Inhibition of mild steel corrosion in 3.5% NaCl medium using 1-butyl-3-methylimidazolium chloride

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Abstract

The inhibition of mild steel corrosion after its immersion in freely aerated 3.5% sodium chloride (NaCl) solutions at different temperatures (308-328K) by different concentrations of 1-butyl-3-methylimidazolium chloride (1-BMIC) was reported. The experimental tests in this work were weight loss measurements, potentiodynamic polarization, electrochemical impedance spectroscopy, scanning electron microscopy and fourier transform infrared spectroscopy. 1-BMIC efficiently inhibits the corrosion of mild steel in 3.5% NaCl medium. The inhibition efficiency of 1-BMIC increases with increasing the concentration of 1-BMIC. Adsorption of 1-BMIC on the surface of mild steel obeys Freundlich adsorption isotherm. 1-BMIC behaves as mixed type inhibitor with anodic predominance. Protective film formation against the chloride attack is confirmed by SEM and FT-IR techniques.

Keywords: *Corrosion inhibition, imidazolium chloride, mild steel, adsorption isotherm, EIS, Polarisation*

1. INTRODUCTION

Corrosion of metals is a major industrial problem that has attracted a lot of investigations in recent years [1-4]. Most metals are inherently unstable and have the natural tendency to react with their environments to obtain lower energy by forming a chemical compound in a more stable state. Steel materials which are very susceptible to attack in aggressive media are the commonly exposed metals in industrial environments [5]. Mild steel is particularly used in most structural shapes such as beams, plates, bars, and pipes used in seawater which contains chlorides that are detrimental to the steel. Heterocyclic compounds such as azole derivatives have been reported to be effective inhibitors, especially those containing nitrogen, sulfur or oxygen atoms and aromatic rings or multiple bonds in their molecular structure are well known to inhibit the corrosion of metals in corrosive media [6-14]. The effectiveness of these compounds as corrosion inhibitors mainly depends on its functional groups, steric effects, electronic density of donor atoms, and p-orbital character of donating electrons. The inhibition mechanism usually invokes their interactions with the metallic surfaces via their adsorption sites where polar functional groups are usually regarded as the reaction centers. Here, the inhibitor molecules get bonded to the metal surface by chemisorption, physisorption, or complexation with the polar groups acting as the reactive centers in the molecules [15].

The objective is to study the effects of 1- butyl-3-methylimidazolium chloride (1-BMIC) on the inhibition of mild steel corrosion in 3.5% NaCl medium. The chemical structure of 1-BMIC is shown in Fig. 1. The presence of imidazolium cation in 1-BMIC has been reported to facilitate and increase the adsorption probability of these compounds, which in turn protects the mild steel surfaces through reducing the severity of the surrounding environment.
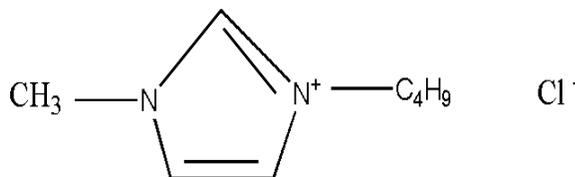

Figure 1: Chemical structure of 1-BMIC

2. Experimental

2.1. Materials preparation

Mild steel strips with the composition C, 0.13%; Mn, 0.39%; Si, 0.18%; S, 04%; P, 0.40% and Fe, reminder and size of 3x3x0.6 cm were used for weight loss and the effect of temperature studies. Mild steel cylindrical rods of the same composition embedded in araldite with exposed area of $0.5cm^2$ were polished using a sequence of emery papers of different grades and then degreased with acetone and it was used for electrochemical studies. 1-butyl-3-methylimidazolium chloride (1-BMIC) was purchased from Sigma Aldrich Company. The concentration of the 1-BMIC is ranges from 0.0002M-0.001M in 3.5% NaCl medium. The chemical structure of 1-BMIC is shown in Figure 1. The corrosive solution of 3.5% NaCl (AR grade) was prepared in distilled water and used for all studies.

2.2. Weight loss measurements

The weight loss measurements were performed at different temperature (from 308K to 328K) maintained in a thermostated water bath. The temperature maintenance in all immersion was around 24h. The solution volume was 100ml. The mild steel rods were withdrawn from the tested solution, washed thoroughly with distilled water followed by acetone and dried with air, then weighed again. Weight loss was used to calculate the corrosion rate (ρ) and inhibition efficiency (IE) as follows:

$$\rho = \frac{Wb - Wa}{St} \tag{1}$$

where W_b and W_a are the specimen weight before and after immersion in the test solution respectively, S is the surface area of the specimen and t is the end time of each experiment. The IE (%) values were being calculated from WL data by using Equation (2):

$$IE_{WL}(\%) = [\rho° - \rho/\rho°] \times 100 \tag{2}$$

where $\rho°$ is a corrosion rate without inhibitor and ρ is a corrosion rate with inhibitor.

2.3. Electrochemical measurements

Electrochemical measurements were carried out using H & CH electrochemical workstation impedance Analyzer Model CHI 604D provided with iR compensation facility, using three-electrode cell assembly. A double wall one-compartment cell with a three-electrode configuration was used. Mild steel was used as a working electrode and platinum electrode as counter electrode and calomel as reference electrodes. During the polarization study, the scan rate (v/s) was 0.005; Hold time at Ef (s) was zero and quiet time (s) was 2. AC impedance spectra were recorded in the same instrument using three-electrode cell assembly. The real part and imaginary part of the cell impedance were measured in ohms for various frequencies. The charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) values were calculated using the relation:

$$R_{ct} = (R_s + R_{ct}) - R_s \tag{3}$$
$$C_{dl} = \tfrac{1}{2}\pi R ct f_{max} \tag{4}$$

Where f_{max} = maximum frequency and R_s = Solution resistance. AC impedance was recorded with initial E(v) = 0 High frequency (Hz) = 1×105, Low frequency (Hz) = 0.1, Amplitude (v) = 0.005 and Quiet time (s) = 2. The working surface area was $0.5cm^2$, abraded with emery paper (grade 600-1200) on test face, rinsed with distilled water, degreased with acetone, and dried with a cold air steam. Before measurement the electrode was immersed in test solution at open circuit potential (OCP) for 10 min to be sufficient to attain a stable state. All electrochemical measurements were carried out at 308 K using 100ml of electrolyte (3.5% NaCl) in stationary condition. Each experiment was repeated at least three times to check the reproducibility. The inhibition efficiency is calculated by using the following formula:

$$IE\% = (i^0{}_{corr} - i_{corr} / i^0{}_{corr}) \times 100 \tag{5}$$

where $i^0{}_{corr}$ and i_{corr} are the corrosion current density values without and with inhibitor respectively.

2.4. Scanning electron microscopy

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The mild steel specimens were immersed in acid and chloride solutions in the presence and absence of inhibitor for a period of 24 h. After 24 h, the specimens were taken out and dried. The nature of the surface film formed on the surface of the mild steel specimen was analyzed by using JEOL (JSM 6390) Scanning electronic microscopy.

2.5. Fourier Transform Infrared Spectroscopy (FT-IR)

The inhibitors (Coated on KBr disc) were characterized by FT-IR spectroscopy (8400S SHIMADZU spectrometer). After immersion in 3.5% NaCl with addition of inhibitor for 24 h, the specimen was cleaned with distilled water, dried with a cold air. Then the thin adsorption layer formed on steel surface was rubbed and it was characterized by the same spectrophotometer.

3. Results and discussion

3.1. Weight loss measurements

The calculated values of corrosion rates and inhibition efficiencies for mild steel in 3.5% NaCl medium from weight loss measurements are presented in Table 1. The inhibition efficiency of 1-BMIC on mild steel corrosion in chloride medium is illustrated in Fig. 2. The plots show that inhibition efficiency increased with increasing concentration of 1-BMIC. Inhibition or retardation of corrosion is achieved by incorporating compounds that have the capability to deactivate the corrodents and reducing their rate of diffusion to the metal surface. Fig. 2 clearly shows that the inhibitive effect of 1-BMIC on mild steel corrosion in chloride medium is also concentration dependent. These results indicate that inhibitive capability of 1-BMIC is more efficient on mild steel in chloride medium.
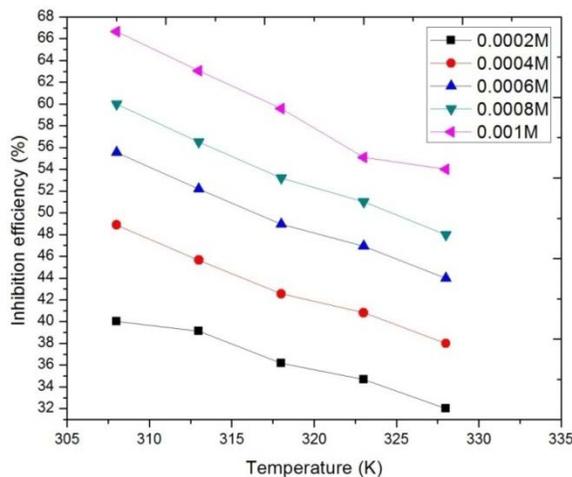

Figure 2 Arrhenius plots for mild steel in the absence and presence of different concentrations of 1-BMIC in chloride medium.

3.2. Electrochemical impedance spectroscopy

The effect of 1-BMIC on the corrosion behaviour of mild steel in 3.5% NaCl medium was studied by EIS measurements are presented in Table 2. Fig 3 shows the Nyquist plots of the impedance data as depressed capacitive semicircles. The diameter found by the intercept of each of these loops and the horizontal Z_{real} axis is related to the charge transfer resistance (R_{ct}). The value of charge transfer resistance obtained is a measure of electron transfer across the metal/solution interface and is inversely proportional to corrosion rate. Hence, a large R_{ct} value indicates strong resistance against corrosion. The inhibition efficiency is calculated from the relation shown in equation (6):

$$IE\% = (R^1_{ct} - R_{ct} / R^1_{ct}) \times 100 \tag{6}$$

where R^1_{ct} and R_{ct} are the values of the charge transfer resistance observed in the absence and presence of inhibitor molecules.

The increase in charge transfer resistance in the presence of 1-BMIC arise from the change in chemical composition of the surface film due to incorporation of 1-BMIC inhibitor molecules into the film especially through

defect sites with low ionic resistance. The incorporated molecules form high resistive compounds. Since the anodic reaction rate is determined by the diffusion of ions through the surface film it can be reasoned that the formation of resistive compounds in the film gave rise to the increase in the charge transfer resistance which is responsible for inhibition.

Table 1 Influence of temperature on the corrosion rate of mild steel in 3.5% NaCl at different concentrations of 1-BMIC and the corresponding corrosion inhibition efficiency.

Temperature(K)	Concentration (M)	Corrosion Rate($\times 10^{-6}$)	Inhibition Efficiency (%)
308	Blank	4.5	-
	0.0002	2.7	40.00
	0.0004	2.3	48.88
	0.0006	2.0	55.55
	0.0008	1.8	60.00
	0.001	1.5	66.66
313	Blank	4.6	-
	0.0002	2.8	37.5
	0.0004	2.5	45.65
	0.0006	2.2	52.17
	0.0008	2.0	56.52
	0.001	1.7	63.04
318	Blank	4.7	-
	0.0002	3.0	36.17
	0.0004	2.7	42.55
	0.0006	2.4	48.93
	0.0008	2.2	53.19
	0.001	1.9	59.57
323	Blank	4.9	-
	0.0002	3.2	34.69
	0.0004	2.9	40.84
	0.0006	2.6	46.93
	0.0008	2.4	51.02
	0.001	2.2	55.10
328	Blank	5.0	-
	0.0002	3.4	32.00
	0.0004	3.1	38.00
	0.0006	2.8	44.00
	0.0008	2.6	48.00
	0.001	2.3	54.00

The results suggest inhibition performance of 1-BMIC on corrosion of mild steel in acid and chloride medium shows that the corrosion of mild steel is mainly controlled by a charge transfer process [16]. The increase in the R_{ct} values points toward enhanced corrosion resistance due to the corrosion-inhibiting potential of 1-BMIC. The decrease in C_{dl} values, resulting from a decrease in the dielectric constant and/or increase in the interfacial thickness, can be attributed to the adsorption of 1-BMIC onto the metal/solution interface and reduces the corrosion rate of the metals in the chloride corrodent [17-18].
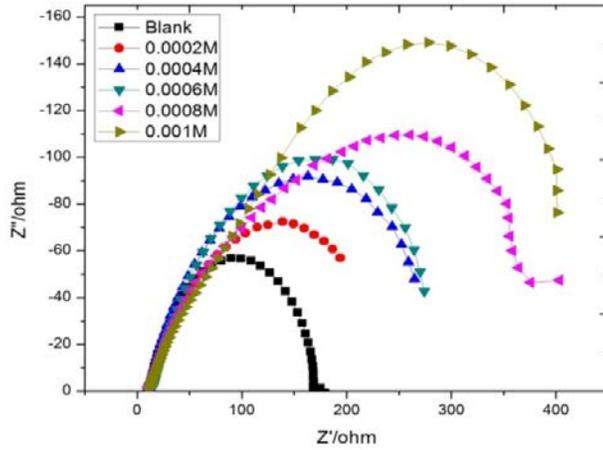
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Figure 3 Nyquist plots for mild steel immersed in 3.5% NaCl containing different concentration of 1-BMIC at 08K.

Table 2 Electrochemical impedance parameters for mild steel 3.5% NaCl in the absence and the presence of 1-BMIC.

C_{inh} (M)	R_s (Ωcm^2)	R_{ct} (Ωcm^2)	C_{dl} (F/cm^2)	%IE
Blank	11.16	159.84	2.206×10^{-5}	-
0.0002	11.27	233.53	1.269×10^{-5}	31.54
0.0004	10.94	253.26	9.305×10^{-6}	36.89
0.0006	14.60	258.80	8.403×10^{-6}	38.24
0.0008	10.86	343.34	5.690×10^{-6}	53.45
0.001	9.467	390.53	3.689×10^{-6}	59.07

3.3. Potentiodynamic Polarization Studies

The calculated values of polarization parameters in 3.5% NaCl are presented in Table 3. Fig 4 presents the anodic and cathodic polarization curves for mild steel corrosion in chloride medium in the absence and presence of 1-BMIC. The inhibition efficiencies are calculated by the following expression:

$$IE\% = (i^0_{corr} - i_{corr} / i^0_{corr}) \times 100 \tag{7}$$

where i^0_{corr} and i_{corr} are the uninhibited and inhibited corrosion current densities, respectively.
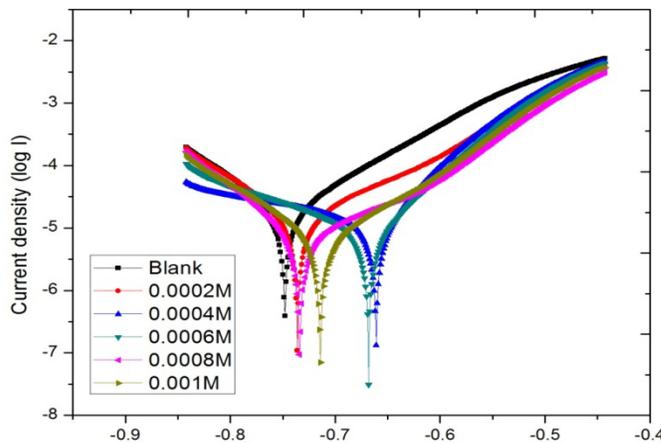

Figure 4 Tafel plots for mild steel immersed in 3.5% NaCl containing different concentration of 1-BMIC at 308K.

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Table 3 Potentiodynamic polarization parameters for the corrosion of mild steel in 3.5% NaCl in the absence and the presence of 1-BMIC.

C_{inh} (M)	E_{corr} (mV)	i_{corr} (Acm^{-2})	b_c	b_a	R_p	%IE
Blank	-0.748	2.250 x10^{-5}	10.83	9.424	954.0	-
0.0002	-0.733	1.742 x10^{-5}	10.56	7.111	1412.8	22.58
0.0004	-0.661	1.567 x10^{-5}	2.786	14.06	1648.0	30.35
0.0006	-0.668	0.943 x10^{-5}	5.310	14.65	2310.9	58.10
0.0008	-0.734	0.889 x10^{-5}	12.31	6.423	2610.0	60.48
0.001	-0.714	0.789 x10^{-5}	9.733	9.886	2809.1	64.93

The anodic and cathodic Tafel lines for mild steel in presence of 1-BMIC are almost parallel upon increasing inhibitor concentrations. This suggests that the inhibitor act by simple blocking the mild steel surface. In the other words, the inhibitor decreasing the exposed surface area for corrosion as well as it doesn't affect the mechanism of mild steel dissolution or hydrogen evolution reaction. The inhibition efficiency increases with increasing the inhibitor concentration in chloride medium as shown in Table 3. The presence of 1-BMIC shift towards the positive corrosion potential (E_{corr}), therefore, 1-BMIC can be arranged as mixed-type inhibitor with anodic predominance in 3.5% NaCl medium.

3.4. Adsorption Consideration

The adsorption of an organic on inhibitor the surface of the corroding metals observed from the impedance data may be regarded as a substitution process between the inhibitor and water molecules adsorbed on the metal surface. Addition of inhibitor molecules adsorbs on the metal surface and interaction between them can be described by adsorption isotherms [19]. In the temperature range studied, the best correlation between the experimental results and the isotherm functions was obtained using Freundlich adsorption isotherm. The adsorption isotherm for adsorption is given by the equation [20-21]:

Freundlich isotherm: $\log \theta = \log K_{ads} + n \log C$ (8)

where C is the inhibitor concentration, θ is the degree of the coverage on the metal surface and K_{ads} and n are the equilibrium constants for the adsorption-desorption process. The correlation coefficient (R^2) was used to choose the isotherm that best fit experimental data (Table 4). A plot of $\log \theta$ vs $\log C$ gave straight line in chloride medium as shown in Figure 5. It is found that all linear correlation coefficients are equal to 1 and all the values of n are same. These isotherms conform to Freundlich type in chloride medium suggesting physisorption of 1-BMIC. From the intercepts of the straight lines θ -axis, K_{ads} values were calculated and given in Table 4 and the values of K_{ads} decreases with increasing temperature suggesting that the inhibitor is physically adsorbed on the mild steel surface in both media. The equilibrium constant of adsorption, K_{ads} is related to the standard free energy of adsorption, ΔG°_{ads} , with the following equation:

$\Delta G^{\circ}_{ads} = -RT \ln(55.5 K_{ads})$ (9)

where R is the gas constant, T is the temperature and 55.5 is the molar concentration of water in solution. From Table 4, the negative value of ΔG°_{ads} ensures the spontaneity of adsorption process and stability of the adsorbed layer on the steel surface. Generally the value of ΔG°_{ads} around -20kJ/mol or lower are consistent with physisorption, while those around -40kJ/mol or higher value involve chemisorptions [21]. The values of ΔG°_{ads} for 1-BMIC are given in Table 4 and these values indicate the molecules are physisorbed. The heteroatom of the inhibitor molecule makes it adsorbed readily on the metal surface forming an insoluble stable film on the metal surface thus decreasing metal dissolution [22].

3.5. Effect of Temperature

Corrosion reactions are usually regarded as Arrhenius processes and the rate (ρ) can be expressed by the relation:

$\log \rho = \log A - E_a/2.303 RT$ (10)

where ρ is the corrosion rate determined from the weight loss measurement, E_a, the apparent activation energy, A the Arrhenius constant, R the molar gas constant and T is the absolute temperature. The regression between $\log \rho$

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and 1/T was calculated by computer, and the parameters were calculated and given in Table 5. Arrhenius plots of log ρ and 1/T for the blank and different concentrations of 1-BMIC in chloride medium are shown in Fig. 6. The slope of the line is ($-E_a$ / 2.303R) and the intercept of the line extrapolated (1/T=0) gives log A.

Table 4 Thermodynamic data for studied 1-BMIC from experimental adsorption isotherm

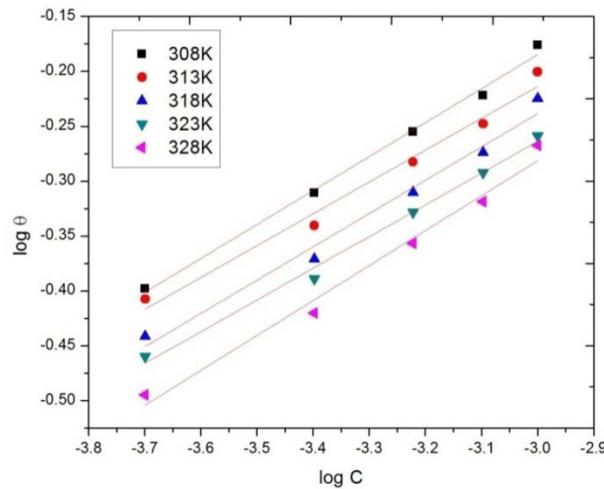| Temperature (K)	K_{ads}	ΔG_{ads} (kJ/mol)	n	R^2
308	5.5335	-13.902	0.309	0.995
313	5.4325	-14.858	0.315	0.993
318	4.6989	-14.712	0.303	0.983
323	3.9994	-13.752	0.288	0.993
328	4.7206	-15.187	0.318	0.984

Figure 5 Plot of log C versus log θ for mild steel in 3.5% NaCl containing different concentrations of inhibitor.
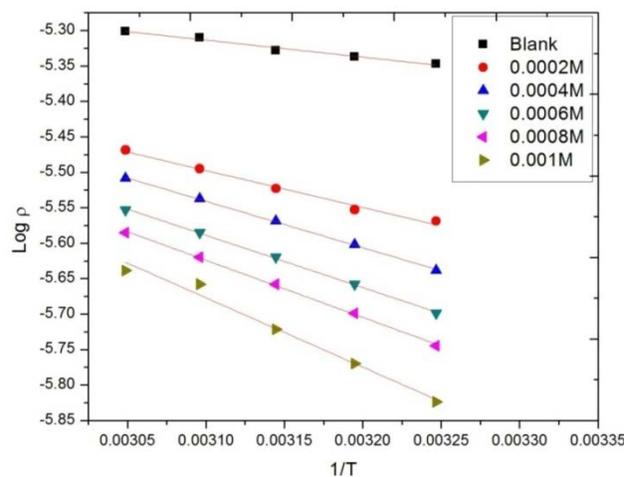

Figure 6 Arrhenius plots for mild steel in the absence and presence of different concentrations of 1-BMIC in chloride medium.

The values of E_a in the presence of inhibitor were greater than its absence clearly indicated that the corrosion reaction of mild steel was inhibited by 1-BMIC. The increase in the activation energy in the presence of inhibitor signified physical adsorption. On the other hand, the change of enthalpy (ΔH°) and entropy (ΔS°) of activation for the formation of activation complex in the transition state can be obtained from the transition state equation:

$$\log \rho/T = [(\log(\frac{R}{hN})) + (\frac{\Delta S^{*}}{2.303R})] - \frac{\Delta H^{*}}{2.303RT} \qquad (11)$$

where h is Plank's constant and N is the Avogadro's number. A plot of log ρ/T vs. 1/T gives straight line in chloride medium as shown in Figure 7. The slope is (-ΔH°/2.303R) and the intercept is [(log(R/ hN) + (ΔS° / 2.303R)], from which the values of ΔH° and ΔS° are calculated, respectively (Table 6). The data show that the thermodynamic parameters (ΔH° and ΔS°) of the dissolution reaction of steel in 3.5% NaCl medium in the presence of the 1-BMIC are higher than those of the non-inhibited solution. The lower values of ΔH° indicates less energy barrier for the reaction in the presence of 1-BMIC and the positive values of the enthalpies reflect the endothermic nature of steel dissolution process. The negative values of entropies imply that the activated complex in the rate determining steps represents the association rather than dissociation step, meaning that a decrease in disorder takes place on going from reactant to the activated complex [23-24]. The inhibition efficiency values obtained from the EIS are good agreement with the results obtained from weight loss and the potentiodynamic polarization measurements (Fig. 8).
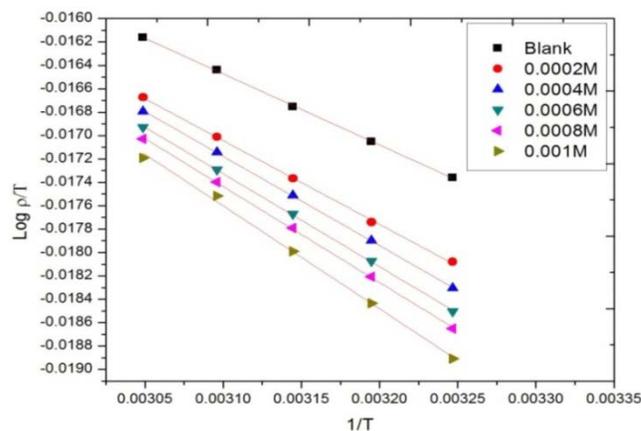

Figure 7 Transition state plots for mild steel in the absence and presence different concentrations of 1-BMIC in chloride medium.

3.6. Scanning electron microscopy

The scanning electron microscope images were recorded to establish the interaction of organic molecules with the metal surface. Fig.9a and Fig. 9b shows SEM images of polished mild steel surface, mild steel immersed in 3.5 % NaCl for 24h with and without inhibitor. The SEM images revealed that the specimens immersed in the inhibitor solutions are in better conditions having smooth surface, while the metal surface immersed in 3.5 % NaCl is rough and covered with corrosion products and appeared like full of pits and cavities. This indicated that the inhibitor molecules hindered the dissolution of iron by forming organic film on the steel surface and there by reduced the rate of corrosion. Hence the inhibitor (1-BMIC) protects mild steel in 3.5% NaCl medium.

Table 5 Activation parameters of the dissolution reaction of steel in 3.5% NaCl in the absence and presence of 1-BMIC

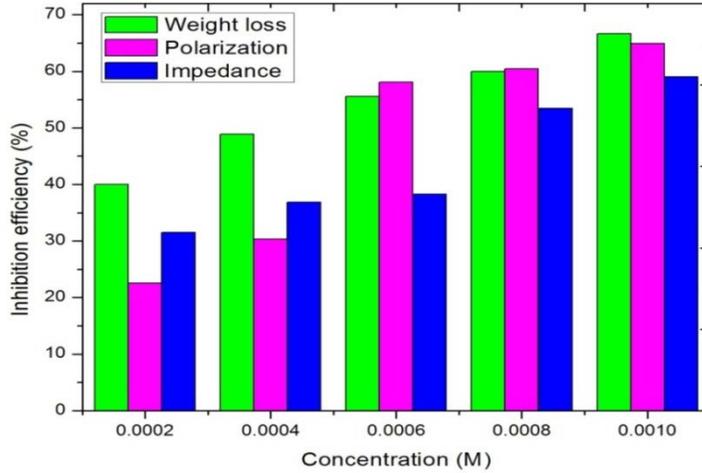| Concentration (M)	A (g cm^{-2} min^{-1})	E_a (kJ/ mol)	ΔH°(kJ/mol)	ΔS°(J/mol/K)
Blank	2.818×10^{-5}	4.6821	0.1197	-13.046
0.0002	1.445×10^{-4}	10.2356	0.1387	-13.049
0.0004	3.508×10^{-4}	12.8822	0.1544	-13.051
0.0006	5.585×10^{-4}	14.4256	0.1585	-13.052
0.0008	8.375×10^{-4}	15.7336	0.1626	-13.052
0.001	2.618×10^{-3}	19.1193	0.1783	-13.052

Figure 8 Comparison of inhibition efficiency values obtained by weight loss, polarization and impedance measurements of mild steel in 3.5% NaCl containing different concentrations of 1-BMIC at 308K.

3.7. FT-IR Studies

Several researchers [25] have confirmed that FTIR spectrometer is a powerful instrument that can be used to determine the type of bonding of organic inhibitor absorbed on the metal surface. In this study, FTIR spectrometer was used to identify whether there is an adsorption and to provide new bonding information on the steel surface after immersion in NaCl solution.
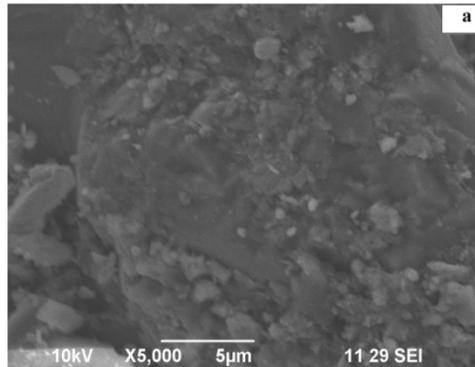

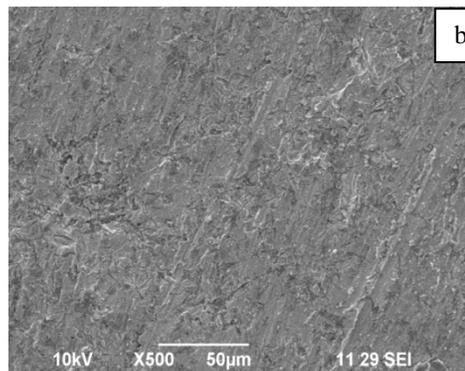**Figure 9a SEM images of mild steel in 3.5% NaCl without inhibitor**

Figure 9b SEM images of mild steel in 3.5% NaCl with inhibitor.

The FT-IR spectrum of 1-BMIC is shown in Fig.10. Original absorption at 3408.22 cm^{-1} is overlapped by the strong stretching mode of N-H. The sharp peak at 1633.71 cm^{-1} is attributed to C=N stretching vibrations The band at 1456.26 cm^{-1} is due to the stretching mode of N-heteroaromatic imidazole ring. The peak at 1168.86 cm^{-1} corresponds to the C-N stretching vibrations. The FT-IR spectrum of adsorbed protective layer formed on mild steel surface after immersion in 3.5% NaCl medium of 1-BMIC are shown in Figure 11. On comparing Figures.10 & 11, all important peaks present in pure compound appear in adsorption layer on the steel surface, which means that most of the functional groups in 1-BMIC are present in the adsorbed surface film. Moreover, some of the peaks for the adsorbed film have diminished or even vanished. The band at 3408.22 cm^{-1} is shifted to 3344.57 cm^{-1} in chloride medium.
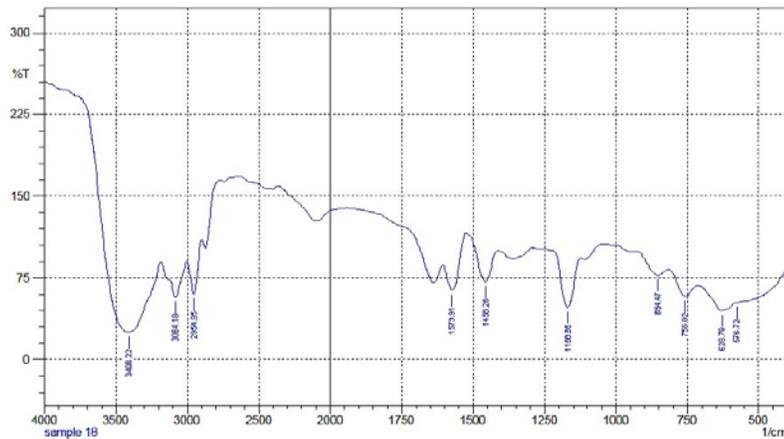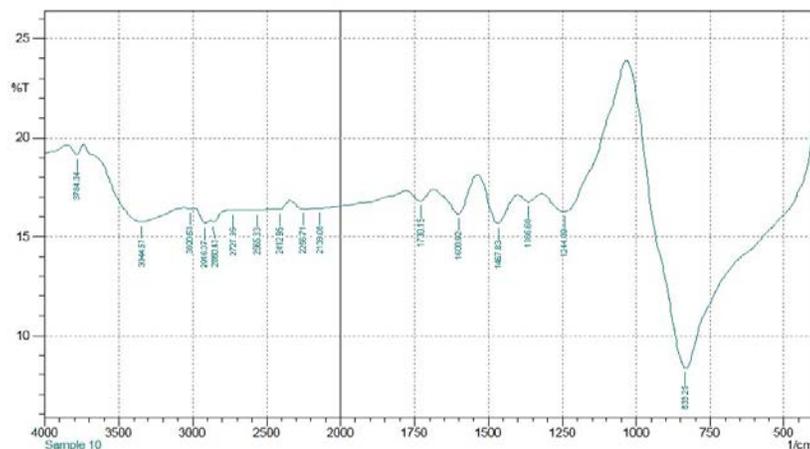

Figure 10 FT-IR spectrum of 1-BMIC

Figure 11 FT-IR spectrum of adsorbed layer formed on the mild steel after immersion in 3.5%NaCl containing inhibitor.

Table 6 Frequencies and assignment of adsorption of FT-IR by 1-BMIC and chloride containing 1-BMIC

1-BMIC (frequency in cm^{-1})	NaCl containing 1-BMIC (frequency in cm^{-1})	Assignment
3408.22	3344.57	N-H stretch
1633.71	1600.92	C=N stretch
1456.26	1467.83	N-heteroaromatic imidazole ring
1168.86	1244.09	C-N stretch

These observations indicate that the 1-BMIC has coordinated with Fe^{2+} through nitrogen atom of the 1-BMIC and also through the π electrons of imidazole ring resulting in the formation of Fe^{2+}- 1-BMIC complex formed on the metal surface. The frequencies and assignment of FTIR by 1-BMIC, chloride containing 1-BMIC were shown in Table 6.

4. Conclusion

The main conclusions drawn from this study are:
1-BMIC efficiently inhibits the corrosion of mild steel in 3.5% NaCl medium. 1-BMIC behaves as mixed type inhibitor with anodic predominance in sodium chloride medium. Adsorption of 1-BMIC on the surface of mild steel obeys Freundlich adsorption isotherm. Reduction in the values of I$_{corr}$ and C$_{dl}$ in the presence of an inhibitor has been dealt. The inhibition efficiency of 1-BMIC increases with increasing the concentration. On increasing the temperature, the corrosion rate increases. The inhibition efficiency values obtained from the EIS are good agreement with the results obtained from weight loss and the potentiodynamic polarization measurements. Protective film formation against the chloride attack is confirmed by SEM and FT-IR techniques.

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