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RESEARCH ARTICLE

CORROSION INHIBITION OF MILD STEEL PIPELINE USING NONIONIC SURFACTANTS

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ABSTRACT

The effect of three different nonionic surfactants on the mild steel in artificial corrosive oil were studied. Potentiodynamic polarization measurements were depicted that the investigated inhibitors acts as mixed type inhibitors. The SEM was used to justify the practical results. The relationship between molecular structure and the inhibition efficiency were evaluated using PM3 semi-empirical method. The inhibition efficiency was matched with the most calculated quantum chemical parameters namely E_{HOMO} (highest occupied molecular orbital), E_{LUMO} (lowest unoccupied molecular orbital), the energy gap ΔE, the Mulliken charges, the hardness η, the dipole moment μ, the total energy change E_T, the electron affinity A, the absolute electronegativity x, the fraction of electron transfer ΔN and the electrophilicity index ω.

1. INTRODUCTION

Mild steel is widely used in different industrial field as petroleum industries due to its low cost and high mechanical properties. The protection of metals from corrosion especially in different aggressive acid media has achieved using different organic compounds containing hetero atom as oxygen, nitrogen and sulpher (Abo-Dief, 2014; Khidr *et al.*, 2013; Abo-Dief *et al.*, 2014; Kumar *et al.*, 2010). The use of surface active materials (surfactants) as corrosion inhibitors have many advantages comparing with other organic compounds due to their unique structure. Surfactants are molecules composed of a polar hydrophilic head, attached to a non-polar lipophilic tail. The inhibitory action of inhibitors may be physisorption due to electrostatic interaction or chemisorption due to charge sharing between inhibitor and metal (Hegazy *et al.*, 2012; Nessim *et al.*, 2014; Hegazy and Zaky, 2010; Elshamy and Al-Ayed, 2013). Recently, quantum chemical calculations, have been found to be effective tool for explaining the inhibition interaction (Xiao-Ci *et al.*, 2000; Bentiss *et al.*, 2005; Issa *et al.*, 2008; Zhao *et al.*, 2005; Yurt *et al.*, 2006; Emregül *et al.*, 2006; El-Naggar, 2007). In this research, the efficiency of three different nonionic surfactants as corrosion inhibitors were investigated experimentally and theoretically. The effect of investigated surfactants on the rheology and other physicochemical properties of the aggressive oil were measured. Quantum chemical calculations were employed to study the effect of the structure on the adsorption of the investigated molecules. In order to determine the adsorption mechanism, some quantum chemical parameters were calculated using PM3, among the other semi-empirical methods (Wang *et al.*, 1999; Hosseini *et al.*, 2011; Amir *et al.*, 2014). All theoretical calculation have been performed after complete geometry optimization.

2. EXPERIMENTAL

2.1. Materials

Tests were performed on mild steel of the following composition (wt%): C = 0.15%, Si = 0.08%, S = 0.025%, P = 0.025%, and Mn = 1.02% and the remainder is Fe.

2.2. Synthesis

The inhibitor $C_8H_{17}phO(C_2H_4O)_nH$, n = 9 (A) were purchased from sigma Aldrich, while the others $(C_{13}H_{27}COO(C_2H_4O)_yH$, y=9 and 14 (B and C) respectively were synthesized according to the method was reported (Khidr *et al.*, 2013).

The analyses of the two synthesized inhibitors were as follows: FTIR: 2887 cm^{-1}(CH_2str.), 1469 cm^{-1} (CH_2 bend.), 1112 cm^{-1} (C-O str.) and 1732 cm^{-1} (C=O).

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2.3. Aggressive solution

0.4 g of dodecyl sulphide (DDS) as one of the most corrosive sulpher compound in petroleum oil was added to a white technical oil (free of sulpher, nitrogen and salt contents) to represent aggressive solution. characteristics of the white technical oil were measured (Mohamed and Abo-Dief, 2012) and the data listed in Table 1.

Table 1. Physico-chemical characteristics of the technical white oil

Specifications	Sulphur Content, ppm	Refractive Index, 20 °C	Specific gravity, 15.6°C	Viscosity, cSt, 40 °C	Mean Mol. Wt.	T. A. N., mg KOH/g sample	Salt Content, wt.%	Nitrogen Content, wt.%	Structure group analysis %
Method	ASTM	ASTM	ASTM	ASTM	ASTM	ASTM	ASTM	Kjeldhal	ASTM
Number	D-4294	D-1747	D-4052	D – 445	D –2502	D-664	D-1275	method	D-3238
Results	Nil	1.47	0.83	15.34	190.00	0.2244	Nil	Nil	$C_{A=}$26.4, $C_{N\ =}$ 2.8 , $C_{P\ =}$ 70.8, $R_{A\ =}$ 0.44, $R_{T\ =}$ 0.80 , $R_{N\ =}$ 0.36

2.4. Weight loss measurements

Mild steel strips of 2cm x 1cm x 0.2cm were abraded with a series of emery paper (grades 320-400-600-800-1200) and then washed with distilled water, cleaned with acetone and then dried. After weighing accurately by a digital balance with sensitivity of ±0.1mg, the strips were immersed in 500ml beaker, which contain 300ml of aggressive oil with and without addition of 0.05W% of the investigated surfactants (A, B and C) separately at room temperature. After different time intervals 150, 300, 450 and 600 hrs, the strips were taken out, washed, dried and weighed accurately. Experiments were carried out in triplicate. The average weight loss of three parallel mild steel sheets was obtained. The corrosion rate (C_r), surface coverage (θ) and inhibition efficiency (IE %) were determined (Nessim *et al.*, 2014) by using Equations 1–3:

$$C_r = \frac{W_0 - W_i}{At} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

$$\theta = \frac{W_0 - W_i}{W_0} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$

$$IE\% = \frac{W_0 - W_i}{W_0}\, x100 \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

where, w_i and w_o are the weight loss values with and without inhibitor, respectively. A is the total area of the specimen and t is the immersion time.

2.5. Potentiodynamic polarization measurements

Potentiodynamic polarization measurements were conducted using an Autolab potentiostat /galvanostat (PGSTAT-302 N, Eco Chemie, and The Netherlands) and Metrohm 797 VA Computrace Model. The inhibition efficiency of the investigated inhibitor molecules (IE %) was calculated using the following equation (Elshamy, 2013; Wang *et al.*, 1999; Hosseini *et al.*, 2011; Amir *et al.*, 2014; Badr *et al.*, 2014; Mohamed *et al.*, 2012; Negm *et al.*, 2011).

$$IE\% = \left(\frac{i_{uninh} - i_{inh}}{i_{uninh}}\right) x100 \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(4)$$

Where, i_{uninh} and i_{inh} are the corrosion current density values without and with inhibitors.

2.6. Scan electron microscope (SEM)

The morphology of mild steel surface with and without optimum inhibitor concentration was examined by scanning electron microscope (JEOL, model JSM-6390 LA).

2.7. Quantum chemical calculation

For theoretical study complete geometry of inhibitor molecules optimized using PM3, among the other semi-empirical methods, using HyperChem 8.0.10, windows program, then quantum chemical parameters obtained from this optimized structure. Frontier molecule orbitals (HOMO and LUOM) may be used to predict the adsorption centers of the investigated inhibitors. According to Koopman's theory (Sastri and Perumareddi, 1997), the electron affinity A and the hardness η of the inhibitor molecules are related to E_{HOMO} and E_{LUMO}. Absolute electronegativity x and absolute hardness η of the inhibitor molecule are given by (Issa *et al.*, 2010):

$$x = \frac{1}{2}(I + A) = -(E_{LUMO} + E_{HOMO})/2 \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(5)$$

$$\eta = \frac{1}{2}(I - A) = (E_{LUMO} - E_{HOMO})/2 \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(6)$$

Where, I (- E_{HOMO}) is the ionization potential of an atom or a molecule is the minimal energy needed for the detachment of an electron. A (-E_{LUMO}) is the electron affinity, it is the energy released upon attachment of an electron to an atom or molecule.

The fraction number of electron (ΔN) from the inhibitor to the metallic atom is also calculated depending on the quantum chemical calculation (El Ashry *et al.*, 2011) using the following equation:

$$\Delta N = \frac{(x_{\eta Fe} - x_{\eta inh})}{2(\eta_{Fe} + \eta_{inh})} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(7)$$

Where xFe and xinh are the absolute electronegativity of iron and inhibitor respectively; Fe and inh are the absolute hardness of iron and inhibitor respectively. The theoretical value for absolute electronegativity and hardness of iron are 7 and 0 eV/mol respectively. Recently, some authors (Robert and László, 1999; Gómez-Zavaglia *et al.*, 2005; Chattaraj *et al.*, 2006) have introduced an electrophilicity index (ω) this was proposed as measure of the electrophilic power of the molecule and it defined as:

$$\omega = \frac{\mu^2}{2\eta} \quad \text{………………………..(8)}$$

3. RESULTS AND DISCUSSION

3.1. Effect of inhibitor concentrations on the oil viscosity

It was detected that as the inhibitor concentration increases, the oil viscosity increases. Also, as the test period increase, the viscosity increases due the heat dissipation increment. For all inhibitors under investigation, it is found that the 0.05Wt.% of the inhibitors have the lower viscosity values. Figure 1. illustrates a different behavior for the studied inhibitors on the viscosity of the oil media at 0.05wt.%. It is clear that the lowest viscosity exhibit in presence of A followed by B and C respectively. As inhibitor A shifted the aggregation concentration of oil and it is clear that oil prefer the interaction with this inhibitor rather than self-aggregation

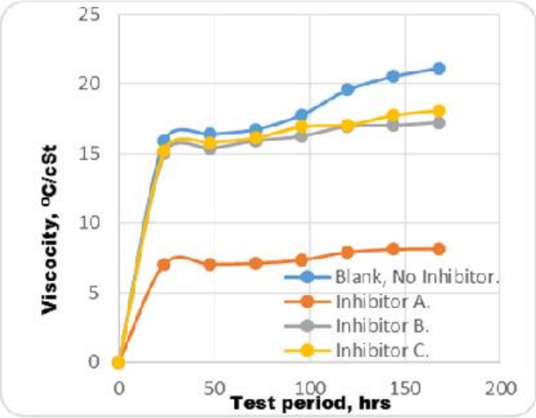


Figure 1. Viscocity/test period relation at 0.05W% for all inhibitors under investigation

3.2. Weight loss measurements

Figure 2. shows the relation between corrosion rates with respect to the investigated inhibitors, it is clear that inhibitor A is the most effective one comparing by B and C. The relation between the surface coverage and the concentration are represented by Figure 3. Inhibitors A show good adsorption on the metal surface (0.90) followed by B (0.86) and C (0.81). phenyl group may be responsible for the good efficiency of A in addition to ethylene oxide units (e.o), where it’s bi electron increase the donation tendency of this inhibitors to the metal surface and hence increase the active sites.

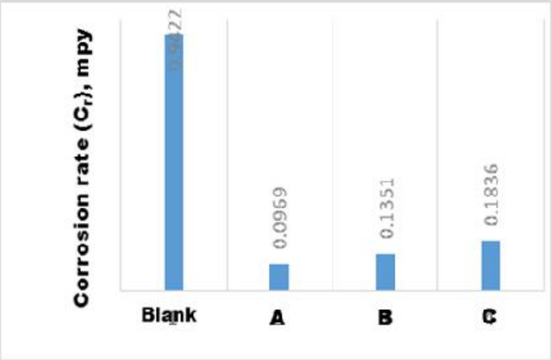


Figure 2. corrosion rate of the investigated inhibitors

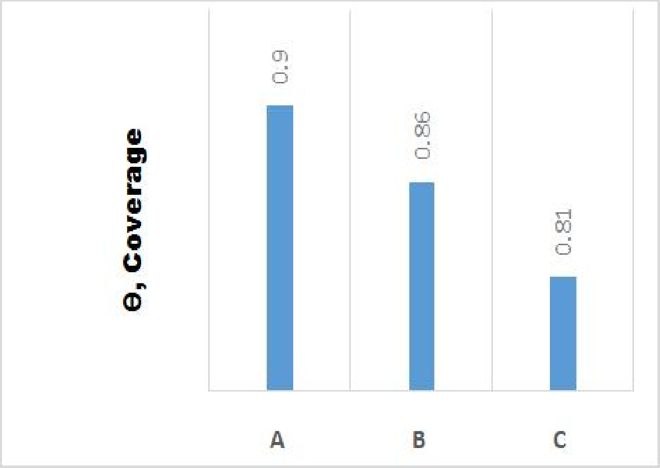


Figure 3. Surface coverage of the investigated inhibitors

Figure 4. shows the inhibition efficiency of the investigated inhibitors, the inhibition action of inhibitor A could be attributed to the adsorption of its components on mild steel surface. The formed layer, of the adsorbed molecules, isolates the metal surface from the aggressive medium leading to decreasing the corrosion rate. Although inhibitor C possesses the largest number of e.o. unit (14), it has the lowest inhibition efficiency, this may explain by the coiling of the long e.o. chain to appear shortest than other inhibitors (e.o. = 9).

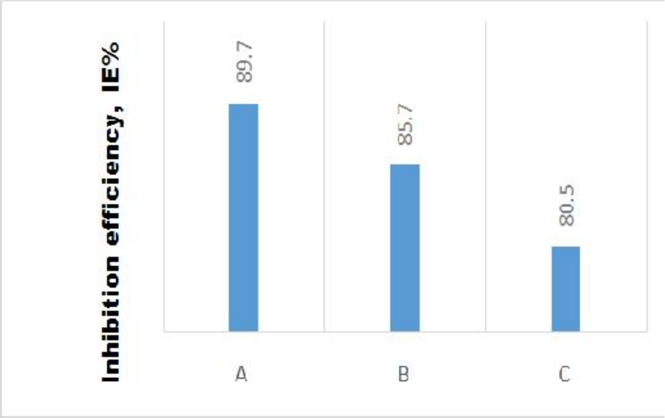


Figure 4. The inhibition efficiency of the investigated surfactants

3.3. Potentiodynamic polarization

Potentiodynamic polarization parameters are very important tool in characterizing the corrosion behavior of the metal surface, which can generally be derived from the polarization curves. Corrosion parameters as corrosion current density (Icorr), and inhibition slopes are given in **Figure 5.** and **6.** respectively. The corrosion current obtained in presence of inhibitors solutions under investigation are lower than inhibitor free solution, i.e. blank. The decrease in the corrosion current for inhibitor solutions imply that the rate of electrochemical reactions are reduced due to the formation of a barrier layer over the mild steel surface by the inhibitor molecules (Khamis *et al.*, 2013).

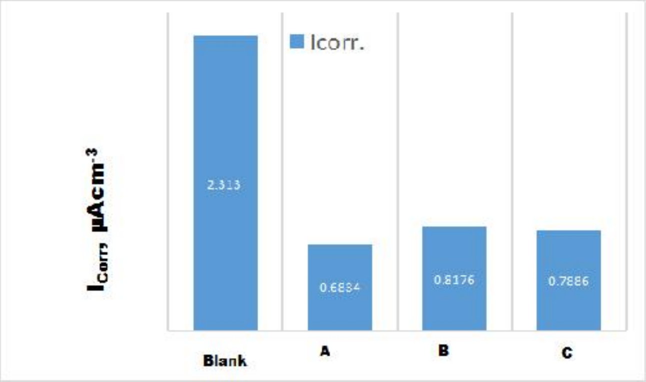


Figure 5. Variation of I_{corr} With and without inhibitors

It is cleared that, both anodic and cathodic reactions of mild steel electrode were decreased with increasing concentration of the investigated inhibitors. These results suggest that not only the addition of studied inhibitors reduce anodic dissolution but also retard the hydrogen evolution reaction. The maximum decrease in Icorr was observed for inhibitor A and the inhibition efficiency of the investigated inhibitors was increased in the following order: A > B > C. The inhibition efficiency was found to be increased as the inhibitor concentration increased which interpreted on the basis of the amount of surface coverage, increases with increasing inhibitor concentration (Mohamed and Abo-Dief, 2012). The Ecorr values of all investigated inhibitors were shifted slightly toward both cathodic and anodic directions, this may be considered due to their mixed-type behavior. The change in Ba and Bc values indicated that the adsorption process modify the mechanism of anodic dissolution as well as cathodic hydrogen evolution (Tang *et al.*, 2012; Fouda *et al.*, 2012). In addition, the cathodic Tafel slopes (Bc) are also found to be smaller than the respective anodic one (Ba), these observations are correlated with the fact that corrosion of the studied metal substrate under cathodic control.

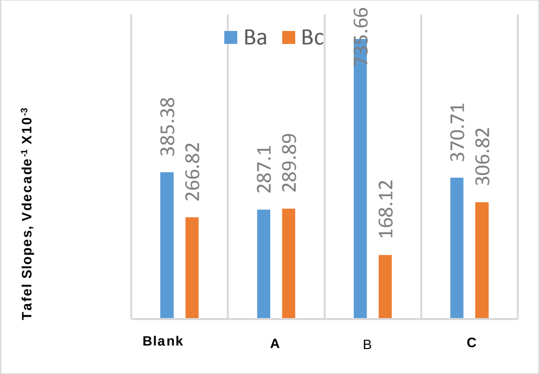
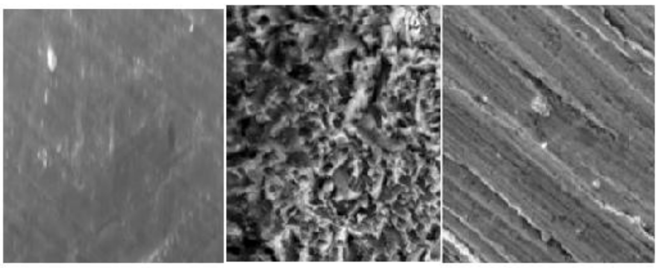


Figure 6. Variation of anodic and cathodic poteniodynamic slopes

3.4. Effect of inhibitor on mild steel morphology

A photograph of the polished mild steel surface before immersion in aggressive solution is shown in **Figure 7.a.** The photograph shows that the surface was smooth and without pits. The resulting scanning electron micrographs reveal that, the surface was strongly damaged in the absence of the inhibitor (**Figure 7.b).** The faceting seen in this figures was a result of pits formed due to the exposure of mild steel to the aggressive oil medium. Figure 7.c declare that the corrosion of mild steel is inhibited in presence of inhibitor A (most effective one). This suggests that the inhibitor is strongly adsorbed on metal surface, hindered the corrosion process.



(a) (b) (c)

Figure 7. SEM micrographs of mild steel surface (a) blank, (b) without surfactants and (c) with Surfactant A

3.5. Quantum chemical parameters

The optimized molecular structure (bond length, bond angles and dihedral angles) of the investigated inhibitors with minimum total energies. The planner geometry of the investigated inhibitors may be suggested that the molecular adsorption probably occurs in such way that the metal surface and the molecular plane were parallel to each other with largest contact area between inhibitor molecule and metal surface confirmed the donation and back donation between the investigated inhibitor molecules and metal surfaces. The quantum chemical method is introduced to study the relationship between molecular structure and inhibition efficiency of the investigated inhibitors.

3.5.1. Energies of frontier molecular orbitals (E_{HOMO} and E_{LUOM})

E_{HOMO} depicts the ability of inhibitor molecules to donate the electron when attacked by electron acceptors (3-d orbital in Fe atom), while E_{LUMO} depicts the ability of the inhibitor molecule to accept charge or electron when attacked by electron donors (4-S orbital in F atom). The value of E_{HOMO}, E_{LUMO} and energy gap $\Delta E = E_{LUMO} - E_{HOMO}$ are listed in **Table 2**.

From chemical adsorption view, an increase in E_{HOMO} cause significant increase in inhibition efficiency of the inhibitor molecule (Sastri and Perumareddi, 1997; Gao and Ling, 2007), while some authors were interpreted the negative sign of E_{HOMO} to be an indication of physical adsorption. The data listed in **Table 2** shows that inhibitor A possesses less negative value of E_{HOMO} while it is the most effective one as corrosion inhibition this may indicated that it adsorbed on metal surface is controlled essentially by chemical adsorption in addition to the physical adsorption.

Figure 8 depict the charge densities, the highest occupied molecular orbital and the lowest unoccupied molecular orbital for inhibitor A.

Table 2. The calculated quantum chemical parameters and experimental efficiency of investigated inhibitors

Inhibitors	E_{HOMO}, eV	E_{LUMO}, eV	ΔE, eV	μ, Debye	E_T, kcal/mol	A, eV	X eV	η, eV	ΔN	ω, eV	IE%
A	-8.986	0.323	9.259	0.413	-175655	8.986	0.216	4.629	0.732	0.019	89.7%
B	-10.658	-0.320	10.339	4.245	-184667	10.658	0.193	5.169	0.658	1.743	85.7%
C	-10.741	0.938	9.803	1.275	-252991	10.741	0.204	4.901	0.693	0.166	80.5%

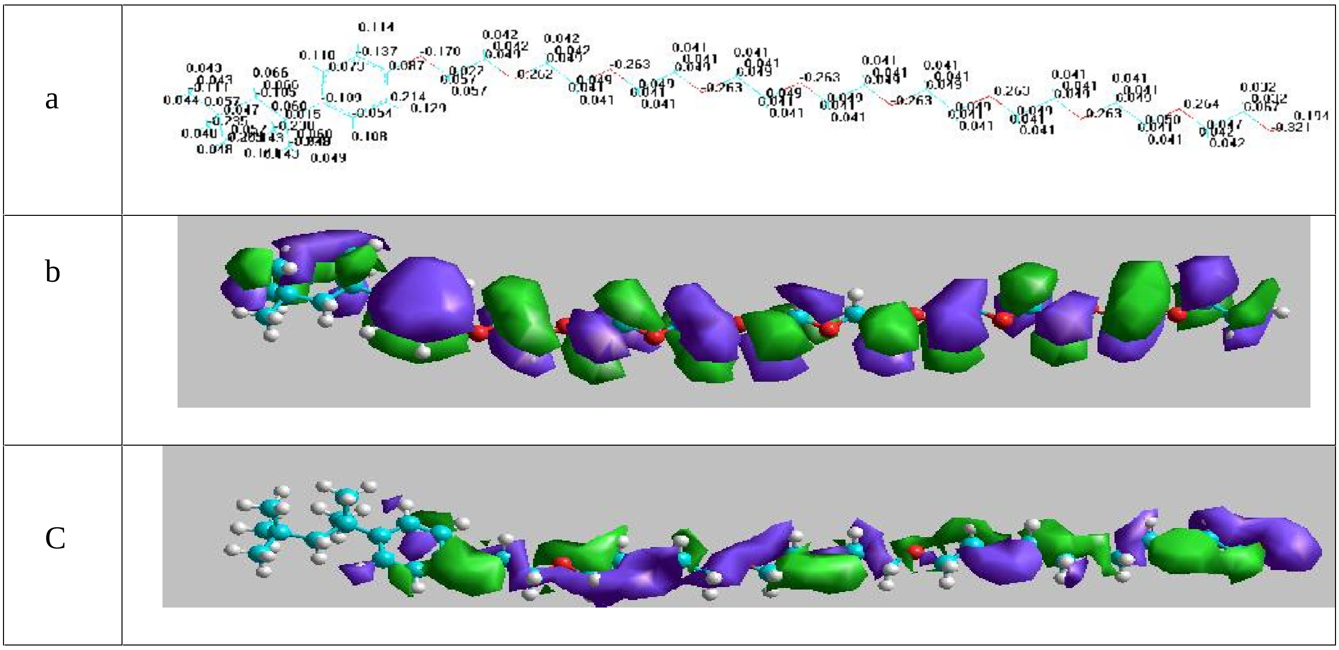


Figure 8. The optimized structure of inhibitor A (a) charge distribution (b) HOMO and (c) LUMO

Lower energy gap values render good inhibition efficiencies, because the energy needed to remove an electron from the last occupied orbital will be low (Obot *et al.*, 2009). The data listed in **Table 2** declare a satisfied agreement between the theoretical and experimental results. The most effective inhibitor A possesses the lowest energy gap value. There was good relation between the energy gap and inhibition efficiency. The inhibition efficiency increase as the energy gap ΔE decrease.

3.5.2. Dipole moment μ

The dipole moment is the electronic distribution in the molecule (Ahmad *et al.*, 2010). There is a lack of agreement in literature of the correlation between the dipole moment values and the inhibition efficiency (Ebenso *et al.*, 2010). Some author proposed linear relation between dipole moment value and efficiency while the others proposed inverse relation (Kokalj, 2010). The most effective inhibitors A has the lowest dipole moment value (inverse relationship). The decrease in the dipole moment cause the metal to favor the interaction of this inhibitor molecule on its surface.

Ju *et al*. (2008) concluded that physical adsorption results from electrostatic interaction between active centers of the molecule and metal surface. Therefore the positive sign of the coefficient of μ suggested physical adsorption. Inhibitor A possesses the lowest dipole moment confirmed that it's inhibition occur via chemical adsorption in addition to physical adsorption.

3.5.3. Total Energy E_T

The total energy of the molecule including all forms of kinetic and potential energy indicated the reactivity and stability of the molecule. As the total energies of the inhibitor molecule increase (in negative) the stability increase and the donating ability decrease. **Table 2** declare that the most effective inhibitor A (IE = 89.7%) possess the lowest value of the total energies (-175655Kcal/mol) while the lowest effective inhibitor C (IE = 80.5 %) possess the highest value of total energy (-252991Kcal/mol) show good matched between theoretical and experimental results.

3.5.4. Hardness η

The global hardness is approximated as ΔE/2. As the value of hardness increase the stability increase and the reactivity of inhibitor molecule decrease. Concerning the data listed in **Table 2** inhibitor A has the lowest hardness value and hence, has the greatest tendency to interact with the metal surface. The theoretical expectation for hardness is agreement with the experimental results

3.5.5. Electron affinity A

The electron affinity of the molecule is directly related to the energy of the lowest unoccupied molecular orbital. The higher the electron affinity the greater the ability of molecule to accept electron from 4-S orbital of the Fe atom. The data listed in **Table 2** show good correlation between the electron affinity and experimental data. Although inhibitor A has the lowest electron affinity value it has the highest inhibition efficiency. This may be due the presence of phenyl group with its bi cloud in addition to the ethylene oxide chain make the interaction of this molecule with the metal surface via its active centers (oxygen atom, л e’s) is the controlling factor of its inhibition mechanism. So, good donation of this inhibitor molecule in addition to the back donation of the metal.

3.5.6. The fraction number of the transferred electron ΔN

According to Lukovit’s study (Lukovits *et al.*, 2001), if the value of $\Delta N < 3.6$ the inhibition efficiency increase and the electron donation ability of the inhibitor increase, according to the calculating data **(Table 2)** it is observed that, the inhibition efficiency increased with the increase in the value of ΔN. From the previous discussion, low hardness value, and high value of the fraction of electron transferred enhances inhibition efficiency.

3.5.7. electrophilicity index ω

Table 2 shows the electrophilicity index values of the investigated molecules. Inhibitor A exhibit the lowest value of electrophilicity which confirm the high capacity to donate electrons. Inhibitor B exhibit lower electrophilicity index than inhibitor C, this is attributed to the large number of e.o unit in B (9) comparing with C (14) and also confirmed the suggestion of the coiling behavior of the long e.o for inhibitor C and hence it gives the lowest efficiency. From the previous discussion the adsorption mechanism of the investigated inhibitors on the metal surfaces can be explained as electron donation from the inhibitor molecules to the metal surface in addition to the back donation from metal surface to the inhibitor molecules. So, the inhibition occur via both chemical and physical adsorption.

4. Conclusion

1- The investigated compounds are efficient inhibitors for corrosion of mild steel in the studied corrosive media.
2- Polarization measurements were shown that all inhibitors act as mixed type inhibitors, inhibiting the corrosion of mild steel by blocking the active site of the metal surface.
3- The SEM results clearly indicate the presence of protective surface layer over the inhibited metal surface.
4- Quantum chemical calculation based on semi-empirical PM3 are confirmed the relation between the molecular structure and the inhibition efficiencies.

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