الحد من تآكل الفولاذ الكاربوني بوسط حامض الهيدرولكوريك باستخدام ثايوسينايد البوتاسيوم

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الملخص:

لقد تمت دراسة تأثير مركب ثايوسينايد البوتاسيوم (KSCN) على الحد من تآكل الفولاذ الكاربوني الموجود في حامض 1M–HCl وعلاقة تراكيز المثبط وذلك باستخدام منحنيات الاستقطاب وطيف الممانعة الكهروكيميائي وقياس نقصان الوزن. ان النتائج التي تم الحصول عليها كشفت ان هذا المركب هو نوع جيد من المثبطات الفعالة. وكذلك تمت دراسة تأثير درجة الحرارة على السلوك التآكلي للفولاذ باستعمال تركيز (KSCN) ملائم، وذلك فى مدى درجات الحرارة (298°K–328°K)، حيث لوحظ نقصان في كفاءة التثبيط بصورة طفيفة مع زيادة درجة الحرارة. ان التغيرات في معاملات الممانعة (مقاومة حركة الشحنات، سعة الطبقات) دلت على ان امتزاز الـ KSCN على سطح المعدن مما ادى إلى تكوين طبقة عازلة حامية. لقد وجد بأن امتزاز KSCN على سطح الفولاذ الكاربوني يخضع لامتزاز لانجمير الآيزوثيرمي. وكما تم تحديد ومناقشة بعض الدوال الثرموديناميكية لعمليات التحلل والأمتزاز.

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ORIGINAL ARTICLE

Inhibition of carbon steel corrosion in 1 M HCl medium by potassium thiocyanate

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Abstract The effect of potassium thiocyanate (KSCN) on the corrosion of carbon steel (C-steel) in 1 M HCl has been investigated in relation to the concentration of the inhibitor by polarization curves, electrochemical impedance spectroscopy and weight loss measurement. The results obtained revealed that this compound is a good mixed type inhibitor with predominant cathodic effectiveness. The effect of temperature on the corrosion behavior with the addition of optimal concentration of KSCN was studied in the temperature range 298–328 K. The value of inhibition efficiency decreases slightly with the increase in temperature. Changes in impedance parameters (charge transfer resistance, R_{ct}, and double layer capacitance, C_{dl}) were indicative of adsorption of KSCN on the metal surface, leading to the formation of a protective film. Adsorption of KSCN on the C-steel surface was found to obey the Langmuir adsorption isotherm. Some thermodynamic functions of dissolution and adsorption processes were also determined and discussed.

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1. Introduction

Iron and iron-based alloys of different grades are extensively used in numerous industrial and engineering applications, including construction and designs, where they are deployed in various service environments containing, acids, alkalis and salt solutions. These service environments readily lead to inevitable corrosion of exposed surfaces of the metal because of their aggressive nature. It is a general consensus that the best method to protect the metal deployed in these corrosive

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environments is to add corrosion inhibitors (Salghi et al., 2000a,b; Larabi et al., 2005; Mihit et al., 2010; Benali et al., 2011; Zarrok et al., 2011). To this end, the use of organic and inorganic substances to inhibit corrosion of metals in many environments is well established. Most of the well-known acid inhibitors are organic compounds containing nitrogen, oxygen and sulfur atoms (Benali et al., 2006; Merah et al., 2008; Ben et al., 2012). But other types of inhibitors, such as inorganic compounds, are used in different acidic media (Robertson, 1951; Cartledge, 1968; Breslin et al., 1994; Bada-wy et al., 1999; Salghi et al., 2000a,b; Kolics et al., 2001; Wu and Tsai, 2003; Ali et al., 2009). In this work, potassium thio-cyanide (KCN) has been studied for its corrosion inhibition of C-steel in 1 M HCl solutions by weight loss and electrochemical methods.

2. Experimental

2.1. Materials

The steel used in this study is a carbon steel (Euronorm: C35E carbon steel and US specification: SAE 1035) with a chemical composition (in wt%) of 0.370% C, 0.230% Si, 0.680% Mn, 0.016% S, 0.077% Cr, 0.011% Ti, 0.059% Ni, 0.009% Co, 0.160% Cu and the remainder iron (Fe).

2.2. Solutions

The aggressive solutions of 1 M HCl were prepared by dilution of analytical grade 37% HCl with distilled water. The inorganic compound tested was potassium thiocyanate. The concentration range of this compound was 10^{-5}–10^{-2} M.

2.3. Weight loss measurements

Coupons were cut to $2 \times 2 \times 0.08$ cm^3 dimensions for weight loss measurements. Prior to all measurements, the exposed area was mechanically abraded with 180, 320, 800, and 1200 grades of emery papers. The specimens were washed thoroughly with bi-distilled water, degreased and dried with ethanol. Gravimetric measurements are carried out in a double walled glass cell equipped with a thermostated cooling condenser. The solution volume was 80 mL. The immersion time for the weight loss was 6 h at 298 K.

2.4. Polarization measurements

2.4.1. Electrochemical impedance spectroscopy
The electrochemical measurements were carried out using Volta lab (Tacussel-Radiometer PGZ 100) potentiostat and controlled by Tacussel corrosion analysis software model (Voltamaster 4) under static conditions. The corrosion cell used had three electrodes. The reference electrode was a saturated calomel electrode (SCE). A platinum electrode was used as auxiliary electrode of a surface area of 0.094 cm^2. The working electrode was carbon steel. All potentials given in this study were referred to this reference electrode. The working electrode was immersed in the test solution for 30 min, to establish a steady state open circuit potential (Eocp). After measuring the Eocp, the electrochemical measurements were performed.

All electrochemical tests have been performed in aerated solutions at 298 K. The EIS experiments were conducted in the frequency range with a high limit of 100 kHz and a different low limit 0.1 Hz at open circuit potential, with 10 points per decade, at the resting potential, after 30 min of acid immersion, by applying 10 mV ac voltage peak-to-peak. Nyquist plots were made from these experiments and the best semicircle can be fitted through the data points in the Nyquist plot using a non-linear least square fit so as to give the intersections with the x-axis.

2.5. Potentiodynamic polarization

The electrochemical behavior of C-steel sample in the inhibited and uninhibited solution was studied by recording anodic and cathodic potentiodynamic polarization curves. Measurements were performed in the 1 M HCl solution containing different concentrations of the tested inhibitor by changing the electrode potential automatically from −800 to −200 mV versus corrosion potential at a scan rate of 1 mV s^{-1}. The linear Tafel segments of anodic and cathodic curves were extrapolated to corrosion potential to obtain corrosion current densities (I_{corr}).

3. Results and discussion

3.1. Polarization curves

Fig. 1, shows the polarization curves of C-steel in 1 M HCl without and with different concentrations (10^{-5}–10^{-2} M) of KSCN. With the increase of KSCN concentrations, both anodic and cathodic currents were inhibited. This result shows that the addition of KSCN inhibitor reduces anodic dissolution and also retards the hydrogen evolution reaction.

The inhibition efficiencies were calculated from I_{corr} values according to the following equation:

$$E\% = \frac{I_{corr} - I'_{corr}}{I_{corr}} \times 100 \tag{1}$$

where I_{corr} and I'_{corr} are the corrosion current densities in the absence and the presence of the inhibitor.
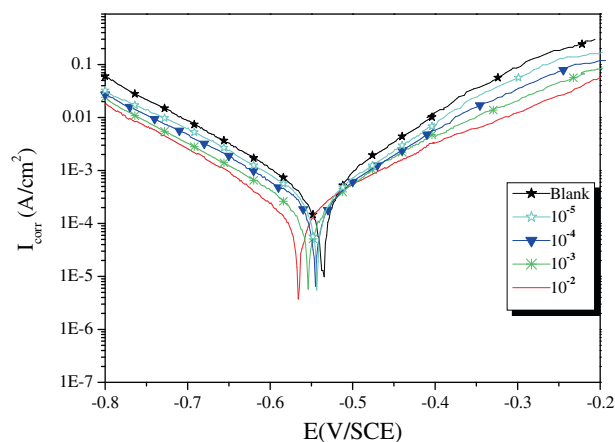

Figure 1 Potentiodynamic polarization curves of C-steel in 1 M HCl in the presence of different concentrations of KSCN.

Table 1 Electrochemical parameters of C-steel at various concentrations of KSCN in 1 M HCl and corresponding inhibition efficiency.

Conc. (M)	$-E_{corr}$ (mV/SCE)	I_{corr} (µA/cm^2)	$-b_c$ (mV/dec)	E (%)
Blank	536	355	150	–
10^{-5}	543	242	164	31.83
10^{-4}	545	195	135	45.07
10^{-3}	553	164	121	53.80
10^{-2}	566	70	123	80.28

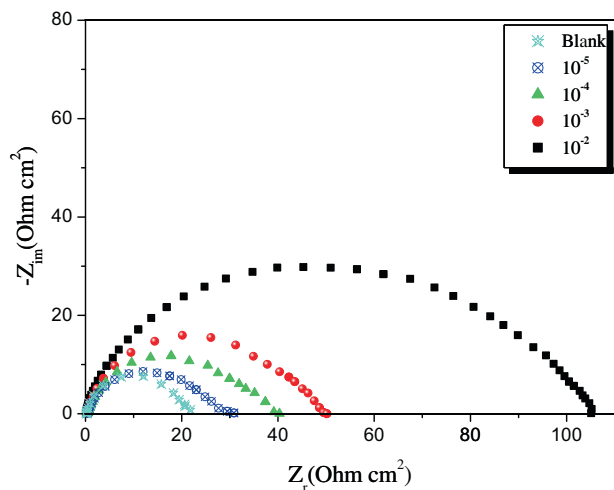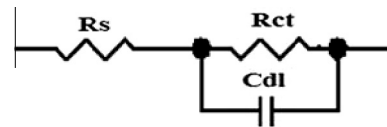**Figure 2** Nyquist diagrams for C-steel electrode with and without KSCN at E_{corr} after 30 min of immersion.

Figure 3 The electrochemical equivalent circuit used to fit the impedance spectra.

Table 1 gives the values of kinetic corrosion parameters as the corrosion potential E_{corr}, corrosion current density I_{corr}, cathodic Tafel slope (b_c), and inhibition efficiency for the corrosion of C-Steel in 1 M HCl with different concentrations of KSCN. The corrosion current densities were estimated by Tafel extrapolation of the cathodic curves to the open circuit corrosion potentials.

From this table, it can be deducted that, the presence of KSCN in the acidic solution results in a slight shift of corrosion potential toward more negative in comparison to that in its absence, and the values of corrosion potential nearly remain constant with the addition of different concentrations of KSCN. These results indicate that KSCN acts as a mixed-type inhibitor with predominant cathodic effectiveness. The addition of KSCN produces slight changes in the values of b_c. This indicates (Benali et al., 2007) that the adsorbed molecules of KSCN do not affect the mechanism of hydrogen evolution. The I_{corr} values decreased in the presence of different concentrations of KSCN. Values of inhibition efficiency were found to increase with an increase in the concentration of KSCN reaching maximum (80.28% value at 10^{-2} M).

3.2. Electrochemical impedance spectroscopy measurements

The corrosion behavior of C-steel in 1 M HCl in the absence and the presence of KSCN was investigated by EIS at 298 K after immersion for30 min. Nyquist plots for C-steel in uninhibited and inhibited 1 M HCl is shown in Fig. 2, that imped-

ance spectra show that a single semicircle and the diameter of the semicircle increases with increasing inhibitor concentration. These diagrams exhibit that the impedance spectra consist of one capacitive loop at high frequency, the high frequency capacitive loop was attributed to charge transfer of the corrosion process.

Values of the charge transfer resistance R_{ct} were obtained from these plots by determining the difference in the values of impedance at low and high frequencies (Benali et al., 2007). Values of the double-layer capacitance C_{dl} were calculated from the frequency at which the impedance imaginary component $-Z_{im}$ is maximum using the equation:

$$f(-Z_{Imax}) = \frac{1}{2\pi C_{dl} R_{ct}} \qquad (2)$$

The equivalent circuit model employed for these systems is presented in Fig. 3.

The percent inhibition efficiency is calculated by charge transfer resistance obtained from Nyquist plots, according to the equation:

$$E_{R_{ct}}\% = \frac{R'_{ct} - R_{ct}}{R'_{ct}} \times 100 \qquad (3)$$

where R_{ct} and R'_{ct} are the charge transfer resistance values without and with inhibitor, respectively.

Table 2 gives the values of the charge transfer resistance R_{ct}, double-layer capacitance C_{dl}, and inhibition efficiency obtained from the above plots. It can be seen that the presence of KSCN enhances the values of R_{ct} and reduces the C_{dl} values. The decrease in C_{dl}, which can result from a decrease in local

Table 2 Electrochemical impedance parameters for corrosion of C-steel in acid medium at various contents of KSCN.

Conc. (M)	R_{ct} (Ohm cm^2)	f_{max} (Hz)	C_{dl} (µF/cm^2)	$E_{R_{ct}}$ (%)
Blank	21	89	85.19	–
10^{-5}	31	100	51.36	32.25
10^{-4}	40	90	44.23	47.50
10^{-3}	50	80	39.81	58.00
10^{-2}	106	40	37.55	80.19

Table 3 Effect of KSCN concentration on corrosion data of C-steel in 1 M HCl.

Conc. (M)	W_{corr} (mg cm^{-2} h^{-1})	E_W (%)	θ
Blank	1.00	–	–
10^{-5}	0.69	31.00	0.31
10^{-4}	0.56	44.00	0.44
10^{-3}	0.43	57.00	0.57
10^{-2}	0.21	79.00	0.79
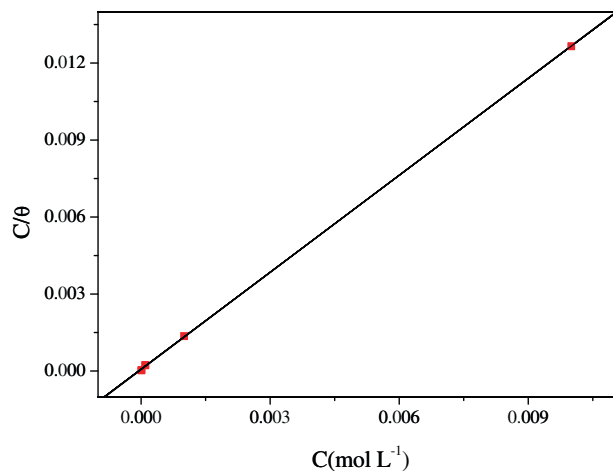
Figure 4 Plot of the Langmuir adsorption isotherm of KSCN on the C-steel surface at 298 K.

dielectric constant and/or an increase in the thickness of the electric double layer (Finley and Hackerman, 1960), suggested that KSCN molecules function by adsorption at the metal/solution interface. Thus, the decrease in C_{dl} values and the increase in R_{ct} values and consequently of inhibition efficiency may be due to the gradual replacement of water by the adsorption of the KSCN molecules on the metal surface, decreasing the extent of dissolution reaction (Hackerman et al., 1966; Benali et al., 2006).
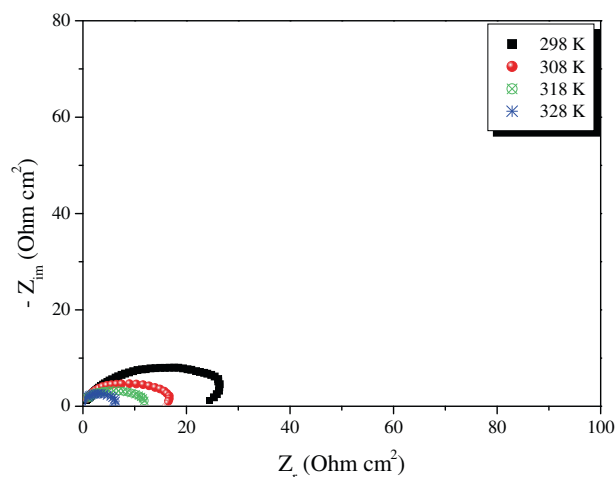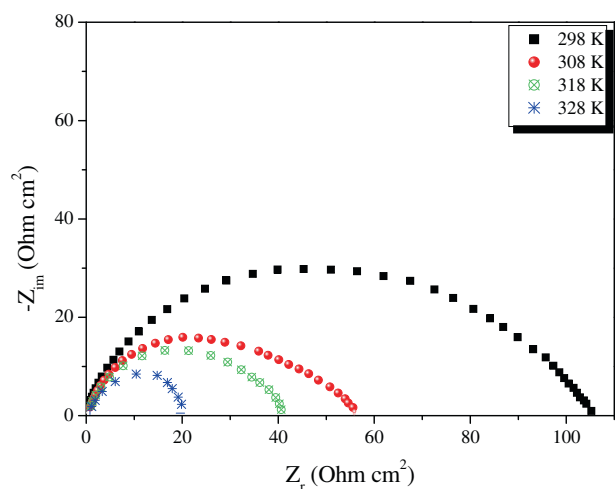
3.3. Weight loss measurements

Values of the inhibition efficiency and corrosion rate obtained from the weight loss measurements of C-steel for different concentrations of KSCN in 1 M HCl at 298 K after 6 h of immersion are given in Table 3.

The inhibition efficiency is defined as follows:

$$E_w\% = \frac{W_{corr} - W'_{corr}}{W_{corr}} \times 100 \qquad (4)$$

where W_{corr} and W'_{corr} are the corrosion rates of C-steel due to the dissolution in 1 M HCl in the absence and the presence definite concentration of inhibitor, respectively.

It can be seen from Table 3, that inhibition of KSCN inhibits the corrosion of C-steel and efficiency increases with the increasing inhibitor concentration. Maximum $E_w\%$ of KSCN was achieved at 10^{-2} M.

Figure 5 Nyquist diagrams for C-steel in 1 M HCl at different temperatures.

Figure 6 Nyquist diagrams for C-steel in 1 M HCl + 10^{-2} M of KSCN at different temperatures.

The results obtained from weight loss measurements were in agreement with the ones obtained from impedance and polarization data.

3.4. Adsorption isotherm

The basic information on the interaction between the inhibitor and the C-steel can be provided by the adsorption isotherm. Two main types of interaction can describe the adsorption of the organic or inorganic compounds: physical adsorption and chemisorption. These are influenced by the chemical structure of the inhibitor, the type of the electrolyte and the charge and nature of the metal.

Attempts were made to fit θ values to various isotherms including Frumkin, Temkin and Langmuir. By far the best fit was obtained with the Langmuir isotherm. According to this isotherm θ is related to concentration inhibitor C via:

$$\theta = \frac{KC}{KC + 1} \qquad (5)$$

Table 4 Thermodynamic parameters for the adsorption of KSCN in 1 M HCl on the C-steel at different temperatures.

	Temp (K)	R_{ct} (Ohm cm^2)	f_{max} (Hz)	C_{dl} (μF/cm^2)	E_{Rct} (%)
Blank	298	21	89	85.19	–
	308	13	152	80.58	–
	318	10	244	65.26	–
	328	5.4	588	50.15	–
KSCN	298	106	40	37.55	80.19
	308	56	158	17.99	76.78
	318	40	125	31.84	75.00
	328	19	400	20.95	71.58

where K is the equilibrium constant for the adsorption process.

$$\log K = -1.74 - \left(-\frac{\Delta G^0_{ads}}{2.303RT} \right) \tag{6}$$

where ΔG^0_{ads} is the free energy of adsorption. Rearrangement of the Eq. (5) yields to:

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{7}$$

It was found that Fig. 4 (plot of $\frac{\theta}{C}$ versus C) gives a straight line with slope near to 1, indicating that the adsorption of compound under consideration on C-steel/acidic solution interface obeys Langmiur's adsorption.

The free value of the energy of adsorption as calculated from Eq. (6) is $\Delta G^0_{ads} = -33.53$ kJ/mol. The negatives values of ΔG^0_{ads} show that the adsorption of KSCN is a spontaneous process (O'M and Swinkels, 1964) under the experimental conditions described. It is well known that values of ΔG^0_{ads} of the order of -20 kJ/mol or lower indicate a physisorption; those of order of -40 kJ/mol or higher are associated with chemisorptions as a result of the sharing or transfer of electrons from organic molecules to the metal surface to form a co-ordinate (O'M and Drazic, 1962; Branzoi et al., 2000). The calculated ΔG^0_{ads} value for KSCN was slightly inferior to -40 kJ/mol, confirming that the adsorption mechanism of KSCN on C-steel surface probably involves two types of interactions, predominant physisorption, and weak chemisorption.

3.5. Effect of temperature

To investigate the mechanism of inhibition and to calculate the activation energies of the corrosion process, EIS measurements were taken at various temperatures in the absence and the presence of different concentrations of KSCN (Figs. 5 and 6). Corresponding data are given in Table 4. In the studied temperature range (298–328 K) the R_{ct} values decrease with increasing temperature both in uninhibited and inhibited solutions and the values of the inhibition efficiency of KSCN decrease with temperatures.

The activation parameters for the corrosion process were calculated from Arrhenius type plot according to the following equation:

$$Ln\left(\frac{1}{R_{ct}}\right) = -\frac{E_a}{RT} + LnA \tag{8}$$

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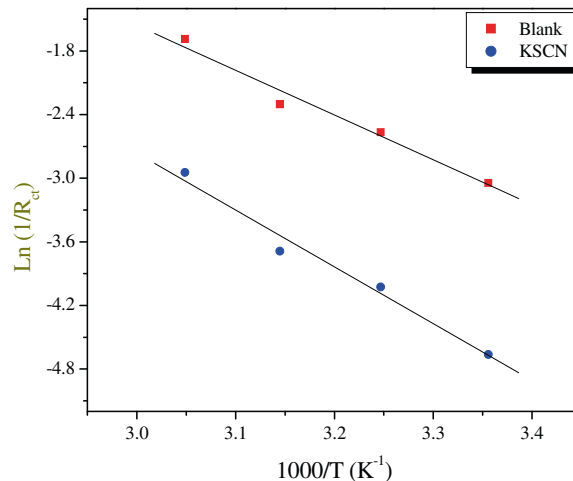

Figure 7 Arrhenius plots of C-steel in 1 M HCl with and without 10^{-2} M of KSCN.
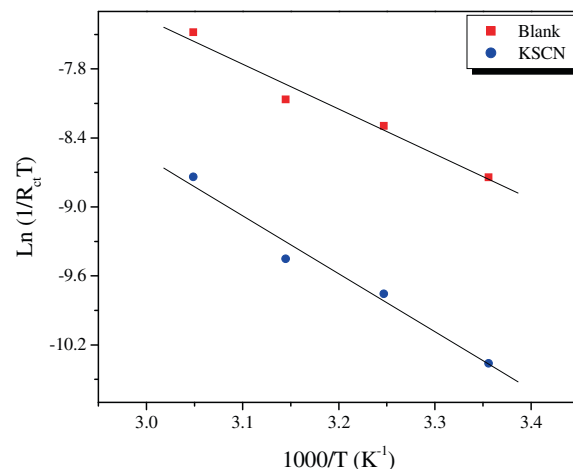

Figure 8 Arrhenius plots of C-steel in 1 M HCl with and without 10^{-2} M of KSCN.

The variations of $Ln(1/R_{ct})$ in 1 M HCl containing various concentrations of KSCN used with the reciprocal of the absolute temperature are presented in Fig. 7. Straight lines with coefficients of correlation (c. c) close to 1 are obtained.

However, the adsorption phenomenon has been successfully explained by thermodynamic parameter, to further elucidate the inhibition properties of inhibitor, the kinetic model

Table 5   The value of activation parameters E_a, ΔH_a^* and ΔS_a^* for C-steel in 1 M HCl in the absence and presence of 10^{-2} M of KSCN.

	E_a (kJ/mol)	ΔH_a^* (kJ/mol)	ΔS_a^* (J/mol K)	E_a -ΔH_a^* (kJ/mol)
Blank	35.13	32.54	−161.04	2.60
KSCN	44.54	41.94	−142.84	2.60

was another useful tool to explain the mechanism of corrosion inhibition for the inhibitor. The activation parameters for the corrosion process were calculated from the Arrhenius equation:

$$\frac{1}{R_{ct}} = \frac{RT}{Nh} \exp\left(\frac{\Delta S_a^*}{R}\right) \exp\left(\frac{\Delta H_a^*}{RT}\right) \qquad (9)$$

where h is Planck's constant, N Avogadro's number, R the universal gas constant, ΔH_a^* the enthalpy of the activation and ΔS_a^* is the entropy of activation.

Fig. 8 shows the plot of $Ln(1/R_{ct}T)$ against $1/T$. Straight lines were obtained with a slope of $(\Delta H_a^*/R)$ and an intercept of $(Ln(R/N\ A\ h) + (\Delta S_a^*/R))$ from which the values of ΔH_a^* and ΔS_a^* were calculated and are listed in Table 5.

From Table 5, it seems that E_a and ΔH_a^* varied in the same fashion. The values of E_a were higher for the inhibited solutions than those for the uninhibited solutions.

On the other hand, the positive signs of ΔH_a^* reflected the endothermic nature of the C-steel dissolution process. The value of ΔS_a^* is higher for the inhibited solution than that for the uninhibited solution. This phenomenon suggested that a decrease in randomness occurred on going from reactants to the activated complex. This might be the result of the adsorption of organic inhibitor molecules from the acidic solution which could be regarded as a quasi-substitution process between the organic compound in the aqueous phase and water molecules at electrode surface (Afia et al., 2012).

Conclusion

The studied KSCN shows excellent inhibition properties for the corrosion of C-steel in 1 M HCl at 298 K, and the inhibition efficiency increases with increasing of the KSCN concentration. The concentration dependence of the inhibition efficiency calculated from weight loss measurements and electrochemical studies were in good agreement. Based on the polarization results, the investigated KSCN acts predominantly as a mixed-type inhibitor. Adsorption of KSCN on the C-steel surface in 1 M HCl obeys the Langmuir adsorption isotherm model and leads to the formation of a protective film. The inhibition efficiency of KSCN is temperature-dependant, and inhibition efficiency decreases slightly with the increase in the temperature. The addition of KSCN leads to a decrease in activation corrosion energy.

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