

Preparation and Study of Some Pyridinium Chloride Derivatives as Corrosion Inhibitors for Mild-Steel in 1M H₂SO₄ Aqueous Solution

Atheer Fadhel Mahmood and Mehdi Salih Shihab

Department of Chemistry, College of Science Al-Nahrain University, Baghdad-Iraq.

E-mail: atheer.chemo@yahoo.com.

Abstract

Pyridinium chloride derivatives, namely: 1-benzyl-4-(2-cyclohexylidenehydrazinecarbonyl) pyridinium chloride, C1; 1-benzyl-4-(2-(1-phenylethylidene) hydrazinecarbonyl) pyridinium chloride, C2; 1-benzyl-4-(2-(diphenylmethylene)hydrazinecarbonyl) pyridinium chloride, C3, were prepared and studied as corrosion inhibitors for mild steel in 1M H₂SO₄ aqueous solution using weighting loss measurements for 24 hours at 30°C. The results showed that inhibition efficiency for all pyridinium chloride derivatives have rapprochement in value with the highest concentration. Increasing in inhibitor concentration accompanies with corrosion rate decreases, inhibition efficiencies increase and surface coverage degree increases. The free energy values of adsorption revealed physisorption effect for (C1, C2, and C3) and gave useful information to predict the interaction between the surface of metal and the organic molecules as corrosion inhibitors.

Keyword: Pyridinium salts, Corrosion, Mild steel, Adsorption isotherm.

1. Introduction

Acidic media was used in various industrial processes for long time, such as industrial acid cleaning, acid descaling, and oil well acidification [1–3]. In these corrosive media, corrosion inhibitors have been added to aggressive environments to protect metallic materials [4]. Organic compounds containing one or more N, O, S and P atoms with lone pair electrons can inhibit the corrosion of metal in aqueous acid solutions [5]. The metal ion acts as an electrophile and organic compound with N, O, S and P atoms with lone pairs have nucleophilic centers and readily available for sharing. Several Schiff bases have also been investigated as corrosion inhibitors for different metals and alloys in acidic media [6,7].

Bereket et al [8] investigated the corrosion inhibition of low carbon steel using quaternary ammonium compounds and surfactant, acetyl tri methyl ammonium bromide. Some works [9] studied the inhibitor behavior of acetyl tri methyl ammonium bromide for mild steel corrosion in 1M HCl and 2M HCl. Vasudevan, T.[10] studied the influence of n- Hexadecyl pyridinium chloride on the corrosion of mild steel in 5N HCl and 5N H₂SO₄.

The present study aimed to investigate the efficiency of some new pyridinium chloride benzyl chloride in (2 mL) of absolute ethanol

was allowed to stand overnight at room temperature in a round-bottomed flask equipped with a condenser. The mixture is then heated for 25 hours. The resulting solution was evaporated and the solid was washed with 3 portions of (10 mL) diethyl ether. The molecular formula of suggested inhibitors is shown in Fig.(1).

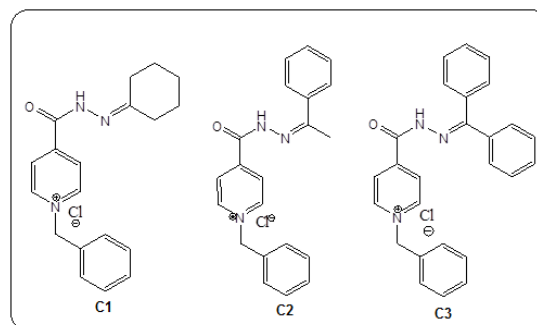


Fig.(1) The molecular formula of suggested inhibitors (C1, C2 and C3).

2.2. Preparation of aggressive solution

Aggressive solution of 1M H₂SO₄ was prepared by dilution of analytical grade 98% H₂SO₄ with distilled water. Inhibitor concentrations of 1x10⁻² to 5x10⁻⁴ M were prepared in 1M H₂SO₄ solution at 30°C.

2.3. Weight loss measurements

A sheet of mild steel used has the composition percentages (0.002 % P, 0.288 %

Mn, 0.03 % C, 0.0154 % S, 0.0199 % Cr, 0.002 % Mo, 0.065 % Cu, and 0.0005 % V) and the remainder was iron. The mild steel sheet was mechanically press-cut into disc shape with diameter (2.5 cm). These disc shapes were polished with emery paper to 4000 grades in order to get a smooth surface. Then they cleaned with distilled water, alcohol, and finally acetone. The treated specimens were then stored in a moisture-free desiccator before their use for corrosion experiments.

Mild steel specimens were initially weighted in an electronic balance. After that, the specimens were suspended and completely immersed in 250 ml beaker containing 1M sulphuric acid in the presence and absence of inhibitors for 24 hrs. The specimens were removed after 24 hours exposure period at 30°C, washed with water to remove any corrosion products and finally washed with acetone. Then they were dried in desiccator for 24 hour and reweighted. Mass loss measurements were performed according to ASTM method described previously [15,16]. The tests were performed in duplicate to guarantee the reliability of the results and the

mean value of the weight loss is reported. Weight loss allowed calculation of the mean corrosion rate in ($\text{mg cm}^{-2} \text{h}^{-1}$). Corrosion rate of mild steel was determined using the relation (1)[17]:

$$W = \frac{\Delta m}{S \cdot t} \dots\dots\dots (1)$$

Where Δm is the mass loss (mg), S is the area (cm^2) and t is the immersion period (hours).

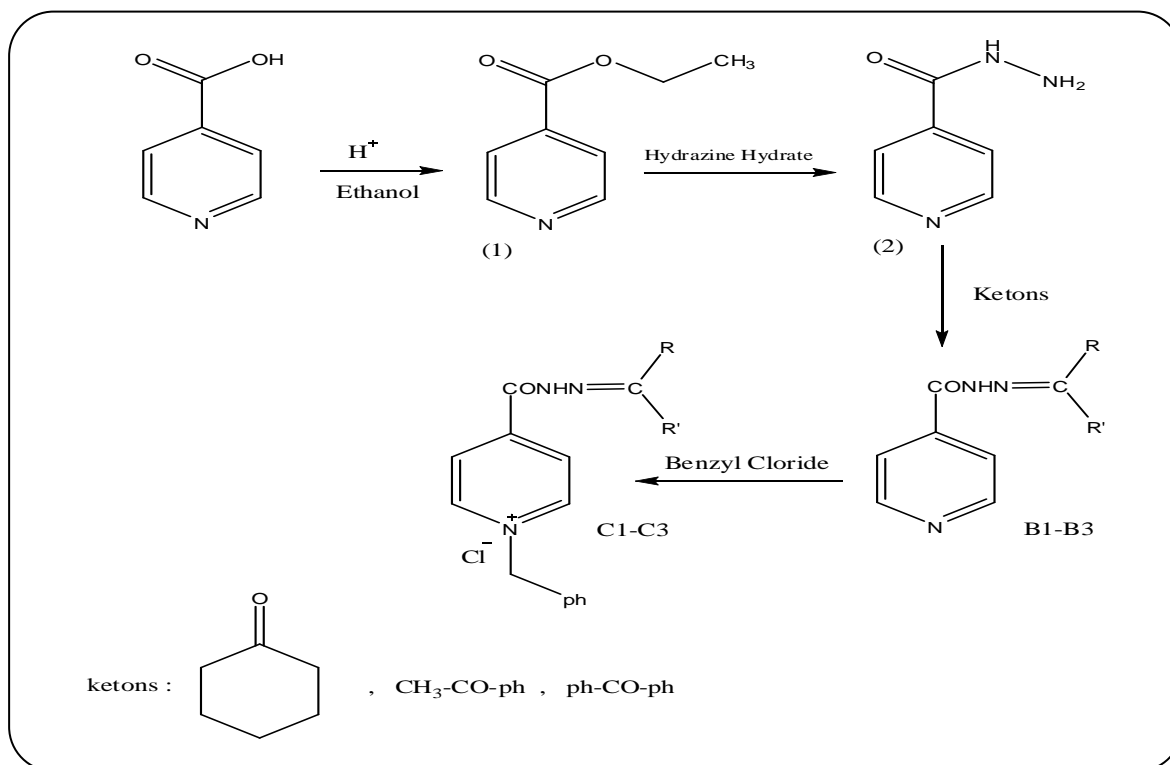
The percentage inhibition efficiency (E(%)) was calculated using the relationship (2) [18]:

$$E\% = \frac{W_{\text{corr}} - W_{\text{corr(inh)}}}{W_{\text{corr}}} \times 100 \dots\dots\dots (2)$$

Where W_{corr} and $W_{\text{corr(inh)}}$ are the corrosion rates of mild steel in the absence and presence of inhibitor, respectively.

3. Results and Discussion

Scheme (1) shows the preparation of pyridinium chloride derivatives (C1-C3) of the present work



Scheme (1) Preparation steps of the pyridinium chlorides (C1, C2, and C3).

The reactions above are included preparation of the pyridinium chloride derivatives (C1, C2 and C3) by using isonicotinic acid hydrazide to carry out some derivatives of Schiff's bases with different ketones. Then the reaction followed by the substitution reaction with benzyl chloride.

The FT-IR spectral data and physical properties of prepared compounds: ethyl isonicotinate (1), isonicotinic acid hydrazide (2), (B1, B2, B3 and C1, C2, C3) and summarized in the Table (1):

Table (1)
Physical properties of prepared compounds and FTIR spectral data.

Compound	Chemical formula	Color	M.W. (g/mol)	M.P. °C	Yield %	FT-IR (cm ⁻¹ , stretching)	
1	ethyl isonicotinate	C ₈ H ₉ NO ₂	Yellow	151.16	(B.P. 223-225)	60	aromatic(C-H) 3035 aliphatic (C-H) 2872 ester (C=O) 1720
2	isonicotinic acid hydrazide	C ₆ H ₇ N ₃ O	White	137.14	164-167	67	aromatic(C-H) 3047, amine (N-H) 3302 and 3221, amide (C=O) 1668
B1	N'-cyclohexylidene isonicotinohydrazide	C ₁₂ H ₁₅ N ₃ O	White	217.27	147-150	83	aromatic(C-H) 3032, aliphatic (C-H) 2858, amide (C=O) 1658, amide (N-H) 3217
B2	N'-(1-phenylethylidene) isonicotinohydrazide	C ₁₄ H ₁₃ N ₃ O	White	239.27	130-133	81	aromatic(C-H) 3028, aliphatic (C-H) 2819, amide (C=O) 1634, amide (N-H) 3178
B3	N'-(diphenylmethylene) isonicotinohydrazide	C ₁₉ H ₁₅ N ₃ O	Pale pink	301.34	186-190	75	aromatic(C-H) 3076, amide (C=O) 1658, amide (N-H) 3255
C1	1-benzyl-4-(2-cyclohexylidenehydrazinecarbonyl) pyridinium chloride	C ₁₉ H ₂₂ ClN ₃ O	Brown	343.85	75-77	85	aromatic(C-H) 3039, aliphatic (C-H) 2855, amide (C=O) 1674, amide (N-H) 3166, imine (N=C) 1635
C2	1-benzyl-4-(2-(1-phenylethylidene)hydrazinecarbonyl) pyridinium chloride	C ₂₁ H ₂₀ ClN ₃ O	Yellow	365.86	68-70	83	aromatic(C-H) 3034, aliphatic (C-H) 2947, amide (C=O) 1681, amide (N-H) 3116, imine (N=C) 1639
C3	1-benzyl-4-(2-(diphenylmethylene)hydrazinecarbonyl) pyridinium chloride	C ₂₆ H ₂₂ ClN ₃ O	Pale yellow	427.93	202-204	83	aromatic(C-H) 3035, aliphatic (C-H) 2827, amide (C=O) 1672, amide (N-H) 3116, imine (N=C) 1639

The ¹H-NMR spectral data of compounds (C1 –C3) in ppm listed in Table (2):

Table (2)
The $^1\text{H-NMR}$ spectral data of compounds (C1 –C3) in ppm.

Comp. No.	Compound structure	$^1\text{H-NMR}$ data of ($\delta\text{-H}$) in ppm
C1	1-benzyl-4-(2-cyclohexylidenehydrazinecarbonyl) pyridinium chloride	4H of pyridine-ring (8.7-9.4); 5H of Phenyl-ring(7.2-8.7) , 2H of $-\text{CH}_2$ group(5.9); 1H of $-\text{OH}$ (4.0 tautomerism of $-\text{CO-NH-}$ moiety)(3); 1.5-2.3 of 10H of aliphatic ring
C2	1-benzyl-4-(2-(1-phenylethylidene) hydrazinecarbonyl) pyridinium chloride	4H of pyridine-ring (8.7-9.5); 10H of Phenyl-rings (7.3-8.7) , 2H of $-\text{CH}_2$ group(5.9); 1H of $-\text{OH}$ (4.1 tautomerism of $-\text{CO-NH-}$ moiety)(3); 1.3 $-\text{CH}_3$ group
C3	1-benzyl-4-(2-(diphenylmethylene)hydrazinecarbonyl) pyridinium chloride	1H of $-\text{NH}$ amid (13.2); 4H of pyridine-rings (8.7-9.4); 15H of Phenyl-ring(7.5-8.7) , 2H of $-\text{CH}_2$ group(5.982); 1H of $-\text{OH}$ (4.01 tautomerism of $-\text{CO-NH-}$ moiety)(3)

The results of corrosion rate and inhibition efficiency that obtained from weight loss measurements with different concentrations of suggested inhibitors (C1, C2, C3) after 24 hours immersion at 30 °C are summarized in Table (3) and depicted in Figs.(2,3).

Table (3) Corrosion rate (W_{corr}), inhibition efficiency (E%), surface coverage (θ) and standard free energy of adsorption ($\Delta G^{\circ}_{\text{ads}}$) for mild steel in 1M H_2SO_4 by using weight loss measurements, where time (T) was 24 hours.

Table (3)
Show Corrosion rate, inhibition efficiency, surface coverage and standard free energy of adsorption of compounds (C1, C2 and C3).

Concentration (M)	Corrosion rate ($\text{mg.cm}^{-2}.\text{h}^{-1}$)	E%	θ	$\Delta G^{\circ}_{\text{ads}}$ (kJ.mol^{-1})
Blank	0.631	-	-	-
(C1)				
5×10^{-4}	0.434	31.16	0.3116	-28.53 ($R^2=0.992$)
1×10^{-3}	0.136	78.47	0.7847	
5×10^{-3}	0.020	96.84	0.9684	
1×10^{-2}	0.019	96.97	0.9697	
(C2)				
5×10^{-4}	0.262	58.55	0.5855	-31.00 ($R^2=0.999$)
1×10^{-3}	0.096	84.86	0.8486	
5×10^{-3}	0.014	97.85	0.9785	
1×10^{-2}	0.011	98.25	0.9825	
(C3)				
5×10^{-4}	0.499	20.86	0.2086	-27.48 ($R^2=0.965$)
1×10^{-3}	0.090	85.73	0.8573	
5×10^{-3}	0.009	98.52	0.9852	
1×10^{-2}	0.004	99.33	0.9933	

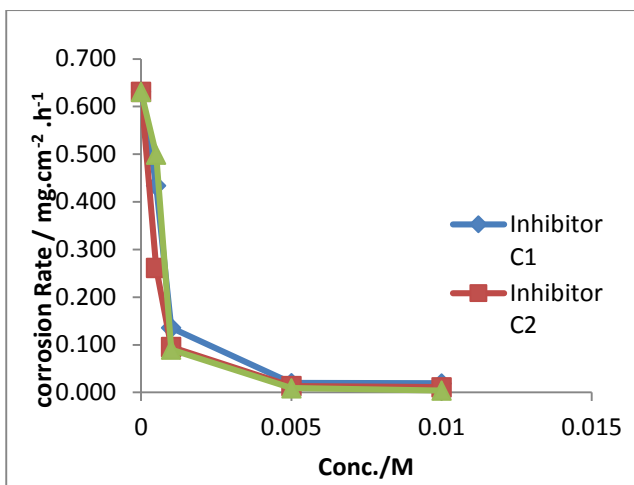


Fig.(2) Effect of inhibitor concentrations on the rate of corrosion for mild steel in 1M H₂SO₄ at 30°C for suggested inhibitors (C1, C2, and C3).

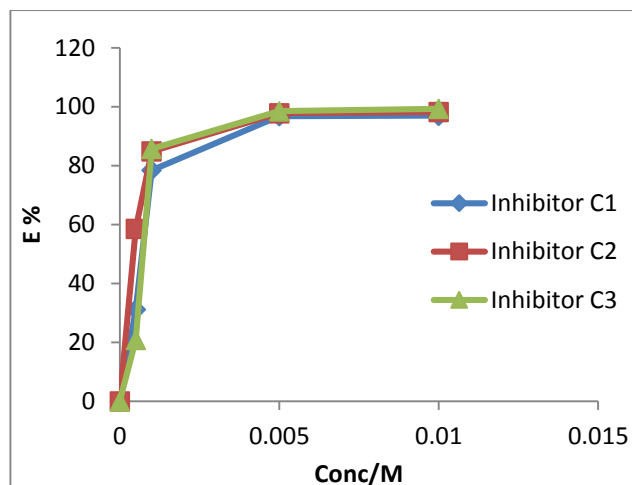


Fig.(3) Effect of inhibitor concentrations on the inhibition efficiency for mild steel 1M H₂SO₄ at 30°C for suggested inhibitors (C1, C2, C3).

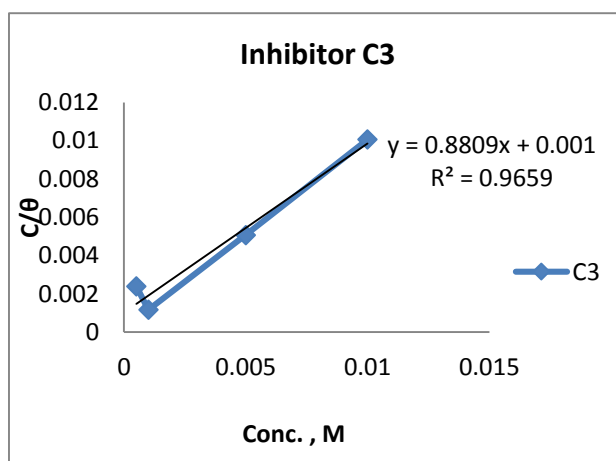
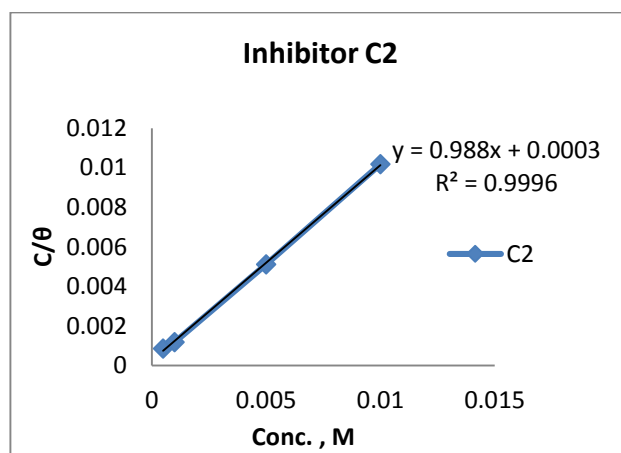
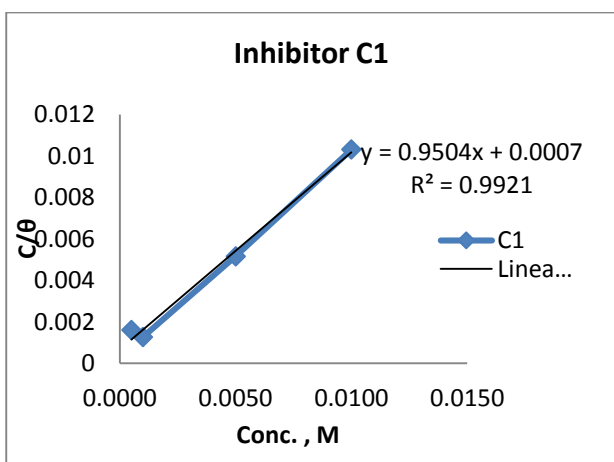


Fig.(4) the linear relationship between C/θ versus C for (C1, C2 and C3).

Table (3) indicates that the protection efficiency increases with increasing the inhibitors concentration and the maximum inhibition efficiencies were achieved at 10⁻² M. Thus, the comparative study reveals

that inhibition efficiency of (C1, C2, and C3) are approximately equal. The rapprochement in the inhibition efficiency could explain by the effect of molecular structure of organic

inhibitors on inhibition efficiency, as well as adsorption process.

Basic information can be provided from the adsorption isotherms to explain the interaction between the organic compounds and metal surfaces. So that, the degree of surface coverage values (θ) for different inhibitor concentrations in 1M H₂SO₄ was achieved from weight loss measurements ($\theta = E (\%) / 100$) (listed in Table (3)) at 30°C and tested with Langmuir isotherm relationship (3) [26]:

$$C/\theta = 1/K_{ads} + C \dots\dots\dots (3)$$

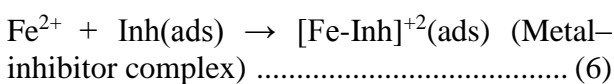
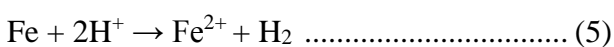
Where C is concentration in M, K_{ads} is the equilibrium constant of the adsorption process.

According to the Langmuir isotherm, K_{ads} values can be calculated from the intercepts of the straight line of plotting C/ θ versus C Fig.(3). K_{ads} is related to the standard free energy of adsorption, ΔG°_{ads} , with the following equation: (The value 55.5 is the molar concentration of water in the solution in M).

$$K_{ads} = \frac{1}{55.5} \exp\left(-\frac{\Delta G_{ads}}{RT}\right) \dots\dots\dots (4)$$

Table (3), show that values of standard free energy of adsorption are negative to indicating that the processes of adsorption of all suggested inhibitors (C1, C2, C3) were spontaneous processes on the mild steel surface after 24 hours immersion at 30°C and that's given sense for remarkable interaction between suggested inhibitors and metal surface. Here, adsorbed molecule moves closer to the surface of metal making electrons start to overlap with that of the surface atoms which causes physisorption for suggested inhibitors [27-30].

It is generally accepted that the adsorption of an organic inhibitor on a metal surface in acidic media usually involves formation of a metal–inhibitor complex by combining an inhibitor with freshly generated Fe²⁺ ions on the steel surface [31]:



Therefore, formation of a metal–inhibitor complex could work as protective layer for anodic sites to reduce formation of Fe²⁺ sites. Thus it could be suggested that probability of formation a compact metal–inhibitor complex is low at low concentrations of suggested inhibitors. The adsorption mechanism for given inhibitors depends on adsorption behavior of organic molecules containing N, O atom. The presence of more than one functional group has been reported to often lead to changes in the electron density of a molecule, which could influence its adsorption behavior [32].

The suggested inhibitors could adsorb with the corroding metal surface via the compact metal–inhibitor complex on anodic sites and that will cause to reduce losing (Fe) atoms from the surface by electrochemical dissolution.

Finally, the suggested inhibitor (C2) showed physisorption with $\Delta G^{\circ}_{ads} = -31 \text{ kJ/mol}$, it's confirmed with the values of E% (58-98) for different concentrations, Table (3) comparing with the others. In spite of low concentration (0.0005M) of (C2) but still E% is higher comparing with the E% values of (C1) and (C3). (C2) has higher electronic density around the -N=CH group comparing with (C1) and (C3). This regular distribution of the electronic density on the surface of organic molecule (C2) improves the interaction between the organic molecule and metal surface.

4. Conclusion

The prepared pyridinium derivative compounds (C1, C2, and C3) were used successfully as corrosion inhibitors on the mild steel surface in 1M H₂SO₄ aqueous solution at 30°C. The results of inhibitive efficiency (E%) showed interesting inhibitive effects of suggested inhibitors. The free energy values of adsorption revealed physisorption effect for (C1, C2, and C3) gave useful information to explain the interaction between the surface of metal and the organic molecules.

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الخلاصة

تم تحضير وتشخيص مشتقات كلوريد البيريدينيوم، وهم (C1) -1 بنزيل -4 (سايلكو هكسايل داين هيدرازين كاربونيل) كلوريد البيريدينيوم، (C2)، 1-بنزيل -4 -2) فينل أثلين داين) كلوريد البيريدينيوم و (C3) 1-البنزيل-4 -2) (ثنائي فنيل مثلين) هيدرازين كاربونيل) كلوريد البيريدينيوم، وتمت دراسة هذه المركبات العضوية كمثبطات لتآكل الحديد الصلب في محلول مائي لحمض الكبريتيك بتركيز 1 مولاري ولمدة 24 ساعة في درجة حرارة 30 درجة مئوية. وباستخدام قياسات فقدان الوزن، حيث أظهرت النتائج أن كفاءة التثبيط لجميع مشتقات كلوريد البيريدينيوم تتناسب طردياً مع تركيز المثبط في المحلول. وجد بأن زيادة تركيز المثبط يؤدي إلى انخفاض معدل التآكل، وارتفاع كفاءة التثبيط تعني زيادة في تغطية سطح المعدن. كما تم حساب قيم طاقة كبس الحرة للامتزاز وتبين ان نوع الامتزاز هو فيزيائي على سطح المعدن للمركبات (C1, C2, C3) وقد تم الحصول على معلومات مفيدة للتنبؤ بالتفاعل الحاصل بين جزيئات المركبات العضوية كمثبطات لتآكل سطح المعدن.