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Synthesis and Study Behavior of Some New Pyridinium Salts as Corrosion Inhibitors for Mild Steel in 1M H_2SO_4

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Abstract

Some new pyridinium bromide derivatives inhibitors, namely; 1-benzyl-4-{N-[(4-hydroxyphenyl) methylidene] hydrazinecarbonyl) pyridine-1-ium bromide, A1; 1-benzyl-4-{N-[(4-ethoxyphenyl) methylidene] hydrazine carbonyl} pyridine-1-ium bromide, A2; 1-benzyl-4-{N-[(4-propoxyphenyl) methylidene] hydrazinecarbonyl} pyridine-1-ium bromide, A3; were synthesized and studied as inhibitors corrosion for meld steel in 1M H_2SO_4 solution at room temperature via loss of weight measure ements for 24 hours. The results showed that inhibition for all pyridineum bromide derivatives have high values with the high concentrate. Increase in inhibition concentrate with corrosion rate decreases, inhibitor increasing and surface coverage degree increases. The free energy value of revealed physisorption effects for (A1, A2, and A3) and gave useful information to predict the interaction nature between the surface of metal and the organic molecules as corrosion inhibitors.

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1. Introduction

Corrosion is an abiquitous phenomenon and there is way to complete stop it. Some new solution can only slow their process. However, the pollution environmental, global warm and climete changed are the direct cause for increase corrosion. just the increasing in global temp. by 2 °C causes an increasing of corrosion rate by up to 15% [1-3]. Corrosion is induced by electrochemical and chemical processes. Chemical corrosion takes in dry gas and conductive liquid where there is electron floweed. The mainly effect of chemical is oxide layer as a result of oxidation in air [4,5].

Schiff's bases are chemical compounds, which have biological effects such as anti-inflammatory, analgesic, antimicrobial, anticonvulsant, antitubercular, anticancer, antioxidant, and anthelmintic, in addition to several Schiff base has investigated as corrosion inhibitor for metals and alloy in acid media [6-9].

On the other side, toxicity and biological of pyridanium salts has well docimented because their increase applications. More specifically, pyridanium salts carrying long alkyl chains were found to be outstanding organic anticorrosion agents [10-17]. In this work, we suggested to study some new Pyridinium bromide derivatives as corrosion inhibitors for mild steel in 1M H_2SO_4 aqueous solution at room temperature via loss of weight measurements for 24 hours.

2. Experimental

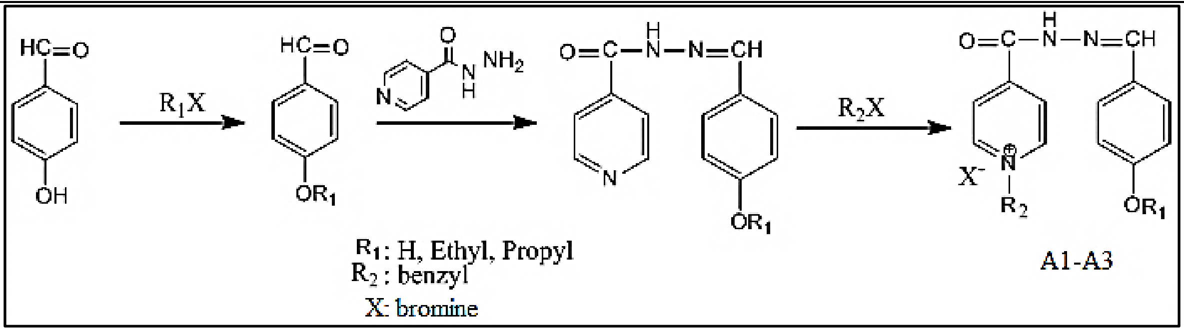
2.1 Materials and preparation:

All the chemicals (reagents and solvents) were use as received. The spectra of prepare compound was recoded using FTiR 8300 transformed infrared spectro photometer of Company as a potassium bromide (kBr) in the Wave numbers range of (4000-400) cm^{-1} and ^1H-NMR (DMSO-d_6). Melting point were determined by the open capillary were used hot Gallenkamp melting point apparatus and were corrected. *SPECTROMAX (*2009, Germany) was used to determined composition of mild steel sample, (State Company for inspection and rehabilitation, Ministry of Industry and Materials, Baghdad).

2.2 Preparation:

Synthesis of suggested inhibitors

Pyridinium bromide derivatives inhibitor, name: namely 1-benzyl-4-{N-[(4-hydroxyphenyl) methylidene] hydrazine-carbonyl) pyridine-1-ium bromide, A1; 1-benzyl-4-{N-[(4-ethoxyphenyl) methylidene] hydrazinecarbonyl} pyridine-1-ium bromide, A2; 1-benzyl-4-{N-[(4-propoxyphenyl) methylidene] hydrazinecarbo-nyl} pyridine-1-ium bromide, A3; were synthesized as follow:



Scheme 1. Steps for synthesis of Pyridium salts A1-A3.

4-alkoxybenzaldehydes were prepared by using bromoethane, 1- Bromopropane, with 4-hydroxybenzaldehde (C1). Added the 4-hydroxybenzaldehyde (0.02 mole, 2.44 g), (K_2CO_3) (0.075 mole, 10.36 g) and corresponding n-alkyl halide (0.03 mol), then added EtOH as a solvent. Refluxed for 24 hours. overnight for the final solution. The formed solution transferred to the separator funnel with D.W (50 ml), the oily layers are formed, dried with anhydrous magnesium sulfate [18], % yield and melting point are listed in Table 1.

Table 1. The physicochemical properties of compounds C2-C3.

No.	**IUPAC names**	**Molecular formula**	**Molecular weight (g/mol)**	**Yield (%)**	**Color**	**Boiling point °C**
C2	4-ethoxybenzaldehyde	$C_9H_{10}O_2$	150	69	Brown	257
C3	4-propoxybenzaldehyde	$C_{10}H_{12}O_2$	164	70	Dark brown	245

A mixture of each compound C1- C3 (0.01 mol) with (0.01 mol) of isonicotinic acid in 20 ml of absolute ethanol, and (2-3) drops of glacial acetic acid. Then, the mixture of solution was refluxed for 3 hours. Finally, the precipitates were formed, collected by filtration, dried and recrystallized with ethanol [19], % yield and melting point of B1-B3 are listed in Table 2.

Table 2. Physical Properties of compound B1-B3.

No	**IUPAC names**	**Molecular formula**	**Molecular weight (g/mol)**	**Yield (%)**	**Color**	**Melting point °C**
B1	N-[(4-hydroxyphenyl) methylidene]pyridine-4-carbohydrazaid	$C_{13}H_{11}O_2N_3$	241.25	85	Yellow	270-272
B2	N-[(4-ethoxyphenyl) methylidene]pyridine-4-carbohydrazaid	$C_{15}H_{15}O_2N_3$	269.30	79	White	263-265
B3	N-[(4-propoxyphenyl) methylidien]pyridine-4-carbohyrazide	$C_{16}H_{17}O_2N_3$	283.33	80	White	222-225

A mixture of each (0.01 mol) Schiff's bases B1-B3 with (0.01 mol) of benzyl bromide and EtOH as solvent. The solution refluxed for 48 hours. and then filtration the solution and collect the solid phase of A1-A3 [20], % yield and melting point are listed in Table 3.

Table 3. Physical properties of compounds A1-A3.

Comp. No.	IUPAC names	Molecular formula	Molecular weight (g/mol)	Yield (%)	Color	Melting point ºC
A1	1-benzyl-4-{N-[(4-hydroxyphenyl) methylidene]hydrazinecarbonyl)pyridine-1-ium bromide	$C_{20}H_{18}O_2N_3Br$	332.38	57	Dark yellow	Gummy
A2	1-benzyl-4-{N-[(4-ethoxyphenyl) methylidene] hydrazinecarbonyl}pyridine-1-ium bromide	$C_{22}H_{22}O_2N_3Br$	360.44	75	Brown	Gummy
A3	1-benzyl-4-{N-[(4-propoxyphenyl) methylidene] hydrazinecarbonyl}pyridine-1-ium bromide	$C_{23}H_{24}O_2N_3Br$	374.46	82	Dark brown	Gummy

2.3 Preparation and weight loss of aggressive solutions:

Aggressive solution of 1M H_2SO_4 was prepare by dilute of analytical graide 98% H_2SO_4 with distil water. Inhibitor concentrate of (1×10^{-2} to 5×10^{-2} M) was prepared in 1 M H_2SO_4 solution at room temp.

2.4 Weight loss measure ements [21]:

The sheets of mild steel use have the composition percent (0.002% P, 0.288% Mn, 0.03% C, 0.0154% S, 0.0199% Cr, 0.002% Mo, 0.065% Cu, 0.0005% V) and the remainder iron. The mild steel was mechanically press-cut into disc shape with diameter (2.5 cm). These shapes were polished with paper to 2000 graides in order to get a surface. Then cleaned with distil water, alcohol, and finally acetone.

The treated specimens were store in a moisture free desiccator before they used in corrosion study. Mild steel was initially weight in an electronic balance. After that, the specimens were suspended and completely immersed in 250 ml beaker contain 1M sulphuric acid in the presence of inhibitor for 24 hours. The specimens were removed after 24 hours. exposure at room temperature, washes with water to remove any corrosion and finally washed with alcohol. Then they were dried and reweighed. Mass loss measure ements was performed as by ASTM methods described [22]. The tests were performed in guarantee the reliability of the results and the main values of the weight is reported. Weight loss allowed calculation of the mean corrosion rate in (mg cm^{-2} h^{-1}). The rate of corrosion of mild steel was determine by using the relations (2.1).

$$W = \frac{\Delta m}{St} \qquad \ldots(2.1)$$

where W is corrosion rates of mild steel, 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 (2.2) [23]:

$$E\% = \frac{W_{corr} - W_{corr(inh)}}{W_{corr}} \times 100 \qquad \ldots(2.2)$$

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

Basic of the information can be provided from the adsorption isotherms to explanation the interaction between metal surfaces and organic compounds. So, the degree of surface values (θ) at different inhibitors concentrations in 1M H_2SO_4 were achieved from weight lost measure ($\theta = E$ (%)/100) at room temperature and test with langmuir isotherms relations (2.3) [24]:

$$C/_{\theta} = 1/_{K_{ads}} + C \qquad \ldots(2.3)$$

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

The langmuir isotherm, K_{ads} value can calculate from the straight line of plotting (C/θ versus C). K_{ads} is relate to the stander free energy of adsorption, ΔG^{o}_{ads}, with the following equation (2.4) (the value 55.5 is the molar conce. of water in the solution in M).

$$K_{ads} = \frac{1}{55.5} \exp\left(\frac{\Delta G^{o}_{ads}}{RT}\right) \qquad \ldots(2.4)$$

3. Results and Discussion

In general, the characteristic stretching bonds of FTIR spectra to a Schiff base compounds (B1-B3) are (C=N) bonds. The stretching (C=N) and (C=O amide) bonds are usually lie within the range (1670-1661 cm^{-1}). There are other characteristic stretching bonds for compounds (B1-B3) , as (C–H) aromatic bond at (3046-3079 cm^{-1}) and as well as , the stretching (-NH_2) of iosnicotinic acid hydraziad is disappeared (3154-3363 cm^{-1}) in all the product compounds [25,26]. FTIR spectral data are shown in Table 4, and Figures 1 - 3.

Table 4. FTIR spectral data for prepared compounds (B1-B3).

Comp. No.	νC–H Aromatic	νC–H Aliphatic	νC=C Aromatic	ν(C=O and C=N Amide)	νN–H Amide
B1	3079	2951, 2870	1540	1670, 1610	3184
B2	3057	2949, 2870	1586	1687,1610	3163
B3	3046	2940, 2870	1561	1661, 1616	3154

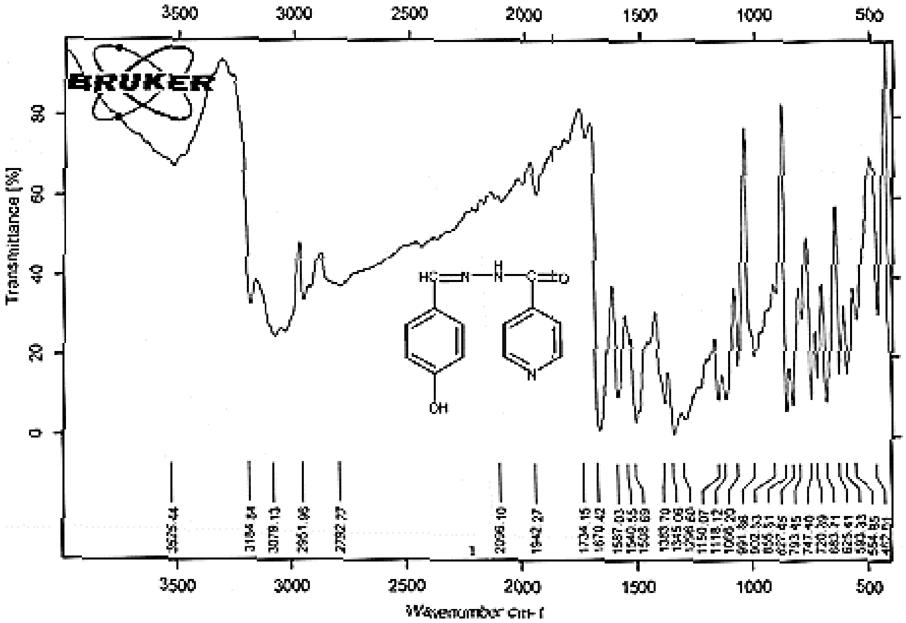


Figure 1. FTIR spectrum of compound (B1).

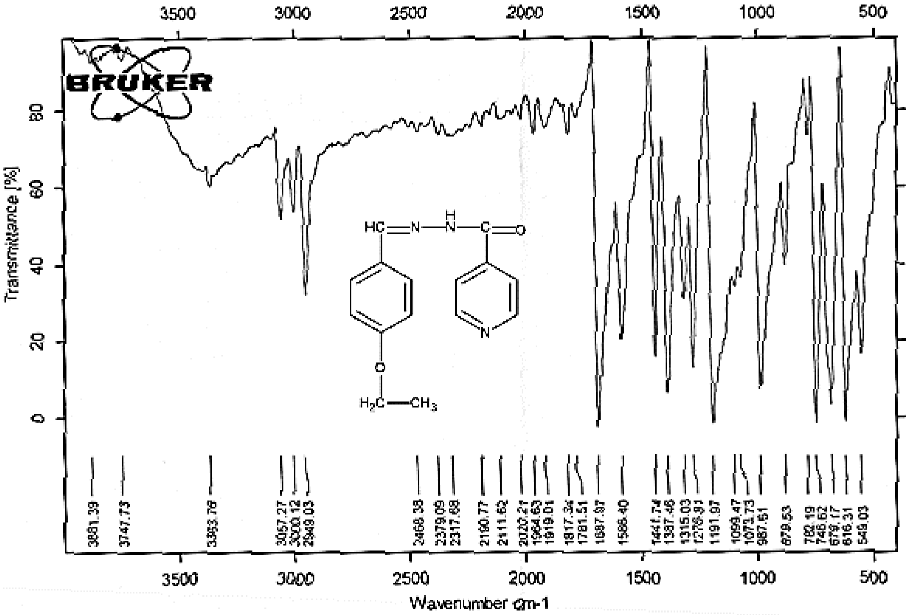


Figure 2. FTIR spectrum of compound (B2).

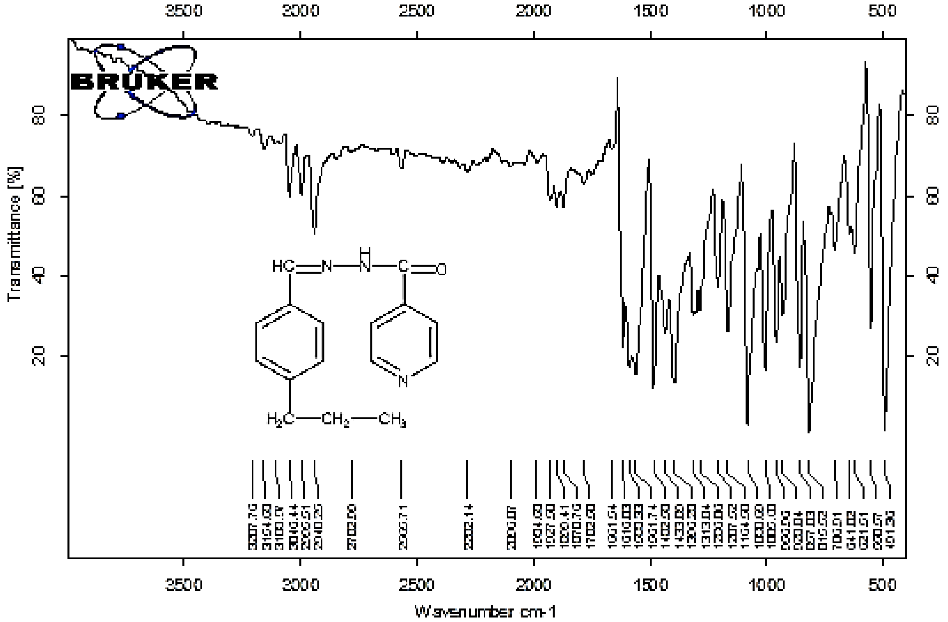


Figure 3. FTIR spectrum of compound (B3).

In general, the characteristic stretching bonds of FTIR spectra for pyridinium salts (A1-A3) are (C=N) bond. The stretching (C=N) bond of the compound (A1-A3) with in the range (1591-1670 cm^{-1}). There are others characteristic stretching bonds, as (C–H) aliphatic bond at (2905-2962 cm^{-1}). Also, (C=O) bond of nicotinaldehyde can be disappeared from the Schiff's base compounds (B1-B3) (27). FT-IR spectral data are shown in Table 4 and Figures 4 - 6 for pyridinium salts compounds (A1-A3).

Table 5. FTIR spectral data for prepared compounds (A1-A3).

Comp. No.	**νC–H Aromatic**	**νC–H Aliphatic**	**νC=C Aromatic**	**νN–H Amide**	**ν(C=O and C=N Amide)**
A1	3075	2962, 2890	1563	3210	1670, 1610
A2	3060	2905, 2890	1516	3233	1650, 1597
A3	3060	2926, 2890	1516	3230	1640, 1591

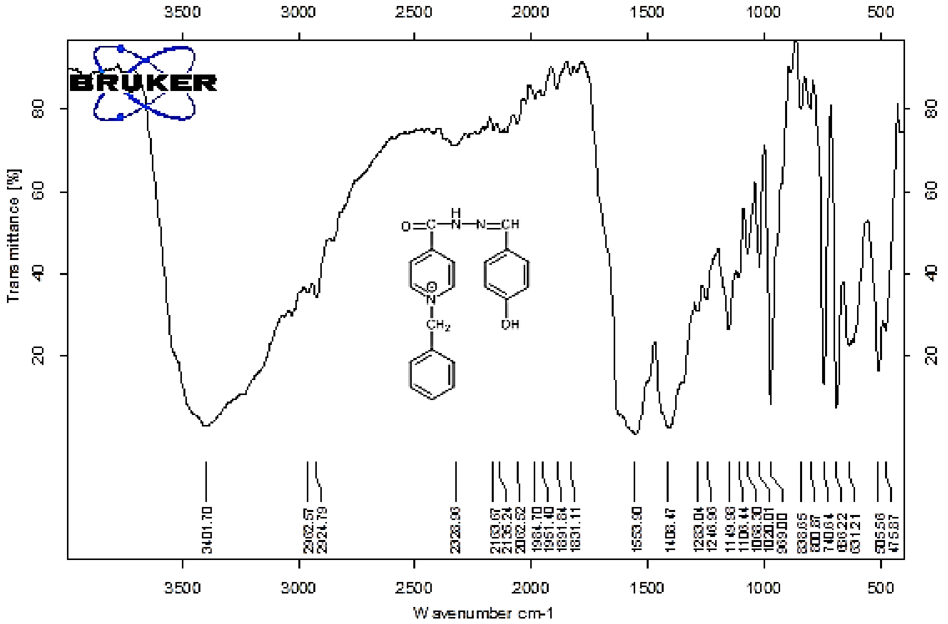


Figure 4. FTIR of compound (A1).

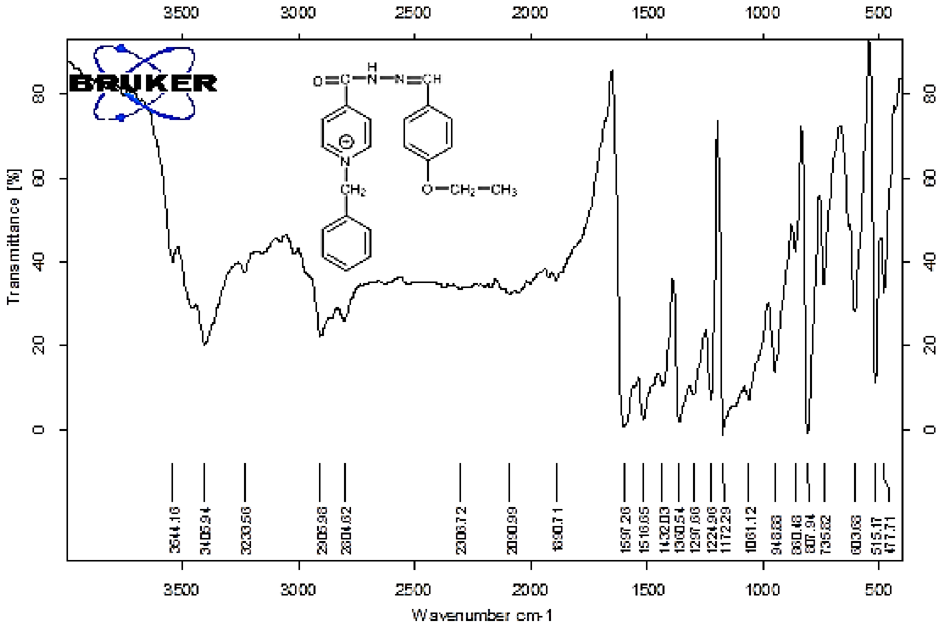


Figure 5. FTIR of compound (A2).

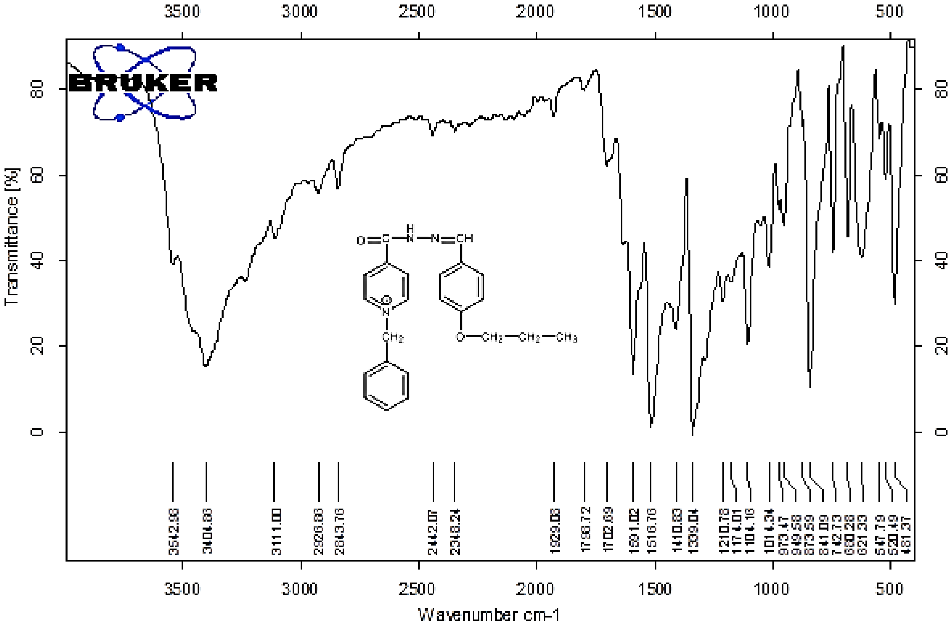
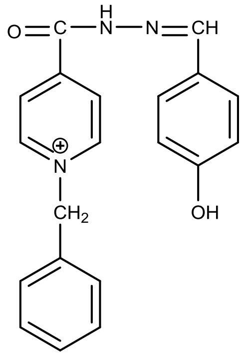
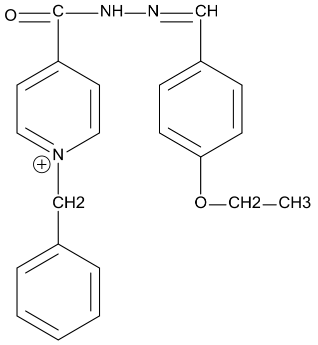
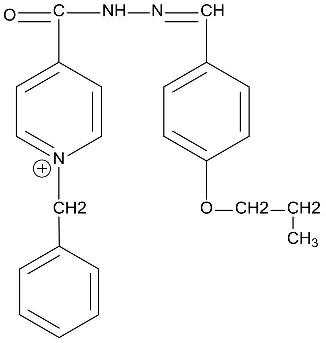
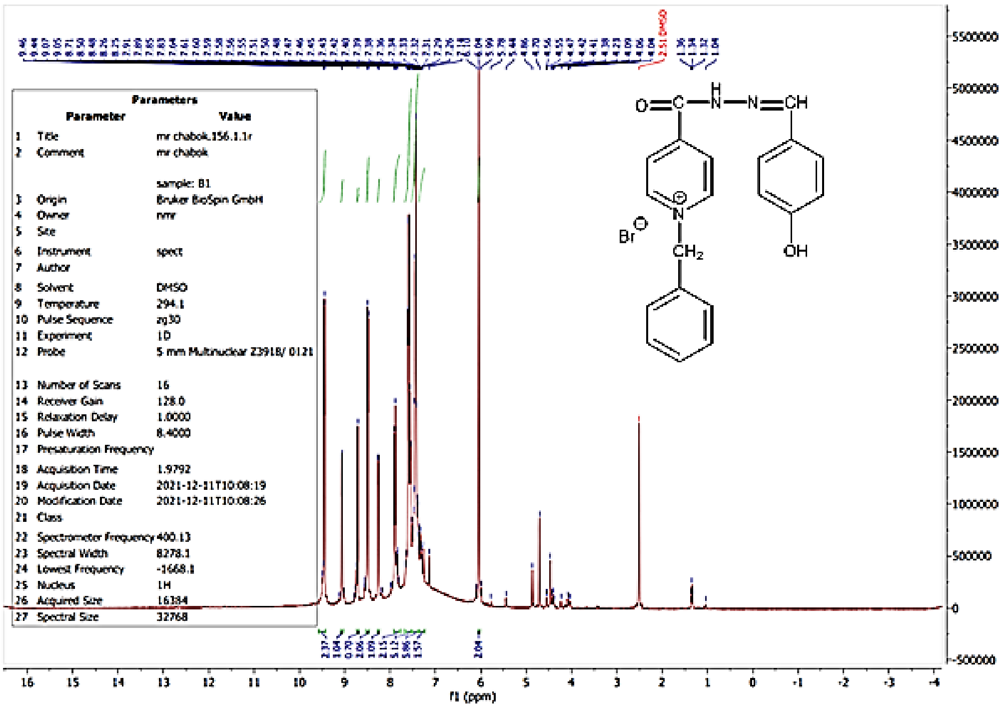


Figure 6. FTIR of compound (A3).

^{1}H-NMR spectra of prepared pyridinium salt derivatives (A1-A3) are also use for confirm the structure of finally product. Table 6 and Figures 7 - 9, show the following characteristic chemical shifts (ppm, δ) for pyridinium salt derivatives (A1-A3) in DMSO-d6 as a solvent [28].

Table 6. ^{1}H-NMR spectral data of prepared compounds (A1-A3) in ppm.

Comp. No.	**Compound Structure**	**^{1}H-NMR data of (δ-H) in ppm**
A1		^{1}H of N–H (–CO–NH– moiety) (9.6); 4H of pyridine-ring (8.5-9.1); ^{1}H of N=CH– (8.4); ^{9}H of phenyl- rings (7.1-8.2); ^{2}H of –N$^+$–CH$_2$ group (6.0)
A2		^{1}H of N–H (–CO–NH– moiety) (9.5); 4H of pyridine-ring (8.5-9.3); ^{1}H of N=CH– (8.4); 9H of phenyl-rings (7.1-8.3); ^{2}H of –N$^+$–CH$_2$ group (6.0); ^{2}H of –O–CH$_2$ group (4.0); 3H of CH$_3$ (1.0)
A3		^{1}H of N–H (–CO–NH– moiety) (9.5); ^{4}H of pyridine-ring (8.5-9.3); ^{1}H of N=CH– (8.4); 9H of phenyl-rings (7.1-8.3); ^{2}H of –N$^+$–CH$_2$ group (6.0); ^{2}H of –O–CH$_2$ group (4.0); ^{5}H of aliphatic chain (0.9-1.5)



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25	Nucleus	1H
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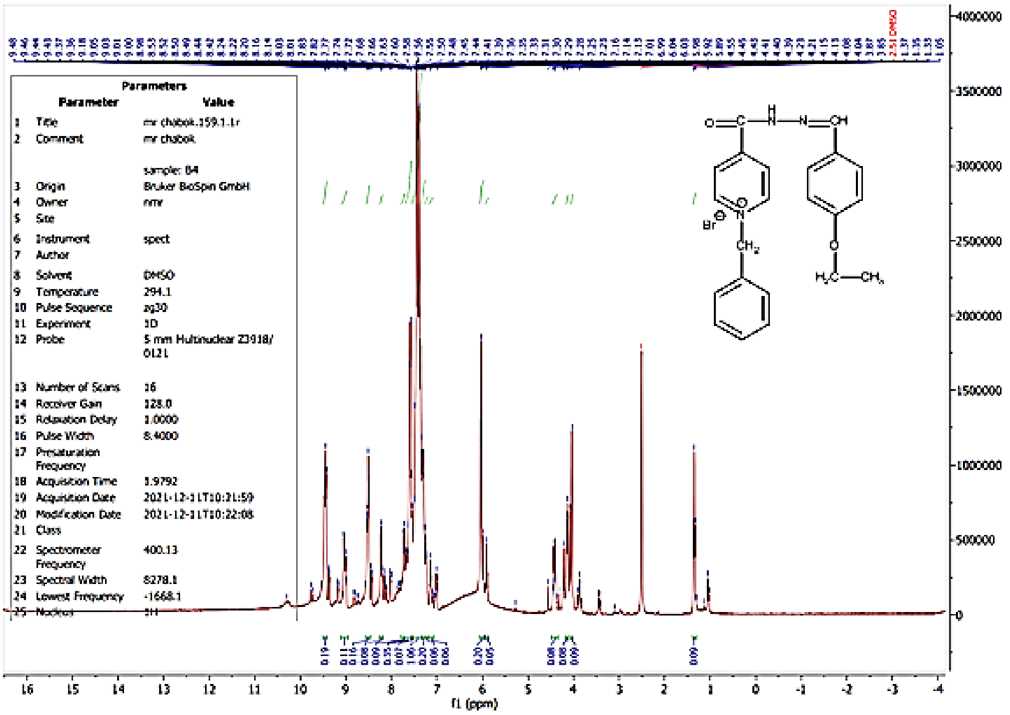
27	Spectral Size	32768

Figure 7. ^{1}H-NMR of compound (A1).



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12	Probe	5 mm Multinuclear Z3918/ 0121
13	Number of Scans	16
14	Receiver Gain	128.0
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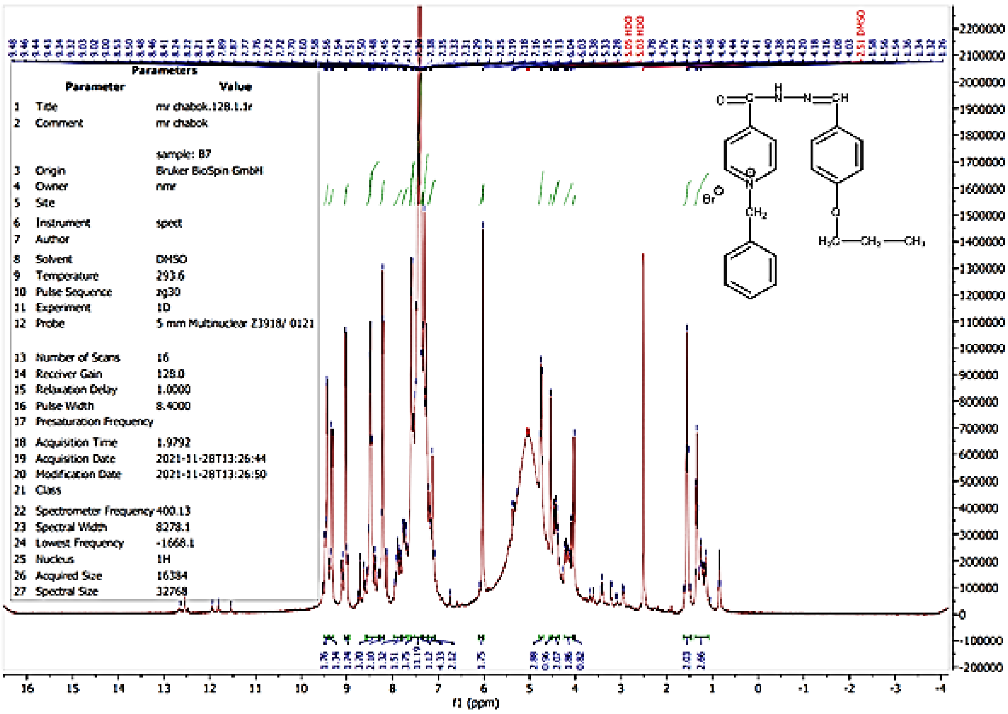
25	Nucleus	1H

Figure 8. ^{1}H-NMR of compound (A2).



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6	Instrument	spect
7	Author	
8	Solvent	DMSO
9	Temperature	293.6
10	Pulse Sequence	zg30
11	Experiment	1D
12	Probe	5 mm Multinuclear Z3918/ 0121
13	Number of Scans	16
14	Receiver Gain	128.0
15	Relaxation Delay	1.0000
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24	Lowest Frequency	-1668.1
25	Nucleus	1H
26	Acquired Size	16384
27	Spectral Size	32768

Figure 9. ^{1}H-NMR of compound (A3).

The rate corrosion results and efficiency inhibition which obtained by weight loss tests at variety concentrations of prepared salts (A1 to A3) within 24 hours. of immersing in 1 M solution of H_2SO_4 at room temperature that are summarizing in Table 7 and are illustrating in Figures 10 and 11.

Table 7. Corrosion rate.

Concentration (M)	**Corrosion rate (mg.cm^{-2} .h^{-1})**	**E%**	**θ**	**ΔG^o_{ads} (kJ. mol^{-1})**
Blank	0.3440	–	–	
(A1)				
0.0005	0.1769	48.576	0.4858	
0.001	0.1564	54.535	0.5453	–28.08 (R^2=0.9911)
0.005	0.0853	75.203	0.7520	
0.01	0.0298	91.337	0.9134	
(A2)				
0.0005	0.132	61.628	0.6163	
0.001	0.099	71.221	0.7122	–32.71 (R^2=0.9997)
0.005	0.0152	95.581	0.9558	
0.01	0.010	97.093	0.9709	
(A3)				
0.0005	0.0599	82.587	0.8259	
0.001	0.0293	91.064	0.9106	–33.32 (R^2=0.9993)
0.005	0.0277	92.366	0.9237	
0.01	0.0131	96.192	0.9619	

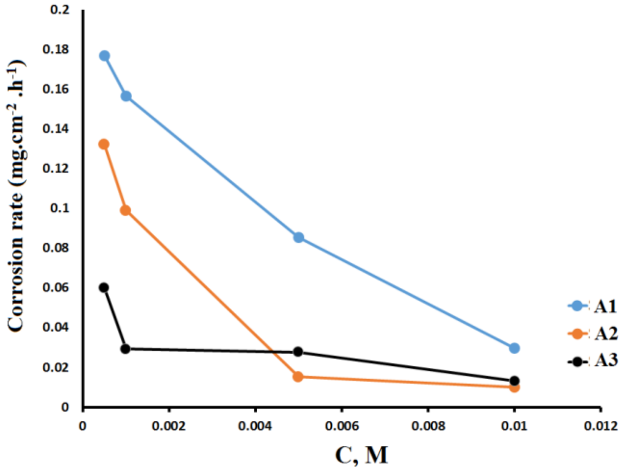

Figure 10. Effect of inhibitor concentration on corrosion rate of mild steel obtained at room temperature in 1 M H_2SO_4.

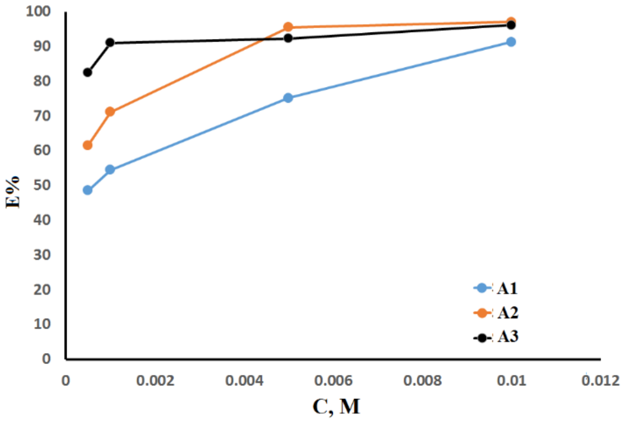

Figure 11. Effect of inhibitor concentration on IE % of mild steel obtained at room temperature in 1 M H_2SO_4.

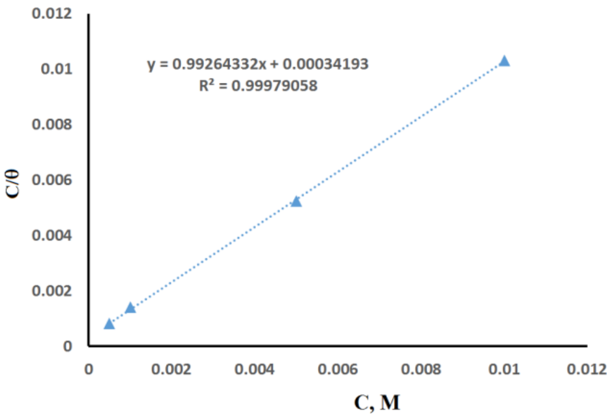

Figure 12. Langmuir adsorption plots for mild steel in 1 M H_2SO_4 solution at different concentration of A2.

Table 7 shows that as the inhibitor concentration is increased, the defense efficiency improves, with the highest inhibition efficiencies reached at 0.01 M. As a result of the comparison, the efficiency inhibition of (A1- A3) is roughly equivalent. The effect of molecular organic structure on inhibition efficiency may explain the in increasing in inhibition efficiency.

The adsorption free energy values (see Table 7 and Figure 12) show negative sign that is indicated the processadsorption of prepared inhibitors (A1-A3) are spontaneously process on metal surface of mild steel within 24 hours. of immersing and this is to give explaining to the natural interaction between metal surface and prepared inhibitors (A1-A3). From this point, the adsorbent molecule is approached nearby the metal surface, causing the electrons of organic molecules begin to be interfered with the empty orbitals of surface atoms causing the formation of metal-organic molecule complex [29-32].

4. Conclusions

The synthesized pyridinium derivative compounds (A1-A3) were used successfully as corrosion inhibitor on the mild steel surface in 1M H_2SO_4 aqueous solution at room temperature. The results of inhibitor efficiency (E%) showed interesting inhibitive effects of inhibitors. The free energy value of absorption revealed physisorption effect for (A1-A3) gave useful information to explain the interaction between the surface of metal and the organic molecules.

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