

Preparation and Assessment of Water Soluble Hyperbranched Polymers Based on Polyamide as Corrosion Inhibitor for Petroleum Application

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Abstract

The effect of addition of hyperbranched poly(amide-ester) (HPAE) generations 3 and 4 (AG3 and AG4) on the corrosion of steel in hydrochloric acid solution has been studied by weight loss measurements and quantum chemical calculations. The inhibition efficiency depended on the concentration of the hyperbranched poly(amide-ester). The experimental results suggest that these compounds are efficient corrosion inhibitors and the inhibition efficiencies increase with increasing their concentrations. The computed quantum chemical properties show good correlation with experimental inhibition efficiencies. Results obtained showed that hyperbranched poly(amide-ester) (HPAE) generations 4 is the best inhibitor, and the protection efficiency follows the order: AG4 > AG3. Inhibition efficiency was found maximum up to 97.05% for AG4 at the highest concentration (1000 ppm).

Keywords

AB₂-type-prepolymerized monomer, hyperbranched poly(amide-ester), corrosion, Steel

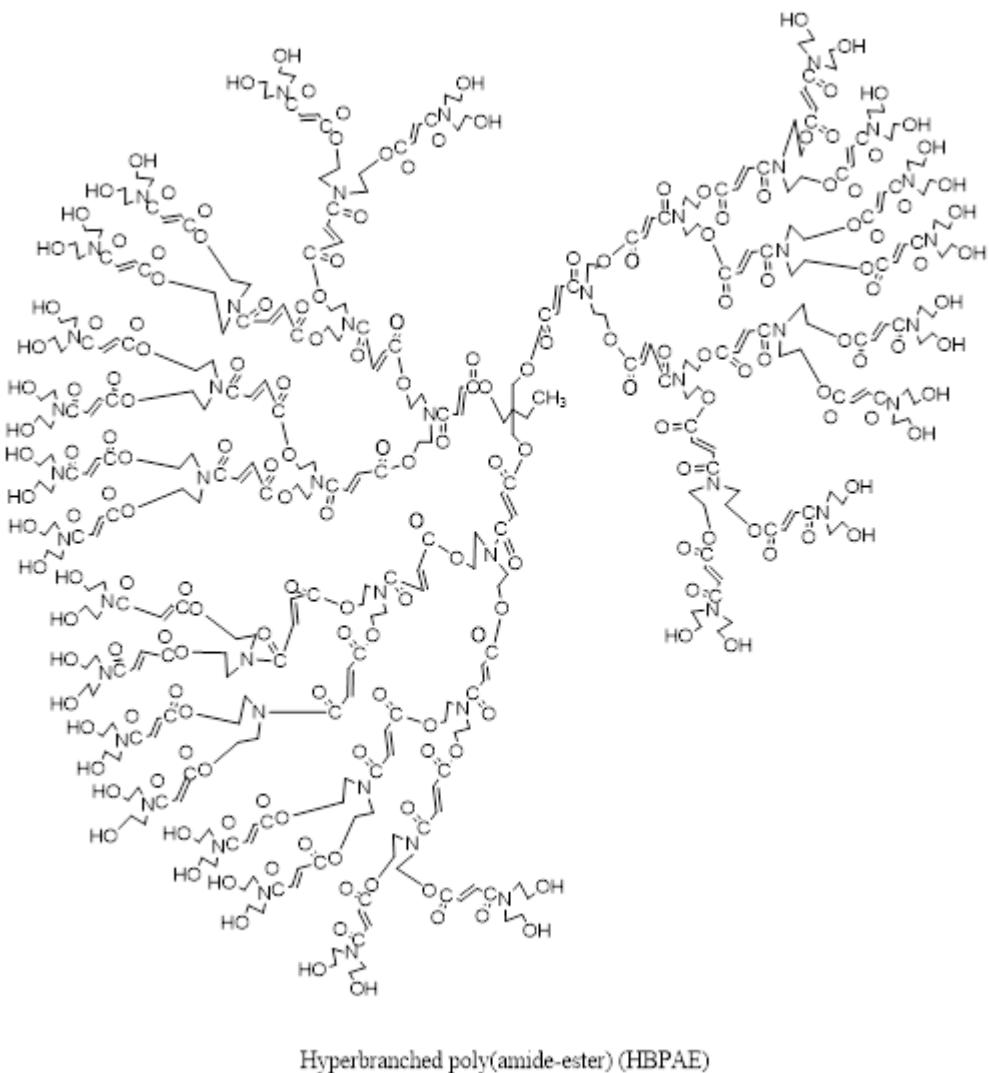
1. Introduction

Corrosion is a naturally occurring phenomenon commonly define as the deterioration of material by chemical interaction with their environment (Rokosz, et al., 2016; Louis et al., 2016; Mathina and Rajalakshmi, 2016). Corrosion of metals has cause huge economic losses involving billions of dollars each year in many industries (Verma and Khan, 2016; Louis et al., 2017; Desai, 2015).

The most efficient corrosion inhibitors are organic compounds containing electronegative functional groups and p electrons in their triple or conjugated double bonds (Emregu et al., 2006). The initial mechanism in any corrosion inhibition process is the adsorption of the inhibitor on the metal surface (El Ashry et al., 2006; Khaled, 2008; Xia et al., 2008). The adsorption of the inhibitor on the metal surface can be facilitated by the presence of hetero atoms (such as N, O, P and S) as well as an aromatic ring. The inhibition of the corrosion of metals can also be viewed as a process that involves the formation of a chelate on the metal surface, which involves the transfer of electrons from the organic compounds to the surface of the metal and the formation of a coordinate covalent bond. In this case, the metal acts as an electrophile while the nucleophilic center is in the inhibitor. Polymers find applications as effective corrosion inhibitors for steel (Olivares et al., 2006). The use of polymers as corrosion inhibitors have drawn considerable attention recently due to their inherent stability and cost effectiveness. Owing to the multiple adsorption sites, polymeric compounds adsorb more strongly on the metal surface compared with their monomer analogues (Ali and Saeed, 2001). Therefore, it is expected that the polymers will be better corrosion inhibitors. The Literature reveals that a wide range of polymeric compounds have been successfully investigated as potential inhibitors for the corrosion of metals in aggressive media. Polymers such as poly ethylenimine and poly vinylpyrrolidone, poly (o-polyvinyl pyridine and poly vinylpyrrolidone, maleic anhydride and N-vinyl-2-pyrrolidone, poly amino-benzoquinone, polyvinyl alcohol, and polyethylene glycol have been reported (Schweinsberg et al., 1996; Abd El Rehim et al., 2010; Shukla et al., 2008)

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The present article is devoted to the study of (HPAE) generation 3 and 4 (AG3 & A G4) as corrosion inhibitors for steel in 1M HCl solution using weight loss measurements and quantum chemical calculations. The structure of (HPAE) is shown in Scheme 1.



2.Experimental

2.1. Solutions

a-Hydrochloric acid solution

1 M HCl was used as a corrosive solution, and was prepared by diluting concentrated HCl (37%) to a required concentration using bidistilled water.

b- Inhibitor solutions

100 ml stock solutions of hyperbranched polymers were prepared by dissolving the desired weight of each compound in 100ml of absolute ethanol, and then the six different concentrations 50,100,200,400,500 and 1000ppm were prepared by dilution with known concentration of hydrochloric acid.

2.2. Carbon steel electrode

Corrosion tests were performed on carbon steel electrode having the following chemical composition (wt.%): 0.09% C, 0.09% Si, 0.46% Mn, 0.03% P and the remainder Fe.

2.3. Method used for Corrosion Measurements

2.3.1. Weight loss measurements

The pre-weighed test specimens were immersed in 1 M HCl solution with and without various concentrations of (HPAE) generation 3 and 4 (G3 & G4). After 12 hours, the test specimens were retrieved, washed with distilled water, rinsed with ethanol, dried with acetone and reweighed using an electronic weighing balance the difference in weight was taken as the corrosion mass loss, the same procedure was repeated after 24, 36, 48,60 and 72 hours exposure. The inhibition efficiency IE% and corrosion rater (CR) from mass loss are calculated by (Al Shamma et al., 1987):

$$\%IE = \left(\frac{W_2 - W_1}{W_2} \right) \times 100 \quad (1)$$

Where W_1 and W_2 are the weight loss of the carbon steel in the presence and absence of inhibitor, respectively.

$$\text{The degree of surface coverage } (\theta) = IE/100 \quad (2)$$

The corrosion rate (CR) was calculated from the following equation

$$CR = (m_1 - m_2) / (S.t) \quad (3)$$

where m_1 is the mass of the specimen before corrosion, m_2 the mass of the specimen after corrosion, S the total area of the specimen, t the immersion time and CR the corrosion rate.

2.3.2. Quantum chemical calculations

Quantum chemical calculations done by using an Unrestricted Hartree Fock (UHF) level which are implemented in Hyperchem 8.0 based on MINDO3 semi-empirical method ever used for organic inhibitor's calculation. The entire system was utilized at 298K, which was controlled by the Andersen thermostat, NVT ensemble, with a time step of 1.0fs and simulation time of 2000ps, using the COMPASS force field. The molecular dynamic simulation was carried out in a simulation box ($25.32 \text{ \AA} \times 25.32 \text{ \AA} \times 43.21 \text{ \AA}$) with periodic boundary conditions. The box included a Fe slab, a self- assembled molecule solution layer and a confined water layer. For the iron surface, a Fe (III) surface was selected for study because the (III) was a densely packed surface and therefore the most stable. Before the simulation, they're on plane was first cleaved from the Fe cell, and then the surface was optimized to the energy minimum and enlarged to an appropriate super cell. The energy of the highest occupied molecular orbital (HOMO), energy of the lowest unoccupied molecular orbital (LUMO), electronegativity, global hardness and softness, electron affinity, ionisation potential, etc. were calculated.

The calculation of other parameters such as logP (hydrophobic parameter), polarizability and hydration energy was obtained by QSAR method from the optimized geometry.

3. Results and discussion

3.1 Study the Protection Efficiency of C-Steel Electrode by the Chemical Testing

Technique (weight loss method).

The corrosion rates, Coverage surface and inhibition efficiency values, of carbon steel calculated using weight loss data, for various concentrations (50,100,200,400,500 and 1000ppm) of (HPAE) generation 3 and 4 (AG3 & AG4) in 1M HCl solutions are presented in Table 1. It can be deduced that there is a significant reduction in the weight loss of test specimens immersed in varying concentrations of (HPAE) in comparison to the blank solution. Additionally, as the concentration of (HPAE) increases, the weight loss reduces. The reduction in the weight loss could be attributed to the adsorption of (HPAE) generation 3 and 4 (AG3 & AG4). The adsorption of these compounds on the metal surface creates a barrier to the dissolution of the metal in corrosive medium (Bahwsar et al., 2015; Kumar et al., 2016). It is also seen from Table 1 that the inhibition efficiency obtained from weight loss method is

found to follow the order: AG4 > AG3, indicating the more beneficial effect of compound AG4 on corrosion inhibition of carbon steel. As seen from Fig. 1. It is very obvious from Fig.1 that the corrosion rate of the steel in the absence of (HPAE) decreases with the increase in concentration of the (HPAE). The stability of inhibitive behavior of (HPAE) by the trend of the inhibition efficiency as a function of time. The values of inhibition efficiency (IE%) for each concentration of (HPAE) generation 3 and 4 (AG3 & AG4) are recorded in Table 1. It was observed that the inhibition efficiency increases with an increase in inhibitor concentration This indicates that the weight loss is highly dependent on the concentration of the inhibitor.

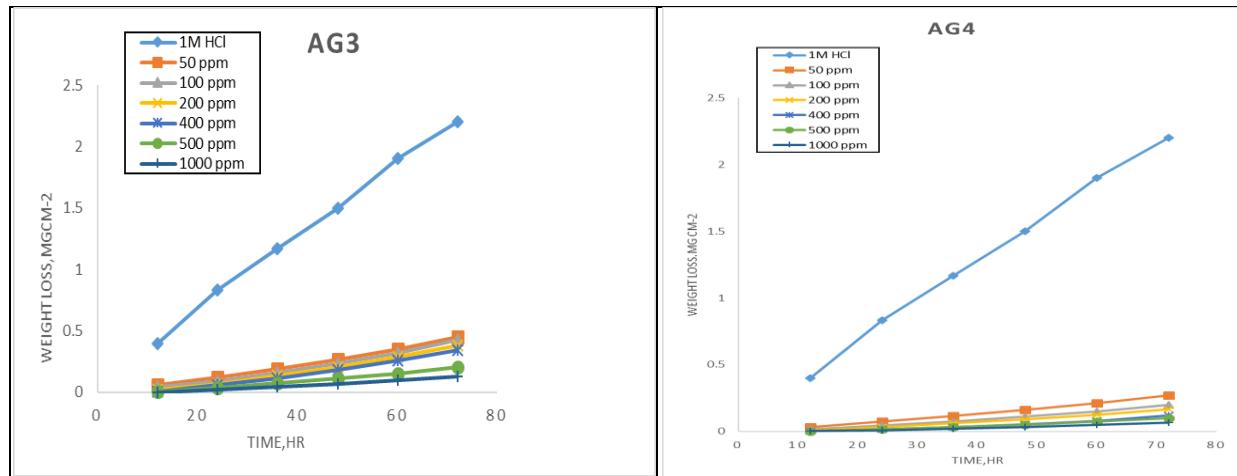


Fig. 1. weight loss method of steel immersed in 1M HCl solution in the absence and presence of different concentrations of G3 and G4 inhibitors at 25°C.

Table1.Corrosion rate (CR), Coverage surface(θ) and the inhibition efficiency (IE %) of steel in 1M HCl solutions in the absence and presence of different concentrations of G3 and G4 inhibitor obtained by weight loss method at 25°C.

Inhibitor	Concentration (ppm)	Rate of corrosion (CR) ($\text{mg cm}^{-2} \text{ hr}^{-1}$)	Coverage surface (θ)	Efficiency (IE %)
G3	Blank	15.28	-	-
	50	3.11	0.7962	79.62
	100	2.98	0.8046	80.46
	200	2.64	0.8270	82.70
	400	2.40	0.8426	84.26
	500	1.40	0.9078	90.78
	1000	0.88	0.9417	94.17
G4	50	1.88	0.8764	87.64
	100	1.39	0.9086	90.86
	200	1.16	0.9236	92.36
	400	0.82	0.9457	94.57
	500	0.70	0.9541	95.41
	1000	0.45	0.9705	97.05

3.2. Theoretical study

Molecular modeling simulation

The chemical adsorption is probably the most important type of interaction between the metal surface and the inhibitor molecule. The inhibition efficiency of inhibitors depends on the adsorption mode of those molecules on the metal surface. The ligation capability of a molecule to a metal surface depends on the electronic charge, the chelating or active atoms, i.e. the more negative charge, and the stronger is the binding capability. The investigated inhibitors have different active sites for adsorption on the metal surface. Therefore, in order to investigate the preferred adsorption site for the interaction between the inhibitor compounds and the Fe (III) surface, molecular dynamics were performed on a system comprising the investigated molecules and iron surface. The structures of the adsorbate components were minimized until they satisfied certain specified criteria. As can be seen from Table 2, inhibitor AG4 molecule showed the maximum adsorption energy found during the simulation process comparing to the other inhibitors which indicates that this molecule has the highest inhibition efficiency, which is in agreement with the experimental observations, the value of adsorption energy decreases with the corresponding decrease in experimental value. The order by which E_{ads} decreases is: AG4 > AG3; (E_{ads}). The values of binding energy were also calculated (E_{bind}), Table 2, which is the negative value of adsorption energy, $E_{bind} = -E_{ads}$ (Bhawsar et al., 2015). All inhibitors have high values of binding energy. The higher the value of binding energy, the easier the inhibitor adsorbed on the metal surface and the higher the inhibition efficiency. This agrees well with the experimental results. The close contact between the studied inhibitors and Fe (III) surface as well as the best adsorption configuration for the studied compounds are shown in Fig. 2.

Quantum chemical calculations:

Computational methods have a potential application towards the design and development of organic corrosion inhibitors in the corrosion field. According to the frontier molecular orbital theory, the formation of a transition state is due to an interaction between frontier orbitals (HOMO and LUMO) of reacting species. Thus, the treatment of the frontier molecular orbitals separately from the other orbitals is based on the general principles governing the nature of chemical reactions. The effectiveness of an inhibitor can be related with its electronic and spatial molecular structure. Certain quantum-chemical parameters that can be related to the interactions of metal-inhibitor, these are: the HOMO energy that is often associated with the capacity of a molecule to donate electrons, the energy gap ΔE (the lower values of energy gap, the better corrosion inhibition), and the dipole moment μ , because low values will favor the accumulation of inhibitor molecules on the metallic surface. A good correlation between the rate of corrosion and E_{HOMO} , as well as with energy gap ($\Delta E = E_{LUMO} - E_{HOMO}$) has been found in previous works (Khaled, 2006).

The planar geometry is clear in Fig. 3 which can offer the largest contact area between the inhibitor molecules and the steel surface.

Highest occupied molecular orbital energy (EHOMO) and lowest unoccupied molecular orbital energy (ELUMO) are very popular quantum chemical parameters. These orbitals, also called the frontier orbitals, determine the way the molecule interacts with other species. The HOMO is the orbital that could act as an electron donor, since it is the outermost (highest energy) orbital containing electrons. The LUMO is the orbital that could act as the electron acceptor, since it is the innermost (lowest energy) orbital that has room to accept electrons. According to the frontier molecular orbital theory, the formation of a transition state is due to an interaction between the frontier orbitals (HOMO and LUMO) of reactants. Fig. 4 shows that the energy of the HOMO is directly related to the ionization potential and the energy of the LUMO is directly related to the electron affinity. The HOMO-LUMO gap,

i.e. the difference in energy between the HOMO and LUMO, is an important stability index. Low values of the energy gap ΔE will render good inhibition efficiencies, because the energy needed to remove an electron from the last occupied orbital will be low. The concept of “activation hardness” has been also defined on the basis of the HOMO–LUMO energy gap. The qualitative definition of hardness is closely related to the polarizability, since a decrease of the energy gap usually leads to easier polarization of the molecule. Moreover, MO level is the gap between the HOMO and LUMO energy levels for the studied molecule. As was reported, some atoms such as N and O have unoccupied d orbitals and so exhibit a tendency to obtain electrons; the electrons in the d orbitals can easily be offered because the applied force they affect is small. From Table 3 it can be seen that compound AG4 have the lower HOMO–LUMO energy gap ΔE of 6.284. The lower the LUMO energy, the easier the acceptance of electrons from metal surface, which decreases LUMO–HOMO energy gap and improves the efficiency of inhibitor.

(ΔN) also calculated depending on the quantum chemical method. Values of ΔN showed inhibition effect resulted from electrons donation. if $\Delta N < 3.6$, the inhibition efficiency increased with increasing electron-donating ability at the metal surface. The fraction of electrons transferred from inhibitor to the iron surface, was calculated. In this study, compound AG4 will be expected to inhibit the iron corrosion through donations of electrons to the iron surface that will be the electron acceptor as indicated in Table 3. compound AG4 has the highest HOMO energy and ΔN values, and it has the greatest ability of offering electrons, while compound AG4 expected to have the lowest inhibition efficiency, for vice versa.

The hydrophobicity of an organic molecule increases with decreasing water solubility. Log P accounts for the hydrophobicity of an actual molecule. It can be shown from Table 3 that the inhibition efficiency for all investigated compounds increases with increasing log P value. Because the log P value depends on the molecule hydrophobicity.

From Table 4, there is a good correlation between the electron affinity and the efficiency for all the compounds. Electron affinity values are all negative, indicating that their inhibition potential may be related to the tendency of the molecules to be electrophilic. As the electron affinity increase along each compound, the affinity of the compounds to accept electrons increase and the energy given off increase. Electronegativity is related to the ability of the molecule to draw electron toward itself as it represents the negative of the electronic chemical potential. Hence, it is related to the efficiency and electron affinity. Thus, the electronegativity and hardness are of course used extensively to make predictions about chemical behavior. In other words, large electronegativity values characterize the acceptor and small electronegativity values are found for the donators. As the electronegativities of the considered compound AG4 (3.08) are smaller than the metallic iron (7 eV mol⁻¹) and conversely, the electronic chemical potential of the compound AG4 are larger than the metallic iron (-7 eV mol⁻¹), this indicates the flow of electrons from the molecule, which has the highest chemical potential (to the metal, which has the lowest chemical potential). Global hardness provides information about the reactive behavior of molecules. A high value of the absolute hardness is, thus, an indication of high stability and low reactivity. As the global hardness decrease, the inhibition efficiency increases and vice versa. The values of increasing softness are in good agreement with the experimental results, since there is a positive relation between the softness and the efficiency. Hence, the best inhibitor is the one which has the highest value of (σ), which also indicates that higher number of electrons is transferred (Ikpi, et al. 2017).

It is confirmed that, the more negative the atomic partial charges of the adsorbed center, the more easily the atom donates its electrons to the unoccupied orbital of the surface atoms of the metal. In Table 4, the oxygen atomic charges are more negative than the

nitrogen atomic charges, so the oxygen atoms more easily adsorbed on the carbon steel surface.

Based on the discussion above, it can be concluded that the compound AG4 will have many active centers for adsorption on iron surface. These are areas containing N and O atoms and are the most possible sites for bonding to iron surface through donating electrons to the iron surface.

The molecular electrostatic potentials (MEPs) are very helpful in the negative regions can be regarded as nucleophilic centers, whereas regions with positive electrostatic potential are potential electrophilic sites. Moreover, the electrostatic potential makes the polarization of the electron density visible. The calculations showed that the oxygen and Nitrogen atom have negative electrostatic potential which means that these sites are the active centers for the binding to the metal surface, Fig. 5. The use of Mulliken population analysis to estimate the adsorption centers of inhibitors have been widely reported and it is mostly used for the calculation of the charge distribution over the whole skeleton of the molecule. There is a general consensus by several authors that the more negatively charged heteroatom is, the more it can be adsorbed on the metal surface through donor–acceptor type reaction.

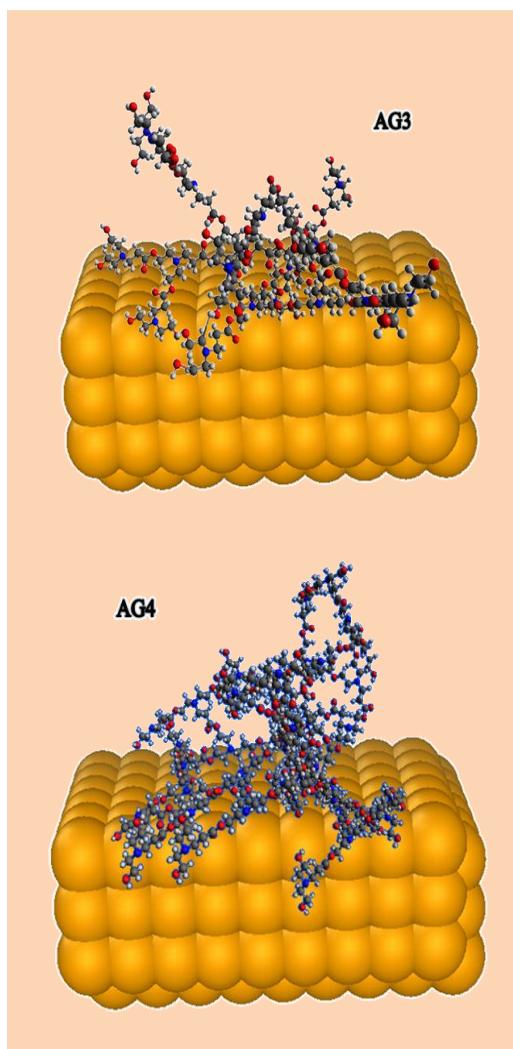


Fig. 2. Equilibrium configuration and most radial distribution function of inhibitor molecules adsorbed on a Fe(III) surface

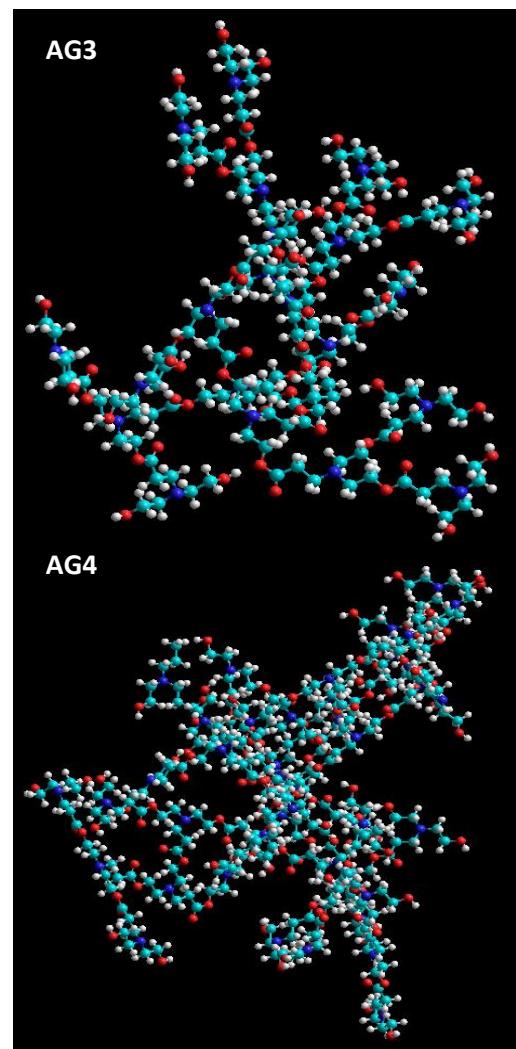


Fig. 3: Optimized geometries of the investigated compounds

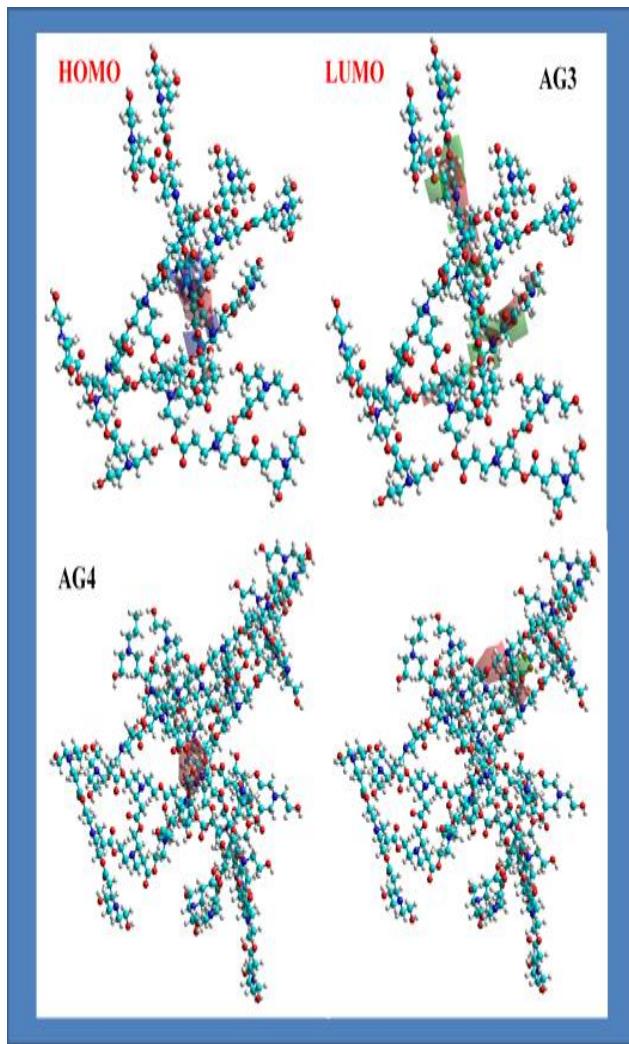


Fig. 4: The frontier molecule orbital density distributions of the investigated compounds

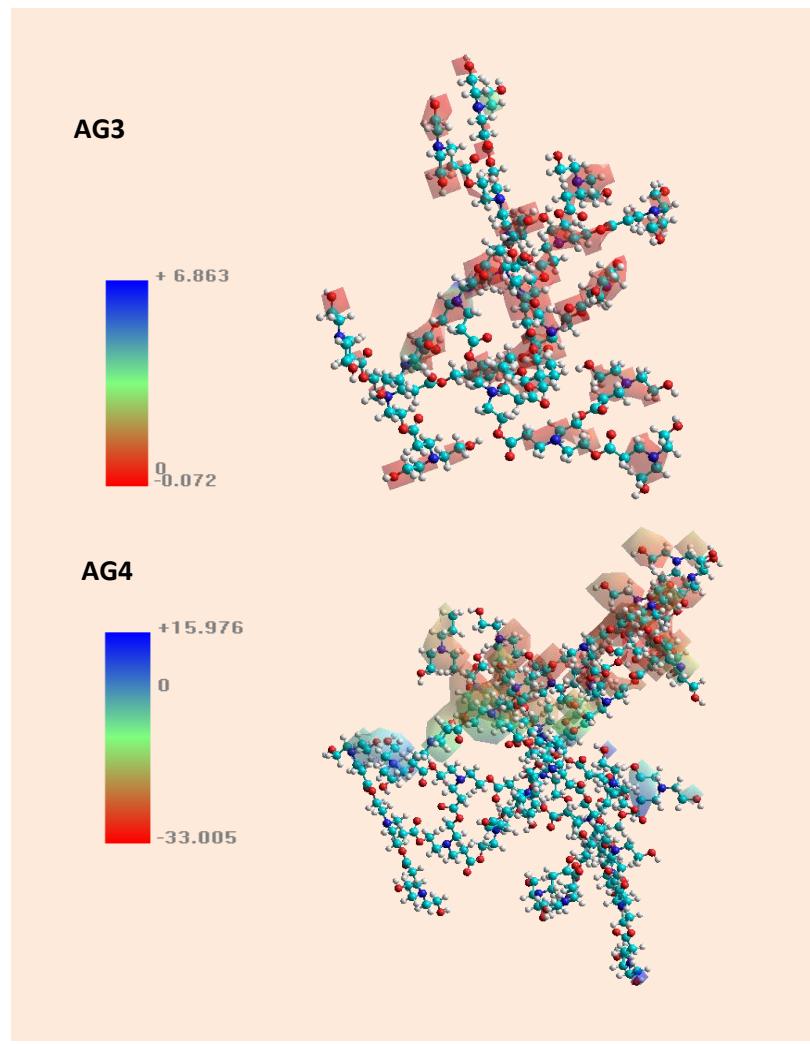


Fig. 5: Molecular electrostatic potential map of investigated compounds

Table 2. Binding energies and adsorption energy of investigated inhibitor

Inhibitor	E _{adsorption} (eV)	E _{binding} (eV)
AG3	-475.21	475.21
AG4	-631.65	631.65

Table 3: Quantum chemical parameters of the investigated compounds.

Compound	E _{HOMO} (eV)	E _{LUMO} (eV)	ΔE(eV)	μ(debye)	LogP	ΔN
AG3	-6.029	1.32	7.349	13.04	-15.05	0.632
AG4	-6.231	0.053	6.284	12.91	-20.32	0.652

Table 4: Other Calculated Quantum Chemical Parameters of the Investigated Inhibitors

Inhibitor	Ionization Potential, I (eV)	Electron Affinity, A (eV)	Electronegativity (eV mol ⁻¹)	Global Hardness, (eV mol ⁻¹)	Softness, $\sigma = 1/\eta_{inh}$ (eV ⁻¹)
AG3	6.029	-1.32	2.354	3.672	0.272
AG4	6.231	-0.053	3.089	3.142	0.318

Conclusions

- (1) The results showed that inhibitors (HPAE) generation 3 and 4 (G3 & G4) have excellent inhibition efficiency for the corrosion of carbon steel in 1 M HCl. The inhibition efficiency increases in the following order G4 > G3.
- (2) The weight loss measurements show that the inhibition efficiency increasing with increasing the inhibitor concentration.
- (3) Inhibition efficiency was found maximum up to 97.05% for G4 at the highest concentration (1000 ppm).
- (4) The calculated quantum chemical parameters were found to give good reasonably good correlation with the efficiency of corrosion inhibition.

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الملخص العربي

تحضير وتقديم بوليمرات عديدة التفرع مبنية على البولي إميد كموانع لتأكل التطبيقات البترولية

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يعد تأكل المعادن مشكلة صناعية تلقى اهتماماً كبيراً ويدخل الصلب الكربوني في العديد من التطبيقات الصناعية مثل التطبيقات البحرية-المعالجة الكيميائية-انتاج وتكرير البترول والبناء ومعدات تجهيز المعادن. هذه التطبيقات تحدث عادة تأكل المعدات وخطوط الأنابيب المصنوعة من الحديد وبسبائكه. وجد ان البوليمرات لها تطبيقات هامة كموانع لتأكل الصلب. ويهدف هذا البحث الى استخدام مركبات بوليمرية عديدة التفرع كمواد مثبتة للتأكل.

وقد تم تقييم مدى كفاءة البوليمرات المحضرة كموانع للتأكل باستخدام تقنيات مختلفة مثل (الفقد في الوزن - حسابات قيم الكم الكيميائية) ووجد ان تلك المركبات لها كفاءة عالية في تثبيط ا للتأكل للصلب الكربوني.