

# Inhibition of mild steel corrosion in 1 M hydrochloric acid by benzimidazolium bromide derivatives

Nada Abdulwali <sup>a\*</sup>, Hamid Ennaji <sup>b</sup>, Rassam Al Subari <sup>d</sup>,  
Abdallah Guenbour <sup>a</sup>, Abdelkbir Bellaouchou <sup>a</sup>, José Garcia Anton <sup>c</sup>,  
El Mokhtar Essassi <sup>b</sup>

<sup>a</sup>*Laboratoire de corrosion-électrochimie, Université Mohamed V Agdal. Faculté des Sciences. Av. Ibn Battouta. BP 1014, Rabat. Maroc.*

<sup>b</sup>*Laboratoire de Chimie Organique Hétérocyclique. Université Mohamed V Agdal. Faculté des Sciences. Av. Ibn Battouta. BP 1014, Rabat. Maroc.*

<sup>c</sup>*Departamento de Ingenieria Quimica y Nuclear, E.E.S.I. Industriales, Universidad Politecnica de Valencia, P.O. Box 221012, E-46071 Valencia, Spain.*

<sup>d</sup>*University Mohammed V- Souissi, Faculty of Dentistry, Rabat, Morocco.*

*Correspondent author: nada\_alwajih@hotmail.fr*

## Abstract

Corrosion inhibition by some benzimidazolium bromide derivatives on mild steel in 1 M hydrochloric acid solutions at 298 K has been investigated by Potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and scanning electronic microscope analysis. These techniques were applied to study the metal corrosion behavior in the absence and presence of different concentrations of these inhibitors under the influence of various experimental conditions. The studies have shown that 1,3-Dioctylbenzimidazolium bromide was the most efficient inhibitor reaching values of inhibition efficiency (IE%) more than 96% at a concentration of  $10^{-3}$  M. The results indicate that these compounds act as mixed type inhibitors retarding the anodic and cathodic corrosion reactions. The results obtained from polarization and electrochemical impedance spectroscopy (EIS) are in good agreement. The thermodynamic parameters for adsorption process were calculated.

**Keywords:** benzimidazolium bromide derivatives; Corrosion; Inhibition; Mild Steel; Hydrochloric acid

## 1. Introduction

Acid solutions are widely used in industry, Some of important fields of application being acid pickling, industrial cleaning, acid descaling, oil-well acidizing and petrochemical processes [1–3]. The use of inhibitors is the most practical methods for protecting metals against corrosion and chemically efficient corrosion inhibitors [4–8]. During the past decade, the inhibition of mild steel corrosion in acid solutions by various types of organic inhibitors has attracted much attention [9–11], many researchers report that the inhibiting effect mainly depends on some physicochemical and electronic properties of the organic compound molecule which related to its functional groups, steric effects, electronic density of donor atoms, and  $\pi$  orbital character of donating electrons, and so on [12,13]. It has been shown that organic compounds contain hetero atoms with high electronic density such as nitrogen, sulphur, and oxygen, or those containing multiple bonds which are considered as adsorption centers, are effective as corrosion inhibitors [14–21]. Examples are benzimidazol-type compounds [22–24], containing several heterocyclic structures, which have excellent corrosion properties for the corrosion of many metals in various aggressive media [25,26]. However, the disadvantages of some inhibitors are their toxicity. In line with environmental protection requirement, the use of organic inhibitors is nowadays quite limited, so toxic inhibitors that are widely used in industrial processes should be replaced with new environment friendly inhibitors [27]. The Non-toxic benzimidazole and its derivatives have been proved excellent inhibitors for mild steel [28–30]. The objective of the present work was to study the inhibition effect of the synthesized non-toxic benzimidazole derivatives on the corrosion of mild steel in hydrochloric acid solutions. In order to evaluate compounds as corrosion inhibitors and to design novel inhibitors, the inhibition performance was evaluated by potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and scanning electronic microscopy (SEM).

## 2. Experimental method.

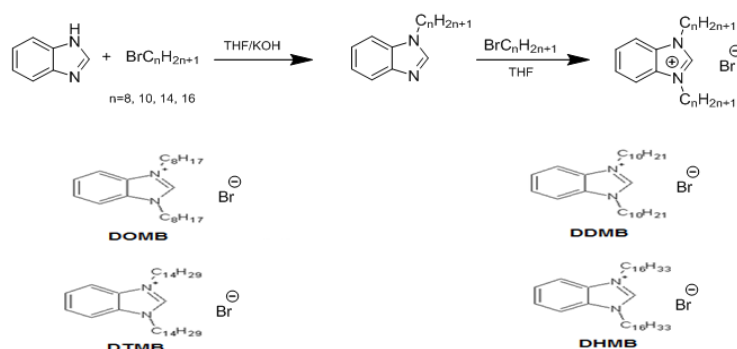
### 2.1. Materials

The mild steel were of the following composition (wt); C (0.2%), Mn (0.54%), P (0.16%), S(0.24%), Cr (0.37%), Si (0.53%) and the remainder is Fe. The mild steel specimens were mechanically cut in to 1 cm<sup>2</sup> coupons used in the electrochemical studies. Prior to all measurements, the samples were polished with emery paper from 400 to 1200 grades. They were cleaned with acetone, washed with double-distilled water, and finally dried at room temperature before being immersed in the acid solution.

### 2.2. Inhibitors

Four benzimidazolium bromide derivatives, namely 1,3-Dioctylbenzimidazolium bromide (DOMB); 1,3-didecylbenzimidazolium bromide (DDMB); 1,3-Ditetradecylbenzimidazolium bromide (DTMB) and 1,3-Dihexadecylbenzimidazolium bromide (DHMB), respectively, Were synthesized in laboratory, The 1,3-dialkyl-Benzimidazolium bromide were synthesized as follows : In a three-neck round bottom flask equipped with condenser, 5 g (42.4 mmol) of 1H-benzimidazole, was dissolved in 100 mL THF, 3.18 g (63.6 mmol) of potassium hydroxide and 50.8 mmol of 1-bromoalkan were

added. The mixture was refluxed for 5 h, then, after cooling and filtration the solvent was removed under reduced pressure. The crude crystals were crystalized from ethanol. In the second step, 20 mmol of 1-alkanbenzimidazole was dissolved in 100 mL THF and 25 mmol of 1-bromoalkan was added. After 3h of heating the solvent was removed, the solid product was recrystallized from ethanol. The synthesis and structures of benzimidazolium bromide derivatives is shown as follows.



### 2.3. Solutions

The aggressive solutions of 1 M HCl were prepared by dilution of analytical grade 37% HCl with doubly distilled water. The concentration range of the inhibitors employed was  $1 \times 10^{-5}$  to  $1 \times 10^{-3}$  M, and the solution in the absence of inhibitors was taken as blank for comparison.

### 2.4. Electrochemical measurements

Before each polarization experiments, the working electrode was first immersed into the test solution for 1h at room temperature (25°C) in order to attain its free corrosion potential was recorded as a function of time. After this time a steady-state, corresponding to the corrosion potential ( $E_{\text{corr}}$ ) of the working electrode, was obtained. The electrochemical experiments were carried out in a conventional three electrodes electrochemical cell with a platinum counter electrode and saturated calomel electrode (SCE) as reference electrode. Scan rate of potential was  $1.0 \text{ mV s}^{-1}$  and potential was scanned in the range of  $-500$  to  $-250 \text{ mV}$  relative to the corrosion potential. Polarization data were recorded using a digital potentiostat model Voltalab PGZ301 piloted by Volta Master soft-ware. The polarization curves were carried out in the absence and presence of different concentrations of inhibitors. The IE (%) was calculated from:

$$\text{IE\%} = \left( \frac{I_{\text{corr}} - I'_{\text{corr}}}{I_{\text{corr}}} \right) \times 100$$

where  $I_{corr}$  and  $I'_{corr}$  are the uninhibited and inhibited corrosion current densities respectively. The electrochemical impedance spectroscopy (EIS) measurements were carried out at the open circuit potential ( $E_{ocp}$ ), using a computer-controlled potentiostat (Votalab). Impedance spectra were obtained in the frequency range of 100 kHz to 10 mHz after 1h of immersion in the test solutions. A sine wave with 10 mV amplitude was used to perturb the system. The impedance diagrams are given in the Nyquist representation.

## 2.5. Surface analysis

The surface morphology of the mild steel samples after 3h of immersion in 1M HCl solution with and without inhibitor, were investigated by scanning electron micrograph (SEM) using a Quanta 200 FEG model No.D7860 (XTM @ 2001 FEI Company).

## 3. Results and discussion

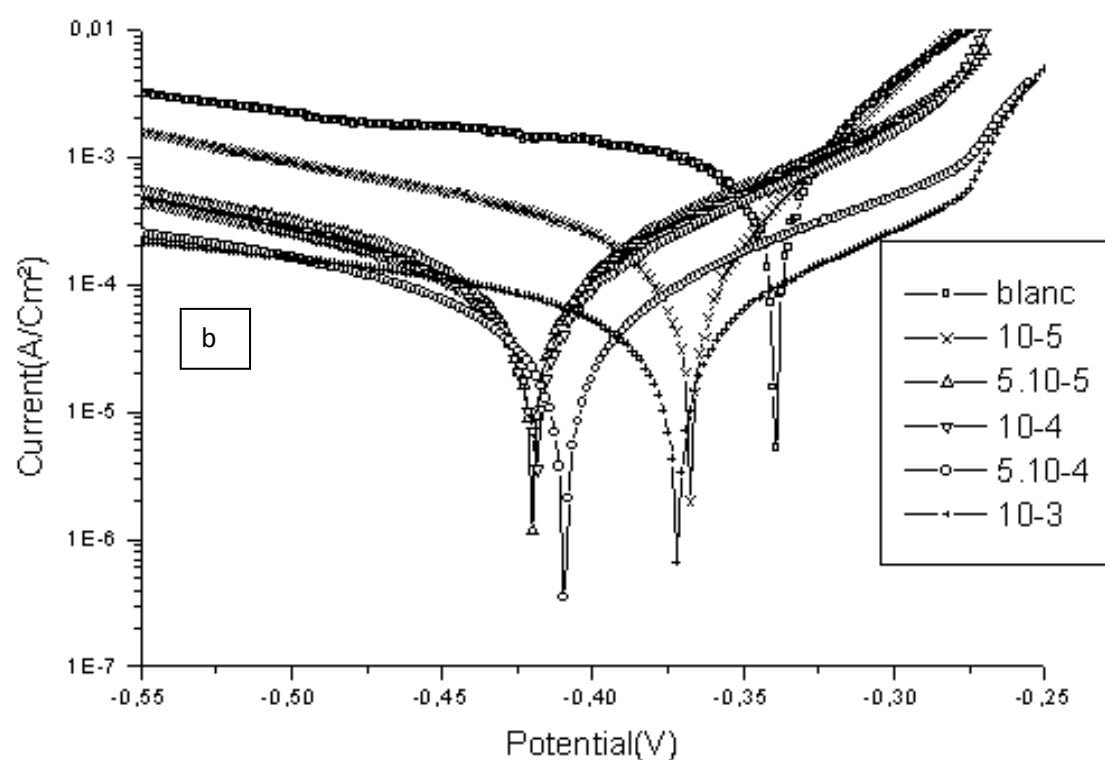
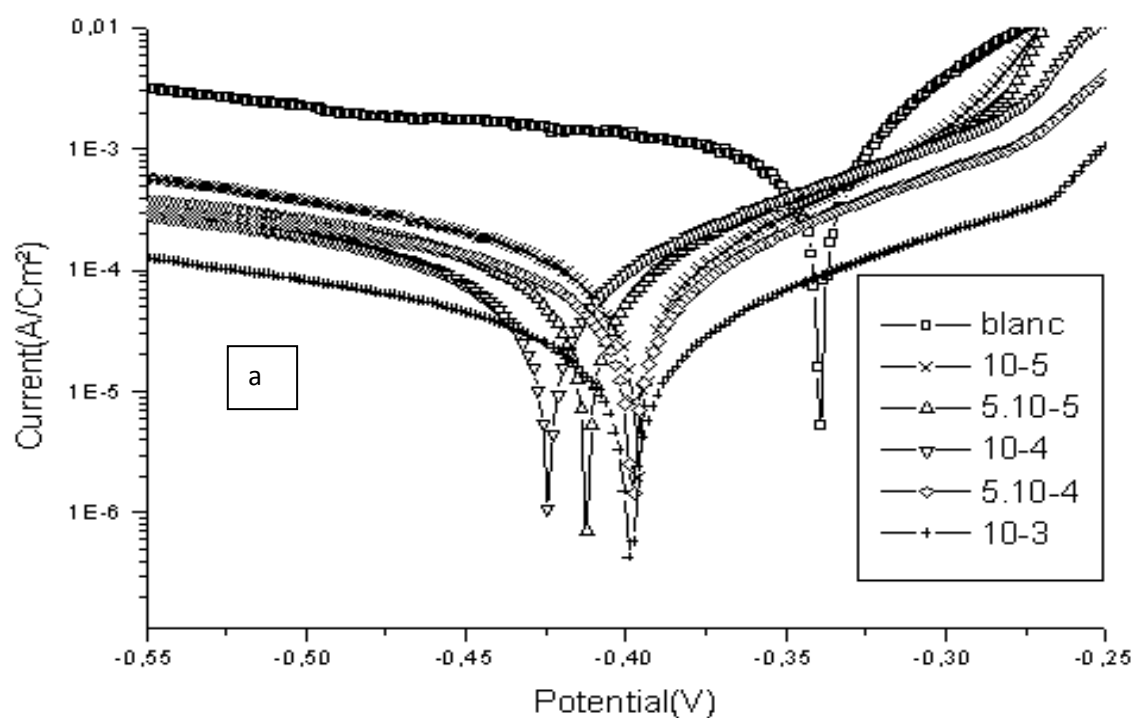
### 3.1. Polarization measurements

The Fig. 1 show potentiodynamic curves obtained for the mild steel electrode in 1M HCl solution with and without different concentrations of compounds studied. The values of corrosion potential ( $E_{corr}$ ), corrosion current density ( $I_{corr}$ ), obtained by extrapolation of the Tafel lines, cathodic tafel slope(bc) and percentage inhibition efficiency (IE (%)) for different concentrations of inhibitors in 1M HCl are given in Table 1. From fig. 1, it is clear that both anodic metal dissolution and cathodic reduction reactions were inhibited when the inhibitors compounds were added to the acid solution and this inhibition was more pronounced with increasing inhibitor concentration. The presence of inhibitors in HCl solution leads to a slight shift of the corrosion potential towards cathodic direction in comparison that obtained in the absence of inhibitor. These results indicate that all the inhibitors compounds act as mixed type inhibitor with a predominance control of the cathodic reaction. The values of cathodic Tafel slope (bc) for all compounds are found to increase with increasing the concentration of inhibitors. The Tafel slope variations suggest that all compounds influence the kinetics of the hydrogen evolution reaction [31]. This indicates an increase in the energy barrier for proton discharge, leading to less gas evolution [32]. The value of %IE increased with increase in concentration of inhibitor, which indicates higher surface coverage of the metal. The action to corrosion resistance is related to the formation of a passive film on the steel surface, which was further supported by SEM images of the electrode surface.

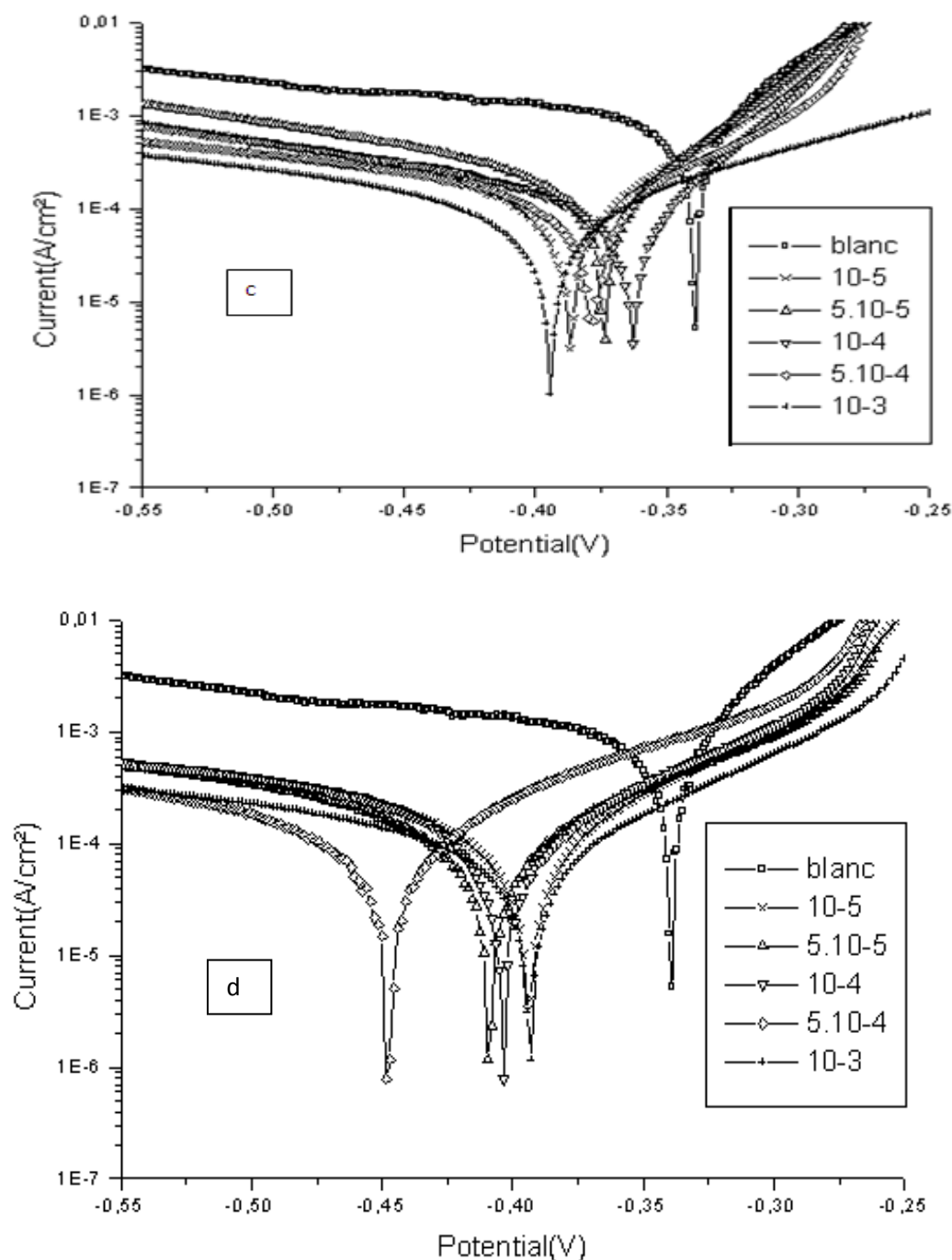
Table 1

Polarization parameters and the corresponding inhibition efficiency for the corrosion of mild steel in 1 M HCl containing different concentrations of benzimidazolium bromide derivatives at 25 °C.

Inhibitors	C <sub>inh</sub>	E <sub>corr</sub> (mV/ECS)	I <sub>corr</sub> (μA/cm <sup>2</sup> )	– bc(mV/dec)	E%
HCl 1M	0	–338	849	185.8	----
DOMB	10 <sup>–5</sup>	–396	125	232.5	85.3
	5.10 <sup>–5</sup>	–412	111	381.2	86.9
	10 <sup>–4</sup>	–420	98	358.5	88.5
	5.10 <sup>–4</sup>	–397	102	326.7	88.0
	10 <sup>–3</sup>	–398	30	367.3	96.5
DDMB	10 <sup>–5</sup>	–367	218	214	74.3
	5.10 <sup>–5</sup>	–420	130	216	84.7
	10 <sup>–4</sup>	–418	100	197	88.2
	5.10 <sup>–4</sup>	–409	62.8	314	92.6
	10 <sup>–3</sup>	–371	68.4	364	91.9
DTMB	10 <sup>–5</sup>	–386	142	237	83.3
	5.10 <sup>–5</sup>	–373	214	226	74.8
	10 <sup>–4</sup>	–362	125	236	85.3
	5.10 <sup>–4</sup>	–378	143	290	83.2
	10 <sup>–3</sup>	–394	103	288	87.9
DHMB	10 <sup>–5</sup>	–393	181	167	78.7
	5.10 <sup>–5</sup>	–408	157	240	81.5
	10 <sup>–4</sup>	–403	189	283	77.7
	5.10 <sup>–4</sup>	–447	125	221	85.3
	10 <sup>–3</sup>	–392	110	286	87.0







**Fig.1.** Polarization curves for mild steel in 1 M HCl containing different concentration of (a) DOMB,(b) DDMB,(c) DTMB and (d) DHMB.

### 3.2. Electrochemical impedance spectroscopy

The corrosion behaviour of mild steel in 1 M HCl in the presence and absence of inhibitors compounds was investigated by EIS at 298K after immersion in solutions for 1 h. Nyquist plots of steel in uninhibited and inhibited acidic solutions containing various concentrations of inhibitors display one capacitive loop at high frequency, the high frequency capacitive loop was attributed to charge transfer of the corrosion process [33] which are shown in Fig. 2. The impedance spectra show that a single semicircle and the diameter of semicircle increases with increasing inhibitor concentration. The impedance diagrams obtained are not perfect semicircles this is attributed to the difference in the frequency dispersion [34], due to the heterogeneity of the electrochemical system. This heterogeneity results from the roughness, impurities, dislocations, the adsorption of the inhibitor and the formation of porous layers [35,36]. It can be seen from the plots that the impedance response of steel has significantly changed on addition of the inhibitors. Various parameters such as charge-transfer resistance ( $R_{ct}$ ), double layer capacitance ( $C_{dl}$ ) are given in table 2.  $R_{ct}$  values were calculated from the difference in impedance at lower and higher frequencies as suggested by Tsuru et al. [37].  $C_{dl}$  values were calculated from the frequency at which the imaginary component of impedance was maximum ( $Z_{im\ max}$ ) using the relation:

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}}$$

where  $f_{max}$  is the frequency at which the imaginary component of impedance is maximum. The inhibition efficiency got from the charge-transfer resistance is calculated by the following relation:

$$IE(R)\% = \frac{R_{ct} - R_{ct}^0}{R_{ct}} \times 100$$

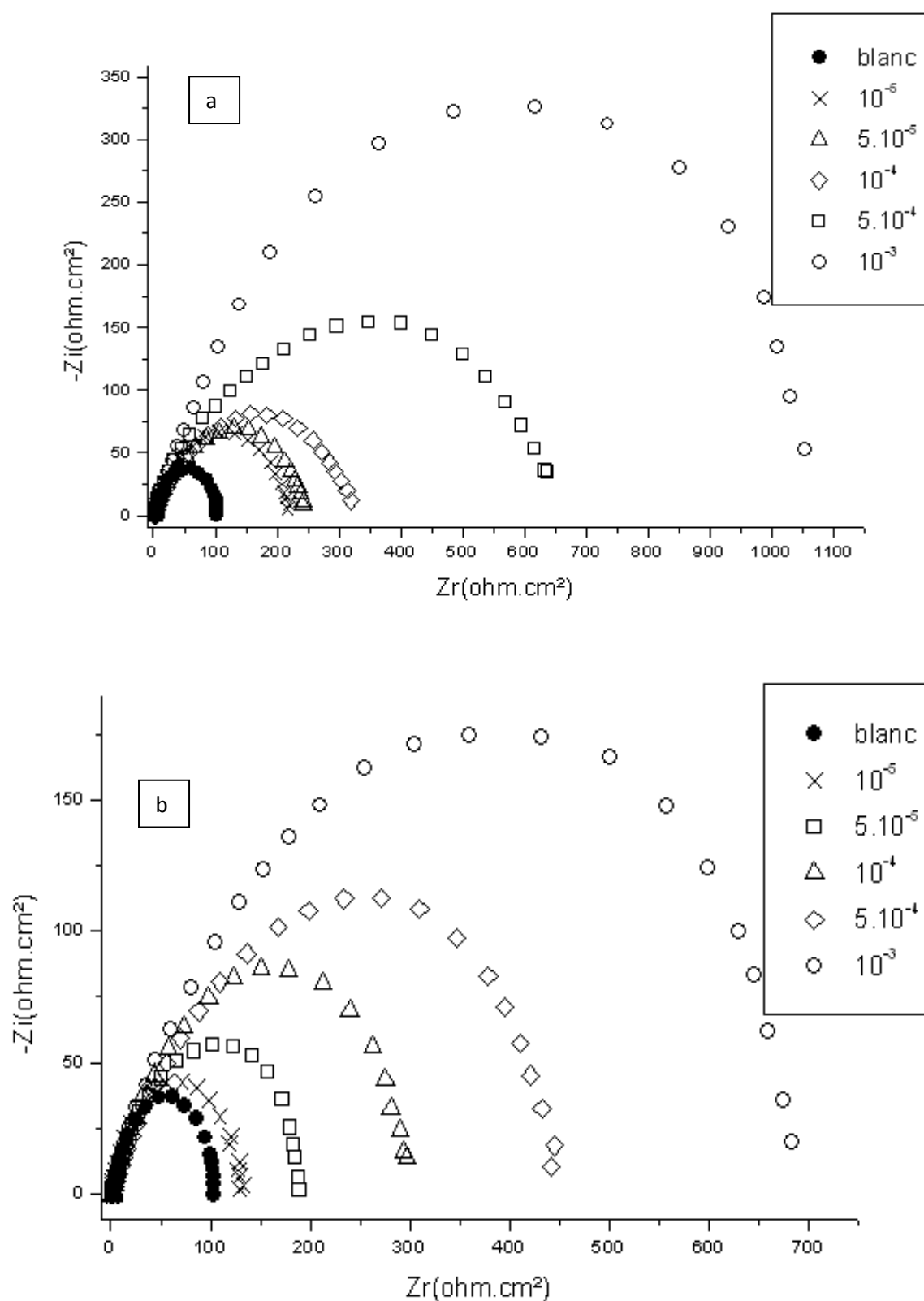
where  $R_{ct}$  and  $R_{ct}^0$  represent the resistance of charge transfer in the presence and absence of inhibitor, respectively.  $R_{ct}$  is the diameter of the loop. As it can be seen from Table 2, the  $R_{ct}$  values of all investigated benzimidazolium bromide derivatives increased with the increasing concentrations of the inhibitors. On the other hand, the values of  $C_{dl}$  decreased with an increase in the inhibitors concentration thus with inhibition efficiencies. The decrease in the  $C_{dl}$ , which can result from a decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer, suggested that the benzimidazolium bromide derivatives molecules function by adsorption at the metal/solution interface. Thus, the change in  $C_{dl}$  values was caused by the gradual displacement of water molecules by the adsorption of the organic molecules on the metal surface, decreasing the extent of the metal dissolution [38].

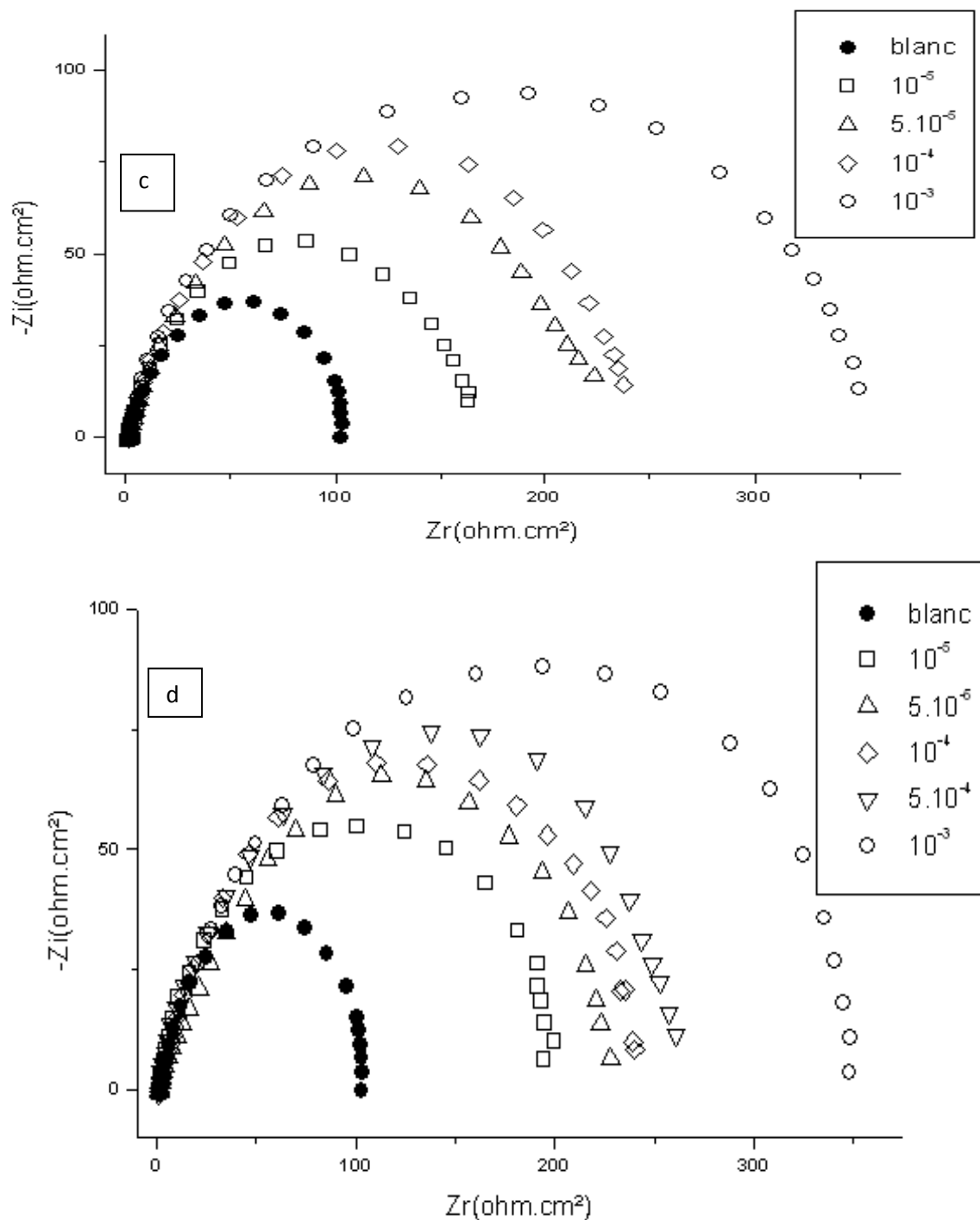


Table 2

Corrosion parameters obtained by impedance measurements for mild steel in 1 M HCl at various concentrations of benzimidazolium bromide derivatives at 25 °C.

Inhibitors	C inh	Rs	Rct	Cdl( $\mu\text{F}/\text{cm}^2$ )	Ez%
	0	1.44	102	104	–
DOMB	$10^{-5}$	1.684	223	28.55	65
	$5.10^{-5}$	1.491	244.4	26.05	68.2
	$10^{-4}$	1.673	325.4	32.61	84.3
	$5.10^{-4}$	0.1	676.3	23.53	88.5
	$10^{-3}$	12.08	1085	23.28	92.8
DDMB	$10^{-5}$	0.8	131.8	80.50	41
	$5.10^{-5}$	2.184	197.0	53.86	60.5
	$10^{-4}$	1.626	301.6	35.18	74
	$5.10^{-4}$	3.823	458.5	55.09	83
	$10^{-3}$	5.613	690	36.09	88.9
DTMB	$10^{-5}$	0.8	165	96.46	52.9
	$5.10^{-5}$	1.069	222.7	71.47	65.1
	$10^{-4}$	1.353	237.4	67.04	67.2
	$10^{-3}$	–0.5	362	43.97	78.5
DHMB	$10^{-5}$	0.7	201	79.18	61.3
	$5.10^{-5}$	2.221	231.6	68.69	66.4
	$10^{-4}$	0.2	248.3	40.49	68.7
	$5.10^{-4}$	1.505	267.4	59.52	70.9
	$10^{-3}$	2.471	351.8	43.99	78.5





**Fig.2.** Nyquist plots for mild steel in 1 M HCl containing different concentration of (a) DOMB,(b) DDMB,(c) DTMB and (d) DHMB.

### 3.4. Adsorption isotherm

The inhibition of corrosion of metals by organic compounds is explained by their adsorption isotherms. the last one is described by two main types of adsorption to be known, physical adsorption and chemisorption. The values of surface coverage,  $\theta$ , corresponding to different concentrations of inhibitors at 298 K have been used to explain the best isotherm to determine the adsorption isotherm. The most frequently used adsorption isotherms are Langmuir, Temkin, and Frumkin. So several adsorption isotherms were checked for the description of adsorption behavior of the studied compounds, and it is found that Langmuir adsorption isotherm is the best approximate between them for the adsorption of benzimidazolium bromide derivative compounds on mild steel surface in HCl solution. The Langmuir adsorption isotherm given by the following equations [39]:

$$\frac{C_{inh}}{\theta} = C_{inh} + \frac{1}{K_{ads}}$$

$$K_{ads} = \frac{1}{55.5} \exp \left( \frac{-\Delta G^{\circ}_{ads}}{RT} \right)$$

where  $C_{inh}$  is the inhibitor concentration,  $\theta$  is the fraction of the surface covered,  $K_{ads}$  is the equilibrium constant of the adsorption process, and  $\Delta G^{\circ}_{ads}$  is the standard free energy of adsorption. The value 55.5 in the above equation is the molar concentration of water in solution in mol/l [40]. the surface coverage ( $\theta$ ) can obtain from polarization measurements, it calculated by the following equation :

$$\theta = \frac{I^{\circ} - I}{I^{\circ}}$$

The thermodynamic parameters for adsorption process obtained from Langmuir adsorption isotherms for the studied benzimidazole derivatives are given in Table 3.

The result shows that all the linear correlation coefficient ( $R^2$ ) can be obtained from the regressions between  $C/\theta$  and  $C$  as shown in Fig. 3, in the other hand all the slopes are close to one, there is confirm that the adsorption of benzimidazolium bromide derivatives compounds in 1 M HCl follows the Langmuir adsorption isotherm. The values of  $K_{ads}$ , which indicate the binding power of the inhibitor to the steel surface, can lead to calculate the adsorption energy. Values of  $\Delta G^{\circ}_{ads} = -38.8$ ,  $-41.1$ ,  $-38.8$  and  $-39.2$  kJ mol<sup>-1</sup> for compounds DOMB, DDMB, DTMB and DHMB, respectively. The negative value of  $\Delta G^{\circ}_{ads}$  means that the adsorption of benzimidazolium bromide derivatives compounds on mild steel surface is a spontaneous process, and furthermore the negative values of  $\Delta G^{\circ}_{ads}$  also show the strong interaction of the inhibitor molecule onto the mild steel surface [41,

42]. It generally accepted that the values of  $\Delta G^\circ_{\text{ads}}$  up to  $-20 \text{ kJ mol}^{-1}$ , the types of adsorption were regarded as physisorption, the inhibition acts due to the electrostatic interactions between the charged molecules and the charged metal, while the values around  $-40 \text{ kJ mol}^{-1}$  or smaller, were seen as chemisorption, which is due to the charge sharing or a transfer from the inhibitor molecules to the metal surface to form a covalent bond [43, 44]. The values of  $\Delta G^\circ_{\text{ads}}$  in our measurements range from  $-41$  to  $-39 \text{ kJ mol}^{-1}$  (in Table 3), it is suggested that the adsorption of these benzimidazole derivatives involves two types of interaction, chemisorption and physisorption.

Table 3

Thermodynamic parameters for the adsorption of benzimidazolium bromide derivatives in 1 M HCl on the mild steel at 25 °C.

inhibitors	$R^2$	$K_{\text{ads}}$	$\Delta G^\circ_{\text{ads}}(\text{KJ/mol})$
DOMB	0.9998	112765.8	$-38.8$
DDMB	0.9999	291840.4	$-41.1$
DTMB	0.9996	118306.3	$-38.8$
DHMB	0.9999	135185.0	$-39.2$

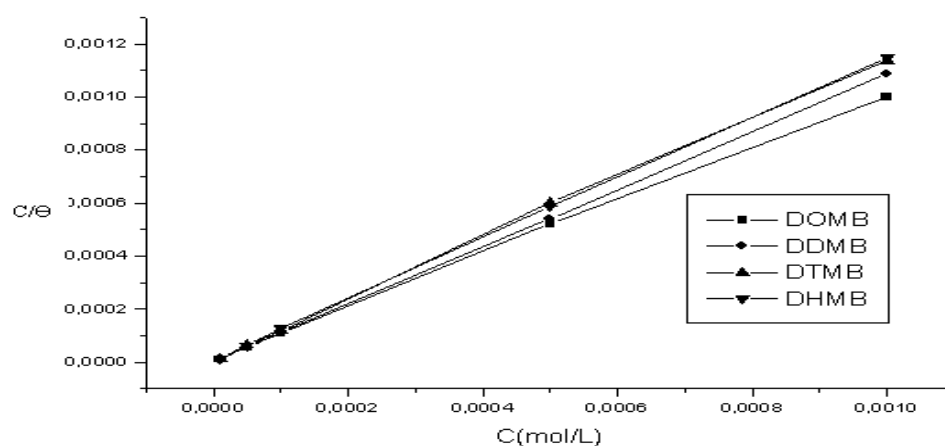
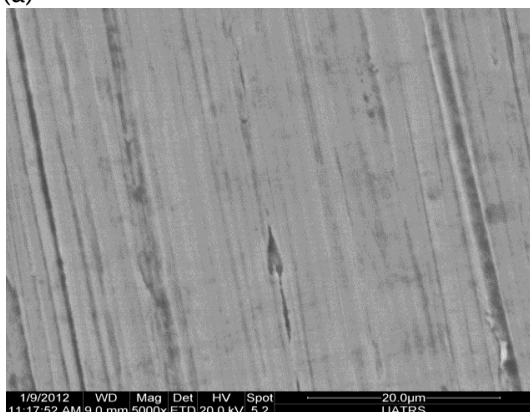


Fig.3. Langmuir isotherm adsorption of benzimidazolium bromide derivatives on the surface of mild steel in 1 M HCl at 25°C.

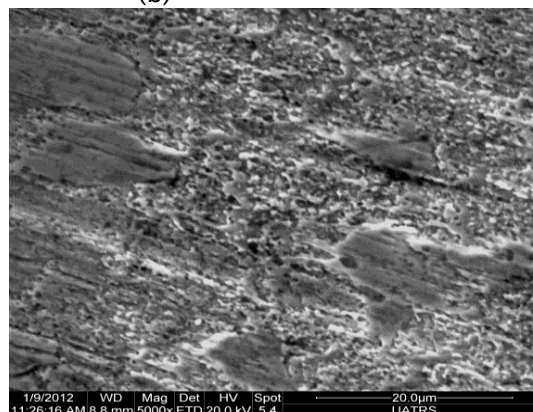
## SEM

In order to evaluate the conditions of the steel surfaces in contact with hydrochloric acid solution, a superficial analysis was carried out. SEM photographs obtained from mild steel surface after specimens immersion in 1 M HCl solutions for 4 h in the absence and presence of  $1.0 \times 10^{-3}$  M of benzimidazolium bromide derivatives compounds are shown in Fig. 4 (a) shows the polished surface of steel before being exposed to the testing environment. The polishing lines of the steel surface are visible in Fig. 4 (a). Fig. 4 (b) shows the SEM image surface of steel after immersion in 1 M HCl, this reveals a severe damage on surface due to metal dissolution SEM images of the mild steel surface after immersion in 1 M HCl with  $1.0 \times 10^{-3}$  M inhibitor of DOMB and DDMB, are shown in Fig. 4 (c) and Fig. 4 (d), it can be seen that in presence of the inhibitor, the rate of corrosion is suppressed, it revealed that there is a good protective film adsorbed on specimens surface, which is responsible for the inhibition of corrosion.

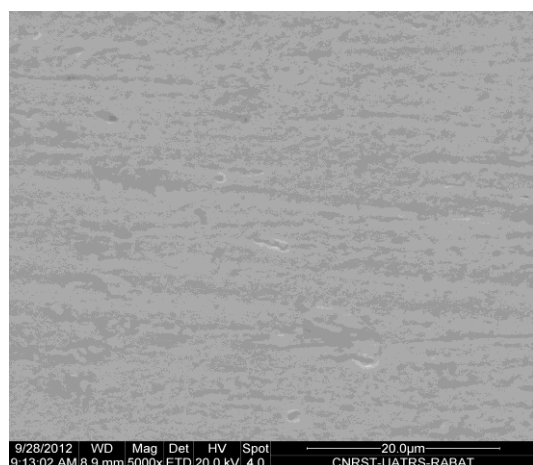
(a)



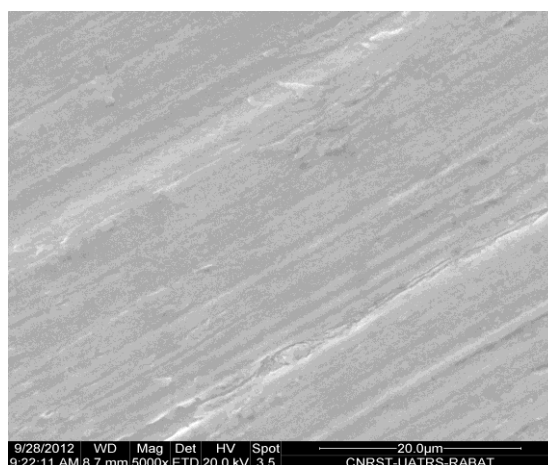
(b)







(d)



(c)

Fig. 4 . SEM micrographs of mild steel samples at 298 K (a) only surface polishing, (b) after immersion in 1 M HCl without inhibitor, (c) after immersion in 1 M HCl in presence of  $1.0 \times 10^{-3}$  M DOMB and (d) after immersion in 1 M HCl in presence of  $1.0 \times 10^{-3}$  M DDMB.

## Conclusion

1. All studied benzimidazolium bromide derivatives shown excellent inhibition properties for the corrosion of mild steel in 1 M HCl solutions, and the inhibition efficiency increases with increasing the concentration of the inhibitors;
2. The results obtained from electrochemical measurements show that the inhibiting properties increase with inhibitor concentration. The IE increases in accordance to the order: DOMB > DDMB > DTMB > DHMB, and the inhibitor efficiencies determined by polarization and EIS methods are in good agreement.
3. The double layer capacitances decrease with respect to blank solution when the benzimidazolium bromide derivatives compounds added. This fact may be explained by adsorption of benzimidazolium bromide derivatives molecules on the mild steel surface.
4. The adsorption model obeys the Langmuir adsorption isotherm at 298 K, and the negative values of free energy of adsorption indicated that the adsorption of the benzimidazolium bromide derivatives molecule is a spontaneous process.
5. The SEM micrographs confirm the protection of the mild steel corrosion in 1M HCl solution by the benzimidazolium bromide derivatives studied.

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