



## New organic ammonium salts as corrosion inhibitors for mild steel in saline solution



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### Abstract

New organic ammonium salts, namely: 4-oxo-4-(2-(pyridin-4-yl)hydrazineyl)but-2-enoate tetraethyl ammonium, B1, 2-(2-(pyridin-4-yl)hydrazine-1-carbonyl)benzoate tetraethyl ammonium, B2, 4-oxo-4-(2-(pyridin-4-yl)hydrazineyl)butanoate tetraethyl ammonium, B3, 5-oxo-5-(2-(pyridin-4-yl)hydrazineyl)pentanoate tetraethyl ammonium, B4, were successfully synthesized and confirmed structures by spectral techniques. The new organic ammonium salts were used for mild steel as a protective corrosion inhibitor. Organic salts were tested with 3.5% saline solution for 24 hours at room temperature and carried out with the method of weight loss. The inhibition efficiency results for organic inhibitors (B1-B4) were increased with increasing the concentrations of organic ammonium salts and that also accompanied with the corrosion rate to be decreased. By increasing the inhibition efficiency, the coverage surface degree factor was increased. The values of thermodynamic parameter (free energy) were predicted the physisorption effect with all organic ammonium salts (B1-B4).

**Keywords:** Ammonium salts, adsorption phenomenon, weight loss method.

### 1. Introduction

Because of its fairly high strength, both low cost and wide range accessibility, significant infrastructure components like storage tanks, and the majority of the mild steel used in steel structures and pipelines [1]. Corrosion is one of the primary causes of depreciation for such valuable products, especially in environments like those found in acidic and saline environments, for example, are aggressive media. Inhibitors typically create protective layer of coatings on the surface of metal in order to slow down the corrosion processes [2]. Different researches were reported for applying of the organic inhibitors for corrosion, which was provided to control or prevent the harmful defect of corrosion of the surface of mild steel within saline media [3], [4]. Usually, the use of inhibitors as a thin layer like a kind of protective mechanisms to reduce the corrosion phenomena is basically depended on the nature of the molecular structure of inhibitor which could make adsorption on the surface of metal and that caused protection effects [5-7]. Many sorts of protective inhibitors could be provided as useful tools to inhibit or prevent corroding processes within various corrosive electrolytes concerning iron metal [8-10].

In the present work, we did report the effects of saline solution on the mild steel corrosion surface in the absence and presence of organic ammonium salts (B1-B4) (see Figure 1). We determined the inhibition performance of each organic inhibitors (B1-B4) by applying weight loss investigating. The weight loss test as a simple mean to show how much protective efficiency of organic salts are effect as an inhibitor on the corrosion process or corrosion rate of mild steel in saline media

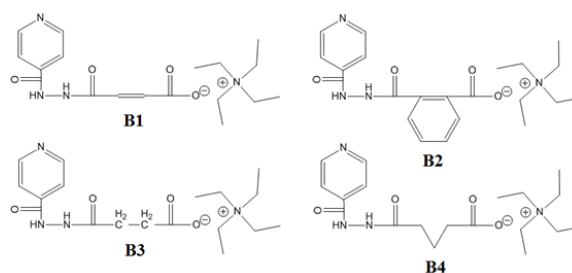


Figure 1. New organic ammonium salts (B1-B4)

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## 2. Experimentals

### 2.1 Chemicals

All chemical reagents and organic solvents were used directly without purifications.

### 2.2 Instrumentation

The new organic salts were confirmed structures by Bruker/FTIR spectrophotometry and has wave number range (4000-400cm<sup>-1</sup>). Bruker /1H-NMR was used by applying (DMSO/d6) solvent.

### 2.3. General method for synthesis of tetraethyl ammonium salts

The synthesized tetraethyl ammonium salts had names: 4-oxo-4-(2-(pyridin-4-yl)hydrazineyl)but-2-enoate tetraethyl ammonium, B1, 2-(2-(pyridin-4-yl)hydrazine-1-carbonyl)benzoate tetraethyl ammonium, B2, 4-oxo-4-(2-(pyridin-4-yl)hydrazineyl)butanoate tetraethyl ammonium, B3, 5-oxo-5-(2-(pyridin-4-yl)hydrazineyl)pentanoate tetraethyl ammonium, B4. These organic ammonium salts (B1-B4) were synthesized as below :

A mixture of isonicotinic acid hydrazide (0.01 mol) with (maleic anhydride, phthalic anhydride, succinic anhydride or glutaric anhydride) (0.01 mol) in 15 mL of THF as a solvent was stirred for four hours at room temperature to obtain amic acid compounds (A1-A4). Then left the mixture 24 hours at room temperature. The crude compound that obtained was filtered, then washed two times by 5 mL THF solvent. Then, equivalent molar of each amic acid (A1-A4) and tetraethyl ammonium hydroxide (35%) was mixed with 5 mL CH<sub>3</sub>OH as a solvent and stirred at room temperature for 3 hours. The mixture was left for 48 hours at 37 °C. The final organic salt (B1-B4) was washed with 5 mL of methanol two times then dried.

- A1, 4-oxo-4-(2-(pyridin-4-yl)hydrazineyl)but-2-enoic acid, mp: 157-160°C, Yield %:87, yellow powder, FTIR data (cm<sup>-1</sup>): N-H amide 3369, =C-H olefinic 3120, =C-H aromatic 3039, -C=O carboxylic 1702, -C=O amide 1648.

- A2, 2-(2-(pyridin-4-yl) hydrazine-1-carbonyl) benzoic acid, mp: decomp. at 170°C, Yield %:76, yellow powder, FTIR data (cm<sup>-1</sup>): N-H amide 3366, =C-H aromatic 3068, -C=O carboxylic 1700, -C=O amide 1640.

-A3, 4-oxo-4-(2-(pyridin-4-yl) hydrazineyl) butanoic acid, mp: 192-195°C, Yield %:95, yellow powder, FTIR data (cm<sup>-1</sup>): N-H amide 3331, -C-H aliphatic 2939, 2810, =C-H aromatic 3101, -C=O carboxylic 1729, -C=O amide 1679.

- A4, 5-oxo-5-(2-(pyridin-4-yl) hydrazineyl) pentanoic acid, mp: 150-152°C, Yield %:95, yellow powder, FTIR data (cm<sup>-1</sup>): N-H amide 3377, -C-H aliphatic 2944, 2810, =C-H aromatic 3068, -C=O carboxylic 1710, -C=O amide 1644.

- B1, 4-oxo-4-(2-(pyridin-4-yl)hydrazineyl)but-2-enoate tetraethyl ammonium, bp: 167°C, Yield %:89, yellow liq., FTIR data (cm<sup>-1</sup>): -N-H amide 3398, -C-H aliphatic 2982, 2880, =C-H olefinic and aromatic 3100, 3070, -C=O carboxylate 1721, -C=O amide 1677, 1H-NMR data (ppm): N+(12H, -CH<sub>3</sub>, m, 1.1; 8H, -CH<sub>2</sub>, m, 3.1), unsaturated alkane (2H, d, 5.8, 6.1), pyridine ring (4H, m, 6.2-7.7), -N-H amide (2H, s, 7.8)

- B2, 2-(2-(pyridin-4-yl)hydrazine-1-carbonyl) benzoate tetraethyl ammonium, bp: 244°C, Yield %:87, yellow liq., FTIR data (cm<sup>-1</sup>): -N-H amide 3435, -C-H aliphatic 2998, 2844, =C-H aromatic 3071, -C=O carboxylate 1716, -C=O amide 1633, 1H-NMR data (ppm): N+(12H, -CH<sub>3</sub>, m, 1.1; 8H, -CH<sub>2</sub>, m, 3.1), pyridine and aromatic rings (8H, m, 6.9-7.9), N-H amide (2H, s, 8.8).

- B3, 4-oxo-4-(2-(pyridin-4-yl)hydrazineyl) butanoate tetraethyl ammonium, bp: 219°C, Yield %:93, yellow liq., FTIR data (cm<sup>-1</sup>): -N-H amide 3426, -C-H aliphatic 2977, 2841, =C-H aromatic 3043, -C=O carboxylic 1700, -C=O amide 1652, 1H-NMR data (ppm): N+(12H, -CH<sub>3</sub>, m, 1.1; 8H, -CH<sub>2</sub>, m, 3.1), alkane (4H, m, 2.2, 2.5), pyridine ring (4H, m, 6.5-7.7), N-H amide (2H, s, 11.0).

- B4, 5-oxo-5-(2-(pyridin-4-yl)hydrazineyl) pentanoate tetraethyl ammonium, bp: 223°C, Yield %:90, brown liq., FTIR data (cm<sup>-1</sup>): -N-H amide 3433, -C-H aliphatic 2987, 2841, =C-H aromatic 3084, -C=O carboxylate 1737, -C=O amide 1660, 1H-NMR data (ppm): N+(12H, -CH<sub>3</sub>, m, 1.1; 8H, -CH<sub>2</sub>, m, 3.1), alkane group (6H, m, 2.1-2.5), pyridine ring (4H, m, 6.6-7.9), N-H amide (2H, s, 8.2).

### 2.4. Preparation of aggressive solution

Analytical grade NaCl with distilled water was dissolved to yield an aggressive solution of 3.5% saline solution. The inhibitor concentrations in between (5x10<sup>-4</sup> – 1x10<sup>-2</sup> M) were achieved with 3.5% saline solution.

### 2.5 Weight loss measurements

The mild steel specimen has composition: 0.002% P, V, 0.002 % Mo, 0.03 % C, 0.0154 % S, 0.288 % Mn, 0.065 % Cu, 0.0199 % Cr, and 0.0005 % and remaining Fe element. The mild steel sheet was cut to a disk shape (with diameter 2.5 cm). These disc shapes were treated to have smooth surface, by using different emery paper grades. Then the smooth surface specimens were washed by distilled water, and then with alcohol, finally washed with acetone. The freshly cleaned specimens have been placed in a proper desiccator that containing a drier. For using weight loss method, firstly, the specimen disc is placed in the electronic balance for fixing the weight. Second step, the specimen disc is immersed

completely in a proper vessel containing 3.5% saline solution without and with presence organic inhibitor that has known concentration. Then the content vessel is left for 24 hours at room temperature. After that, the specimen disc is removed, and washed with distilled water and followed by acetone. The specimen disc is reweighed again. The weight loss test was achieved according to ASTM investigation [11]. The test of weight loss was repeated two times to find an average value. The results of weight loss test were used to determine the average corrosion rate (mgcm<sup>-2</sup>h<sup>-1</sup>). The average rate of corrosion for mild steel tests could be calculated by the formula (1) [12]:

$$W = \Delta m / St \dots \dots \dots (1)$$

Where  $W$  = corrosion rate (mg cm<sup>-2</sup> h<sup>-1</sup>),  $\Delta m$  = weight loss difference (mg) before and after specimen immersion,  $S$  = area of specimen (cm<sup>2</sup>) and  $t$  = time of immersion (hrs).

The inhibition efficiency (IE%) could be calculated by using the formula (2) [13]:

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

### 3. Results and discussions

**3.1. Experimental Investigation,** The preparation of tetraethylammonium salts (B1-B4) of the present work were involved synthesis of the amic acid compounds (A1-A4) by using isonicotinic acid hydrazide by reacting with a proper acid anhydride. Then the amic acids achieved a reaction of acid-base with tetraethylammonium hydroxide to prepare tetraethylammonium salt compounds (B1-B4). The spectral data of FT-IR for amic acid compounds (A1-A4) was shown clear new stretching bands for (N-H and C=O amide), as well as, clear new stretching bands for (C=O carboxylate) for synthesized compounds (B1-B4) and the 1H-NMR spectral data was shown clear peaks at (1.1 and 3.1 ppm) for (C-H) of tetraethylammonium fragment of compounds (B1-B4).

**3.2. Corrosion investigation,** the deterioration of mild steel was determined at room temperature and after 24 hrs immersing in 3.5% aqueous NaCl solution. The results of (corrosion rate and inhibition efficiency) were determined by using the tests of weight loss measurements with different concentrations of tetraethylammonium salts (B1-B4) that are shown in Table 1.

Table 1 is revealing that by increasing the concentration of inhibitor (B1-B4), the efficiency of inhibition increases, which shows at 0.01 M the highest efficiencies of inhibition. The order of the inhibition efficiency (B4>B3>B1>B2) would be comparable. The effects of molecular organic structures (flexible structure with B4 and rigidity with B1, B2 and B3) on inhibition efficiency maybe

explained by values of the inhibition efficiency [14], that means the successful adsorption process of organic inhibitor molecules on the mild steel surface. Basic informations of method adsorption can give to elutriate the natural interacting between organic inhibitor molecules and metal surface. Therefore, the value of coverage surface degree, ( $\theta$ ), with different inhibitor concentrations was achieved by weight loss method with 3.5% aqueous NaCl ( $\theta = IE(\%)/100$ ) (see Table 1) at room temperature [15]:

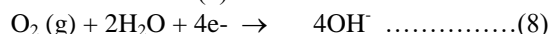
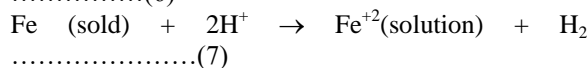
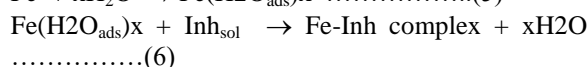
$$C/\theta = 1/K_{\text{ads}} + C \dots \dots \dots (3)$$

Where:  $C$  (concentration, M),  $K_{\text{ads}}$ , equilibrium constant of adsorption process (M<sup>-1</sup>).

By applying the Langmuir isotherm, that value  $K_{\text{ads}}$  is estimated from straight line intersection of  $C$  versus  $C/\theta$ . The values of  $\Delta G_{\text{ads}}^{\circ}$  was determined by equation (4) [15]: (55.5 value: water molar concentrations, M).

$$K_{\text{ads}} = 1/55.5 \exp(-(\Delta G_{\text{ads}}^{\circ})/RT) \dots \dots \dots (4)$$

Table 1 is shown values of free energy for adsorption process have negative sign to reveal that processes of adsorption for (B1-B4) are spontaneously processes on the surface of mild steel in 24 hrs of immersing in 3.5% aqueous NaCl solution at room temperature and that is given a clear picture that the effective interaction between organic molecules (B1-B4) and metal surface. In addition, organic inhibitor molecules can be moved into and approach the surface of metal to interfere organic molecules electrons with available atomic empty orbitals on the surface of metal [16,17], as well as, interfering the retro-donation mechanism [18]. From above, the adsorption of organic inhibitor molecules (by donor atoms) on surface of metal can be caused formation a type of complexation between organic inhibitor molecules and the metal surface, as shown in Equations below (5,6) [19, 20]:



Consequently, the complex of metal-inhibitor can be served like a thin layer film on anode sites, to reduce or prevent formation of Fe<sup>2+</sup> ions (see Equis. 7,8). It is worthy to mention, that the metal-inhibitor complex amount on the surface of metal is decreased with decreasing the organic inhibitor concentration. The adsorption mechanism of inhibitor molecules is explained by the effective of organic molecules via electrons of nitrogen and oxygen atoms that organic molecular inhibitor are containing. It is reported that

adsorption process could be affected by the functional groups in structural molecule via changing electrons density of molecule [21]. The organic inhibitor salts (B1-B4) that adsorb on surface of mild steel via formation of thin layer complex can be blocked the active sites of anode that caused to prevent or reduce the reaction of electrochemical dissolutions that causes losing process of metal atoms from its surface.

The values of  $\Delta G_{\text{ads}}^{\circ}$  for organic inhibitors (B1-B4) are shown in Table 1 revealed adsorption process that carries out electrostatically and physically, by the interactions between the neutral and charged centers on the metal surface electrode and the organic inhibitor molecules [22]. The organic inhibitor (B3) showed value  $\Delta G_{\text{ads}}^{\circ} = -33$  kJ/mol, that verified with

IE% values (69-82) with different inhibitor concentrations, as listed in Table 1. At the highest concentration of inhibitor, IE% value of (B4) is higher than IE% values for inhibitors (B1-B3), but the values of  $\theta$  Goads, follows the order  $B3 > B2 > B4 > B1$ . We believe the reason behind is molecular structure of variant carbon chain between the two carbonyl groups and the spatial geometry of (B1-B4) that cause the electron density around the amide group and acetate group to be differed in intensity. Therefore, the interactions between the surface of metal and the organic inhibitor molecule (B3) are to be improved by the effective distributions of electrons density of inhibitor molecules with the metal surface that comparing with inhibitor molecules (B1, B2 and B4).

Table 1. Rate of corrosion, inhibition efficiency (IE%), surface coverage ( $\theta$ ) and  $\Delta G_{\text{ads}}^{\circ}$  for test of mild steel in 3.5% aqueous NaCl solution with weight loss method for 24 hrs and at room temperature.

Concentrations (M)	Rate of corrosion (mg.cm <sup>-2</sup> .h <sup>-1</sup> )	IE%	$\theta$	$\Delta G_{\text{ads}}^{\circ}$ (kJ.mol <sup>-1</sup> )
Blank	0.1225			
<b>B1</b>				
1 × 10 <sup>-2</sup>	0.0089	0.8169	81.69	-30.47
5 × 10 <sup>-3</sup>	0.0102	0.7901	79.01	(R <sup>2</sup> =0.9972)
1 × 10 <sup>-3</sup>	0.0110	0.7737	77.37	
5 × 10 <sup>-4</sup>	0.0119	0.7551	75.51	
<b>B2</b>				
1 × 10 <sup>-2</sup>	0.0102	0.7901	79.01	-31.85
5 × 10 <sup>-3</sup>	0.0119	0.7551	75.51	(R <sup>2</sup> =0.9991)
1 × 10 <sup>-3</sup>	0.0132	0.7284	72.84	
5 × 10 <sup>-4</sup>	0.0144	0.7037	70.37	
<b>B3</b>				
1 × 10 <sup>-2</sup>	0.0085	0.8251	82.51	-32.85
5 × 10 <sup>-3</sup>	0.0123	0.7469	74.49	(R <sup>2</sup> =0.9997)
1 × 10 <sup>-3</sup>	0.0132	0.7284	72.84	
5 × 10 <sup>-4</sup>	0.0149	0.6934	69.34	
<b>B4</b>				
1 × 10 <sup>-2</sup>	0.0081	0.9609	96.09	-31.53
5 × 10 <sup>-3</sup>	0.0085	0.9588	95.88	(R <sup>2</sup> =0.9999)
1 × 10 <sup>-3</sup>	0.0102	0.7901	79.01	
5 × 10 <sup>-4</sup>	0.0144	0.7037	70.37	

### 3.3. The corrosion inhibition mechanism

Finally, the corrosion inhibition mechanism for this work is to be physical characteristics on mild steel surface. We suggested that the changing electronic environment of the metal surface may be used to explain adsorption process depended on theoretical calculations and the experimental study. Figure 2 shows the inhibition effect of the organic ammonium salt molecule (B1) on the surface of mild steel. Some studies revealed that the surface of metal has electrons deficient in acidic media [23]. The inhibitor molecules with presence of active function groups are capable for interacting with the metal surface that illustrates the tendency of inhibitor

molecule to be adsorbed on the surface of metal. When the concentration of inhibitor is increased, that a protective layer of inhibitor molecules is formed at the solution/metal interface which inhibition effectiveness can be improved. The change of the electronic environment for iron surfaces in solution that encourages organic inhibitor molecules to be adsorbed, and resulting to form a thin layer of organic inhibitor molecules on iron surface. The organic salt derivatives (B1-B4) with the unshared electron pairs of the O and N atoms are interacted with mild steel surface in solution. Physisorbed effect on the metal surface can be formed via electrostatic interaction with active sites on organic inhibitor molecules [24]. The organic inhibitor molecule maybe adsorbed on

the metal surface in solution can be predicted with an imaginary Figure 2 includes: (a) physisorption electrostatic interaction of inhibitor molecules with adsorbed ions, (b) chemisorption or physisorption via unshared electron pairs of heteroatoms and charged atoms and empty d-orbital of iron atoms, and (c) retro-donation via interaction of d-electron of iron atom to the empty inhibitor molecule orbital.

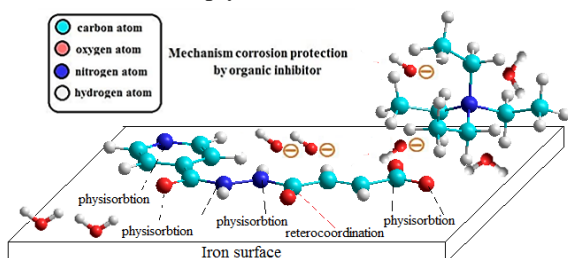


Figure 2. Suggested mechanism of interaction between inhibitor molecules (B1) and mild steel surface in solution.

#### 4. Conclusion

Tetraethyl ammonium organic salts (B1– B4) were successfully synthesized and confirmed their structures using spectral techniques. They applied as corrosion organic inhibitor molecules for mild steel surface in solution of aqueous saline for 24 hrs at room temperature. The results of efficiency inhibition (IE%), were revealed considerable inhibitory effects of the prepared tetraethyl ammonium organic salts as corrosion organic inhibitor molecules for mild steel. The values of free energy adsorption were predicted physical process adsorption for compounds (B1, B2, B3 and B4) on the metal surface and given important data to explain the natural interaction between the organic inhibitor molecules and the surface of mild steel.

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#### Conflicts of interest

There is no conflict to be declared

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