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Electrochemical Studies of Mild Steel Corrosion Inhibition By 1-Butyl-3-Methylimidazolium Chloride In 2M H_2SO_4 medium

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ABSTRACT

The influence of 1-butyl-3-methylimidazolium chloride (1-BMIC) on corrosion inhibition of mild steel in 2M H_2SO_4 was studied by weight loss, effect of temperature, potentiodynamic polarization and electrochemical impedance spectroscopy. The experimental results showed that the inhibition efficiency increases with increasing of 1-BMIC concentrations but decreases with increasing temperatures. The adsorption of 1-BMIC on the mild steel surface obeyed the Freunlich adsorption isotherm. Potentiodynamic polarization curves showed that 1-BMIC acted as mixed type inhibitor in acid medium. This was supported by the impedance which showed a change in the charge transfer resistance and double layer capacitance indicating adsorption of 1-BMIC on the mild steel surface. Scanning electron microscopy (SEM) technique is used to confirm the effectiveness of inhibition of mild steel in sulphuric acid medium. The effects of nitrogen atom in 1-BMIC on the ability to act as a corrosion inhibitor were investigated by theoretical calculations. The thermodynamic functions of the adsorption processes were calculated from the weight loss and the effect of temperature data.

Keywords: Inhibitor, Mild steel, Corrosion, Polarization, Impedance, Adsorption.

INTRODUCTION

The corrosion of metals remains a world-wide scientific problem as it affects the metallurgical, chemical and oil-industries. The increasing interest in the manufacture of sulphuric acid has created the need for obtaining information on the corrosion resistance of mild steel to sulphuric acid attack [1]. Of particular importance also is the need to introduce certain organic compounds as inhibitors into the mild steel-corrodent system to prevent corrosion of the mild steel [2-3]. Most of the well known acid inhibitors are organic compounds containing nitrogen, oxygen and sulphur atoms [4-6]. It has been reported that many heterocyclic compounds containing N, O and S have been proved to be effective inhibitors for the corrosion of mild steel in acid media [6-15. Heterocyclic compounds especially imidazolium compounds are expected to be good corrosion inhibitors because of their environmentally friendly characteristics in addition to their unique properties [16-18], little investigations have been found in the literature [19-20]. Zhang et al [21] have investigated the behavior of alkyl imidazolium ionic liquids for steel in acidic medium. It was found that imidazolium liquids exhibited excellent inhibition performance for mild steel in acidic solution. Imidazolium compounds are reported to show corrosion resistant behavior of copper [22], steel [23-24] and aluminum [25]. It is found that the action of such inhibitors depends on the specific interaction between functional group and the metal surface, due to the presence of the –C=N- group and the electronegative nitrogen in the molecule. The adsorption of organic molecules at the metal/surface is of great interest in surface science and can markedly change the corrosion resisting properties of metals. The protection of corroding surfaces prevents the waste of both resources and money during the industrial applications and it is vital for the extension of the lifetime of the equipment and limiting the dissolution of metals from the components into the environment. Therefore, the prevention of corrosion of metals used in industrial applications is an important issue that must be dealt with. The corrosion inhibition efficiency of organic molecules is mainly dependent on their ability to be adsorbed on the metal surface, which results with the replacement of water molecules at the corroding interface [26].

The main objective of this work is to investigate the corrosion inhibiting capability of 1-butyl-3-methylimidazolium chloride (1-BMIC) against corrosion of mild steel in sulphuric acid solution by various methods.

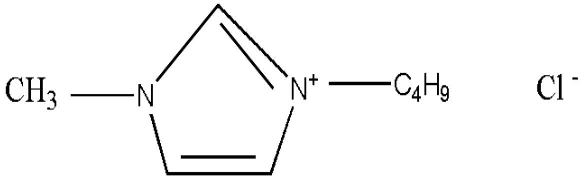

Figure 1: Chemical structure of 1-BMIC

MATERIALS AND METHODS

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 0.002M-0.01M in 2M H_2SO_4 medium. The chemical structure of 1-BMIC is shown in Figure 1. The corrosive solution of 2M H_2SO_4 (AR grade) was prepared in distilled water and used for all studies.

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 50ml. 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:

$$\rho = \frac{Wb - Wa}{St} \qquad (1)$$

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):

$$IE_{WL}(\%) = [\rho^\circ - \rho/\rho^\circ] \times 100 \qquad (2)$$

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

Electrochemical measurements: Electrochemical measurements were carried out using H and 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:

$$R_{ct} = (R_s + R_{ct}) - R_s \qquad (3)$$

$$C_{dl} = ½\pi Rctf_{max} \qquad (4)$$

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 50ml of electrolyte (2M H_2SO_4) 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:

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

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

Scanning electron microscopy: 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.

Quantum chemical calculations:

Quantum chemical calculations were carried out using density functional theory (DFT) with basis set for all atoms with Gaussian 09W program. Some electronic properties such as energy of the highest occupied molecular orbital (E_{HOMO}), energy of the lowest unoccupied molecular orbital (E_{LUMO}), energy gap (ΔE) between LUMO and HOMO on the backbone atoms for 1-BMIC was determined.

RESULTS AND DISCUSSION

Weight loss measurements: Table 1 gives the values of inhibition efficiency for different concentrations of 1-BMIC in 2M H_2SO_4. It can be seen from this table that 1-BMIC efficiently inhibits the corrosion of mild steel in 2M H_2SO_4 solutions. The corrosion rate decreased considerably with an increase in concentration of the inhibitor. This is due to the presence of heteroatom and the extent of inhibition depends upon the nature and mode of adsorption of inhibitor on the metal surface. Weight loss measurements revealed that corrosion inhibition efficiency of the 1-BMIC increases with increasing the concentration.

Table 1 Influence of temperature on the corrosion rate of mild steel in 2M H_2SO_4 at different concentrations of 1-BMIC and the corresponding corrosion inhibition efficiency.

Temperature(K)	Concentration (M)	Corrosion Rate($x10^{-5}$)	Inhibition Efficiency (%)
308	Blank	9.8	-
	0.002	8.66	11.6
	0.004	6.52	33.45
	0.006	5.09	47.99
	0.008	3.81	61.13
	0.01	2.06	78.97
313	Blank	9.86	-
	0.002	8.73	11.46
	0.004	6.64	32.7
	0.006	5.3	46.29
	0.008	3.86	60.86
	0.01	2.51	74.55
318	Blank	9.9	-
	0.002	8.93	9.85
	0.004	6.8	31.35
	0.006	5.53	44.19
	0.008	4.07	58.86
	0.01	2.72	72.58
323	Blank	9.94	-
	0.002	9.09	8.526
	0.004	7.02	29.37
	0.006	5.71	42.51
	0.008	4.26	57.15
	0.01	2.92	70.61
328	Blank	10.07	-
	0.002	9.27	7.865
	0.004	7.22	28.32
	0.006	5.91	41.32
	0.008	4.46	55.71
	0.01	3.13	68.89

Effect of Temperature: In optic to get more information about the performance of 1-BMIC and the nature of adsorption isotherm and thereafter to evaluate the adsorption and activation processes, the influence of temperature is studied. For this purpose, weight loss measurements are determined in the range of temperature 308K-328K, in the absence and presence of inhibitor at various concentrations during 24h of immersion. The corresponding data are shown in table 1. We remark that the rise in temperature leads to an increase in corrosive rate with and without inhibitor. The variation of inhibition efficiency with temperature for mild steel in 2M H_2SO_4 with different concentrations of 1-BMIC is shown in figure 2.

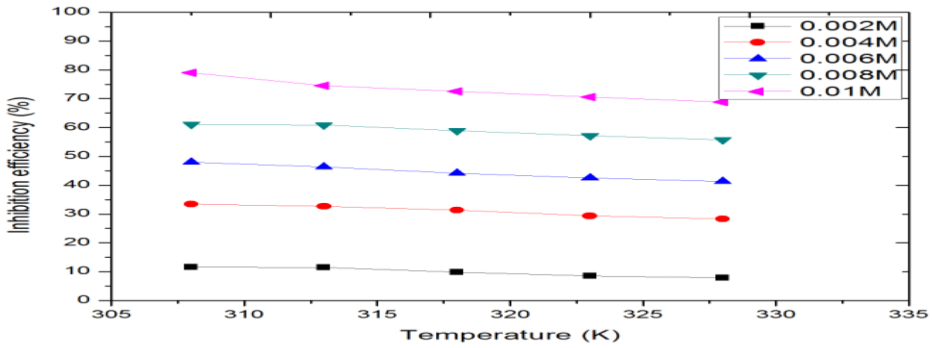

Figure 2: Effect of temperature on the inhibition efficiency of 1-BMIC at different concentrations.

To calculate activation thermodynamic parameters of the corrosion reaction such as apparent activation energy (E_a), the frequency factor (A), the entropy (ΔS°) and the enthalpy of activation (ΔH°), Arrhenius suggested the famous equation which evaluates the temperature dependence of the rate constant as follows [27]:

$$\log \rho = \log A - E_a / 2.303RT \qquad (6)$$

Here, A is the frequency factor and E_a is the apparent activation energy, R is the gas constant (R=8.314 J/mol/K) and T is absolute temperature. Eq. (6) predicts that a plot of log ρ vs. 1/T should be a straight line as shown in figure 3. The slope of the line is ($-E_a$ / 2.303R) and the intercept of the line extrapolated (1/T=0) gives log A.

Table 2 Activation parameters of the dissolution reaction of steel in 2M H_2SO_4in 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	1.51x10^{-4}	1.108	0.0871	-13.045
0.002	4.97x10^{-4}	5.947	0.0991	-13.046
0.004	1.11x10^{-4}	8.612	0.1189	-13.049
0.006	2.72x10^{-3}	11.145	0.1189	-13.049
0.008	4.37x10^{-3}	12.599	0.1267	-13.050
0.01	2.42x10^{-3}	18.158	0.1506	-13.054

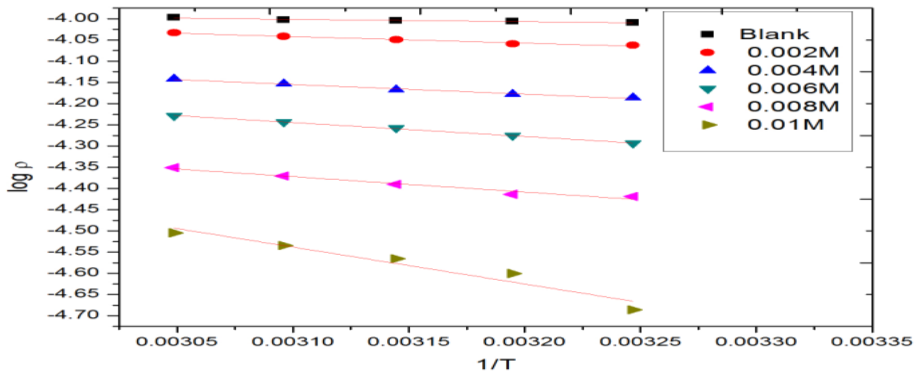

Figure 3: Arrhenius plots for log ρ vs 1/T for mild steel in 2M H_2SO_4 at different concentrations of 1-BMIC.

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 [28-29]. On the other hand, the change of enthalpy ($\Delta H^{\circ)}$ and entropy (ΔS°) of activation for the formation of activation complex in the transition state can be obtained from the transition state equation [27]:

$$\log \rho/T = [(\log(R/hN)) + ((\Delta S^{\circ})/2.303R)] - (\Delta H^{\circ})/2.303RT \qquad (7)$$

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 as shown in Figure 4. The slope is ($-\Delta H^{\circ}/2.303R$) and the intercept is [($\log(R/hN) + (\Delta S^{\circ}/2.303R)$)], from which the values of ΔH° and ΔS° are calculated, respectively (Table 2). The data show that the thermodynamic parameters (ΔH° and ΔS°) of the dissolution reaction of steel in 2M H_2SO_4 in the presence of the 1-BMIC is 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. Similar observation has been documented [30]. 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 [31].

Adsorption isotherm: Adsorption plays a very important role in the inhibition of metallic corrosion by organic molecules. Many investigators have used the adsorption isotherms to study inhibitor characteristics assuming that the inhibitors adsorbed on the metal surface decrease the surface area available for electrode reactions to take place [32-33]. The values of surface coverage related to different concentrations of the inhibitor 1-BMIC in 2M H_2SO_4 obtained from weight loss measurements in the temperature range from 308K-328K have been used to explain the best isotherm to determine the adsorption process.

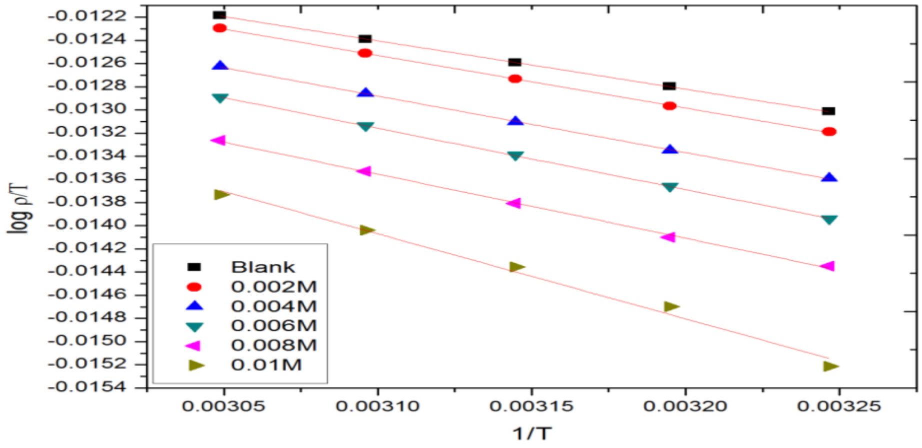

Figure 4: The relation between log ρ/T vs 1/T for mild steel at different concentrations of 1-BMIC.

The adsorption of organic corrosion inhibitor onto the metal-solution interface is a substantial adsorption process between organic molecules and the metallic surface. In the temperature range studied, the best correlation between the experimental results and the isotherm functions was obtained using Freunlich adsorption isotherm.

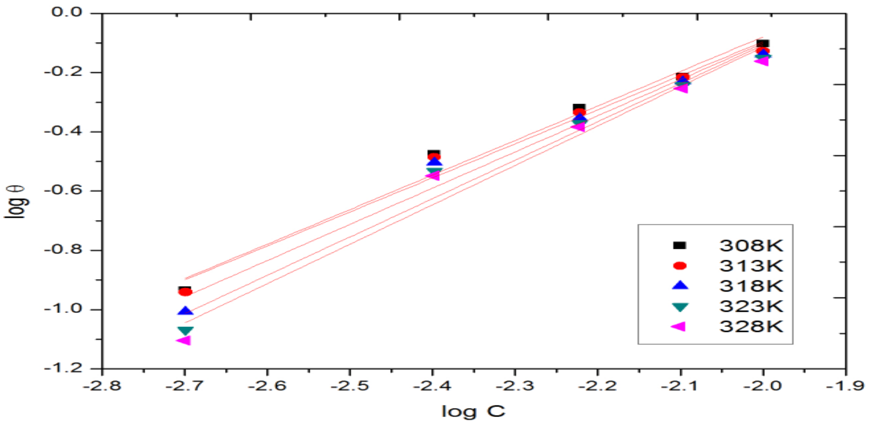

Figure 5: Fitting Freunlich adsorption isotherm model for 1-BMIC in 2M H_2SO_4 at different temperatures.

The Freunlich isotherm for adsorption is given by the equation:

$$\theta = K_{ads} C^n \qquad (8)$$

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 3). A plot of log θ vs log C gave Straight line 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, suggesting physisorption of 1-BMIC. From the intercepts of the straight lines θ-axis, K_{ads} values were calculated and given in table 3 and the values of K_{ads} decreases with increasing temperature suggesting that the inhibitor is physically adsorbed on the mild steel surface. Similar observation has been documented [34].

Table 3 Thermodynamic data for studied 1-BMIC from experimental adsorption isotherm

Temperature (K)	**K_{ads}**	**ΔG_{ads} (kJ/mol)**	**n**	**R^2**
308	2.582	-12.72	0.268	0.953
313	2.239	-12.35	0.245	0.916
318	2.079	-12.16	0.237	0.899
323	2.046	-12.12	0.243	0.899
328	1.972	-12.03	0.243	0.905

The equilibrium constant of adsorption, K_{ads} is related to the standard free energy of adsorption, ΔG°_{ads} , with the following equation:

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

where R is the gas constant, T is the temperature and 55.5 is the molar concentration of water in solution. From Table 3, 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 [35-41]. The values of ΔG°_{ads} for 1-BMIC are given in Table 3 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 [42-43].

Potentiodynamic polarization studies: The potentiodynamic polarization curves of the mild steel in 2M H_2SO_4 solution with various concentrations of 1-BMIC are presented in figure 6. The electrochemical parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), and Tafel slope (b_c and b_a) derived from polarization curves are listed in Table 4. Inspection of tables reveals that the addition of 1-BMIC shifted the E_{corr} to less negative values and no definite trend was observed in the shift of E_{corr} values in the presence of various concentrations of inhibitor. The values of I_{corr} of mild steel in the inhibited solution were smaller than those for the inhibitor free solution. The decrease of corrosion current may be explained by the action of inhibitor on both anodic and cathodic reactions [44].

Table 4 Potentiodynamic polarization parameters for the corrosion of mild steel in 2M H_2SO_4 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	-0.525	$4.693x10^{-3}$	5.413	7.467	7.2	-
0.0002	-0.527	$3.727x10^{-3}$	5.594	8.086	8.5	20.58
0.0004	-0.523	$2.746x10^{-3}$	5.491	8.783	11.1	41.49
0.0006	-0.518	$2.529x10^{-3}$	5.456	8.879	12.0	46.11
0.0008	-0.533	$2.181x10^{-3}$	5.636	8.732	13.9	53.53
0.001	-0.529	$1.229x10^{-3}$	5.762	9.749	22.8	73.81

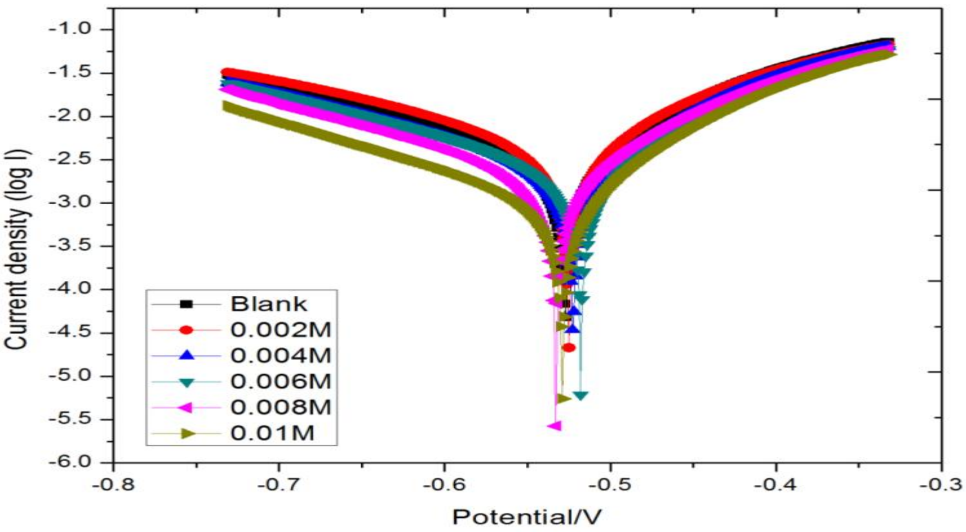

Figure 6: Tafel plots for mild steel immersed in 2M H_2SO_4 containing different concentration of 1-BMIC at 308K.

In the case of 1-BMIC in 2M H_2SO_4 medium, the anodic and cathodic Tafel slope value was slightly affected and indicates that the 1-BMIC behaves as a mixed type inhibitor. The inhibitor molecules are first adsorbed onto the mild steel surface and therefore, impeded by merely blocking the reaction sites of the mild steel surface. In this way, the surface area available for H^+ ions is decreased while the actual reaction mechanism remains unaffected [45]. A higher coverage of the inhibitor on the surface was obtained in solutions with higher inhibitor concentrations. The presence of defects on the metal surface permits a free access of H^+ ions to the mild steel surface [46-47].

Electrochemical impedance studies: Electrochemical impedance spectroscopy provides a rapid and convenient way to evaluate the performance of the organic-coated metals and has been widely used for investigation of protective properties of organic inhibitors on metals. It does not disturb the double layer at the metal/solution interface. The corrosion kinetic parameters such as solution resistance (R_s), charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) have been derived from Nyquist plot and percentages of inhibition efficiency are given in table 5. The semicircle in the Nyquist plot (Figure 7) indicates that the corrosion of inhibitors is mainly controlled by a charge transfer process [48].

Table 5 Electrochemical impedance parameters for mild steel in 2M H_2SO_4 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	1.581	2.844	$4.200x10^{-2}$	-
0.0002	1.661	3.072	$3.767x10^{-2}$	7.422
0.0004	1.563	3.824	$2.381x10^{-2}$	25.63
0.0006	1.661	4.870	$1.539x10^{-2}$	41.60
0.0008	1.661	6.406	$0.918x10^{-2}$	55.60
0.001	1.749	17.12	$0.133x10^{-2}$	83.39

The presence of inhibitors enhances the values of charge transfer resistance. This indicates that 1-BMIC do not alter the electrochemical reaction responsible for corrosion but inhibit corrosion primarily through their adsorption on to the metal surface. The decrease in double layer capacitance values could be attributed to the adsorption of the inhibitor molecule at the metal surface [49].

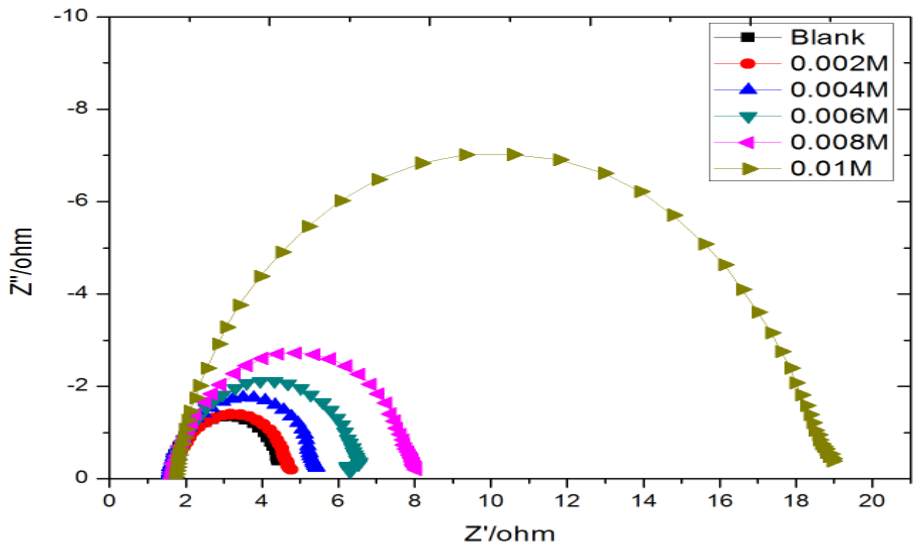

Figure 7: Nyquist plots for mild steel immersed in 2M H_2SO_4 containing different concentration of 1-BMIC at 308K.

The variation of inhibition efficiency, determined by the three methods (weight loss, polarization curves and impedance methods), as a function of concentration of 1-BMIC in 2M H_2SO_4 as shown in figure 8. The results thus obtained show a good agreement with the three methods used in this investigation, significantly in high concentrations.

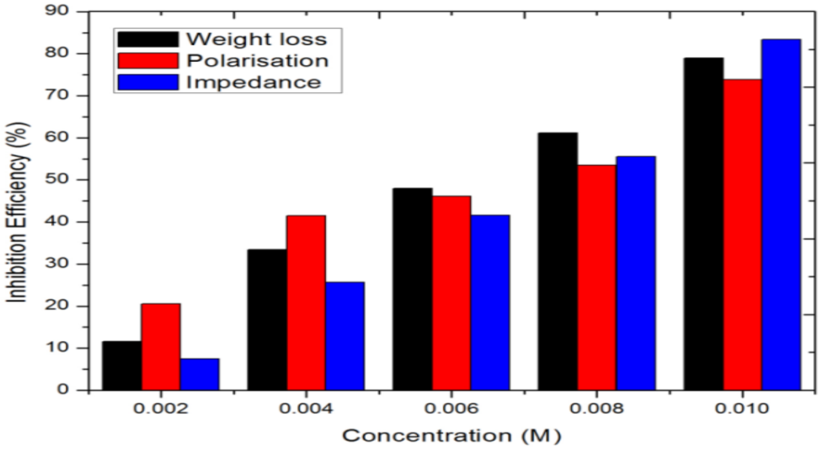

Figure 8: Comparison of inhibition efficiency values obtained by weight loss, polarization and impedance measurements of mild steel in 2M H_2SO_4 containing different concentration of 1-BMIC at 308K.

Scanning electron microscopy (SEM) : The SEM micrographs of the corroded mild steel in 2M H_2SO_4 in the absence and presence of inhibitor are shown in Fig 9 (a&b). Figure 9a of bare mild steel electrode surface, defects and notches were observed, while a layer of closely packed film was 9 (a&b) that corrosion does not occur in presence of inhibitor (1-BMIC) and hence corrosion was present in the solution, then scanning electron microscopy (SEM) observation, confirmed the existence obtained in Figure 9b, and the surface was free from pits and it was smooth. It can be concluded from of an absorbed protective film on the metal surface.

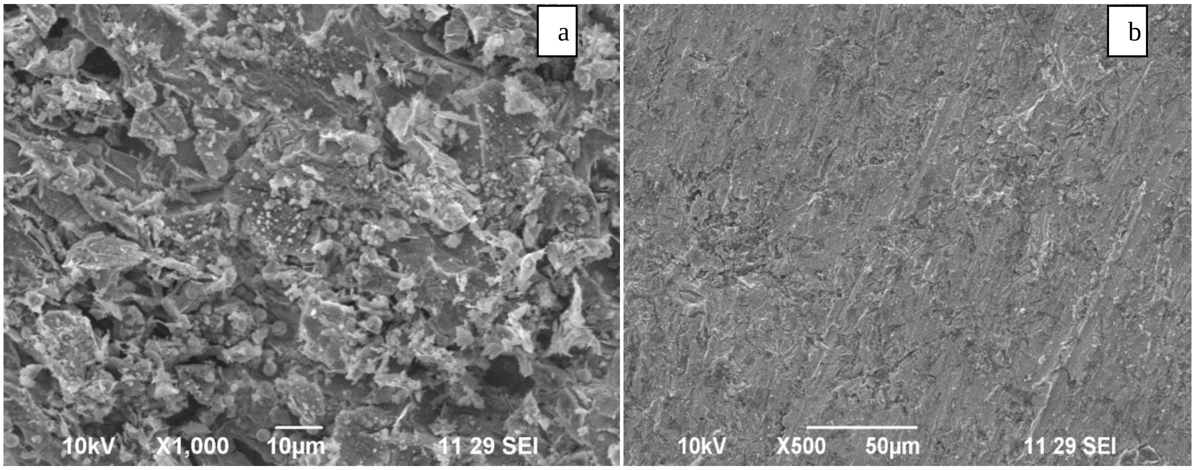

Figure 9(a&b): SEM images of mild steel in 2M H_2SO_4 a) without inhibitor b) with inhibitor.

Quantum chemical studies: It was shown from experimental results that it is possible to get better performance with 1-BMIC as corrosion inhibitor. The effect of the presence of N atom in 1-BMIC on the ability to act as corrosion inhibitor was investigated by quantum chemical calculations. The electronic properties such as energy of the highest occupied molecular orbital (E_{HOMO}), energy of the lowest unoccupied molecular orbital (E_{LUMO}), energy gap (E) between HOMO and LUMO on the backbone atoms were determined by optimization. The optimized molecular structures are given in fig.10 and 11(a, b) and the electronic properties in table 6. The E_{HOMO} is often associated with the electron donating ability of a molecule. Therefore, increasing values of E_{HOMO} indicate higher tendency for the donation of electrons to the appropriate acceptor molecule with low energy and empty molecular orbital. The increasing values of facilitate the adsorption of the inhibitor. The negative sign of the E_{HOMO} value obtained and other thermodynamic parameters indicates that the data obtained supports physical adsorption mechanism. The energy gap, $\Delta E = E_{LUMO} - E_{HOMO}$ as well as Dipole moment, μ favour 1-BMIC implying its effectiveness as a corrosion inhibitor.

Table 6 Quantum chemical parameters for 1-BMIC

Quantum chemical parameters	Value
E_{HOMO} (eV)	-4.7144
E_{LUMO} (eV)	-1.9007
ΔE (E_{HOMO}-E_{LUMO}) (eV)	2.8137
Dipole moment (μ) (D)	12.4980
Ionization Potential (eV)	-4.7144
Electron affinity (eV)	-1.9007
Molecular weight (amu)	174.67
Global hardness (ΔN) (eV)	1.4068

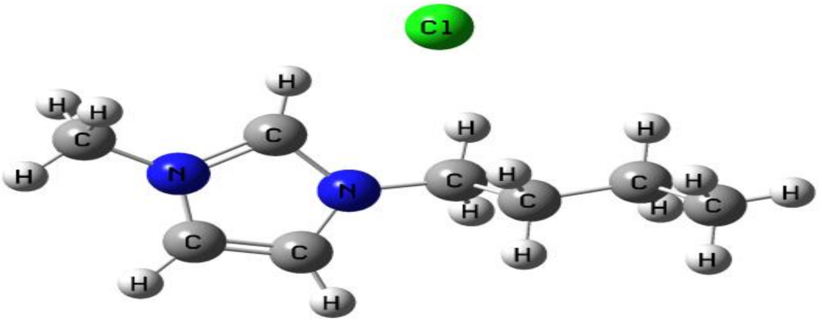

Fig. 10: Optimized molecular structure of 1-BMIC

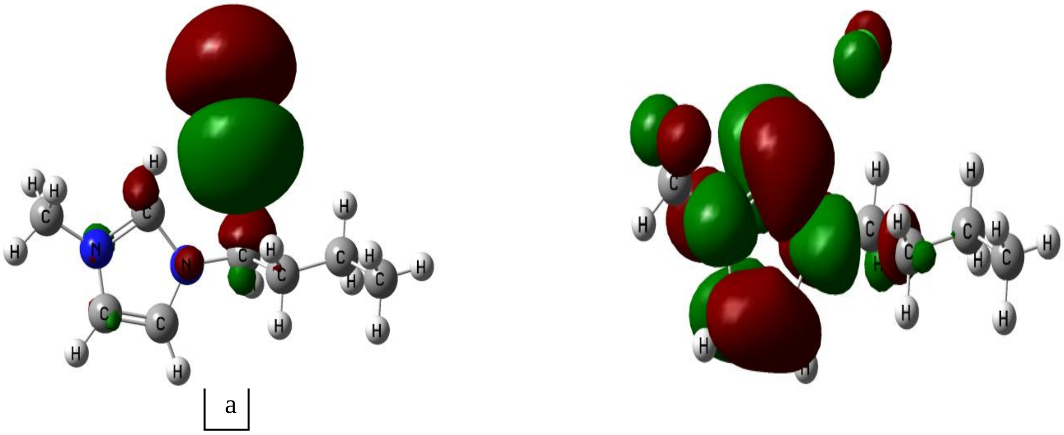

Figure 11: a) HOMO b) LUMO surfaces for 1-BMIC molecule.

APPLICATIONS

In this study, environmentally friendly character of 1-BMIC is useful to protect mild steel from corrosion and this is applicable as an acid corrosion inhibitor in the industrial field as components in acid descaling, oil well acidizing, acid pickling, acid cleaning, etc.

CONCLUSIONS

The main conclusions drawn from this study are 1-BMIC efficiently inhibits the corrosion of mild steel in 2M H_2SO_4 medium. 1-BMIC behaves as mixed type inhibitor. Adsorption of 1-BMIC on the surface of mild steel obeys Freunlich 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 acid attack is confirmed by SEM. The effects of nitrogen atom in 1-BMIC on the ability to act as a corrosion inhibitor were investigated by theoretical calculations.

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