

## **Corrosion Inhibition of Type 430 Stainless Steel in HCl Solution by Dipyridinium Salts**

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*Abstract:* The inhibitive actions of three sensitized derivatives of *N,N'*-diquaternized 4,4'-dipyridinium salts (TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub>) on general and pitting corrosion of 430 stainless steel (SS) in aerated 0.1M HCl have been studied at different temperatures. Corrosion potential and polarization measurements proved that the three compounds act as mixed type inhibitors with dominant anodic and their adsorption on the steel surface obey Langmuir adsorption isotherm. The diquaternized salts inhibited the corrosion and improved the passivation of the SS, their inhibition efficiencies (except for TMdPyBr<sub>2</sub>) increased with temperature. The mechanism of corrosion inhibition was discussed in the light of the molecular structure of the dipyridinium salts.

*Keywords:* Adsorption; Dipyridinium salts; Corrosion inhibition; Stainless steel

### **1. Introduction**

In an environment with the presence of chloride ions, localized corrosion such as pitting and crevice corrosion is still a serious problem for the stainless steel. For this reason, the research study for the passive film of stainless steels and their stability particularly in chloride solutions has a technological importance<sup>[1]</sup>.

The use of inhibitor is one of the most practical methods for protection of the metal against the corrosion but few compounds can be applied usefully against the pitting corrosion<sup>[2]</sup>. The mechanism of corrosion inhibition by organic molecules is mostly attributed due to their adsorption onto metallic surfaces. The adsorption phenomenon depends, principally, on the chemical composition and structure of the inhibitors, nature of the metallic surface, and the acidic properties of the medium where the inhibitor-surface interaction takes place<sup>[3]</sup>.

Surfactants, which consist of one polar group (hydrophilic) and one hydrophobic moiety have been used for several occasions by a large number of investigators and reported that surfactants acts as a good corrosion inhibitors<sup>[4-12]</sup>. Popova *et al.*,<sup>[4,5]</sup> investigated four quaternary ammonium bromides of different heterocyclic compounds as corrosion inhibitors of mild steel in 1 M HCl and 1M H<sub>2</sub>SO<sub>4</sub>. Viologens (*N,N'*-diquaternized 4,4'-dipyridinium salts), which have been employed as herbicides, redox mediators, electrochromic materials, electron-transfer quenchers, and as redox probes in self-assembled monolayers<sup>[13,14]</sup>, could be used as efficient corrosion inhibitors<sup>[15,16]</sup>. However, the details of dipyrindinium dihalides role in the corrosion of stainless steel (type 430) are not yet known. The purpose of this article is to investigate the effect of three substituted dipyrindinium dihalides on the corrosion in acidic medium of HCl. In addition, substituents effects have also been studied and discussed in terms of their molecular structure.

## 2. Experimental Works

Three dipyrindinium dihalide derivatives with chemical structure as shown in Fig.1 were synthesized as described elsewhere<sup>[16]</sup>. A standard corrosion glass cell was used for the polarization and impedance measurements. The material of the working electrode is a sheet with area 1 cm<sup>2</sup> from ferritic type 430 SS, it was cut from cold rolled annealed sheet (containing 17-18.5 wt% Cr and <1500 ppm C). The counter and reference electrodes are platinum sheet and silver-silver chloride (Ag/AgCl, saturated KCl), respectively. Prior to each experiment the working electrode was wet polished with emery papers up to grade 600, rinsed with bi-distilled water, acetone, bi-distilled water. Then, it was transferred to the glass cell which was filled by 200 ml of 0.1M HCl solution. The electrolyte solution was prepared from concentrated analytical reagent HCl and bi-distilled water. The inhibitor solution was prepared by dissolving the appropriate weight in 0.1M HCl solution. All experiments were conducted thermostatically at a given temperature and in an aerated condition without stirring. Electrochemical experiments were recorded using a potentiostat of type Autolab PGSTAT30, coupled to a computer equipped with GPES software for potential and polarization measurements. Potentiodynamic measurements were performed with 0.001 Vs<sup>-1</sup>.

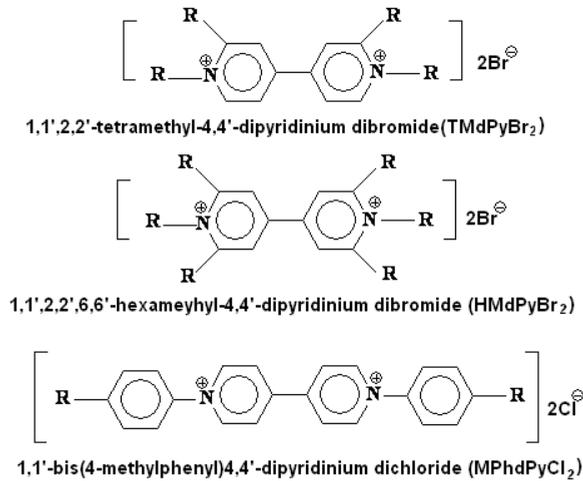


Fig. 1: Structure of dipyrindinium dihalide derivatives (R = CH<sub>3</sub>).

### 3. Result and Discussion

#### 3.1 Corrosion Potential Measurement

After immersion the working electrode in the polarization cell,  $E_{\text{cor}}$  was recorded versus time for  $\sim 30$  min. The average value for  $E_{\text{cor}}$  of the stainless steel in 0.1M HCl at 30°C was 0.508 V (Ag/AgCl). The shifts of  $E_{\text{cor}}$  from the blank value caused by various concentrations of the studied compounds are shown in Table 1. Addition of any inhibitor from the three investigated

Table 1: Effect of inhibitor concentration on the polarization parameters.

Inhibitor	Solution (M)	bc ba (mV/decade)	$-E_{\text{cor}}$ (V)	$i_{\text{cor}}$ $\mu\text{A.cm}^{-2}$	$-E_{\text{pp}}$ (V)	$i_{\text{crit}}$ $\mu\text{A.cm}^{-2}$
	Pure	90 62	508	599	417	2568
TMdPyBr <sub>2</sub>	$5.0 \times 10^{-5}$	125 60	0.503	384	0.421	1887
	$1.0 \times 10^{-4}$	130 48	0.492	244	0.418	1740
	$2.5 \times 10^{-4}$	118 50	0.490	221	0.414	1635
	$5.0 \times 10^{-4}$	112 38	0.486	194	0.431	1479
	$1.0 \times 10^{-3}$	115 40	0.483	174	0.412	1289
HMdPyBr <sub>2</sub>	$5.0 \times 10^{-5}$	115 66	0.495	511	0.415	2452
	$1.0 \times 10^{-4}$	143 64	0.492	340	0.410	1369
	$2.5 \times 10^{-4}$	105 45	0.484	226	0.415	1560
	$5.0 \times 10^{-4}$	128 40	0.480	142	0.400	1307
	$1.0 \times 10^{-3}$	111 38	0.472	172	0.405	1284
MPhdPyCl <sub>2</sub>	$5.0 \times 10^{-5}$	137 65	0.505	397	0.411	2033
	$1.0 \times 10^{-4}$	121 63	0.501	292	0.407	1732
	$2.5 \times 10^{-4}$	114 66	0.498	246	0.387	1704
	$5.0 \times 10^{-4}$	101 50	0.492	220	0.405	1537
	$1.0 \times 10^{-3}$	93 58	0.493	240	0.406	1582

compounds caused systematic displacement of the potential in the anodic direction, indicating that the anodic reaction is somewhat more inhibited than the cathodic reaction. The shift of  $E_{\text{cor}}$  at any concentration increased in the order  $\text{MPhdPyCl}_2 < \text{TMdPyBr}_2 < \text{HMdPyBr}_2$ .

### 3.2 Polarization Measurement

Cathodic polarization and anodic polarization to a potential slightly more positive than the primary passive potential ( $E_{\text{pp}}$ ) were carried out in 0.1M HCl at 30°C and in presence of various concentrations ( $5 \times 10^{-5}$ - $1 \times 10^{-3}$ M) of the inhibitors. Figure 2 shows representative polarization curves in the absence and presence of HMdPyBr<sub>2</sub>. Values of corrosion current density ( $i_{\text{cor}}$ ) associated with the polarization curves were calculated by extrapolation of both anodic and cathodic branches (within Tafel regions) back to  $E_{\text{cor}}$ . The critical current density ( $i_{\text{crit}}$ ) which corresponds to the  $E_{\text{pp}}$  was also obtained. It is a criterion for surface oxide formability on the SS. The estimated parameters were recorded in Table 1. Although the positive shift of  $E_{\text{cor}}$ , both anodic and cathodic branches shifted to lower current density by addition of the inhibitors indicating inhibition of both the cathodic and anodic reactions. The investigated compounds most likely are mixed type inhibitors with dominant anodic, particularly for HMdPyBr<sub>2</sub>. The shifting in the polarization curves runs parallel to that of the blank solution. So that, the addition of inhibitor may doesnot alter the mechanism of either hydrogen evolution or steel dissolution. It was also observed that these inhibitors decreased significantly  $i_{\text{crit}}$  and didn't affect on  $E_{\text{pp}}$  of the steel except slight positive shift at high concentrations of HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub>.

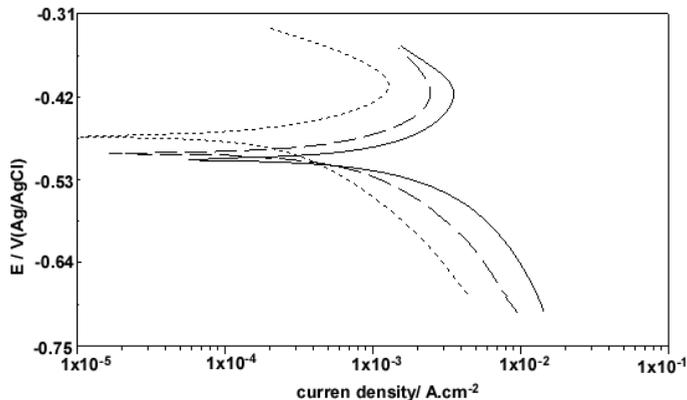


Fig. 2: Cathodic-anodic polarization for the SS in 0.1M HCl (—) at 30°C and in presence of  $5 \times 10^{-5}$  M (--) and  $1 \times 10^{-3}$  M (...) HMdPyBr<sub>2</sub>.

From the  $i_{\text{cor}}$  values the inhibition efficiency (IE) of the additives was calculated according to:

$$\text{IE} = \left\{ \frac{[i_{\text{cor}}^{\circ} - i_{\text{cor}}]}{i_{\text{cor}}^{\circ}} \right\} \times 100 \quad (1)$$

where  $i_{\text{cor}}^{\circ}$  and  $i_{\text{cor}}$  are corrosion current densities in the absence and presence of the inhibitor, respectively. By replacing  $i_{\text{cor}}$  with  $i_{\text{crit}}$  in the above equation suppression efficiency (SE) of the additives for the active dissolution of the SS was calculated.

Figure 3 A shows the IE against inhibitor concentration. For all inhibitors, the IE increased with increasing the concentration and reached fast to a maximum

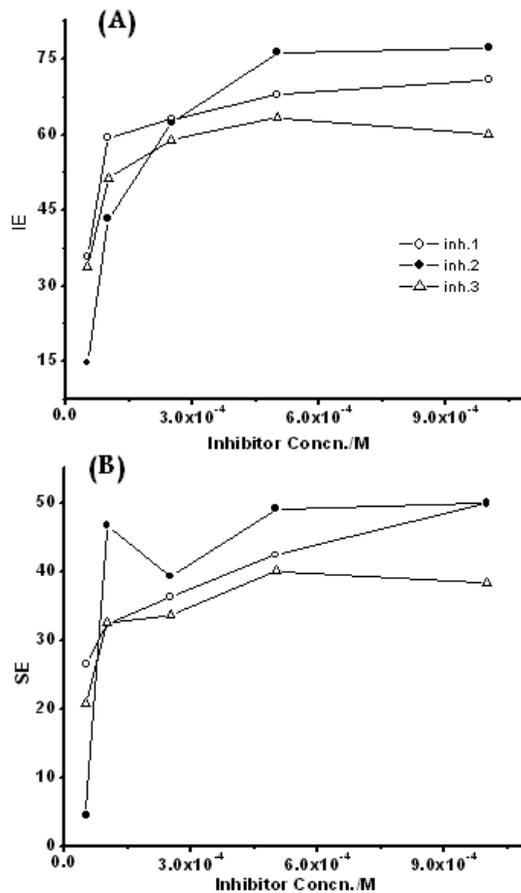


Fig. 3: Inhibition efficiency calculated from  $i_{\text{cor}}$  (A) and  $i_{\text{crit}}$  (B) against the concentration for TMdPyBr<sub>2</sub> (○), HMdPyBr<sub>2</sub> (●) and MPhdPyCl<sub>2</sub> (△).

or plateau. Similar behavior were observed for the same compounds with carbon steel in sulfuric acid<sup>[16]</sup> and other surfactants with steel in HCl<sup>[17]</sup>. In presence of the lowest concentration ( $5 \times 10^{-5} \text{M}$ ) maximum efficiency was 35.9% by TMdPyBr<sub>2</sub>, while it was 77.3% by HMdPyBr<sub>2</sub> at the highest concentration ( $1 \times 10^{-3} \text{M}$ ). The results indicate two different arrangements for the inhibitors in the studied concentration range. The IE decreased in the order TMdPyBr<sub>2</sub> > MPhdPyCl<sub>2</sub> > HMdPyBr<sub>2</sub> at the low concentrations, while it decreased in the order HMdPyBr<sub>2</sub> > TMdPyBr<sub>2</sub> > MPhdPyCl<sub>2</sub> at the high concentrations. Fig.3B shows the SE against inhibitor concentration. The decreasing of  $i_{\text{crit}}$  in presence of the additives was less than that of  $i_{\text{cor}}$  but reflects significantly improvement in the passivation of the SS in the acidic chloride solution. The arrangement for the efficiencies of the inhibitors in Figure 4 is generally similar to that in Figure 3.

### 3.3 Effect of Temperature

The effects of temperature on the inhibitive action of the studied inhibitors were performed. The electrochemical measurements of SS were carried out in 0.1M HCl and in absence and presence of  $1 \times 10^{-3} \text{M}$  inhibitor at different temperatures (30-50°C). Firstly, it is observed that  $E_{\text{cor}}$  of the SS in the pure medium increased with increasing temperature, it shifted from -508 mV at 30°C to -480 mV at 50°C. This is attributed to partial suppression of corrosion by accumulation of the corrosion product on the steel surface. In presence of  $1 \times 10^{-3} \text{M}$  inhibitor the increase of temperature didn't affect the potential,  $E_{\text{cor}}$  were -480, -468 and -490 mV at 30°C and -478, -470 and -486 mV at 50°C for TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub>, respectively. This reflects a strong adsorption of the inhibitors on the SS surface. The changes of the IE and SE with temperature are shown in Table (2). The IE generally decreased with increasing temperature in presence of TMdPyBr<sub>2</sub> but it increased in case of HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub> up to maximum value at 45°C. At this temperature the HMdPyBr<sub>2</sub> exerted efficiency 85%, the highest value in this study.

**Table 2: Inhibition and suppression efficiencies for the inhibitors.**

$t/^\circ\text{C}$	TMdPyBr <sub>2</sub>		HMdPyBr <sub>2</sub>		MPhdPyCl <sub>2</sub>		
	IE	SE	IE	SE	IE	SE	SE
30	76	49.8	71.3	50	60		38.4
35	61.1	46.3	75.8	58.6	54.2		33.9
40	62.3	46.7	84.6	62.1	65		39.8
45	48.4	31.5	84.7	54.6	76		31.2
50	40.7	35	79.4	52.2	68.7		30.8

The SE, in presence of  $1 \times 10^{-3} \text{M}$  HMdPyBr<sub>2</sub>, increased with increasing temperature and reached maximum value (62.1%) at 40°C, as shown in Table 2. In case of TMdPyBr<sub>2</sub> or MPhdPyCl<sub>2</sub> the SE didn't significantly influence by temperature, it decreased slightly after 40°C. This indicates that the passivation

of SS is improved by these inhibitors up to 40°C. The dependence of corrosion current density on the temperature can be expressed with Arrhenius equation:

$$\log i_{\text{corr}} = \log \lambda - (E_a / 2.303 RT) \quad (2)$$

where  $\lambda$  the pre-exponential factor and  $E_a$  is the apparent activation energy of the corrosion process. Plotting of  $\log i_{\text{cor}}$  vs.  $1/T$  produced a straight line as shown in Fig.4. Values of  $E_a$  for SS in 0.1M HCl in the absence and presence of  $1 \times 10^{-3}$ M inhibitor were determined from the produced lines. The linear regression coefficients were 0.995, 0.995, 0.83 and 0.87 in case of the pure solution and in presence of TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub>, respectively. The values of  $E_a$  in the pure solution and in presence of TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub> were 48.35, 82.14, 29.1 and 29.58 k J mol<sup>-1</sup>, respectively. The higher value of  $E_a$  obtained in presence of TMdPyBr<sub>2</sub>, in comparison with that of the blank solution, and the general decrease of the IE with increasing temperature is indicative for physical adsorption (electrostatic attraction between charged molecules and the charged metal surface) of this compound. The lower values of  $E_a$  in the presence of other inhibitors and the general increase of their IE with increasing temperature are indicative for chemisorption (interaction of unshared electron pairs in the adsorbed molecule with the metal) on the steel surface.

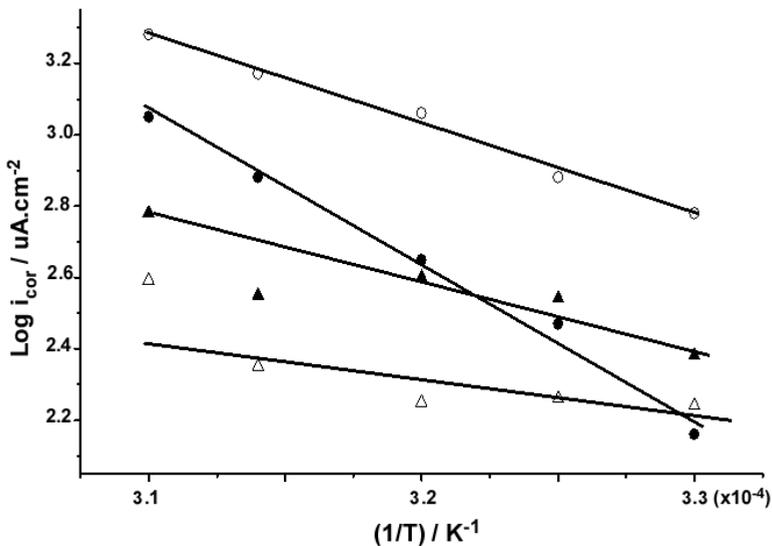


Fig. 4. Arrhenius plots for the corrosion of the SS in 0.1M HCl (○) and in presence of  $1 \times 10^{-3}$  M of TMdPyBr<sub>2</sub> (●), HMdPyBr<sub>2</sub> (△) or MPhdPyCl<sub>2</sub> (▲).

### 3.4 Adsorption Isotherm

The fraction of surface coverage ( $\theta$ ) by inhibitor molecules can be calculated from the equation:

$$\theta = [1 - (i_{corr} / i_{corr}^{\circ})] \quad (3)$$

In an attempt to find the most suitable adsorption isotherm(s),  $\theta$  was subjected to various adsorption isotherms. For all inhibitors, the experimental results were found to fit Langmuir isotherm for monolayer chemisorptions where  $\theta$  and  $C$  (inhibitor's concentration in the bulk of the solution) are related to each other *via* the equation:

$$\theta = KC / (1+KC) \quad (4)$$

Rearrangement gives:

$$C/\theta = (1/K) + C \quad (5)$$

$K$  is the equilibrium constant of the adsorption process. Plotting of  $C/\theta$  against  $C$  gives a straight line with linear regression coefficient  $0.997 \pm 0.001$  for the three inhibitors; Figure 5 shows the plot of MPhdPyCl<sub>2</sub>. The slopes of the straight lines were 1.36, 1.30 and 1.60 for TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub>, respectively. It is larger than one, particularly for MPhdPyCl<sub>2</sub>, indicating the interaction between the adsorbed molecules and/or related to steric hindrance factor<sup>[18]</sup>. The calculated  $K$  values are  $1.02 \times 10^4$ ,  $1.43 \times 10^4$  and  $2.95 \times 10^4$  for TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub>, respectively. This indicating the adsorption process increased in the order TMdPyBr<sub>2</sub> < HMdPyBr<sub>2</sub> < MPhdPyCl<sub>2</sub>.

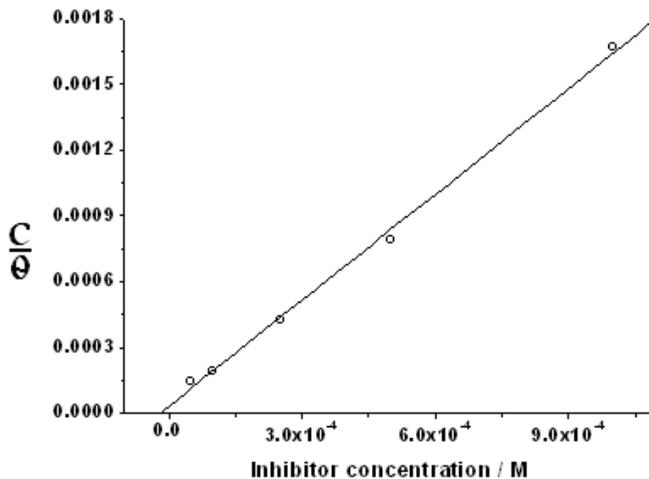


Fig. 5: Langmuir adsorption isotherm of MPhdPyCl<sub>2</sub> on the SS surface.

The constant K is related to the standard free energy of adsorption ( $\Delta G^{\circ}_{\text{ads}}$ ) by the equation:

$$K = (1/55.5) \exp(-\Delta G^{\circ}_{\text{ads}}/RT) \quad (6)$$

where R is the gas constant and T is the absolute temperature. For the investigated inhibitors,  $\Delta G^{\circ}_{\text{ads}}$  values are -33.35, -34.22 and -36.05 kJmol<sup>-1</sup> for TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub>, respectively. Generally, values of  $\Delta G^{\circ}_{\text{ads}}$  around -20 kJ/mol or lower are consistent with electrostatic interaction between the charged molecules and the charged metal surface (physisorption) while those around -40 kJ/mol or higher involve charge sharing or charge transfer from the organic molecules to the metal surface to form a coordinate type bond (chemisorption)<sup>[19]</sup>. It is suggested that the chemical adsorption for the investigated inhibitors increases in the order TMdPyBr<sub>2</sub> < HMdPyBr<sub>2</sub> < MPhdPyCl<sub>2</sub>.

### 3.5 Mechanism of Inhibition

It is suggested that the two ammonium groups and aromatic rings in the dipyridinium molecule can be adsorbed at the different sites on the metallic surface. The molecule is physically adsorbed *via* the two positively charged nitrogen atoms and the aromatic rings are parallel to the surface and the methyl group tail is extended away in the solution. This position is allowed for transfer of the  $\pi$ -electrons of the pyridine rings to the vacant electron orbital of low energy in the metal to form a coordinate type of link (chemical adsorption)<sup>[16]</sup>. The transformation from physical adsorption to chemical type can be occurred gradually<sup>[20]</sup>.

Delocalization of  $\pi$  orbital in the pyridine ring may increase the positive charge on the nitrogen atom of the ring. In other side, the attached methyl groups increase the electron density of the aromatic rings while they may decrease the positive charge of the nitrogen atom. Many arguments in this study prove that the higher methylated molecule HMdPyBr<sub>2</sub> has strong chemisorption with the SS surface while the lower methylated molecule TMdPyBr<sub>2</sub> is acted by weaker chemical adsorption. The chemisorption type inhibitor is important for protection of the stainless steel particularly in aggressive solutions containing chloride ions. The blocking of the active sites on the surface by strong chemical adsorption of the inhibitor enhances the oxide film formation.

The chemical structure of MPhdPyCl<sub>2</sub> molecule is different (Fig.1) by presence of two methyl phenyl groups attached to the nitrogen atom. The presence of additional two benzene rings is expected to increase the covered area and the electronic interaction with the metal surface. MPhdPyCl<sub>2</sub> caused low corrosion activation energy than that in pure medium, and had largest K value and largest

negative  $\Delta G^{\circ}_{\text{ads}}$  value among the investigated compounds, indicating strong chemisorption of this compound. In our previous work<sup>[16]</sup> MPhdPyCl<sub>2</sub> exhibited highest IE, among the same group inhibitors, for low carbon steel in H<sub>2</sub>SO<sub>4</sub> solution. However, in this study, this inhibitor showed the lowest IE of SS in HCl solution and weak inhibitor for pitting initiation. At higher concentration of this inhibitor a red-brown precipitate is formed on the steel surface at the open circuit potential as observed before<sup>[16]</sup>. This inhibitor, most likely, is exposed to irreversible reduction and deposited as precipitate on the electrode surface. Although the deposited layer enhanced the inhibition (secondary inhibition) of mild steel in sulfuric, it is inefficient against the pitting corrosion of the SS. The secondary inhibition may be higher or lower than primary inhibition, depending on the effectiveness of the reaction products<sup>[21]</sup>.

In this study HMdPyBr<sub>2</sub> is superior inhibitor among the studied compound for both the general and localized corrosion of SS in HCl solution. However, this compound exhibited least inhibition efficiency for corrosion of mild steel in H<sub>2</sub>SO<sub>4</sub> and it was attributed to influence of steric hindrance of the attached methyl groups on the adsorption of the molecule<sup>[16]</sup>. But, why the steric hindrance doesn't influence in the stainless steel-HCl system. This is explained by the fact that the changes of the substrate metal and /or the type of aggressive electrolyte are significantly influenced on the adsorption process of the same inhibitor<sup>[21]</sup>.

#### 4. Conclusions

The investigated compounds worked as mixed type inhibitors with dominant anodic against the corrosion of 430 SS in 0.1 M HCl solution. They do not alter the mechanism of either hydrogen evolution or steel dissolution reactions. Also, they improved the passivation of the SS. The IE decreased with increasing temperature in presence of TMdPyBr<sub>2</sub> but increased in case of HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub> up to maximum value at 45°C. The values of  $E_a$  in absence and presence of TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub> were 48.35, 82.14, 29.1 and 29.58 kJ mol<sup>-1</sup>, respectively. The adsorption of these compounds on the steel surface was found to obey Langmuir adsorption isotherm. The calculated K values were  $1.02 \times 10^4$ ,  $1.43 \times 10^4$  and  $2.95 \times 10^4$  and  $\Delta G^{\circ}_{\text{ads}}$  values were -33.35, -34.22 and -36.05 k J mol<sup>-1</sup> for TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyCl<sub>2</sub>, respectively. These arguments suggested that the chemical adsorption for the investigated inhibitors increases in the order TMdPyBr<sub>2</sub> < HMdPyBr<sub>2</sub> < MPhdPyCl<sub>2</sub>. However, the IE and SE increased in the order MPhdPyCl<sub>2</sub> < TMdPyBr<sub>2</sub> < HMdPyBr<sub>2</sub>. The superiority for HMdPyBr<sub>2</sub> as inhibitor was attributed to the higher attached methyl groups which increase the chemisorption property of the dipyrindinium molecule. In spite of its chemisorption on the SS surface, MPhdPyCl<sub>2</sub> showed the lowest IE. This inhibitor, most likely, is exposed to irreversible reduction and deposited as precipitate on the electrode surface.

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## تنشيط تآكل الحديد الصلب من نوع ٤٣٠ في محلول الهيدروكلوريك بواسطة أملاح ثنائي الباريدينيم

فوزية محمد النويصر

قسم الكيمياء - كلية العلوم

جامعة الملك عبدالعزيز - جدة - المملكة العربية السعودية

*المستخلص:* لقد تمت دراسة تأثير ثلاث مشتقات من أملاح:

(N,N'-di-quatarnized-4.4' dipyridinium )

(TMdPyBr<sub>2</sub>, HMdPyBr<sub>2</sub> and MPhdPyC12) وهي

كمثبط للتآكل العام، والثاقب في محلول 0.1M من حمض الهيدروكلوريك عند درجات حرارة مختلفة. وأثبتت نتائج جهد التآكل والقياسات القطبية أن الثلاث مركبات من النوع المثبط الأنودي والكاثودي وذلك بالامتزاز على سطح الصلب وهو يتبع لونجمير ايزوثيرم.

وأن مشتقات أملاح تنشيط التآكل حسنت من خمول الحديد الصلب وأن كفاءة التنشيط زادت مع زيادة درجة الحرارة ماعدا (TMdPyBr<sub>2</sub>). وأن ميكانيكية التنشيط نوقشت من وجهة نظر التركيب الجزئي لأملاح البريديليم.