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Khalid Kabel

Petroleum Applications Department, Egyptian Petroleum Research Institute, Naser City, P.O. 11727, Cairo, Egypt.

L. Kamel

Department of Chemistry, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt.

R. Abou-shahaba

Department of Chemistry, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt.

Azza El-Shenawy

Department of Chemistry, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt.

Seham Shahan

Department of Chemistry, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt.,
hahen.seham@yahoo.com

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BENZOIC ACID DERIVATIVES AS EFFECTIVE CORROSION INHIBITORS FOR CARBON STEEL IN 3.5 % SODIUM CHLORIDE SOLUTION.**Nour. E.A.Abdel-satar¹ A.S.Fouda² E.M.Khalil³ Gamal A.EL-Mahdy³ Ahmed S.Mohamed⁴***1. Chemistry Department, Faculty of Science, Ain Shams University, Cairo, Egypt**2., Chemistry Department, Faculty of Science, Mansoura University, Mansoura-35516, Egypt**3. Chemistry Department, Faculty of Science, Helwan University, Helwan, Cairo, Egypt**4. Refining and Processing Department, Egyptian general petroleum corporation, Cairo, Egypt***ABSTRACT**

New corrosion compounds were prepared based on acryloyl chloride and the efficiency of the prepared compounds to inhibit carbon steel (CS) corrosion in 3.5% sodium chloride solution were studied through different electrochemical techniques [potentiodynamic polarization (PP), electrochemical impedance spectroscopy (EIS) and electron frequency modulation (EFM)] and Quantum chemical calculations. The results showed that the investigated compounds are working as good corrosion inhibitor compounds, the inhibition efficiency (%IE) increases as the compound concentration increase. The polarization data revealed that these compounds act as cathodic and mixed type compounds and are adsorbed on CS surface following Langmuir adsorption isotherm.

Keywords: carbon steel, sodium chloride solution and corrosion inhibitor compound**1. INTRODUCTION**

Corrosion is a material degradation caused by the chemical reaction with other materials and/or the environment [1]. This process often occurs in the industry of a material oil and gas. In the industrial field, Carbon steel plays an important role in a lot of industries specially oil and gas industry [2-5] it is the most widely used engineering material accounts for approximately 85% of the annual steel production worldwide. Carbon steel corrosion in oil and gas industry is a significant problem as it affects not only the production's stability but it also causes economic loss. Organic compounds containing heterocyclic atom is the most widely used corrosion compounds [6-8]. O, N and/or S atoms present in the prepared acryloyl derivatives increase the possibility to use it as corrosion compound [9– 12] due to overlapping of P orbital of hetero atoms with vacant d orbital of metal [13-21]. It can also be said that the compound forms protective layer on the metal surface by the reaction between the solution and the corroded metal surface [22]. Different electrochemical techniques were

applied to study the efficiency of the prepared compounds (compound 1 and compound 2) to prevent carbon steel corrosion in 3.5% NaCl solution.

2. MATERIALS AND SOLUTIONS**2.1. Materials**

The compositions of commercial C steel are as of weight % 0.200 C, 0.91 Mn, 0.007 P, 0.002 Si and the rest is Fe

2.2. Solutions**2.2.1. Acryloyl chloride****2.2.2. Sodium chloride solution 3.5%**

Corrosion solution was attained by mixing 35.24 grams of NaCl and 1000 mL distilled water to get NaCl 3.5%.

2.2.3. The synthesized acryloyl derivatives studied and tested as corrosion compounds with molecular formula and molecular weight are shown as follow.**3. EXPERIMENTAL****3.1. Chemistry:**

The infrared spectra were recorded using potassium bromide disks on a Pye Unicam SP-

H, 4.03; N,
11.19;
Found: C,
54.52; H,
4.07; N,
11.25.

3.1.1.2. 4

3-300 infrared spectrophotometer, the established values of the gas phase frequencies are given between brackets. ¹HNMR spectra were run at 300 MHz, on a Varian Mercury VX-300 NMR spectrometer and Bruker Avance III 400 MHz, using TMS as an internal standard in deuterated dimethylsulphoxide. All the spectral measurements were carried out at the NMR laboratory of the Ministry of Defence Chemical Laboratories, Egypt, at the Micro Analytical Centre of Ain Shams University, Egypt. All the chemical reactions were monitored by TLC.

3.1.1. General method for preparation of compounds 1 and 2

Stirring (0.1 mole) of acryloyl chloride with (0.1 mole) of ammonium thiocyanate in dry acetone (50 ml) for 20 minutes then filter the mixture to remove ammonium chloride, Then reflux the prepared acryloyl thiocyanate mixture with (0.1 mole) of 2-aminobenzoic acid and 4-amino benzoic acid respectively for one hour, pour the solution and evaporate the excess acetone the formed ppt was filtered off, dried.

3.1.1.1. 2-(3-acryloylthioureido)benzoic acid. compound 1

Yield 85 %; yellow semisolid; IR (KBr) broad band at 3494 cm⁻¹ (ν_{OH}), 3378, 3128 cm⁻¹ (ν_{NH}), 1715, 1691 cm⁻¹ (ν_{C=O}). ¹HNMR (300 MHz, DMSO-d₆): δH (ppm) 5.9 (d, 2H, olefinic CH₂), 6.5 (t, 1H, olefinic CH), 7.4-7.8 (m, 4H, Aromatic), 11.2, 11.8 (s, 2H, D₂O exchangeable 2NH), 12 (s, 1H, D₂O exchangeable OH). Anal. Calculated for C₁₁H₁₀N₂O₃S (250.04): C, 52.79;

(3-acryloylthioureido)benzoic acid. compound 2

Yield 89 %; orange semisolid; IR (KBr) broad band at 3444 cm⁻¹ (ν_{OH}), 3352, 3152 cm⁻¹ (ν_{NH}), 1721, 1681 cm⁻¹ (ν_{C=O}). ¹HNMR (300 MHz, DMSO-d₆): δH (ppm) 6.3 (d, 2H, olefinic CH₂), 6.9 (t, 1H, olefinic CH), 7.3-7.9 (m, 4H, Aromatic), 11.3, 11.5 (s, 2H, D₂O exchangeable 2NH), 12.0 (s, 1H, D₂O exchangeable OH). Anal. Calculated for C₁₁H₁₀N₂O₃S (250.04): C, 52.79; H, 4.03; N, 11.19; Found: C, 54.92; H, 4.32; N, 11.50.

3.2. Corrosion inhibition tests

3.5 % sodium chloride solution, Corrosion solution was attained by mixing 35.24 grams of NaCl and 1000 mL distilled water to get NaCl 3.5%. Then preparation of the working solution with the tested compounds with a concentration ranges from 25 ppm to 150 ppm for each compound. All solution were prepared using distilled water.

3.2.1. Electrochemical measurements

Three different electrochemical techniques were applied to study the corrosion characteristics of carbon steel in 100 ml of 3.5 % NaCl in presence and absence of investigated compounds, all of these techniques were performed at 25 °C using three electrode systems present in a glass cell in which a specimen of carbon steel with area equals to 1 cm² act as working electrode and saturated calomel electrode (SCE) act as reference electrode and counter or auxiliary electrode from platinum. The working electrode is prepared by welding the carbon steel specimen

BENZOIC ACID DERIVATIVES AS EFFECTIVE CORROSION ...

with copper rod from one side and perfectly encapsulated into a glass rod so that one face of carbon steel was left to expose the test solution. before starting the experiments the carbon steel specimen is mechanically abraded with emery papers (a coarse paper was used initially and then progressively finer Grades up to 2000 grit size) and degreased using acetone then rinsed with bidistilled water then finally dried between filter paper. Results obtained from electrochemical techniques were utilized by Gamry Instrument (PCI4/750) with a computerized frameworks contain DC105 software for potentiodynamic polarization measurements, EIS300 software for electrochemical impedance (ac) spectroscopy, and EFM140 software for electrochemical frequency modulation techniques. In addition to using Echem Analyst 6.03 software for data fitting, graphing and plotting.

3.2.1.1. Potentiodynamic polarization (PP) measurements

Tafel polarization slopes were obtained through changing the electrode potential automatically from (-0.5 to 0.5 V vs. SCE) [23] at open circuit potential (OCP) with a scan rate of 1 mVs⁻¹. The corrosion current is performed by extrapolation of anodic and cathodic Tafel lines to a point which gives log i_{corr} and the corresponding corrosion potential (E_{corr}) for compound free solution and for each concentration of compound. inhibition efficiency (%IE) and surface coverage (θ) can be calculated using i_{corr} as shown in equation (1)

$$\% IE = 100 \times \theta = [1 - (i_{corr} / i_{corr}^{\circ})] \times 100 \quad (1)$$

Where i_{corr} and i_{corr}° are corrosion current densities in presence and absence of the tested compounds respectively.

3.2.1.2. Electrochemical impedance spectroscopy (EIS) measurements

Kinetics of electrochemical processes and capacitive behaviour of investigated compounds on carbon steel were characterized by EIS measurements at open circuit potential and in a frequency range from 100 kHz to 0.5 Hz. Charge transfer resistance obtained from EIS data used to calculate inhibition

efficiencies (%IE) and surface coverage (θ) as shown in equation (2)

$$\% IE = 100 \times \theta = [1 - (R_{ct}^{\circ} / R_{ct})] \times 100 \quad (2)$$

Where R_{ct}° and R_{ct} are the charge transfer resistance in absence and presence of the tested compounds respectively.

3.2.1.3. Electrochemical frequency modulation (EFM) measurements

Two frequencies 2 and 5 Hz and base frequency 0.1 Hz were used to obtain intermodulation spectra of the electrochemical frequency modulation. The larger peaks were used to calculate the corrosion current density (i_{corr}), the Tafel slopes (β_c and β_a) and the causality factors CF-2 and CF-3. The obtained values of i_{corr} are used to calculate inhibition efficiencies (%IE) and surface coverage (θ) as shown in equation 3:

$$\% IE = 100 \times \theta = [1 - (i_{corr} / i_{corr}^{\circ})] \times 100 \quad (3)$$

Where i_{corr} and i_{corr}° are corrosion current densities in presence and absence of the tested compounds respectively.

4. Quantum Chemical Calculations.

The molecular structures of the investigated compounds were optimized with PM3 semi empirical method. All the quantum chemical calculations were performed with Spartan 10 V1.10.

5. RESULTS AND DISCUSSION

5.1. Chemistry

The reaction of acryloyl chloride with ammonium thiocyanate by stirring in dry acetone for half an hour followed by refluxing the prepared mixture with 2-aminobenzoic acid and 4-amino benzoic acid respectively for one hour the prepared compound accompanied with appearance of 2 v $C=O$ as well as appearance of broad band attributed to presence of COOH this improve the formation compound 1, B respectively which they are not soluble in water so we add Na_2CO_3 to convert it to soluble salt (scheme 1).

5.2. Potentiodynamic polarization (PP) measurements

The kinetics of the cathodic and anodic reactions in the presence and absence of the investigated compounds to evaluate its efficiency had been studied and shown in Figure 2 and figure 3 for compound 1 and 2, respectively. Different electrochemical corrosion kinetics parameters, i.e. corrosion potential (E_{corr}), cathodic and anodic Tafel slopes (β_a , β_c) and corrosion current density (i_{corr}) obtained from the extrapolation of the polarization curves were given in Table 3. It is observed from the obtained data that the inhibition efficiency (%IE) increases by the increase of the compound concentration due to the formation of energy barrier. If the displacement in E_{corr} is more than 85 mV with respect to corrosion potential of blank solution the compound can be worked as anodic or cathodic while if the E_{corr} displacement is less

cathodic type compounds ($\beta_c > \beta_a$) but in inhibitor 1 the displacement is less than 85 mV which indicate that it acts as a mixed type inhibitor.

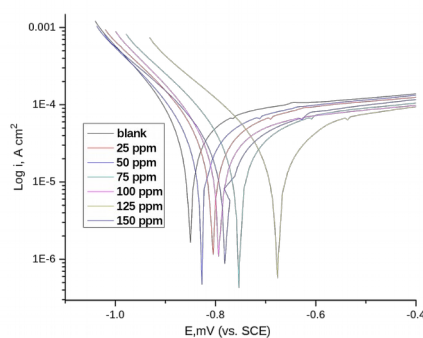
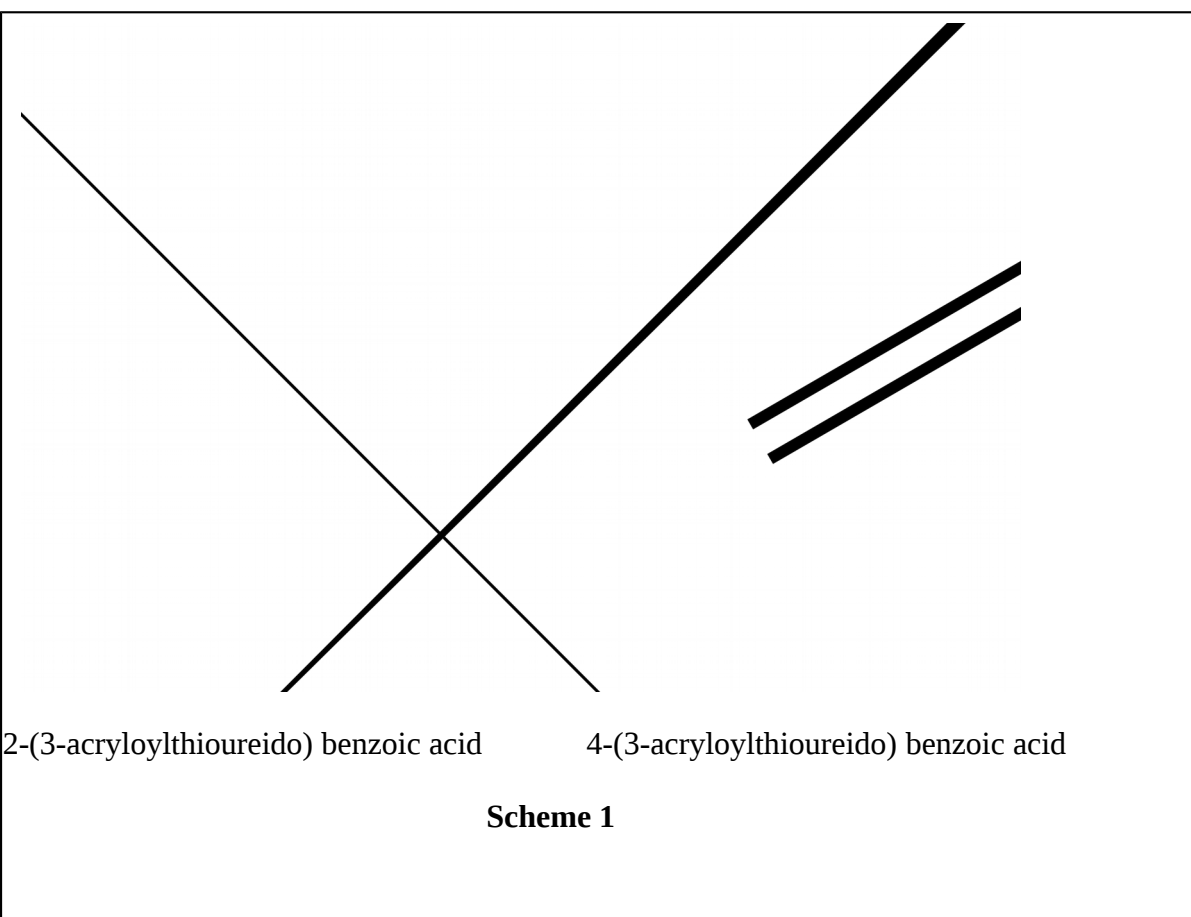


Figure (2): Potentiodynamic polarization curves for the corrosion of CS in 3.5% NaCl in the absence and presence of various concentrations of compound (1) at 25°C



than 85 mV the compound can be worked as mixed type compound [24], in this work E_{corr} displacement is higher than 85 mV in inhibitor 2 which indicate that this compound may be

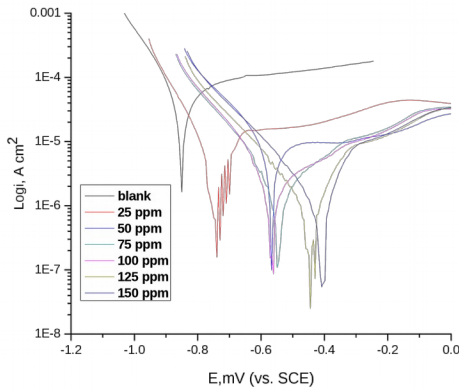


Figure (3): Potentiodynamic polarization curves for the corrosion of C-steel in 3.5% NaCl in the absence and presence of various concentrations of compound (2) at 25°C.

5.3. Electrochemical impedance spectroscopy (EIS) measurements

Surface properties and mechanism of corrosion inhibition can be obtained and examined from EIS diagrams [25-28]. EIS will detect NaCl solution and form a resistance solution (R_s). Once past that, the metal will form a passive layer which tends to be protective for their bond between Fe and functional groups of the investigated compounds. The thicker the passive layer than the value, the higher the R_{ct} . Nyquist and Bode diagrams in the presence and absence of different concentrations of both compounds in 3.5% NaCl solution are shown in Figures 4a, 4b, 5a and 5b respectively, it is observed that Nyquist plots give semicircles due to surface

roughness, impurities and inhomogeneity of electrode surface [29-31]. To analyse the obtained data from EIS measurement an equivalent circuit used as shown in Figure 6 which present charge transfer reaction. Data of capacitance double layer (Cdl) can be measured from parameter CPE data Y_0 and n utilized the expression [32] as shown in equation (4):

$$Cdl = Y_0 (\omega_{max})^{n-1} \tag{4}$$

Where ω_{max} is the angular frequency at which the imaginary component of the impedance reaches its maximum data, $\omega_{max} = 2\pi f_{max}$, f_{max} is the maximum frequency, n is the deviation parameter of the CPE: $-1 \leq n \leq 1$ and Y_0 is magnitude of the CPE. Different electrochemical parameters are shown in table 4

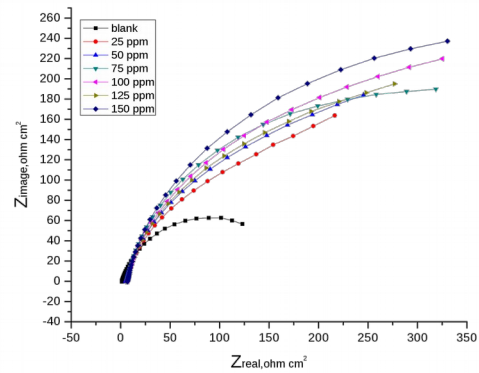


Fig. 4a: The Nyquist plots for corrosion of CS in 3.5% NaCl solutions in the absence and presence of different concentrations of compound (1) at 25°C.

Table (3): Data from potentiodynamic polarization of C steel in 3.5% NaCl containing various concentrations of investigated compounds at 25°C.

Comp	Conc., ppm	- E _{corr} , mV (vs SCE)	i _{corr} , μA cm ⁻²	C.R, mpy	β _c mV dec ⁻¹	β _a mV dec ⁻¹	Θ	% IE
Blank	----	851	94.1	43	176	100	---	---
1	25	806	52.1	36.1	212	110	0.446	44.6
	50	828	46.9	29.4	197	112	0.502	50.2
	75	754	43.3	29.3	217	120	0.540	54.0
	100	753	36.1	28.8	188	123	0.616	61.6
	125	679	30.5	27.5	276	115	0.675	67.6
	150	780	27.7	25.7	221	123	0.706	70.6
2	25	732	26.6	12.2	190	99	0.717	71.7
	50	567	16.4	7.5	210	101	0.826	82.6
	75	546	2.4	1.1	169	113	0.975	97.5
	100	565	1.8	0.835	132	115	0.981	98.1
	125	442	0.43	0.1965	134	113	0.995	99.5
	150	406	0.411	0.188	121	118	0.996	99.6

Table (4): EIS data of C steel in 3.5% NaCl solutions in the absence and presence of different concentrations of investigated compounds at 25 °C

Comp	Conc., ppm	C_{dl} , $\times 10^3 \mu F cm^{-2}$	R_{ct} , Ωcm^2	\bar{E}	%IE
Blank	0	6.12	165.3	---	---
1	25	2.73	371.5	0.555	55.50
	50	2.41	421.1	0.607	60.74
	75	2.25	449.7	0.632	63.24
	100	2.18	465.3	0.645	64.47
	125	2.01	504.5	0.672	67.23
	150	1.81	559.1	0.704	70.43
2	25	0.826	11800	0.986	98.60
	50	0.666	15200	0.989	98.91
	75	0.596	17100	0.990	99.03
	100	0.557	18300	0.991	99.09
	125	0.539	18900	0.991	99.12
	150	0.508	20000	0.991	99.17

Fig. 5b: The Bode plots for corrosion of C-steel in 3.5% NaCl solutions in the absence and presence of different concentrations of compound (2) at 25°C

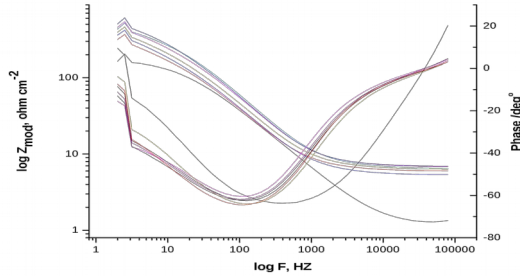


Fig. 4b: The Bode plots for corrosion of C-steel in 3.5% NaCl solutions in the absence and presence of different concentrations of compound (1) at 25°C

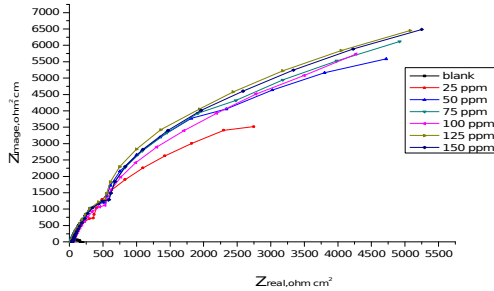


Fig. 5a: The Nyquist plots for corrosion of C-steel in 3.5% NaCl solutions in the absence and presence of different concentrations of compound (2) at 25°C

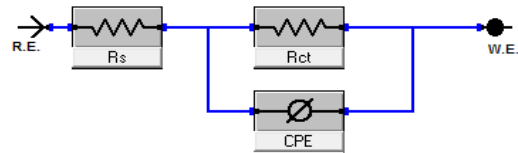
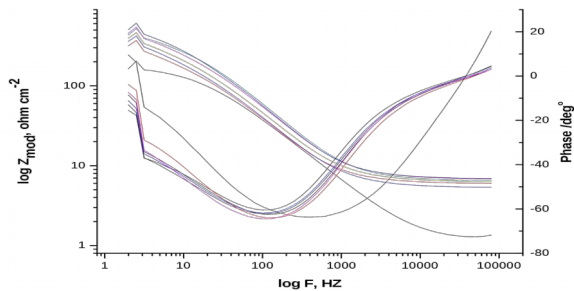


Figure 6: Equivalent electrical circuit model utilized to fit the results of impedance

The given data in Table 4 showed that R_{ct} and %IE increase by increasing compound concentration which is attributed to increase of the adsorbed layer thickness [33] it also shows that the relationship between R_{ct} and C_{dl} is inversely proportional. This occurs due to a decrease in local dielectric constant or increasing the density of electric double layer. The presence of the investigated compounds modifies the structure of the electric double layer by adsorption compound molecules at the interface of a metal with a solution and the replacement of adsorbed water molecules by the inhibitor molecules. Adsorbed compounds on the metal surface forms a barrier layer to prevent mass transfer. With the addition of the compound concentration, the R_{ct} values increased, but the C_{dl} values tended to decrease. This is mainly due to the adsorption of compounds on the metal surface [34].

5.4. Electrochemical frequency modulation (EFM) measurements

EFM is the most convenient and suitable technique for online monitoring as it doesn't need to know Tafel slopes [35, 36], EFM spectra of c-steel in 3.5% NaCl solution in the presence and absence of different

concentrations of both compounds are shown in Figures 7 & 8. Different electrochemical parameters i.e. the corrosion density (i_{corr}), Tafel slopes (β_a , β_c), causality factors (CF-2 and CF-3) and % IE for both compounds are shown in Table 5. The obtained data showed that both

ΔE) of the investigated compounds are shown in table 6. The difference $\Delta E = E_{HOMO} - E_{LUMO}$ is the energy required to move an electron from HOMO to LUMO. Low ΔE facilitate adsorption of the molecule and thus will cause higher inhibition efficiency. The theoretical

values of E_{HOMO} , E_{LUMO} , μ , ΔE indicate that compound 2 > compound 1. This support the

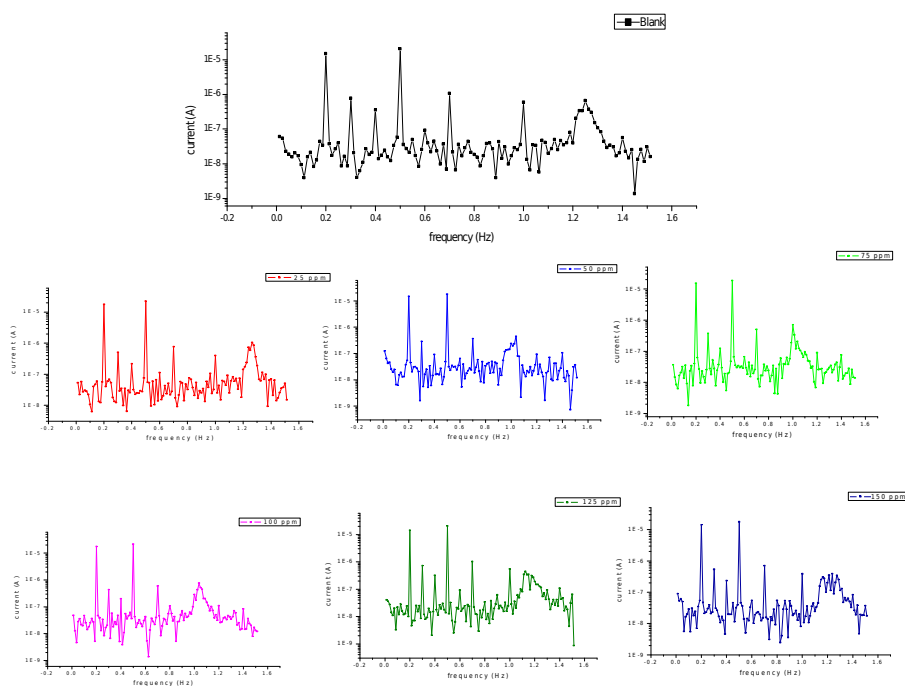


Figure 7: EFM for corrosion of C-steel in 3.5% NaCl solution in the absence and presence of different concentrations of compound (1) at 25°C

compounds are working as good corrosion inhibitors for c-steel corrosion in 3.5% NaCl solution, as shown in Table 5, % IE for both compounds increase with the increase of compound concentration, CF-2 and CF-3 values are close to the theoretical values 2 and 3 respectively which indicate the good quality of the obtained data.

experimental results.

5.5. Quantum chemical calculations

The reactive ability of the inhibitor is related to E_{HOMO} , E_{LUMO} [37]. Higher E_{HOMO} values indicate higher electron donating ability [38], but low E_{LUMO} indicates that the acceptor accepts electrons easily. The calculated quantum chemical indices (E_{HOMO} , E_{LUMO} , μ ,

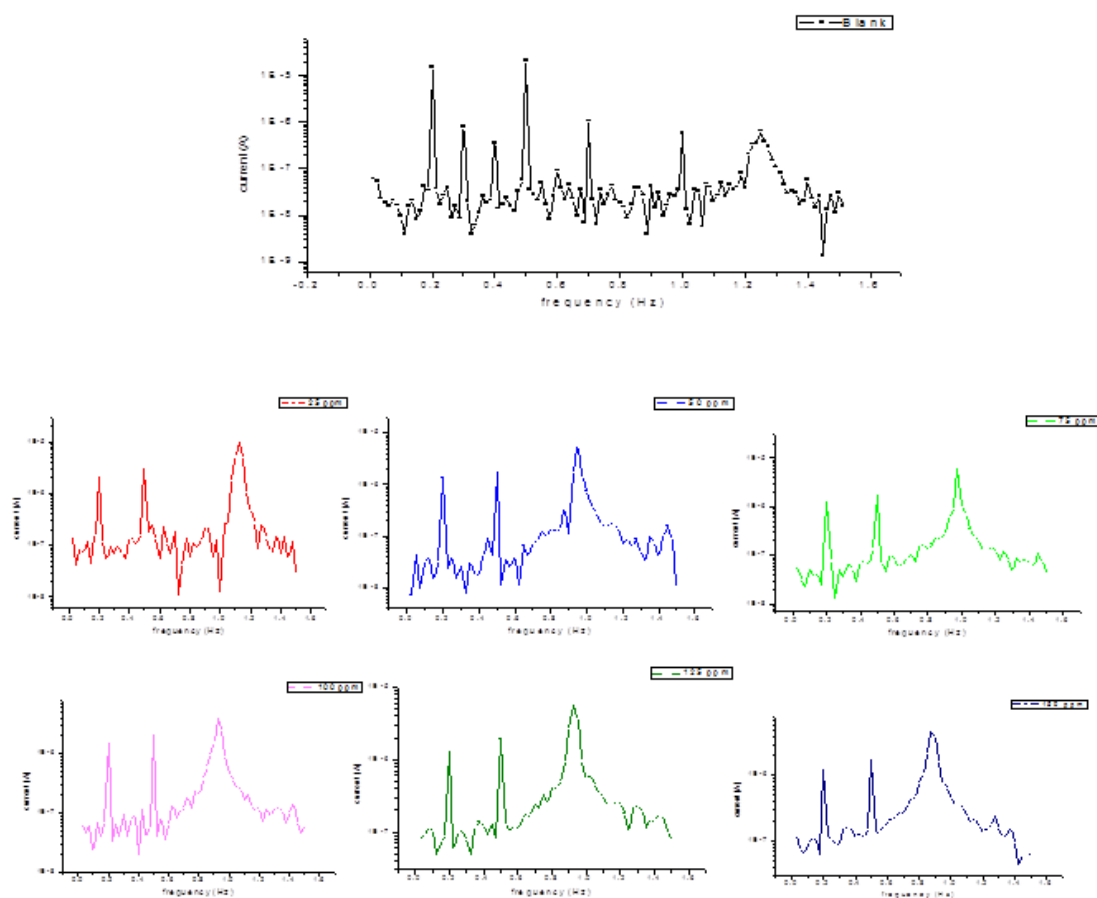


Figure 8: EFM for corrosion of C-steel in 3.5% NaCl in the absence and presence of different Concentrations of compound (2) at 25°C

Table (5): Electrochemical kinetic parameters obtained by EFM technique for C- steel in the absence and presence of various concentrations of investigated compounds in 3.5% NaCl solution at 25 °C

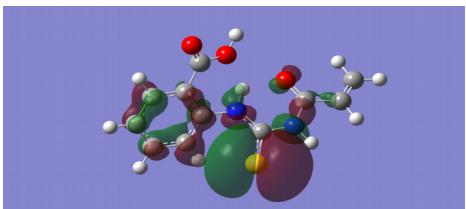
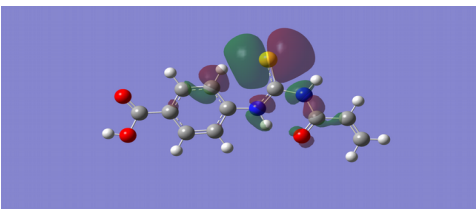
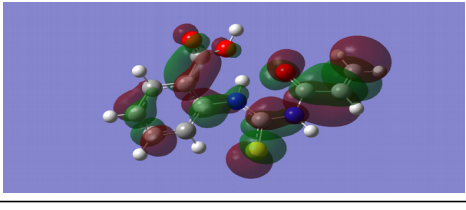
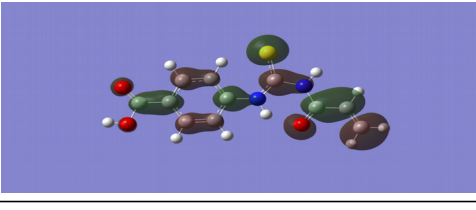
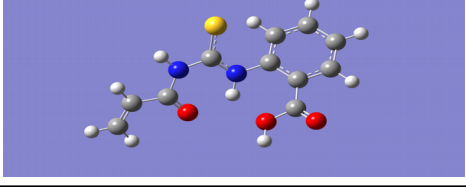
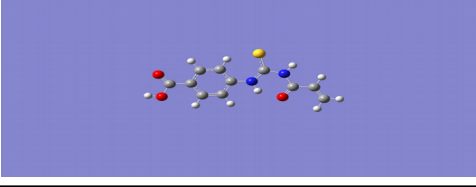
Comp	Conc., ppm	i_{corr} , μAcm^{-2}	\hat{a}_c , $mVdec^{-1}$	\hat{a}_a , $mVdec^{-1}$	C.R, mpy	CF-2	CF-3	\bar{E}	%IE
Blank	----	138	278.3	253	63.1	1.96	2.89	---	---
1	25	76.5	198	318	31	2.06	2.98	0.445	44.5
	50	65.3	223.2	306.2	29.8	1.968	3.12	0.527	52.7
	75	59.6	193.6	280.1	27.2	1.75	3.16	0.568	56.8
	100	54.5	158.1	212.8	24.9	2.12	3.01	0.605	60.5
	125	51.7	149.3	276.9	23.6	2.01	3.26	0.625	62.5
	150	41	139.6	209.9	18.8	2.01	2.83	0.703	70.3
2	25	1.6	38.3	43.4	0.734	1.99	3.05	0.988	98.8
	50	1.22	37.4	52.2	0.555	2.01	3.01	0.991	99.1
	75	0.8693	35.5	39.5	0.0395	1.95	2.9	0.994	99.4
	100	0.6437	22.02	23.9	0.0239	2.01	3.25	0.995	99.5
	125	0.3923	15.2	16.7	0.0179	2.03	2.98	0.997	99.7
	150	0.3013	12.3	13.5	0.0137	1.94	3.26	0.998	99.8

Different

Table (6): The calculated quantum chemical parameters for the investigated compounds using (PM3) method.

Compound	1	2
$-E_{\text{HOMO}}$	0.29542	0.29245
$-E_{\text{LUMO}}$	0.20606	0.20364
ΔE	0.09360	0.08881
μ	4.125	4.178

Table (7): Molecular structure of compounds (1 and 2), and their frontier molecular orbital density distribution (HOMO and LUMO)

	1	2
HOMO		
LUMO		
Optimize structure		

6. Mechanism of inhibition.

Inhibition of c-steel corrosion in 3.5 % NaCl solution in the presence of prepared acryloyl chloride derivatives depends on adsorption of the investigated compound molecules on c-steel surface through interaction of π electrons with the metal and through unshared electrons on heteroatom. The two compounds are the same and contain the same heteroatoms, but the substituted COOH group in compound 2 in the p-position and in compound 1 in the o-position. So, it may form with NH bond and mask the centres NH and C=O, so compound 1 has lesser active centres and molecular areas than compound 2. So, compound 1 has lesser %IE than compound 2.

7. CONCLUSIONS

electrochemical (PP, EIS and EFM) measurements were carried out and proved that the investigated acrylic acid derivative corrosion compounds are good compounds for c-steel corrosion in 3.5 % NaCl solution. Inhibition efficiency (%IE) increases with the increase of compound concentration. PP proved that the investigated compounds are of mixed type inhibitors while causality factors of EFM spectra proved the good quality of the obtained data. The highest efficiency occurs during addition of 150 ppm of compound 2 to reach 99.5%. The mechanism of inhibition that occurs through adsorption of compound molecules on the metal surface to form a thin protective layer. The theoretical values of E_{HOMO} , E_{LUMO} , μ , ΔE indicate that compound 2 >

compound 1. This support the experimental results. The order of %IE of the compounds is: compound 2 > compound 1

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