SYNTHESIS OF CORROSION INHIBITOR BASED ON P-PHENYLENEDIAMINE, FORMALIN, AND ALANINE AND ITS QUANTUM CHEMICAL CALCULATION RESULTS

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Abstract. In this study, the synthesis, physicochemical properties, and reactivity of a new PFA-branded polymer corrosion inhibitor synthesized from p-phenylenediamine, formalin, and the amino acid alanine were comprehensively investigated. The inhibitor was prepared under nitrogen atmosphere using a reflux condenser at various molar ratios and temperatures, and the optimal synthesis conditions were identified. The results showed that the highest product yield was obtained at a molar ratio of 1:2:2 and within a temperature range of 40–65 °C. The yield significantly decreased when the temperature exceeded 65 °C. The structure of the obtained PFA compound was confirmed by IR spectroscopy, where characteristic vibrations of amino, carbonyl, aromatic ring, and methylene groups were observed. The reactivity of the inhibitor molecule was evaluated using semi-empirical (AM1, MNDO, PM3, RM1, MNDO3) and DFT (B3LYP) quantum chemical methods. The calculations indicated that high negative charges were localized on the nitrogen and oxygen atoms, suggesting their potential to form coordination bonds with metal surfaces. The HOMO–LUMO energy gap, chemical hardness, softness, and electrophilicity index revealed that PFA possesses effective adsorption and inhibitory properties. DTA and TGA analyses demonstrated the thermal stability of the PFA inhibitor up to 200–250 °C, confirming its potential applicability under industrial conditions.

Keywords: p-Phenylenediamine, Formalin, Amino acid alanine, IR spectroscopy, Quantum chemical methods

Annotatsiya. Ushbu tadqiqotda p-fenilendiamin, formalin va alanin aminokislotasidan sintez qilingan yangi PFA markali polimer korroziya inhibitori sintezi, fizik-kimyoviy xususiyatlari va reaktivligi har tomonlama o'rganildi.avhum. Ushbu tadqiqotda p-fenilendiamin, formalin va alanin aminokislotasidan sintez qilingan yangi PFA markali polimer korroziya inhibitori sintezi, fizik-kimyohbu tadqiqotda p-fenilendiamin, formalin va alanin aminokislotasidan sintez qilingan yangi PFA markali polimer korroziya inhibitori sintezi, fizik-kimyoviy xususiyatlari va reaktivligi har tomonlama o'rganildi.

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Inhibitor azot atmosferasi ostida turli xil molyar nisbatlar va haroratlarda reflyuks kondensatori yordamida tayyorlandi va optimal sintez sharoitlari aniqlandi. Natijalar shuni ko'rsatdiki, mahsulotning eng yuqori rentabelligi 1: 2: 2 molyar nisbatda va 40-65 harorat oralig'ida olingan. Olingan PFA birikmasining tuzilishi iq spektroskopiyasi bilan tasdiqlandi, bu erda amino, karbonil, aromatik halqa va metilen guruhlarining xarakterli tebranishlari kuzatildi.lingan PFA birikmasining tuzilishi iq spektroskopiyasi bilan tasdiqlandi, bu erda amino karbonil, aromatik halqa va metilen guruhlarining lingan PFA birikmasining tuzilishi iq spektroskopiyasi bilan tasdiqlandi, bu erda amino, karbonil, aromatik halqa va metilen guruhlarining xarakterli tebranishlari kuzatildi. Inhibitor molekulasining reaktivligi yarim empirik (AM1, MNDO, PM3, RM1, MNDO3) va dft (B3LYP) kvant kimyoviy usullari yordamida baholandi. Hisob-kitoblar shuni ko'rsatdiki, azot va kislorod atomlarida yuqori manfiy zaryadlar lokalizatsiya qilingan, bu ularning metall yuzalar bilan koordinatsion aloqalarni hosil qilish imkoniyatlarini ko'rsatadi. HOMO-LUMO energiya bo'shlig'i, kimyoviy qattiqlik, yumshoqlik va elektrofillik indeksi PFA samarali adsorbsiya va inhibitiv xususiyatlarga ega ekanligini aniqladi.OMO-LUMO energiya bo'shlig'i, kimyoviy qattiqlik, yumshoqlik va elektrofillik indeksi PFA samarali adsorbsiya va inhibitiv xususiyatlarga ega ekanligini aniqladi.

Kalit so'zlar: p-Fenilendiamin, Formalin, aminokislota alanin, IQ spektroskopiyasi, kvant kimyoviy usullari

Аннотация. В этом исследовании были всесторонне исследованы синтез, физико-химические свойства и реакционная способность нового полимерного ингибитора коррозии марки PFA, синтезированного из п-фенилендиамина, формалина и аминокислоты аланина. Ингибитор был получен в атмосфере азота с использованием обратного холодильника при различных мольных соотношениях и температурах, и были определены оптимальные условия синтеза. Результаты показали, что наибольший выход продукта был получен при мольном соотношении 1:2:2 и в диапазоне температур 40-65°C. Выход значительно снижался, когда температура превышала 65°C. Структура полученного соединения PFA была подтверждена с помощью ИК-спектроскопии, где наблюдались характерные колебания амино-, карбонильных, ароматических кольцевых и метиленовых групп. Реакционную способность молекулы ингибитора оценивали с использованием полуэмпирических (AM1, MNDO, PM3, RM1, MNDO3) и DFT (B3LYP) квантово-химических методов. Расчеты показали, что на атомах азота и кислорода локализованы высокие отрицательные заряды, что свидетельствует об их способности образовывать координационные связи с

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металлическими поверхностями. Энергетический разрыв HOMO–LUMO, химическая твердость, мягкость и индекс электрофильности показали, что PFA обладает эффективными адсорбционными и ингибирующими свойствами. Анализы DTA и TGA продемонстрировали термическую стабильность ингибитора PFA до 200-250 °C, что подтверждает его потенциальную применимость в промышленных условиях.

Ключевые слова: п-фенилендиамин, Формалин, аминокислота аланин, ИК-спектроскопия, квантово-химические методы.

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].. 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[2]. 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[3]. 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[4,5]. 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[5-8].

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This work aims to study the synthesis and structure of a new type of corrosion inhibitor based on p-phenylenediamine, as well as its inhibition efficiency using an electrochemical method.

EXPERIMENTAL PART

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 (PFA)

The synthesis of the corrosion inhibitor was carried out by adding 0.1 mol of p-phenylenediamine**,** 0.2 mol of formalin (37% aqueous formaldehyde)**,** and 0.2 mol of alanine in a molar ratio of 1:2:2**.** The reaction was performed under an inert nitrogen atmosphere to prevent oxidation of *p-phenylenediamine* by atmospheric oxygen. The mixture was stirred at 40–45 °C for 45 minutes in the presence of a reflux condenser. During the reaction, the temperature was maintained within the range of 55–65 °C**.** The yield of the reaction mainly depended on the mole ratio of the starting materials and the reaction temperature table 1.

Table 1.

Dependence of the synthesis yield of PFA brand corrosion inhibitor on the mole ratio of starting materials and temperature.

p-phenylenediamine formalin+alanin	Temperature ^{0}C	Yield %	Temperature ^{0}C	Yield %
1:1:1	40÷65	49.67	65≤t	23.35
1:2:2		88,42		51.36
2:2:1		56.15		32.26
2:1:3		32.62		29.56
1:2:1		39.43		33.25

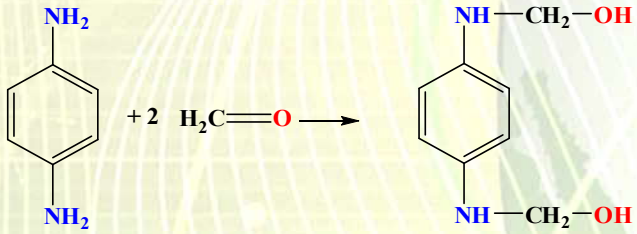
As shown in Table 1, the highest yield (89.4%) was obtained at a temperature range of 40–65 °C and a mole ratio of 1:2:2**.** Increasing the temperature above 65 °C resulted in a noticeable decrease in product yield. The obtained compound was

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well soluble **in** water, ethanol, and dimethylformamide (DMF) at temperatures above 40 °C**.** After completion of the reaction, the mixture was cooled to room temperature, and the solid product was separated by filtration, washed several times with distilled water and ethanol to remove unreacted residues, and then dried under vacuum in an inert nitrogen atmosphere at 50 °C until constant weight**.** The obtained compound was readily soluble in water, ethanol, and dimethylformamide above 40 °C.

IR spectrum analysis of inhibitor

It has been determined that the effectiveness of the inhibitors depends on the fact that they are adsorbed on the surface of steel and form chemical compounds with iron ions that are poorly soluble due to the functional groups in the composition. In addition, the structure of the molecule, the length of the oligomer chain is also important. Therefore, in order to discuss the mechanism of inhibition, we need to know the structure of the molecules of the obtained inhibitors. The structure of substances was studied using the IR spectroscopy method. For this purpose, IR spectra of the first reactant (1,4-phenylenebis(azanedyl)) dimethanol were obtained, newly formed bonds and groups were identified through absorption lines.



Synthesis 1. Reaction of (1,4-phenylenebis(azanedyl)) dimethanol

In Figure 1, the IR spectrometer results of (1,4-phenylenebis(azanedyl)) dimethanol show that in the IR- spectrum of (1,4-phenylenebis(azanedyl)) dimethanol, according to the results of the IR-spectrum, ν(OH) group's valence symmetric ν_s and 1458.81 cm^{-1} field, deformation d vibration frequency was formed. In the 3158.36 cm^{-1} and 1178.36 cm^{-1} regions, valence symmetric ν_s and deformation δ vibrations of the ν(NH_2) group were formed. At the same time, the frequency of valence asymmetric ν_{as}, valence symmetric ν_s and deformation δ vibrations of the ν(CH) bond belonging to the benzene ring at 2981.24 cm^{-1} and 1214.28 cm^{-1}, 2874.35 cm^{-1} and 1443.87 In cm^{-1} fields, the frequency of valence asymmetric ν_{as}, valence symmetric ν_s and deformational δ vibrations of ν(CH_2) group was formed figure 1.

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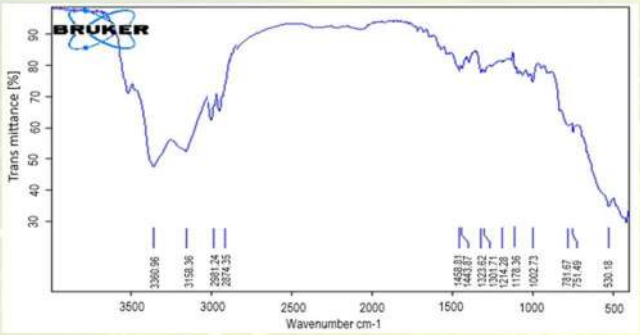


Figure 1. IR-spectrum of (1,4-phenylenebis(azanedyl)).

According to the figure 2 IR-spectrometer results of PFA show that the IR-spectrum of PFA has 3480.52 cm^{-1}, 3366.81 cm^{-1}, 3217.83 cm^{-1}, and 1505.29 cm^{-1}, 1469.20 cm^{-1}, the frequency of valence symmetric ν_s and deformation δ vibrations of ν(NH) group was observed. At the same time, the frequency of valence asymmetric ν_{as}, valence symmetric ν_s and deformation δ vibrations of the ν(CH) bond belonging to the benzene ring at 3100.07 cm^{-1} and 1294.89 cm^{-1}, 2912.10 cm^{-1} and 1444.04 frequency of valence asymmetric ν_{as}, valence symmetric ν_s and deformation δ vibrations of $\nu(CH_3)$ group in cm^{-1} fields, valence asymmetric ν_{as}, valence symmetric ν_s and deformation δ vibrations of $\nu(CH_2)$ group in 2841.93 cm^{-1} and 1182.27 cm^{-1} fields δ is the frequency of oscillations. In the region of 1630.09 cm^{-1}, the valence ν vibration of the n(C=O) group was formed, as well as the frequency of the deformation δ vibration in the region of 836.57 cm^{-1}.

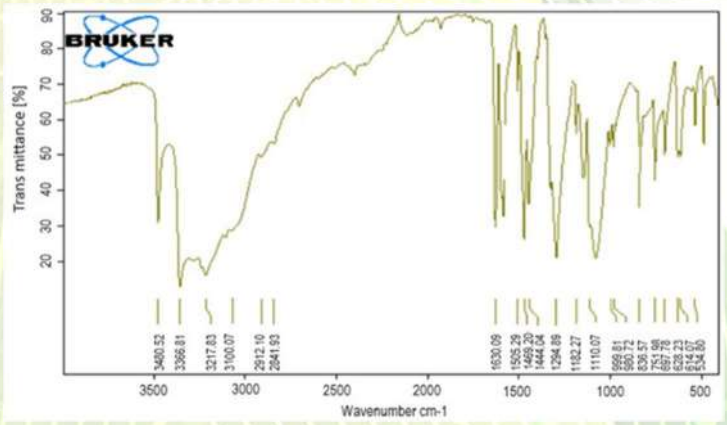
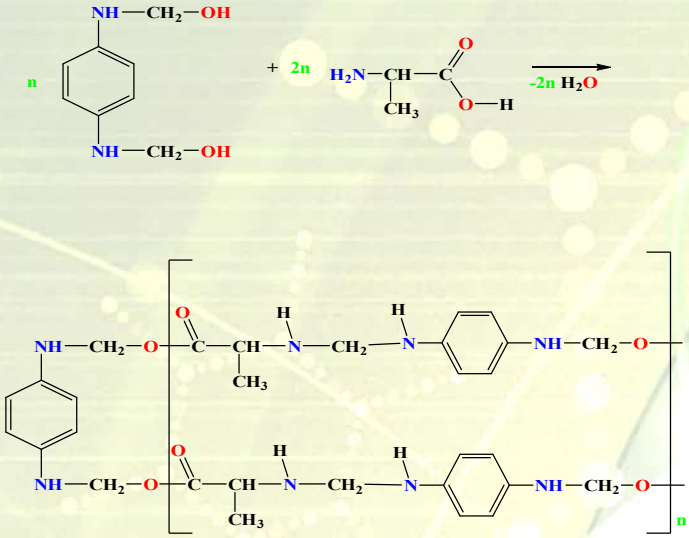


Figure 2. IR- spectrum of PFA brand corrosion inhibitor.

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Synthesis 2. Reaction of PFA brand corrosion inhibitor

Quantum Chemical Analysis of the Reaction Capability of the PFA Inhibitor

Geometric optimization of the PFA was fully optimized using the Popl basis set - RHF/6-311G(d, p) using GaussView 6.0.16 software using the out file generated by the Avogadro software package. Results of GaussView 6.0.16 calculations using the DFT (B3LYP) method The Mulliken method and the frontier molecular orbital (ChegMO) approximation were used to calculate the charges on all atoms[15-18].

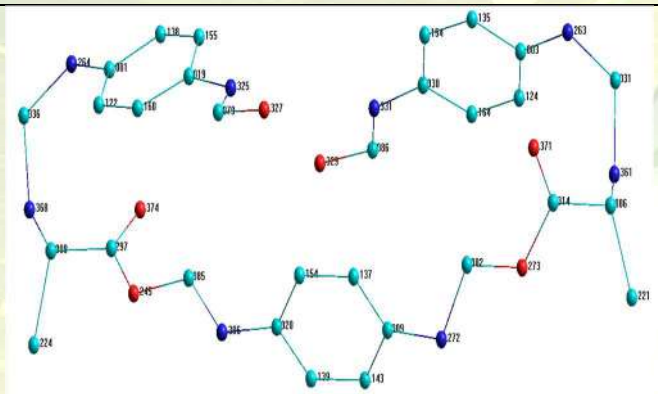
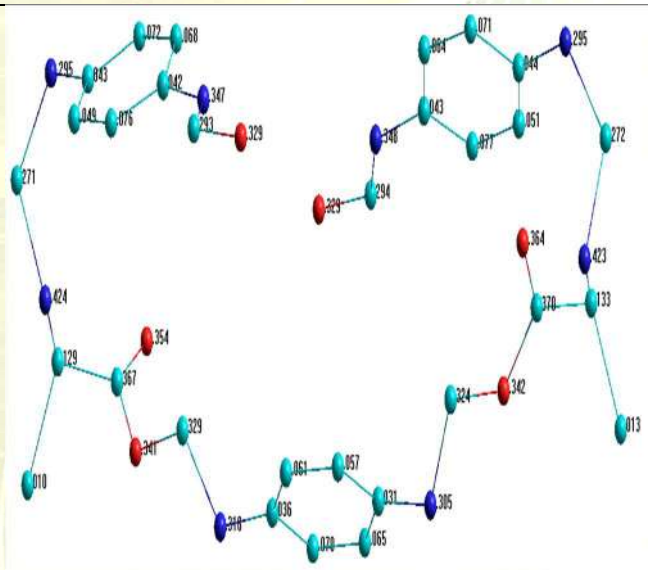
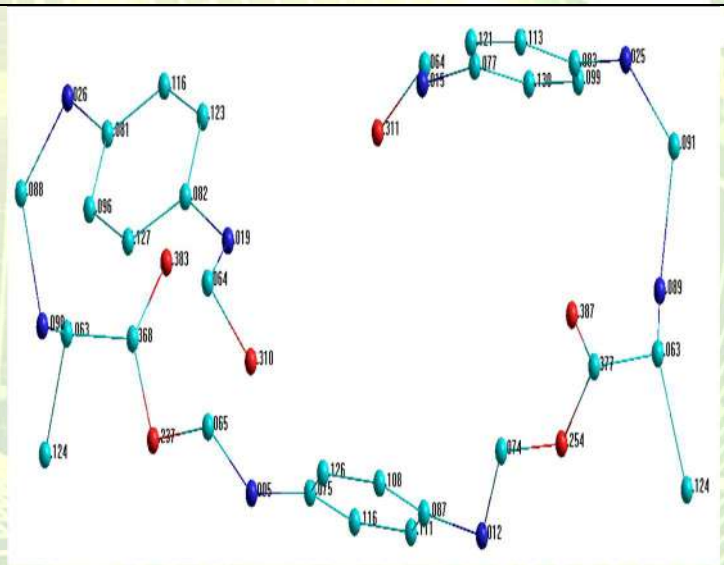
The charge distributions on the atoms of the molecule play an important role in reactivity, that is, they play an important role in predicting the electrophilic and nucleophilic centres of the molecule. Taking this into consideration, the total charges of atoms of PFA molecules were calculated according to the widely used method of charge calculation - Mulliken's method. The calculation results show the localization of the largest negative charge on the nitrogen and oxygen atoms of the PFA molecule (Fig. 3)

One of the main indices used to evaluate the reactivity of organic compounds is the electron density in marginal MOs (ChegMO). The upper occupied molecular orbital (HOMO) and the lower unoccupied molecular orbital (LUMO) are frontier molecular orbitals. An atom with a high electron density in HOMO is an electron-donating atom and is an electrophilic reaction centre. In LUMO, an atom with a high electron density are an electron acceptor atom and a nucleophilic reaction center[19-21]. Based on quantum chemical calculations using semi-empirical methods (AM1, MNDO, PM3, RM1, and MNDO3), it can be concluded that the highest negative effective charges in the PFA molecule are localized on the oxygen atoms of the C=O and C–O–C groups, as well as on the nitrogen atoms of the primary and secondary amino N–H groups. This indicates that these atoms are likely to form coordination bonds with metal surfaces and generate complexes.

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Table-1.

Distribution of Effective Charges on Donor Atoms in PFA Molecules

Calculation method	PFA
AM1	
MNDO	
PM3	

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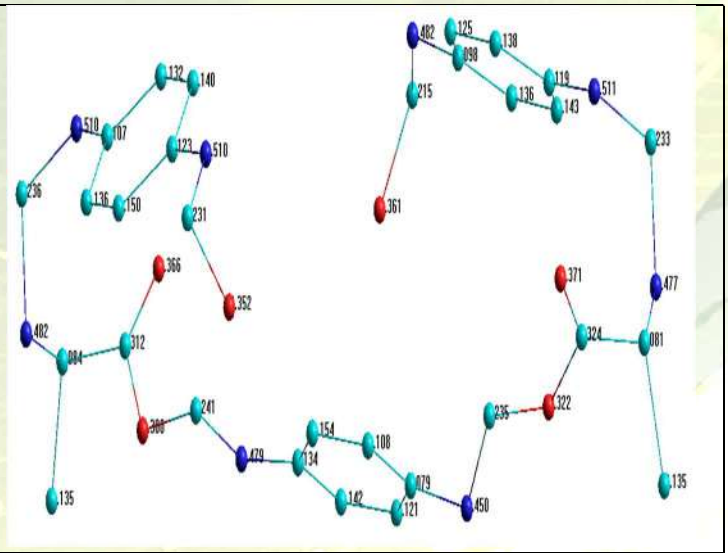
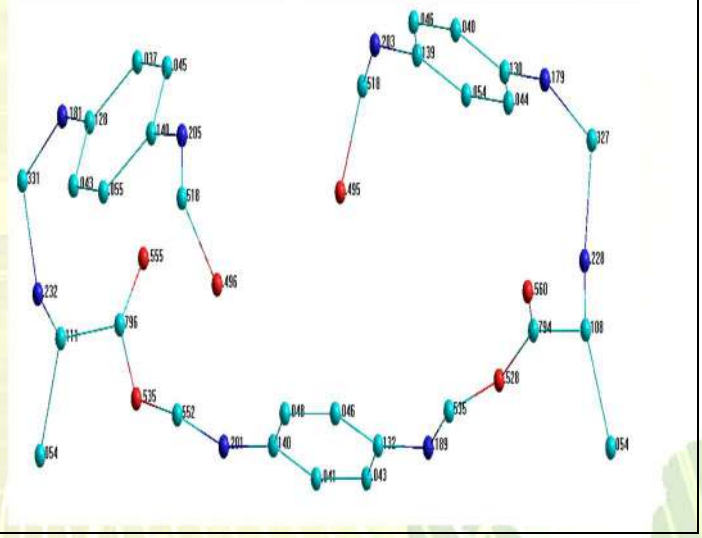
RM1	
MNDO3	

Table 2

Effective Charge Values of Donor Atoms in the Inhibitor Molecules

Atoms	AM1, eV	MNDO, eV	PM3, eV	RM1, eV	MNDO3, eV
PFA					
$\delta_q O^1_{(C-O)}$	-0,329	-0,329	-0,311	-0,361	-0,495
$\delta_q N^1_{(NH)}$	-0,331	-0,348	-0,015	-0,482	-0,203
$\delta_q N^2_{(NH)}$	-0,263	-0,295	-0,025	-0,511	-0,179
$\delta_q N^3_{(NH)}$	-0,361	-0,423	-0,089	-0,477	-0,228
$\delta_q O^1_{(C=O)}$	-0,371	-0,364	-0,387	-0,371	-0,560
$\delta_q O^1_{(C-O-C)}$	-0,273	-0,342	-0,254	-0,322	-0,528
$\delta_q N^4_{(NH)}$	-0,272	-0,305	-0,012	-0.,450	-0,189
$\delta_q N^5_{(NH)}$	-0,306	-0,310	-0,005	-0,479	-0,201

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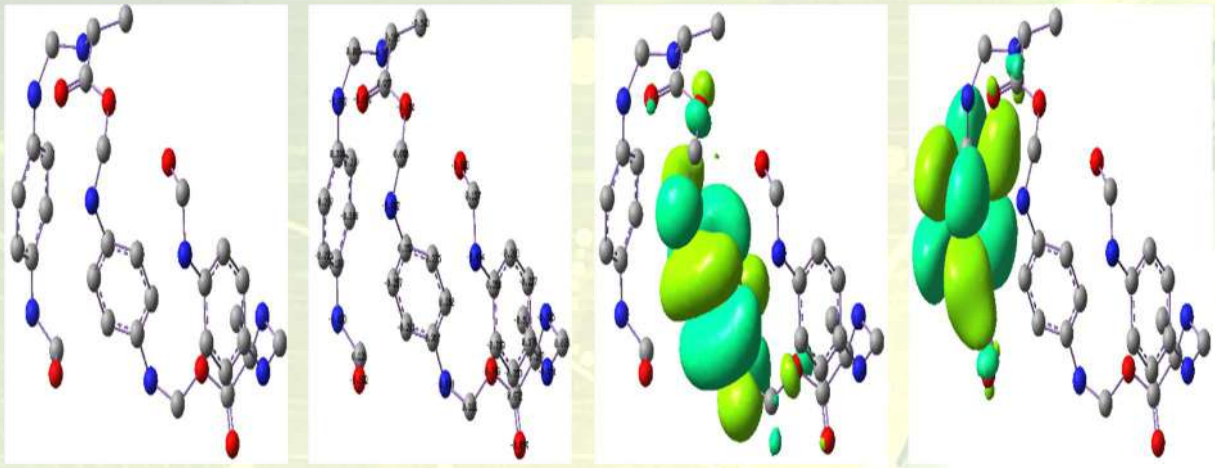
$\delta_q O^2_{(C\text{-}O\text{-}C)}$	-0,245	-0,341	-0,237	-0,300	-0,535
$\delta_q O^2_{(C=O)}$	-0,374	-0,354	-0,383	-0,366	-0,555
$\delta_q N^6_{(NH)}$	-0,368	-0,424	-0,090	-0,482	-0,232
$\delta_q N^7_{(NH)}$	-0,264	-0,295	-0,026	-0,510	-0,181
$\delta_q N^7_{(NH)}$	-0,325	-0,347	-0,019	-0,510	-0,205
$\delta_q O^1_{(C\text{-}O)}$	-0,327	-0,329	-0,310	-0,352	-0,496
E	-8723,6944 (kcal/mol)	-8721,3063 (kcal/mol)	-8745,7442 (kcal/mol)	-7390,0470 (kcal/mol)	-8761,1093 (kcal/mol)

The geometric optimization of PFA was performed using an output file generated by the Avogadro software suite and fully optimized in GaussView 6.0.16 with the Popl basis set at the RHF/6-311G(d,p) level. The results of DFT (B3LYP) calculations carried out in GaussView 6.0.16 were used for Mulliken population analysis and for calculating the charges on all atoms based on the frontier molecular orbital (FMO) approach.

Charge distribution in molecular atoms plays a crucial role in reaction capability, as it helps predict electrophilic and nucleophilic centers of the molecule. Considering this, the total charges of atoms in PFA molecules were calculated using the widely used Mulliken method. The calculation results show that the most negative charges are localized on the nitrogen and oxygen atoms of the PFA molecule (Figure 3b).

One of the key indices used to evaluate the reactivity of organic compounds is the electron density in frontier molecular orbitals (FMOs). The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) are considered as frontier orbitals. Atoms with high electron density in the HOMO are regarded as electron donor atoms and electrophilic reactive centers, while atoms with high electron density in the LUMO are considered electron acceptor atoms and nucleophilic reactive centers.

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a **b** **c** **d**

HOMO = -7,02 *eV* **LUMO = 3,85 *eV***

Figure 3. Effective charge distribution on donor atoms in PFA corrosion molecules.

According to the charge density of the upper occupied molecular orbital (HOMO) and the lower empty molecular orbital (LUMO), the largest charge density distribution belongs to the -O-, C=O and N-H groups (Fig. 3c.d). The orbital energies of HOMO and LUMO and their difference ($\Delta E=E_{LUMO} - E_{HOMO}$, eV) are also important. An increase in the LUMO energy (increasing the occupied level towards the LUMO) in the series of compounds indicates an increase in the electron-donating property of the compound. LUMO energy determines the electron acceptor ability (electron propensity) of compounds. The hardness (ŋ) and softness (σ) of molecules are determined based on ChegMO energies. The hardness is equal to the difference between the ionization potential (I) and electron affinity (A) divided by ŋ=(I-A)/2; softness is found as follows: σ=1/2ŋ. Absolute electronegativity is also found based on ChegMO: (I+A)/2=X. The results are presented in Table 3.

According to the charge density of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO), the largest distribution of charge density belongs to the -O-, C=O and N-H groups (Fig. 3c.d).

Table 3.

Calculated quantum chemical parameters for PFA

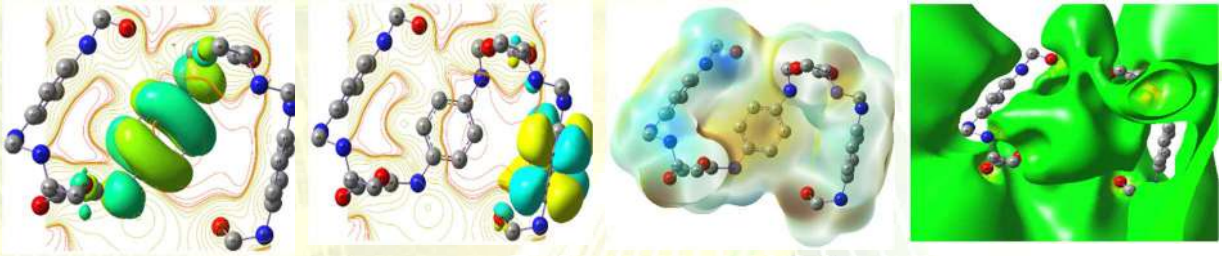
Quantum-chemical parameters	PFA
E_{HOMO}, eV	-7,02
E_{LUMO}, eV	3,85
$\|\Delta E\| = E_{LUMO} - E_{HOMO}$ (eV)	10,87
Ionization potential, $I=-E_{HOMO}$, (eV)	7,02
Electron susceptibility, $A=-E_{LUMO}$, (eV)	-3,85

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Electronegativity, $\chi = (I + A)/2$ (eV)	1,59
Chemical hardness, $\eta = (I - A)/2$ (eV)	5,43
Chemical potential, $\mu_p = -(I + A)/2$ (eV)	-1,59
Chemical softness, $\sigma = 1/(2\eta)$ (eV-1)	0,09
Electrophilic index, $\omega = \mu_p^2/2\eta$ (eV)	0,23
Dipole moment, μ (Debeye)	3,82

E – total energy (eV).

It is known that the level of electrostatic potential is used to determine the electron donor and electron acceptor parts of a molecule. MEP (molecular electrostatic potential) plots in red, blue, and green are negative, positive, and neutral electrostatic potentials, respectively (Figure 4).



a **b** **c** **d**

Figure 4. ESP- HOMO (a), ESP- LUMO (b) and ESP(c, d) of PFA

This PFA forms low-spin complexes due to the high splitting energy in the Fe ion orbital.

Results of DTA and TGA Analyses

The differential thermal analysis (DTA) and thermogravimetric (TG) curves of the PFA corrosion inhibitor (a polymer based on p-phenylenediamine, formalin, and glycine) are crucial for evaluating its thermal stability. These results reveal how the inhibitor behaves at elevated temperatures and determine its applicability in corrosive environments such as oil–gas and metallurgical industries.

The TG curve indicates that the thermal degradation of the PFA inhibitor occurs in three stages: the first stage (99.75–220.88 °C, 11.265%) is associated with the release of water and NH_3, reflecting the moisture resistance of the protective film; the second stage (215.43–234.49 °C, 67.813%) corresponds to the main decomposition of polymer chains, producing CO, CO_2, and organic residues, which weakens adsorption; the third stage (234.49–320.03 °C, 12.659%) involves the elimination of residual groups (phenylene rings), confirming the inhibitor's environmentally safe thermal stability up to 200–250 °C.

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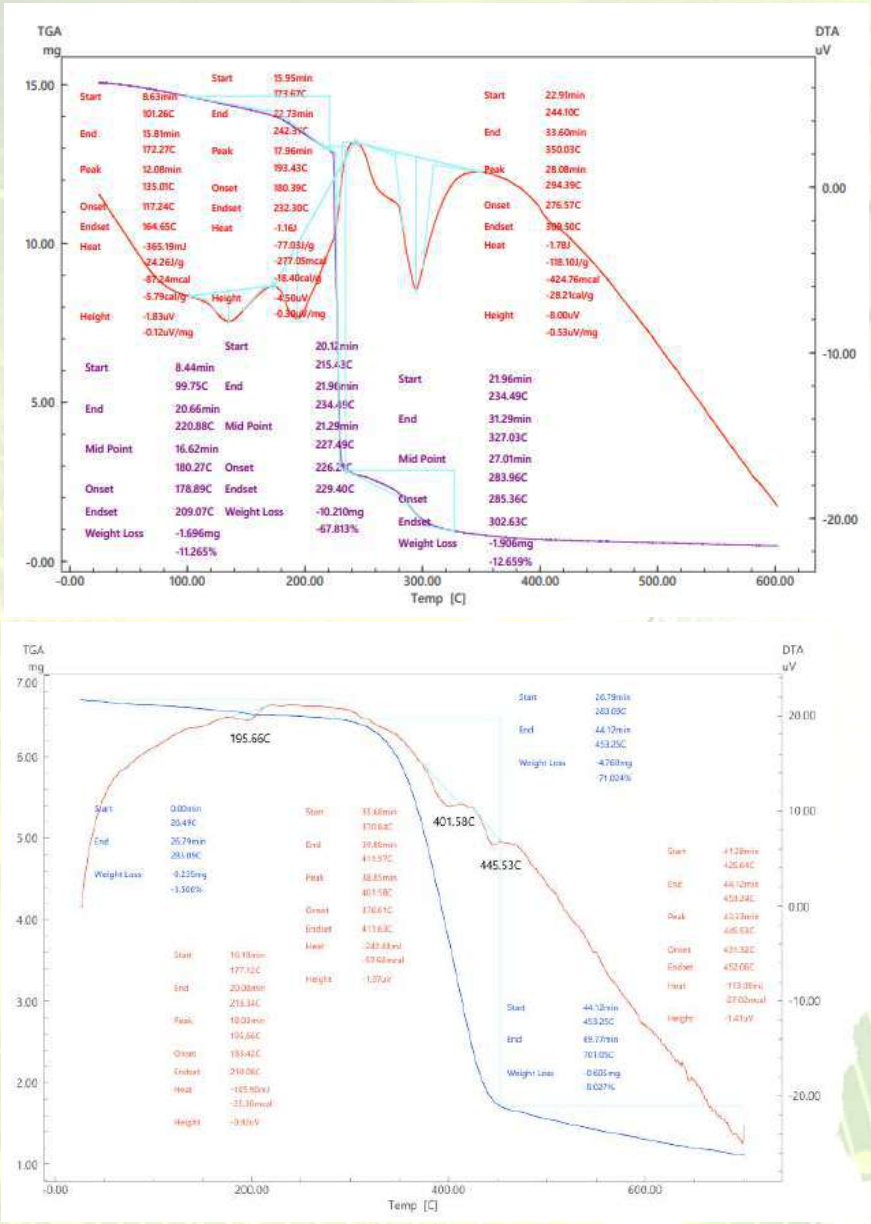


Figure 4. DTA and TGA thermal analysis of the PFA corrosion inhibitor

CONCLUSION

The comprehensive study revealed the optimal synthesis conditions for the PFA corrosion inhibitor synthesized from p-phenylenediamine, formalin, and amino acids, demonstrating that the product yield strongly depends on the molar ratio of the starting materials and the synthesis temperature. IR spectroscopy, quantum chemical calculations, and thermal analyses confirmed the presence of active electron-donor centers, such as nitrogen and oxygen atoms, in the PFA molecule, indicating their ability to form stable coordination bonds with metal surfaces and establish a protective film. The HOMO–LUMO energy characteristics, chemical hardness and softness parameters, as well as DTA–TGA analyses, indicated high adsorption activity and sufficient thermal stability up to 200–250 °C. These findings substantiate the potential application of PFA as an

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effective, stable, and promising corrosion inhibitor in aggressive environments within the oil–gas, chemical, and metallurgical industries.

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