



Synthesis New Triethylammonium Salts as Corrosion Inhibitors for Mild Steel in 1 M H_2SO_4

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Article's Information	Abstract
Received: 09.02.2023 Accepted: 07.05.2023 Published: 30.06.2023	Some new triethylammonium salts, namely triethylammonium 4-(cyclohexyl- amino)-4-oxobut-2-enoate(B1), triethylammonium 5-(cyclohexylamino)-5- oxopentanoate (B2), triethylammonium 4-(cyclohexylamino)-4-oxobutanoate (B3) were synthesized as a corrosion inhibitor for mild steel. These salts could test in one molarity sulfuric acid at room temperature for 24 hours by using weight of losing method. The results revealed that the efficiency of inhibition of all triethylammonium salts were high at the maximum concentration of
Keywords: Triethylammonium salts Corrosion reaction Surface of mild steel Adsorption process	triethylammonium salt. The increase concentration of inhibitors is associated with decreasing the rate of corrosion, but the increasing of efficiencies of inhibition, and a greater degree of surface covering. Adsorption values of the free energy were used to illustrate the effects of physisorption for (B1, B2, and B3), which was helpful in forecasting the natural interaction between organic corrosion inhibitor molecules and the metal surface of steel.

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1. Introduction

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Corrosion inhibitors have been thoroughly researched in a range of industries to reduce the rate of corrosion of metal surfaces in contact with hostile environments [1-3]. Acidic solutions are often applied in a variety of sectors, including petrochemical operations, oil-well acidification, industrial cleaning, acid rescaling, and acid pickling [4].

One of the most effective ways to prevent corrosion, particularly in acid solutions to stop metal evaporation and acid consumption, is to use inhibitors [5].

Many industrial processes employ mild steel in acid solutions, and corrosion of mild steel is known to happen in this setting.

The use of organic inhibitors is one of the most efficient ways to stop corrosion [6].

The topic of mild steel corrosion inhibitors in acidic environments has been the focus of several scientific investigations [7–12].

Surfactant efficiency as corrosion inhibitors has recently been extensively researched in various media and for the protection of multiple metals, leading to studies on organicbased corrosion inhibitors [13-15]. Gemini surfactants, which are new generation quaternized salt surfactants composed of two surfactant monomers joined together by a spacer group, have been thoroughly investigated in corrosion investigations [16,17]. For instance, innovative cationic Gemini surfactants produced for C-steel pipeline corrosion inhibition in 1 M HCl media [18] shown a potent inhibition effectiveness. Additionally, the impact of a long spacer chain on enhancing surface coverage was demonstrated by three cationic Gemini surfactants with different spacer lengths, namely G-12, G-6, and G-2 [19]. Additionally, in oil well formation water with existing sulfide ions, additional cationic Gemini surfactant compounds demonstrated corrosion inhibition efficiency between 76 to 81% at 300 ppm [20]. Inhibition of mild steel corrosion by new green surfactants made from erucic acid was examined in a 15% HCl solution [21]. At 90 °C, they had a 98% inhibitory efficiency. In a solution containing 3.5% NaCl and 0.5 M HCl at temperatures between 30 and 60 °C, cationic surfactants produced with a Schiff base group achieved 95% protection efficacy for C-steel [22]. The goal of the current investigation was to determine the effectiveness of certain triethylammonium salts (see Figure 1) as corrosion inhibitors for mild steel that would be tested in 1M sulfuric acid at room temperature for 24 hours.

ANJS, Vol.26 (2), June, 2023: 6-18



Figure 1. The formula of prepared inhibitor molecules.

2. Experimental

2.1 Materials:

Reagents and solvents were both utilized in their identical original form.

2.2 Instrumentation:

All there were prepared organic compounds measured by Brucker Company FTIR spectrophotometry with range of the wave number (4000-400 cm⁻¹) and ¹H-NMR was applied by using (DMSO-d6) as a solvent. The melting points were calculated using the open capillary technique employing apparatus of Gallenkamp hot stage, but all the measurements were usually uncorrected. The mild steel specimen's composition was determined by SPECTROMAX device (2009, Germany) in Ministry Industry and Materials/State Company for Inspections and Rehabilitation.

2.3 General procedure for synthesized triethylammonium salts:

The synthesis of triethylammonium salts were consisted two steps: namely triethylammonium 4-(cyclohexylamino)-4-oxobut-2-enoate (B1), triethylammonium 5-(cyclohexylamino)-5-oxopentanoate (B2), triethylammonium 4-(cyclohexylamino)-4-oxobutanoate (B3), were synthesized as follow:

A mixture of each compound cyclohexyl amine (0.01 mol) with (0.01 mol) of each (maleic anhydride, glutaric anhydride, succinic anhydride) in 10 mL of THF at room temperature was stirred for four hours to formation amic acids (A1-A3). Then left the mixture 24 hours at 25 °C. The sold compound was filtered, precipitated, and cleaned with 5 mL of THF two times. In the next step, (0.01 mol) of each amic acid and triethylamine in 15 mL methanol was stirred 1 hour at room temperature. Then left the mixture 24 hours at room temperature.

and washed with 5 mL of methanol two times. Tables 1 and 2 show structural data of prepared compounds.

Al-Nahrain Journal of Science ANJS, Vol.26 (2), June, 2023: 6-18

Table 1. Structural data of prepared compounds.								
Compounds	Name	MW, (g/mol)	Color	M.P. °C	Yield %	FTIR data, v, cm ⁻¹		
A1	4-((4-aminocyclohexyl) amino)-4-oxobut-2-enoic acid	212.25	Yellow	158-160	92	C-H aliphatic 2925,2850 C-H olefinic 3050 C=O carboxylic 1664 C=O amide 1637 N-H amide 3249		
A2	5-((4-aminocyclohexyl) amino)-5-oxopentanoic acid	286.13	White	145-147	74	C–H aliphatic 2925,2853 C=O carboxylic 1700 C6O amide 1635 N–H amide 3330		
A3	4-oxo-4-(phenylamino) butanoic acid	193.20	Off- white	159-161	70	C–H aliphatic 2925,2855 C=O carboxylic 1690 C=O amide 1636 N–H amide 3242		
B1	triethylammonium 4-((4- aminocyclohexyl) amino)- 40xobut-2-enoate	313.44	Brown	179-181	87	C-H aliphatic 2929,2855 C-H olefinic 3050 C=O carboxylic 1660 C=O amide 1631 N=H amide 3296 3400 for -N ⁺ -H		
B2	triethylammonium 5-((4- aminocyclohexyl) amino)- 50x0pentanoate	329.48	Yellow	219-221	65	C–H aliphatic 2930,2854 C=O carboxylic 1738 C=O amide 1626 N–H amide 3278 3437 for –N ⁺ –H		
B3	triethylammonium 4-((4- aminocyclohexyl) amino)- 40xobutanoate	315.46	White	220-218	84	C-H aliphatic 2930,2854 C=O carboxylic 1704 C=O amide 1634 N-H amide 3289 3436 for -N ⁺ -H		

Table 2. The ¹H-NMR spectral structural data in ppm for compounds (B1 to B3).

B1	1H of N-H (-CO-NH-) (s, 8.1); 2H of H-N ⁺ - (s, 10.9); 2H of CO -CH=CH-CO (d, 5.8, 6.1); 1H of – NH-CH- (m, 3.5); 6H of –N ⁺ -CH ₂ (m, 3.0); 9H of CH ₃ (m, 1.5); 10H of CH ₂ (m, 1.0-1.7);
B2	1H of N-H (-CO-NH-) (s, 7.7); 1H of H-N ⁺ - (s, 7.4); 6H of CO-CH ₂ -CH ₂ -CH ₂ -CO (m, 2.2-2.7); 1H of –NHCH- (m, 3.9); 6H of –N ⁺ -CH ₂ (m,3.5); 9H of CH ₃ (m, 1.5); 10H of CH ₂ (m, 1.0-1.7);
В3	1H of N-H (-CO-NH-) (s, 7.7); 1H of H-N ⁺ - (s, 7.2); 4H of CO-CH ₂ -CH ₂ -CO (m, 2.2); 1H of –NH-CH- (m, 3.9); 6H of – N ⁺ -CH ₂ (m, 3.5); 9H of CH ₃ (m, 1.5); 10H of CH ₂ (m, 1.01.7);

ANJS, Vol.26 (2), June, 2023: 6-18

2.4. Preparation of aggressive solution:

Dilution of analytical grade (98%) H_2SO_4 with distilled water yielded an aggressive solution of 1M H_2SO_4 . The inhibitor concentrations ranging between (0.0005-0.01 M) were achieved with 1M H_2SO_4 aqueous solution.

2.5 Weight loss measurements:

Composition: 0.288% Mn, 0.0154 % S, 0.03 % C, 0.0199 % Cr, 0.065 % Cu, 0.002 % P, 0.0005 % V, 0.002 % Mo. The mild steel sheet was being formed into a disk (2.5 cm as diameter). These disc forms were polished using energy paper of a grade 2000 to obtain a flawless surface. They then disinfected with distilled water, alcohol and acetone. The treated specimens were dried out in a desiccator before being utilized in corrosion testing. The mild steel disc is initially weighted using an electronic balance. In both the absence and presence of the known concentration of produced organic inhibitor, the mild steel disc was then fully submerged in a suitable beaker containing 1 M sulfuric acid. After 24 hours at room temperature, the specimens were removed and cleaned with a mixture of water and acetone. The specimens were dried and maintained in a desiccator before being reweighted. The weight reduction

technique was used in accordance with ASTM test results [23,24]. To guarantee the validity of the results, the tests were repeated twice. The average value of the weight loss allowed for the calculation of the average rate of corrosion (mgcm-2h-1). Using formula (1), the rate of corrosion of mild steel test was determined as follows [25]:

$$W = \frac{\Delta m}{s.t} \qquad \dots (1)$$

where:

m: a weight loss (grams),

S: area (squaremeters), and

t: immersing time (hours).

The efficiency of inhibition (IE%) was calculated by using the equation (2) as a following [25]:

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

where:

 W_{corr} : corrosion rates in absence of the inhibitor, and $W_{corr(inh)}$: corrosion rates in the presence of the inhibitor.

3. Results and Discussion

Scheme 1 shows the preparation of synthesized triethylammonium salts (B1-B3) of the present work.



Scheme 1. Synthesis steps of the triethylammonium salts (B1, B2 and B3).

The reactions above are included preparation of the amic acids (A1, A2 and A3) by using cyclohexylamine to react with an acid anhydride. Then the reaction followed by the acid-base reaction with triethylamine to prepare triethylammonium salts (B1, B2 and B3).

The FT-IR spectral data and physical properties of the prepared compounds are summarized in the Table (1, see Fig. (2-7)) and the 1H-NMR spectral data of compounds (B1, B2, and B3) in ppm listed in Table 2 (see Figures 8-10).



Figure 3. FT-IR of compound (A2).





Figure 7. FT-IR of compound (B3).



ANJS, Vol.26 (2), June, 2023: 6-18



After 24 hours and at room temperature the immersing, the rate of corrosion results and efficiency of inhibition has obtained using measurements of weight loss with concentrations in different of organic inhibitor molecules (B1-B3) are listed within Table 3 and showed in Figures 11 and 12.

Table 3. Corrosion rate, efficiency of inhibition (E %), coverage of surface (θ) and ΔG^{o}_{ads} for mild steel test by 1 M H₂SO₄ with measurements of weight loss at room temperature in 24 hours.

Concentration (M)	Concentration (M) Corrosion rate (mg.cm ⁻² .h ⁻¹)		θ	ΔG°_{ads} (kJ. mol ⁻¹)
Blank	0.1225			
B1				
5×10 ⁻⁴	0.0233	80.97	0.8097	
1×10 ⁻³	0.0133	89.14	0.8914	-33.74
5×10 ⁻³	0.0095	92.24	0.9224	$(R^2 = 0.9999)$
1×10 ⁻²	0.0072	94.14	0.9414	
B2				
5×10 ⁻⁴	0.0157	87.18	0.8718	_
1×10 ⁻³	0.0038	96.90	0.9690	-34.53
5×10 ⁻³	0.0028	97.71	0.9771	$(R^2 = 0.9999)$
1×10 ⁻²	0.0010	99.18	0.9918	
B3				
5×10 ⁻⁴	0.0049	96.00	0.9600	
1×10 ⁻³	0.0031	97.46	0.9746	-38.79
5×10 ⁻³	0.0024	98.04	0.9804	$(R^2 = 0.9999)$
1×10 ⁻²	0.0022	98.20	0.9820	



Figure 11. Effect of inhibitor concentrations on the corrosion rate for mild steel 1 MH₂SO₄ at room temperature for suggested inhibitors (B1-B3).



Figure 12. Effect of concentrations inhibitor on mild steel 1MH₂SO₄ inhibition efficiency at room temperature for organic inhibitor molecules (B1-B3).

ANJS, Vol.26 (2), June, 2023: 6-18



Figure 13. Langmuir adsorption isotherm plot for mild steel in 1MH₂SO₄ solution in the presence of various concentrations of inhibitor (B1).

The corrosion effectiveness increases with increasing inhibitor concentrations, as shown in Table 3, with 10-2 M having the highest inhibition efficiencies. The analysis reveals that the effectiveness of inhibition (C1 to C3) is roughly equivalent. The values of efficiency inhibition and the process adsorption may be used to describe how molecular organic structures (flexible structures with B2 and B3) affect efficiency inhibition. Basic knowledge about the adsorption process may be used to remove the organic molecule compound's natural contact with the metal surface. In order to acquire the values of surface covering degree, with various doses of inhibitor, weight loss experiments using 1M sulfuric acid were conducted $\theta = E(\%)/100$ (see Table 3) at room temperature and determined by relationship Langmuir isothermal, (3) [26]:

$$\frac{c}{\theta} = \frac{1}{K_{ads}} + C \qquad \dots (3)$$

where C is concentration, (M), K_{ads} , adsorption process of equilibrium constant.

The Langmuir isotherm states that values for K_{ads} are determined by intersections between a straight line and C, (see Figure 13), ΔG^{o}_{ads} , is calculated by equation (4): (55.5 value: water molar concentrations, M).

$$K_{ads} = \frac{1}{55.5} exp\left(\frac{-\Delta_{ads}^0}{R}\right) \qquad \dots (4)$$

Table 3 displays the free energy values of the process adsorption with a negative sign to demonstrate that the adsorption of (B1-B3) is occurring spontaneously over the surface of mild steel after 24 hours of immersion at room temperature. This is done to explain the interaction between the metal surface and the substance being adsorbed (B1-B3). With this point, organic molecules are moving nearby metal surface causing interfering electrons of organic molecules with the empty orbitals of metal surface atoms [27-30]. With this idea, the organic inhibitor adsorption on metal by donor atoms leads to formation kind of complexation between the metal and organic inhibitor as showing in (5, 6) [31]:

$$Fe + 2H^{+} \longrightarrow Fe^{2+} + H^{2} \qquad \dots(5)$$

$$Fe^{2+} + Inh(ads) \longrightarrow [Fe-Inh]^{+2}(ads) \text{ (Metal-inhibitor complex)} \qquad (6)$$

In order to stop the formation of Fe^{2+} ions, the mineral inhibitor complex will act as a thin film layer on anode sites. As a result, it's probable that with decreasing concentrations of the suggested inhibitor, metal inhibitor complexes are lower. The interaction of organic molecular adsorption molecules with N or O atoms controls the process of adsorption of a specific inhibitor. It has been documented that functional groups within molecule is causing to change molecule electrons density, which can be affected adsorption process [32]. The proposed inhibitors (B1-B3) that adsorbing with metal surface through thin film complex can be allocated on the site's anode, that to reduce or prevent to losing metal atoms of surface via the reaction of electrochemical dissolutions.

physical adsorption, as shown in Table 3, was discovered [32]. As indicated in Table, the suggested inhibitor (B3) displayed a value of Goads = 38.79 kJ/mol, which was confirmed by IE% values (96-99) for the same dosages (3). E% is greater than E% values for (B1) and (B2) despite the low concentration of (B3) (0.0005M) (B2). The amide group and acetate group have a greater electron density because of the chemical structure of B3. By evenly distributing the electron density on the surface metal with organic molecules (B3) as well as for B1 and B2, the

ANJS, Vol.26 (2), June, 2023: 6-18

interaction between the organic molecule (B3) and the surface metal will be improved.

Last but not least, Figure 14 illustrates the reaction mechanism of the interaction between the organic molecule (B3) and the surface metal. This figure also depicts the aromatic groups of the suggested inhibitor molecules on the iron surfaces as well as a modeling representation of the process of nitrogen and oxygen atoms adsorption.



Figure 14. Suggested interaction mechanism of inhibitor molecules on the mild steel surface.

4. Conclusion

Triethylammonium salts (B1-B3) were successful using like, corrosion organic inhibitors for surface of iron metal within solution of 1 M sulfuric acid within room temperature. The efficacy inhibitory, IE %, results were showed considerable effects inhibitory of the prepared inhibitor molecules. The free energy adsorption values were showing effect within physical process adsorption (B1-B3) and obtained important informations to elucidate the natural interactions between the molecules of biological matter and the metal surface.

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ANJS, Vol.26 (2), June, 2023: 6-18

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