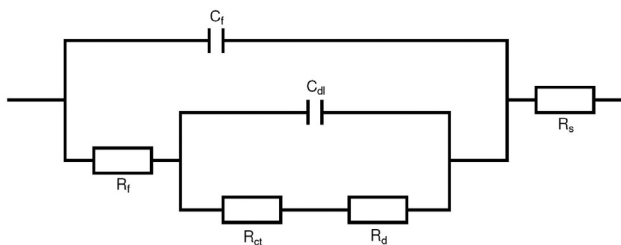


Figure 3 Equivalent electrical circuit diagram for EIS results of LAS in inhibited solutions, R_{ct} , charge transfer resistance; C_{dl} , double layer capacitance; R_s , solution resistance; R_d , diffuse layer resistance; C_f , film capacitance; R_f , film resistance.

capacity of the electrical double layer at the electrode/solution interface and, therefore, decreases the values of C_{dl} (Mansfield, 1987; McCafferty and Hackerman, 1972).

Since the electrochemical theory assumes that the reciprocal of polarization resistance ($1/R_p$) is directly proportional to the corrosion rate, the inhibition efficiency (IE%) was calculated from R_p values using the following equation:

$$IE\% = \left[1 - \left(\frac{R_p^0}{R_p} \right) \right] \times 100 \quad (3)$$

where R_p^0 and R_p are the polarization resistance values in the absence and presence of inhibitor, respectively.

The calculated values of the inhibition efficiency (IE%) for different concentrations of Tryp at different temperatures are also presented in Table 3. The values of IE% increase with the increase of the inhibitor concentration, but decrease with the increase of solution temperature.

The produced results from weight loss and EIS measurements were comparable with those obtained from the EFM method.

3.2. Apparent activation energy

The temperature dependence of low alloy steel dissolution in sulfamic acid solution and in the presence of Tryp was employed using the following Arrhenius equation:

$$\log(\text{Corrosion Rate}) = \frac{-E_a}{2.303RT} + A \quad (4)$$

where E_a is the apparent activation energy, R is the universal gas constant, T is the absolute temperature, and A is the Arrhenius pre-exponential factor.

A plot of logarithm of the corrosion rate of LAS obtained from EFM and EIS techniques versus $1/T$ gives straight lines; and its slope is the $-E_a/2.303R$ (curves are not attached).

Table 3 Electrochemical kinetic parameters obtained from EIS technique for LAS in 0.6 M sulfamic acid with various concentrations of Tryp at different temperatures.

Temp (°C)	Tryp conc. (M)	C_{dl} ($\mu\text{F cm}^{-2}$)	R_p ($\Omega \text{ cm}^2$)	IE%	θ
25	0.00	453.1	40.68	0.00	0.000
	0.005	335.2	67.38	39.63	0.396
	0.01	312.2	93.21	56.36	0.564
	0.02	223.4	199.3	79.59	0.796
	0.04	70.8	389.5	89.56	0.896
40	0.00	877.5	23.7	0.00	0.000
	0.005	684.7	35.16	32.59	0.326
	0.01	542.6	52.55	54.90	0.549
	0.02	306.4	80.77	70.66	0.707
	0.04	187.6	139.8	83.05	0.830
50	0.00	964.3	17.69	0.00	0.000
	0.005	715.4	24.7	28.38	0.284
	0.01	625.3	34.57	48.83	0.488
	0.02	275.6	50.32	64.84	0.648
	0.04	238.6	78.41	77.44	0.774
60	0.00	1686.5	11.29	0.00	0.000
	0.005	1263.2	14.38	21.49	0.215
	0.01	982.8	18.82	40.01	0.400
	0.02	738.9	26.08	56.71	0.567
	0.04	436.8	41.06	72.50	0.725

The literature reported that the higher activation energy value of corrosion process in the presence of inhibitor rather than the absence of inhibitor is attributed to its physical adsorption, while it is found to be opposite in the case with chemical adsorption (Popova et al., 2003; Mora-Mendoza and Turgoose, 2001; Jovancevic et al., 1999; Hirozawa, 1995).

The calculated values of E_a extracted from EFM and EIS techniques, are given in Table 4. The analysis of E_a values shows that an increase in corrosion activation energy in the presence of Tryp compared to its absence with a decrease in inhibition efficiency with the rise in temperature indicates that the type of adsorption of Tryp on the steel surface in sulfamic acid solutions belongs to physical adsorption.

3.3. Adsorption Isotherm

The experimental data for the tested inhibitor have been applied to different adsorption isotherm equations. The best-fitted straight line was obtained for the plot of surface coverage ($\theta = \text{IE}\%/100$) versus logarithm of inhibitor concentration (C_{inh}). This assumes that the adsorption of Tryp at the steel surface, in sulfamic acid solutions, was found to follow the Temkin adsorption isotherm according to the following equation (Oguzie et al., 2004; Duong, 1998):

$$\text{Temkin isotherm: } \theta = \frac{1}{2a} \ln K_{ads} + \frac{1}{2a} \ln C_{inh} \quad (5)$$

where K_{ads} is the equilibrium constant of the adsorption process, C_{inh} is the bulk concentration of inhibitor and θ is the degree of surface coverage.

Fig. 4 is an example of Temkin adsorption isotherm for various concentrations of Tryp at different temperatures; using the data obtained from EFM measurements.

The values of equilibrium constant (K_{ads}) are listed in Table 5. These values were calculated from the intercepts and slopes of the straight lines of Temkin isotherm curves. Inspection of this table reveals that the values of K_{ads} were relatively small confirming the suggestion that Tryp is physically adsorbed on the metal surface (Keera and Deyab, 2005). Additionally, values of K_{ads} decreased with increase in the temperature. This result confirmed the suggestion that the strength of the adsorption decreased with temperature and the inhibitor species are easily removable by the solvent molecules from the steel surface (Keera and Deyab, 2005; Abd El Rehim et al., 2002; El Azhar et al., 2002).

3.4. Surface examinations

The formation of a protective surface film of inhibitor was further confirmed by optical microscopy observations of the electrode surface.

Fig. 5a–c shows an array of optical microscopy images for low alloy steel samples before and after immersion for 24 h in stagnant 0.6 M sulfamic acid solution devoid of and containing 0.04 M Tryp at 25 °C, respectively.

The morphology of specimen surface in Fig. 5b reveals that in the absence of Tryp, the surface is highly corroded with areas of localized corrosion. However, in presence of the inhibitor (Fig. 5c), the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The electrode surface is almost free from corrosion due to the formation of an adsorbed protective film of the inhibitor at the electrode surface.

Table 4 Activation energies of Tryp using the obtained data from the two techniques.

Tryp conc. (M)	E_a (kJ mol^{-1})	
	EFM	EIS
0.00	27.21	29.40
0.005	34.40	35.51
0.01	34.22	36.68
0.02	43.02	47.04
0.04	53.64	52.33

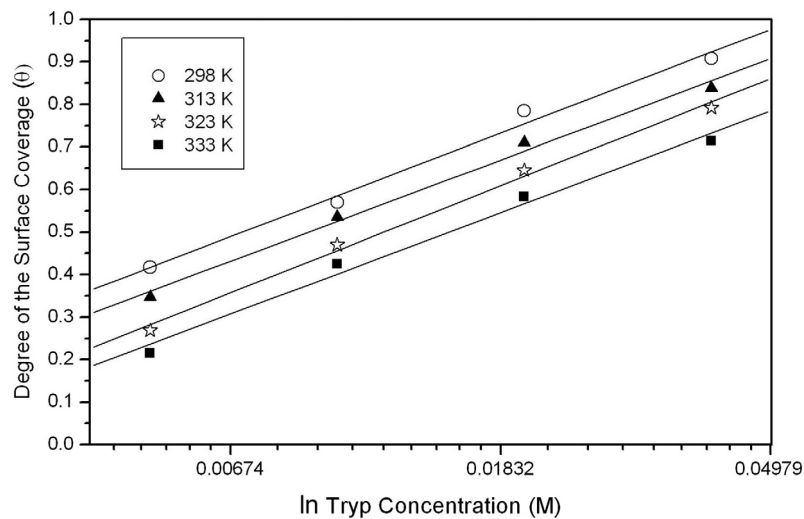


Figure 4 Temkin isotherm plots for LAS in 0.6 M sulfamic in presence of various concentrations of Tryp at different temperatures (data obtained from EFM technique).

3.5. Mechanism of inhibition

All the obtained results support that Tryp actually inhibits the corrosion of LAS in sulfamic acid solutions, to an appreciable extent. The corrosion inhibition is due to its physical adsorption and formation of protective film of the adsorbate on the steel surface.

It is known that the ionization state of an amino acid in aqueous solution depends on the environment pH. In aqueous acid solution, the amino acids exist either as neutral molecules or in the form of cations (protonated) (Amin et al., 2009; Bockris and Yang, 1991; Abdallah and Megahed, 1995).

Table 5 The calculated equilibrium constant of adsorption for Tryp.

Technique	Equilibrium constant of adsorption, K_{ads}			
	25 °C	40 °C	50 °C	60 °C
EIS	1004.32	863.83	728.54	495.60
EFM	1107.35	909.49	611.53	537.34

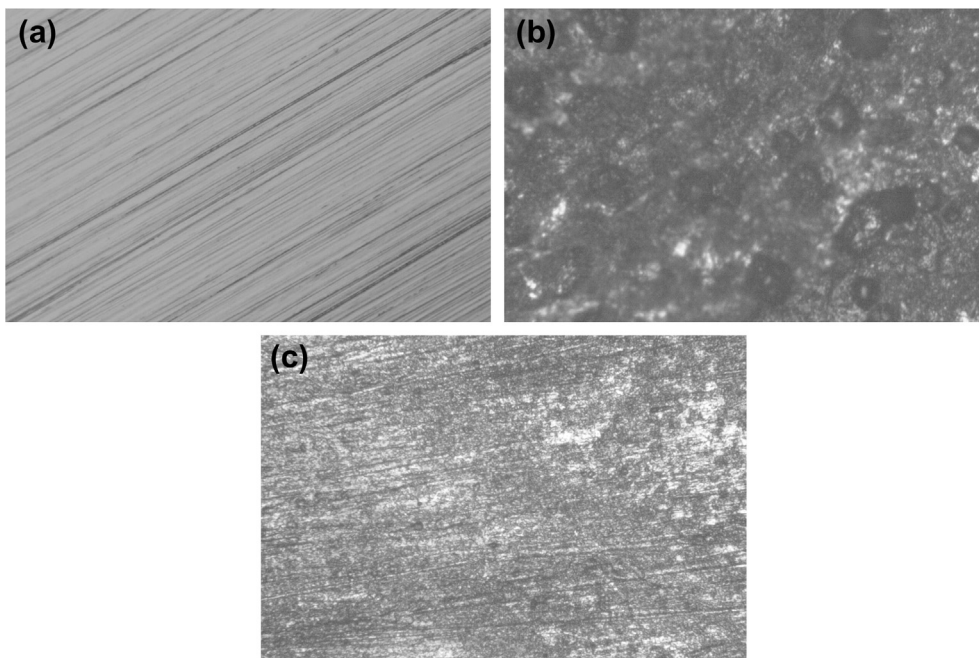


Figure 5 Optical microscopy images for LAS (a) before immersion, (b) after immersion for 24 h in 0.6 M sulfamic acid at 25 °C (c) after immersion for 24 h in 0.6 M sulfamic solution containing 0.04 M Tryp at 25 °C.

It has been reported previously that, in aqueous acid solutions, the surface of steel samples is positively charged (Lagrenée et al., 2002; Solmaz et al., 2008; Wahdan et al., 2002). Therefore, the amino acid may be adsorbed on the positively charged surface in the form of neutral molecules, involving the displacement of water molecules from the metal surface and sharing electrons between nitrogen atoms and the metal surface (Bentiss et al., 1999). Furthermore, adsorption can occur via adsorbed sulfamate anions at the positively charged metal surface. These anions form a negatively charged double layer and consequently, the adsorption capability of protonated amino acids increases (Wahdan et al., 2002; Larabi et al., 2004). Therefore, the inhibition efficiency of Tryp may be ascribed to the presence of a phenylic ring, which provides some of its electronic density to the inhibitor structure (Olivares et al., 2006).

4. Conclusions

- Tryptophan has shown good inhibiting properties for LAS in 0.6 M sulfamic acid solutions.
- The inhibition efficiency was directly proportional to inhibitor concentration, while it was inversely proportional to temperature.
- Adsorption of the inhibitor molecule, onto the steel surface followed the Temkin adsorption isotherm.
- All of the data obtained from the three techniques were in close agreement and their results followed almost the same trends. Therefore, the EFM technique is apparently a promising technique with which to measure corrosion behaviour under the studied conditions.

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