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SYNTHESIS OF THE PFAQ CORROSION INHIBITOR AND RESULTS OF QUANTUM CHEMICAL CALCULATIONS

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Abstract. A novel oligomer-type corrosion inhibitor (PFAQ) based on p-phenylenediamine, formalin, and Alpha amino succinate was successfully synthesized under an inert nitrogen atmosphere. The synthesis conditions were systematically optimized, revealing that the highest product yield (≈86–88%) was achieved at a molar ratio of 1:2:2 and a temperature range of 40–65 °C, while higher temperatures led to a significant decrease in yield. The chemical structure of the synthesized inhibitor was confirmed by FT-IR spectroscopy, which revealed characteristic absorption bands corresponding to –NH, –CH, –CH_2, and C=O functional groups, indicating successful polymer formation. The reactivity and inhibition mechanism of the PFAQ molecule were investigated using semi-empirical quantum chemical methods (AM1, MNDO, PM3, RM1, and MNDO3) as well as DFT calculations at the B3LYP/6-311G(d,p) level. Mulliken charge analysis demonstrated that the highest negative effective charges are localized on oxygen atoms of C=O and C–O–C groups and on nitrogen atoms of secondary amine (N–H) groups, confirming their role as active donor centers capable of forming coordination bonds with metal surfaces. Frontier molecular orbital (HOMO–LUMO) analysis revealed significant electron density on –O–, C=O, and N–H groups, supporting strong adsorption behavior. The calculated global reactivity descriptors, including energy gap, chemical hardness and softness, electronegativity, electrophilicity index, and dipole moment, indicate high adsorption capability and effective corrosion inhibition performance. Additionally, molecular electrostatic potential analysis confirmed the presence of distinct electrophilic and nucleophilic regions, favoring stable metal–inhibitor complex formation. Overall, the obtained experimental and theoretical results demonstrate that PFAQ is a thermodynamically favorable, electronically active, and promising corrosion inhibitor for steel protection in aggressive acidic environments.

Keywords: p-phenylenediamine, formalin, alpha amino succinate, FT-IR spectroscopy, semi-empirical quantum chemical methods, HOMO–LUMO.

Introduction

Metal corrosion is a widespread problem that reduces service life and increases maintenance costs. Traditional synthetic inhibitors can be toxic and environmentally harmful, motivating the search for greener solutions. Plant extracts and plant-based nanoparticles offer promising, sustainable corrosion inhibition due to their natural active constituents and low toxicity[1,2].. Three amino acids with different functional groups—L-methionine, L-arginine, and L-aspartic acid—were investigated as corrosion inhibitors for carbon steel in 0.5 M HCl in the presence of potassium iodide. Electrochemical measurements, surface analyses, and theoretical calculations revealed a pronounced synergistic inhibition effect between the amino acids and KI, leading to significantly enhanced inhibition efficiency, particularly at elevated temperatures. The synergistic mechanism and the influence of amino acid molecular structure on inhibition performance were systematically analyzed[3,4]. Two amino acid–based green corrosion inhibitors, 4-(tert-butylbenzoyl)methionine and 4-(tert-butylbenzoyl)cysteine, were synthesized

via a one-step acylation method and evaluated for Q235 steel in 1 M HCl. Both inhibitors significantly reduced corrosion rates by chemical adsorption, with P-Meth exhibiting higher inhibition efficiency, confirmed by electrochemical, surface, and molecular dynamics analyses[5,6]. Amino acid alkali salts are eco-friendly, stable, and low-cost additives for acidic corrosion inhibitors. This study shows that taurine and aminohexanoic acid reduce Co and Ni leaching and enhance the performance of carboxylic and phosphonic acid inhibitors. Thus, they are promising sustainable alternatives to conventional alkaline additives[7,8]. Metal corrosion causes significant economic losses and safety hazards, yet effective and water-soluble inhibitors remain limited. This study evaluates two quinoxaline derivatives, BrSQX and MeSQX, as inhibitors for mild steel in 1.0 M HCl using weight loss, electrochemical, surface, and theoretical analyses. Both compounds showed high inhibition efficiency, with MeSQX performing slightly better, and their adsorption followed the Langmuir isotherm, indicating chemisorption and protective film formation[9,10].

This work aims to study the synthesis and structure of a new type of corrosion inhibitor based on p-phenylenediamine.

Materials. Compounds of p-paraphenylenediamine, formalin, and alanin were used for this study. All chemical reagents were purchased as "chemically pure" from "Merit Chemicals" company.

Methods.

IR- analysis. The IR spectra of the synthesized compounds were recorded using a Bruker Tensor 27 FTIR spectrometer (Germany) in the range of 4000–400 cm^{-1}.

Quantum chemical analysis. Quantum-chemical calculation of the reactivity of the corrosion inhibitor molecule using Avogadro, Hyper Chem 8.01, Asselrys MS Modeling 3.0.1 using the constrained calculations were carried out using the Intel Pro Pentium 1.40 GHz computer.

Synthesis of the corrosion inhibitor (PFAQ)

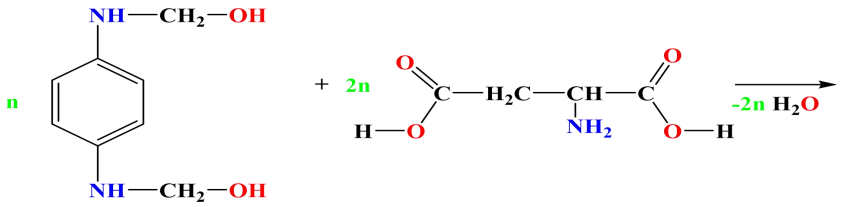
In the synthesis of this corrosion inhibitor, 0.1 mol of p-phenylenediamine and 0.2 mol of formalin were stirred under reflux in a nitrogen atmosphere for 30 minutes at a temperature range of 44–50 °C. Then, 0.1 mol of the initially formed compound and 0.2 mol of alpha amino succinic acid solution dissolved in double-distilled water were slowly added dropwise while stirring. During this process, the temperature was maintained in the range of 55–65 °C.

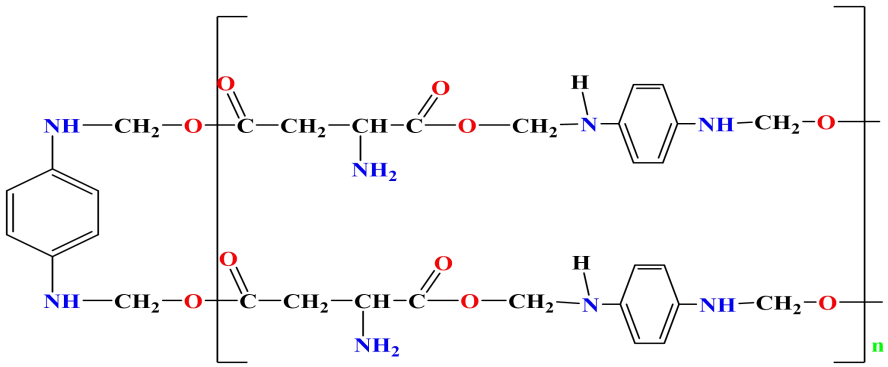
Table-1.

Dependence of the yield of PFAQ corrosion inhibitor synthesis on the molar ratio of the initial reactants and temperature

p-phenylenediamine *formalin*+ alpha amino succinate	Temperature ^{0}C	Yield %	Temperature ^{0}C	Yield %

1:1:1	40÷65	56.67	65≤t	41.15
1:2:2		86.37		82.36
2:2:1		73.21		61.26
2:1:3		55.62		32.56
1:2:1		69.43		42.25





As shown in Table 1, the highest yield was obtained at temperatures in the range of 40–65 °C and an initial molar ratio of 1:2:2. When the temperature exceeded 65 °C, the product yield decreased significantly.

Results and Its Discussion

FT-IR spectral analysis of the PFAQ corrosion inhibitor

Figure 1 shows the FT-IR (BRUKER) spectrum of PFAQ, in which characteristic absorption bands confirm its molecular structure. The bands observed at 3414.21, 3307.62, 3184.19, 3134.33, 1599.69, 1549.72, 1489.05, and 1470.46 cm^{-1} correspond to the symmetric stretching (νs) and deformation (δ) vibrations of the ν(NH) groups. Absorption bands at 3089.19 and 1275.31 cm^{-1} are attributed to the asymmetric (νas), symmetric (νs), and deformation (δ) vibrations of aromatic ν(CH) bonds, while those at 2860.44, 2833.78, and 1423.04 cm^{-1} correspond to ν(CH_2) group vibrations. In addition, a strong band at 1623.59 cm^{-1} is assigned to the stretching vibration of the ν(C=O) group, and the band at 840.43 cm^{-1} corresponds to its deformation vibration.

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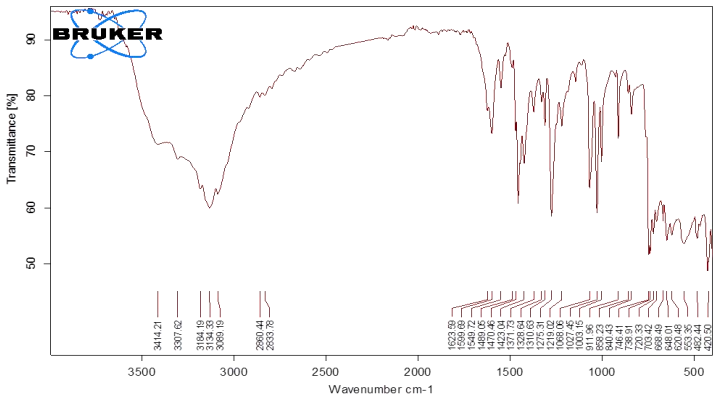

Figure 1. FT-IR spectrum of the PFAQ corrosion inhibitor

Quantum chemical analysis of the reactivity of PFAQ

The reactive properties of the PFAQ molecule were investigated by quantum chemical calculations using the Avogadro, HyperChem 8.01, and Accelrys MS Modeling 3.0.1 software packages. Semi-empirical (UHF) methods with the SCF-MO approach were applied, employing the AM1, MNDO, PM3, RM1, and MNDO3 methods on an Intel Pro Pentium 1.40 GHz computer. Geometry optimization of the molecule was performed using the Polak–Ribiere (conjugate gradient) algorithm. These methods enable the determination of the total molecular energy, electron density distribution of molecular orbitals, and full geometric optimization of the studied molecule[11,12].

One of the key electronic characteristics is the Mulliken atomic charges (CHARGES) and the total energy of the system (TOTAL ENERGY) (Table 2). Based on the results of the semi-empirical AM1, MNDO, PM3, RM1, and MNDO3 calculations, it can be concluded that high negative effective charges in the PFAQ molecule are localized on the oxygen atoms of the C=O and C–O–C groups and on the nitrogen atoms of secondary amine (N–H) groups. This indicates the ability of these atoms to form coordination bonds with metal surfaces, leading to the formation of stable complexes[13,14].

Table-2.

Effective charge distribution on donor atoms of PFAQ molecules

Calculation method	PFAQ

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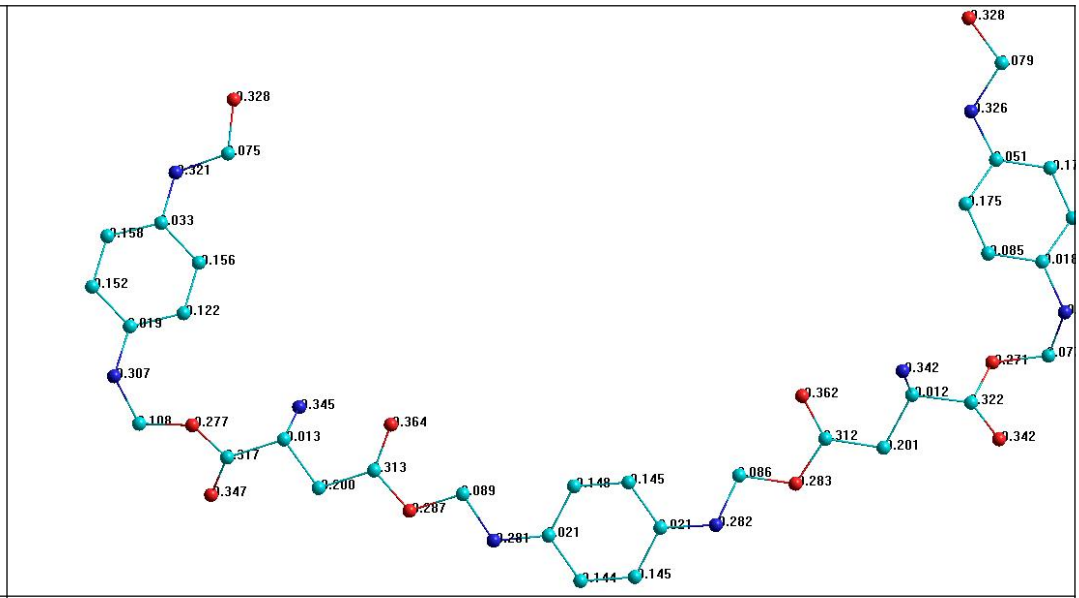
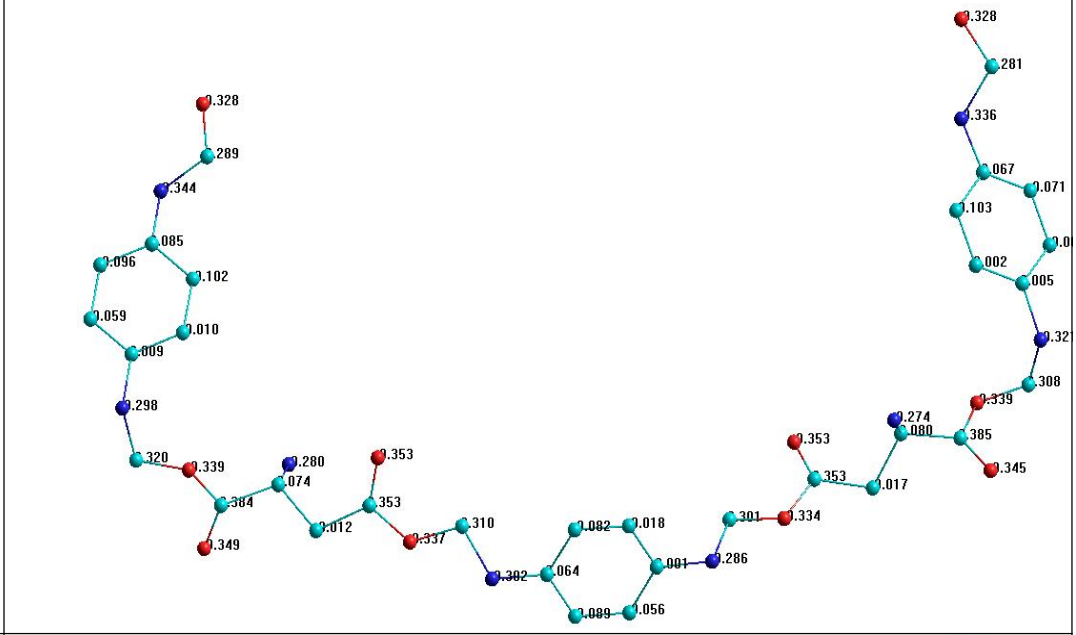
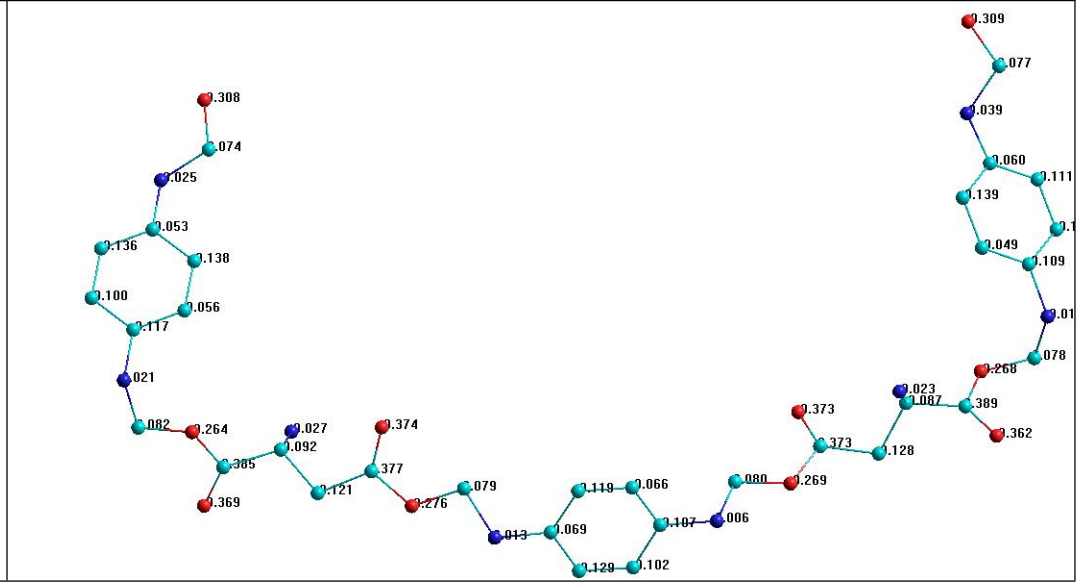
AM1	
MNDO	
PM3	

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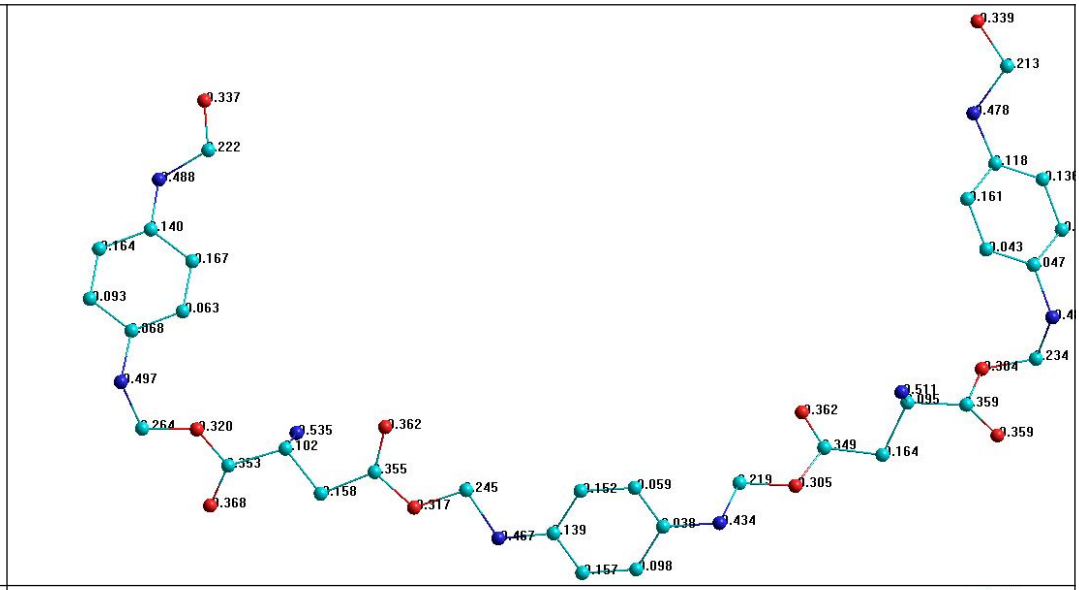
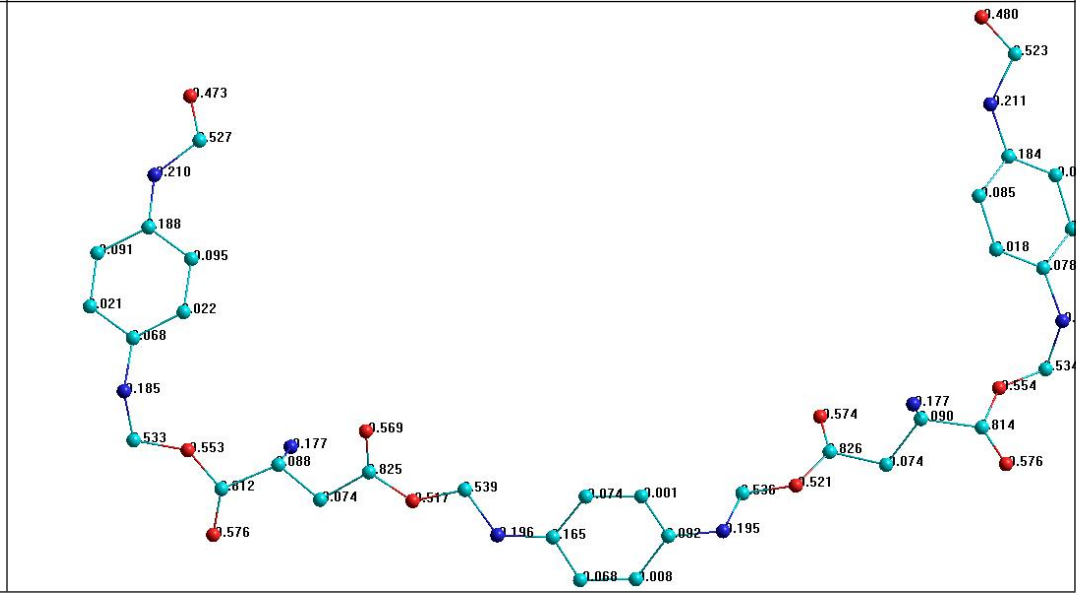
RM1	
MNDO 3	

Table-3.

Effective charge values of donor atoms in PFAQ molecules

Atoms	AM1, eV	MNDO, eV	PM3, eV	RM1, eV	MNDO3, eV
PFAQ					
$\delta_q O^1$(C-O)	-0,328	-0,328	-0,309	-0,339	-0,480
$\delta_q N^1$(NH)	-0,326	-0,337	-0,039	-0,478	-0,211
$\delta_q N^2$(NH)	-0,288	-0,326	-0,015	-0,483	-0,202

$\delta_q O^1$(C-O-C)	-0,271	-0,343	-0,268	-0,304	-0,554
$\delta_q O^1$(C=O)	-0,342	-0,343	-0,362	-0,359	-0,576
$\delta_q N^3$(NH)	-0,342	-0,275	-0,023	-0,511	-0,177
$\delta_q O^2$(C=O)	-0,362	-0,353	-0,373	-0,362	-0,574
$\delta_q O^2$(C-O-C)	-0,283	-0,337	-0,269	-0,305	-0,521
$\delta_q N^4$(NH)	-0,282	-0,289	-0,006	-0,434	-0,195
$\delta_q N^5$(NH)	-0,281	-0,304	-0,013	-0,467	-0,196
$\delta_q O^3$(C-O-C)	-0,287	-0,339	-0,267	-0,317	-0,517
$\delta_q O^3$(C=O)	-0,364	-0,353	-0,374	-0,362	-0,569
$\delta_q N^6$(NH)	-0,345	-0,283	-0,027	-0,535	-0,177
$\delta_q O^4$(C=O)	-0,347	-0,347	-0,369	-0,368	-0,576
$\delta_q O^4$(C-O-C)	-0,277	-0,343	-0,264	-0,320	-0,553
$\delta_q N^7$(NH)	-0,307	-0,304	-0,021	-0,497	-0,185
$\delta_q N^8$(NH)	-0,321	-0,346	-0,025	-0,488	-0,210
$\delta_q O^2$(C-O)	-0,328	-0,328	-0,308	-0,337	-0,473
E	-9466,5252 (kcal/mol)	-9448,0230 (kcal/mol)	-9473,9782 (kcal/mol)	-7642,2949 (kcal/mol)	-9531,4473 (kcal/mol)

Geometric Optimization and Quantum Chemical Analysis of PFAQ

The geometric optimization of PFAQ was carried out using the output file generated by the Avogadro software package. The structure was fully optimized in GaussView 6.0.16 using the RHF/6-311G(d,p) basis set. Density Functional Theory (DFT) calculations were performed with the B3LYP method, and the results obtained in GaussView 6.0.16 were used to compute atomic charges using the Mulliken population analysis. The charge distribution of all atoms was calculated based on the frontier molecular orbital (FMO) approximation[15,16].

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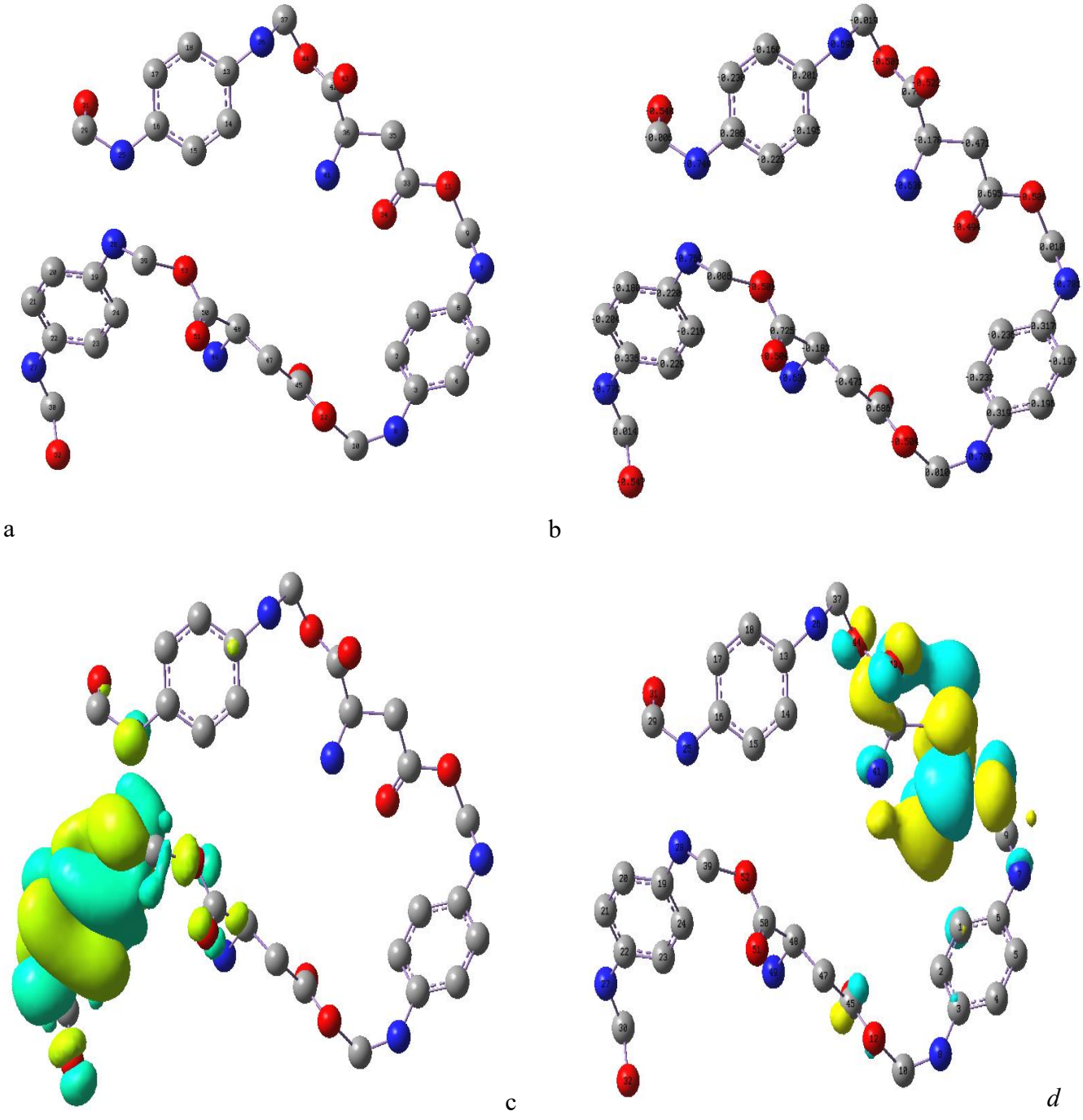
Charge distribution on molecular atoms plays a crucial role in reactivity, as it helps predict electrophilic and nucleophilic centers within the molecule. Considering this, the widely used Mulliken method was applied to compute the total charges of atoms in PFAQ molecules. The calculation results indicate that the most negative charge localization occurs on nitrogen and oxygen atoms in the PFAQ molecule (Figure 2b).

One of the main indices used to evaluate the reactivity of organic compounds is the electron density distribution in frontier molecular orbitals (FMOs). The Highest Occupied Molecular Orbital (HOMO) and the Lowest Unoccupied Molecular Orbital (LUMO) are considered the frontier molecular orbitals. In the HOMO, atoms with higher electron density act as electron-donor centers and are considered electrophilic reactive sites. In the LUMO, atoms with higher electron density act as electron-acceptor centers and are considered nucleophilic reactive sites[17,18].



a b c *d*

YuBMO = -7,02 *eV* QBMO = 3,85 *eV*

Figure 2. Distribution of Effective Charges on Donor Atoms in PFAQ Molecules

The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) exhibit the largest charge density distribution on the -O-, C=O, and N-H groups (Figures 2c,d). The energies of the HOMO and LUMO orbitals, as well as their difference (ΔE = ELUMO – EHOMO, eV), are also of significant importance. An increase in HOMO energy (raising the band level toward the LUMO) indicates enhanced electron-donor ability of the compound, while the LUMO energy determines the electron-acceptor capability (affinity) of the compound. The ionization potential (I = -EHOMO, eV), electron affinity (A = -ELUMO, eV), electronegativity (χ = (I + A)/2, eV), chemical hardness (η = (I – A)/2, eV), chemical potential (μp = -(I + A)/2, eV), chemical softness (σ = 1/(2η), eV^{-1}), electrophilicity index (ω = μp^2 / 2η, eV), and dipole moment (μ, Debye) were calculated. The results are presented in Table 4[18,20].

Table 4.

Quantum Chemical Parameters Calculated for PFAQ

Parameter	Formula	Qiymat (eV)
E_{HOMO}	–	−4.92
E_{LUMO}	–	−0.11
$\Delta E = E_{LUMO} - E_{HOMO}$	−0.11 − (−4.92)	4.81
Ionization potential (I)	−EHOMO	4.92
Electron affinity (A)	−ELUMO	0.11
Electronegativity (χ)	(I + A)/2	2.52
Chemical hardness (η)	(I − A)/2	2.41
Chemical potential (μp)	−(I + A)/2	−2.52
Chemical softness (σ)	1/(2η)	0.21
Electrophilicity index (ω)	$\mu p^2/(2\eta)$	1.32
Dipole moment (μ)	–	5.42 D

It is known that the electrostatic potential surface is used to determine the electron-donating and electron-accepting regions of a molecule. In MEP (molecular electrostatic potential) maps, red, blue, and green colors correspond to negative, positive, and neutral electrostatic potentials, respectively. Negative electrostatic potentials were observed on atoms O12, O31, O32, O34, O43, O46, O51, N25, and N49**,** while positive electrostatic potentials were detected on atoms N7, N8, N41, N25, N27, and N28 (Figure 3).

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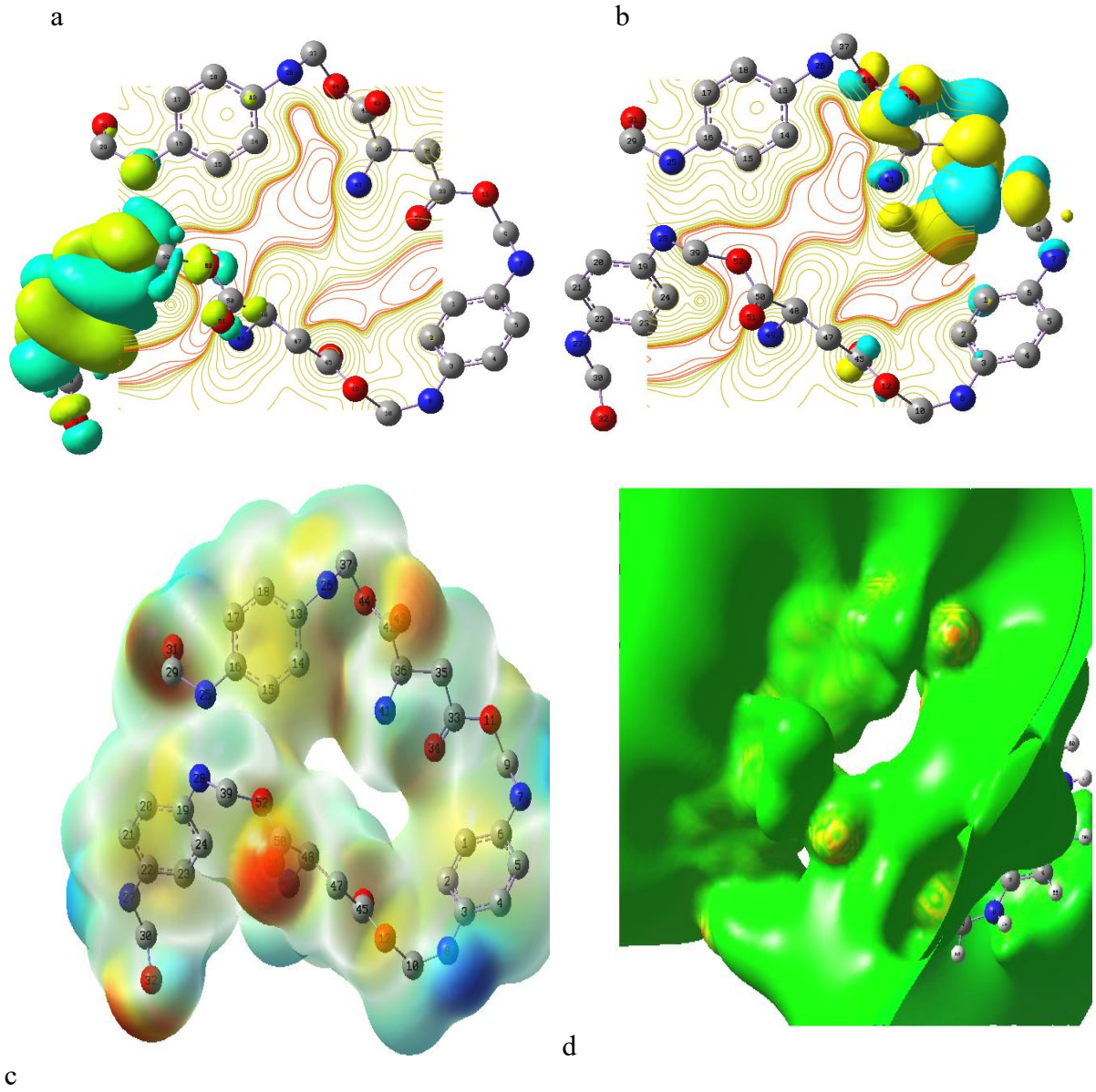

Figure 3. ESP–HOMO (a), ESP–LUMO (b) and ESP (c, d) of PFAQ.

The calculated EHOMO and ELUMO values of the PFAQ molecule indicate its electron donor and acceptor properties, and the relatively small ΔE (4.81 eV) suggests strong adsorption potential on metal surfaces, while the η and χ values are important for assessing its chemical stability and reactivity[21-24].

Conclusion

The novel oligomer-type corrosion inhibitor PFAQ was successfully synthesized from p-phenylenediamine, formalin, and alpha amino succinate under an inert nitrogen atmosphere, with optimized reaction conditions yielding a high product yield (≈86–88%) at a molar ratio of 1:2:2 and a temperature range of 40–65 °C. FT-IR spectral analysis confirmed the formation of the polymeric structure, showing characteristic absorption bands of –NH, –CH, –CH_2, and C=O functional groups, which indicate successful polymerization and the presence of active heteroatom centers. Quantum chemical calculations using semi-empirical methods (AM1, MNDO, PM3, RM1, MNDO3) and DFT (B3LYP/6-311G(d,p)) demonstrated that the most negative effective charges are localized on oxygen atoms of C=O and C–O–C groups and on

nitrogen atoms of secondary amine groups, identifying them as primary donor sites for coordination with metal surfaces. HOMO–LUMO analysis revealed significant electron density on –O–, C=O, and N–H groups, while a relatively small energy gap (ΔE = 4.81 eV) indicates high chemical reactivity and strong adsorption potential. Furthermore, global reactivity descriptors such as chemical hardness, softness, electronegativity, electrophilicity index, and dipole moment, together with MEP mapping, confirm the existence of distinct nucleophilic and electrophilic regions, favoring stable metal–inhibitor complex formation. Overall, the combined experimental and theoretical results indicate that PFAQ is a thermodynamically favorable, electronically active, and promising corrosion inhibitor for steel protection in aggressive acidic media.

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