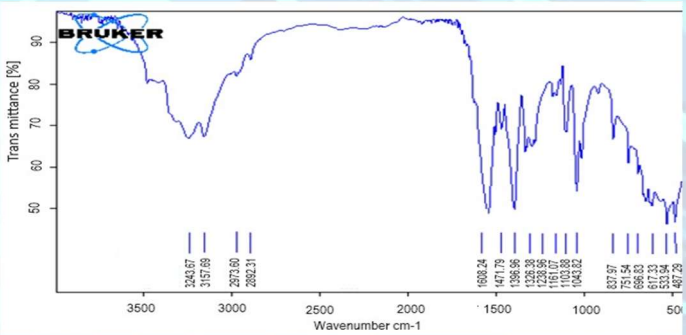
SYNTHESIS OF CORROSION INHIBITOR BASED ON P-PARAPHENYLENEDIAMINE, FORMALIN, AND ALANIN

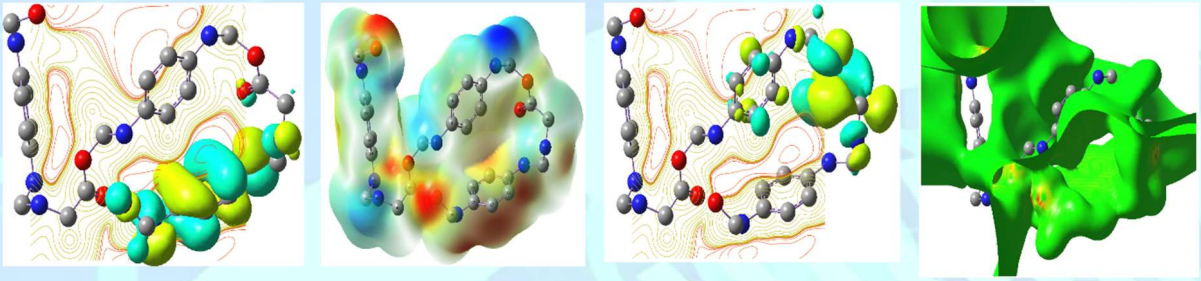
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Abstract. In this article, the optimal conditions for the synthesis of a new type of corrosion inhibitor based on p-paraphenylenediamine, formalin, and alanin are studied. In this case, it was determined that the mole ratio of the initial substances is 1:2:2 mol, and the optimal temperature is 40÷65. The efficiency of the reaction when carried out under these optimal conditions was 89.4%. The structure of the synthesized corrosion inhibitor was analyzed using IR spectra. In addition, the quantum chemical calculation of the reaction property of this synthesized Inhibitor molecule was carried out in Avogadro, Hyper Chem 8.01, Asselrys MS Modeling 3.0.1 by the constrained Semi-empirical (UHF) method, using SCF-MO using semi-empirical AM1, MNDO, PM3, Calculations were performed on an Intel Pro Pentium 1.40 GHz computer using the RM1 and MNDO3 methods. The effectiveness of this synthesized corrosion inhibitor in inhibiting St3 grade steel was studied by gravimetric method, and its steel surface morphology and element composition were studied using SEM analysis.

Graphical abstract.



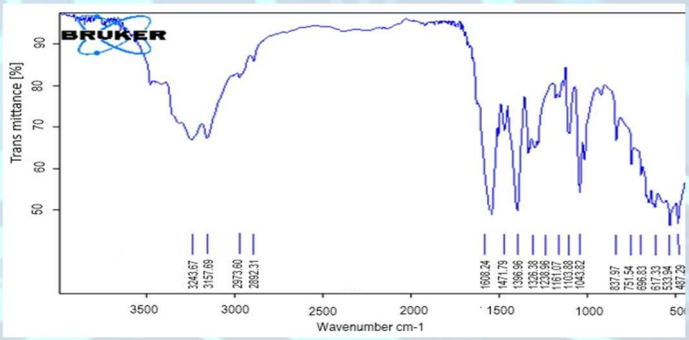
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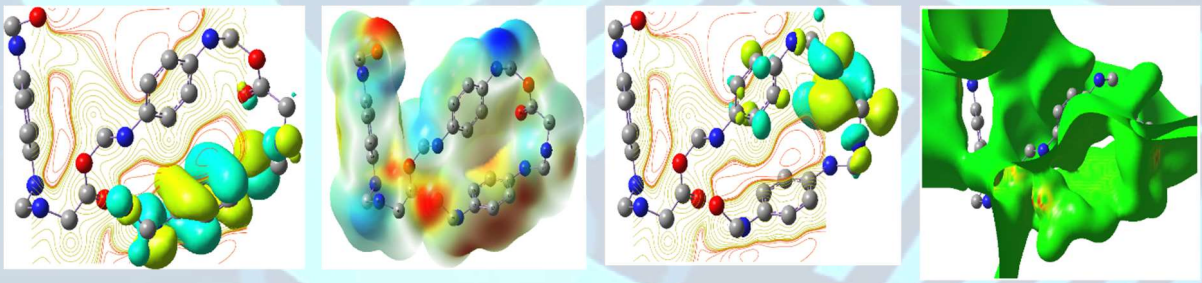


Keywords: p-paraphenylenediamine, formalin, and alanin, corrosion inhibitor, IR spectrium.

Annotatsiya. Ushbu maqolada p-parafenilendiamin, formalin va alaninga asoslangan yangi turdagi korroziya inhibitori sintezi uchun maqbul sharoitlar o'rganilgan. Bunday holda, dastlabki moddalarning mol nisbati 1:2:2 mol, optimal harorat esa 40.65. Ushbu optimal sharoitlarda amalga oshirilganda reaktsiyaning samaradorligi 89,4% ni tashkil etdi. Sintezlangan korroziya inhibitori tuzilishi IQ spektrlari yordamida tahlil qilindi. Bundan tashqari, ushbu sintezlangan Inhibitor molekulasining reaktsiya xususiyatini kvant kimyoviy hisoblash Avogadro, Hyper Chem 8.01, Asselrys MS modellashtirish 3.0.1 da cheklangan yarim empirik (UHF) usuli bilan amalga oshirildi, SCF-MO yordamida yarim empirik AM1, MNDO, PM3 yordamida hisob-kitoblar amalga oshirildi. Intel Pro Pentium 1.40 GHz kompyuter RM1 va MNDO3 usullari yordamida. Ushbu sintezlangan korroziya inhibitori St3 navli po'latni inhibe qilishda samaradorligi gravimetrik usul bilan o'rganildi va uning po'lat yuzasi morfologiyasi va element tarkibi SEM tahlili yordamida o'rganildi.

Grafik annotatsiya



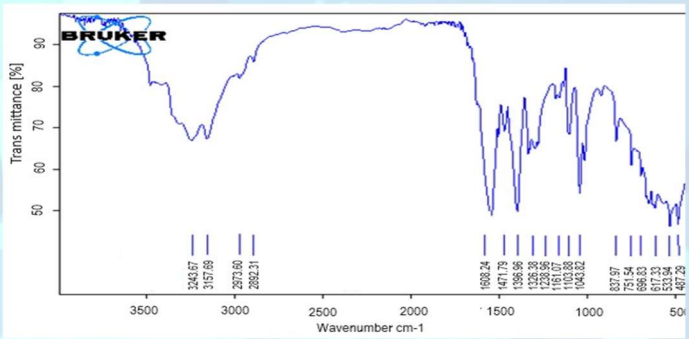


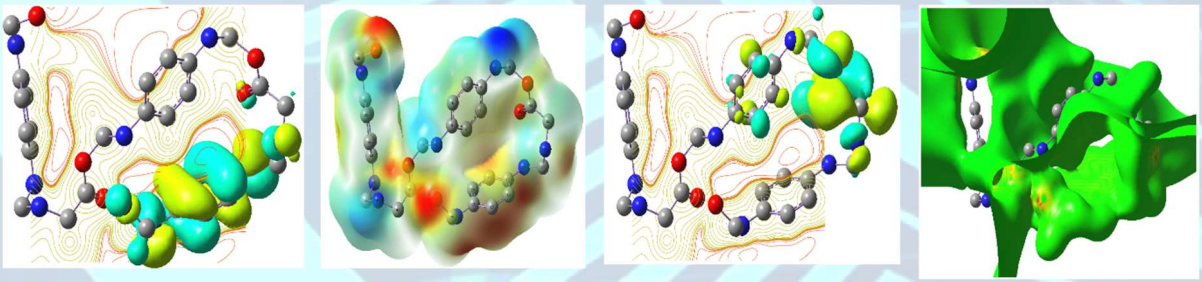
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Kalit so'zlar: p-parafenilendiamin, formalin va alanin, korroziya inhibitori, IQ spektri.

Аннотация. В этой статье исследуются оптимальные условия для синтеза нового типа ингибитора коррозии на основе п-парафенилендиамина, формалина и аланина. При этом молярное соотношение исходных веществ составляет 1:2:2 моль, а оптимальная температура-40,65. При проведении в этих оптимальных условиях эффективность реакции составила 89,4%. Синтезированная структура ингибитора коррозии была проанализирована с использованием ИК-спектров. Кроме того, квантово-химический расчет реакционной природы этой синтезированной молекулы ингибитора был выполнен методом ограниченного полуэмпирического (УВЧ) в Avogadro, Hyper Chem 8.01, Asselrys MS Modeling 3.0.1, расчеты проводились с использованием SCF-mo с использованием полуэмпирического AM1, mndo, pm3. Компьютер Intel Pro Pentium 1,40 ГГц с использованием методов RM1 и mndo3. Эффективность этого синтезированного ингибитора коррозии в ингибировании стали марки ST3 изучалась гравиметрически, а морфология и элементный состав ее стальной поверхности изучались с помощью SEM-анализа.

Графическая аннотация





Ключевые слова: Р-парафенилендиамин, формалин и аланин, ингибитор коррозии, ИК-спектр.

Introduction

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Corrosion inhibitors are widely used to protect metals against various corrosive environments [1,2]. A corrosion inhibitor is a compound that is added in low concentrations to a corrosive solution to reduce and/or minimize the corrosion rate [3.4]. Amines are the main part of organic substances that are among the inhibitors that reduce the process of corrosion of metals under the influence of the external environment. The inhibitory properties of various amines have been widely studied [5]. Complexes of 4-[(2,4-dihydroxy-benzylidene)-amino]-1, 5-dimethyl-2-phenyl-1, 2-dihydroxy-pyrazole-3- with various metals exhibit mixed inhibitory properties in 2 M HCl acid medium shows. It showed inhibition efficiency of 85.1% at a concentration of 11·10-6 mol/l [6]. The inhibition properties of N,N-dipropinoxy methyl amine trimethylphosphonate were studied using potentiometric polarization curves and electrochemical methods. In this study, at a temperature of 298 K, concentrations ranging from 40 mg/l to 320 mg/l, this inhibitor was classified as a mixed type inhibitor [7]. Also, a number of ammonium compounds, namely N [isononylphenoxypoly (ethyleneoxy) carbonylmethyl] ammonium chloride, are cationic surfactants: 1-methyl-3-tetradecyl imidazolium bromide, 1-methyl-3-hexadexyl imidazolium bromide, N,N-tetradecyl pyridinium. bromide, N,N-hexadecyl pyridinium bromide, N,N-dimethyl ammoyl-dimethyl bromide, N,N-dimethyl-N-ethylbenzyl ammonium laurate and N,N-dimethyl-N-ethylbenzyl ammonium acetate compounds from various compounds (10, 25, 50, 100 and 200 mg) concentrations of API 5L X52 grade steel in 2 M hydrochloric acid inhibition efficiency of more than 90%, imidazoline-based dissymmetric and bis-symmetric ammonium salt (DBA) and thiourea modification N-hydroxy ethylated ammonium salts have been used to prevent hydrogen-sulfide corrosion, showing an efficiency of Z = 95 to 97 % [8,9]. Pyrazol[3,4-d]pyrimidnone compound was used as an effective corrosion inhibitor for corrosion protection of carbon steel in 1.0 M HCl solution[10]. From the following diblock copolymers based on ethyl methacrylate and amines: poly[2(dimethylamino)ethyl methacrylate] (PDMA), poly[2-(diethylamino) ethyl methacrylate] (PDEA), poly[2-(diisopropylamino)ethyl methacrylate] (PDPA) homopolymers and polydimethylamino (poly [2-)ethylmethacrylate]-β-poly[methylmethacrylate] (PDMA-β-PMMA), poly[2-(diethylamino)ethylmethacrylate]-β-poly[methylmethacrylate] (PDEA-β-PDMA) and poly[2-(used diisopropylaminolate)-β-poly[2-(dimethylamino)ethyl methacrylate] (PDPA-β-PDMA) compounds as effective corrosion inhibitors for corrosion protection of AISI 304 steel [11].

Experimental part

Materials and methods

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

IR- analysis. In the analysis of these compounds, the IR-spectra produced by the Bruker farm in Germany were used.

SEM and AFM analysis-Surface morphology and microstructure studies of the samples were carried out using a scanning electron microscope SEM - EVO MA 10 (Carl Zeiss, made in Germany) and Agilent 5500 (Agilent, USA) atomic force microscope (AFM).

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 Semi-empirical (UHF) method, semi-empirical AM1, MNDO, PM3, RM1 and MINDO3 using SCF-MO calculations were carried out using the Intel Pro Pentium 1.40 GHz computer.

Synthesis of inhibitor.

In the synthesis of this corrosion inhibitor, 0.1 mol of amine and 0.2 mol of formalin were added slowly from 0.2 mol of alanin with stirring at 40 and 45 ^{0}C for 45 minutes in the presence of a reflux refrigerator in a nitrogen atmosphere. During this process, the temperature was in the range of 55-65 ^{0}C. The yield of this reaction mainly depends on the mole ratio of the starting materials and the temperature of the reaction medium. 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+glycine	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

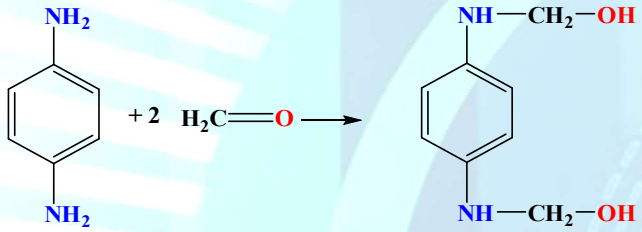
As can be seen from Table 1, the highest productivity was achieved when the temperature was in the range of 40÷65 ^{0}C and the mole ratio of the starting materials was 1:2:2. As the temperature increased to 65 ^{0}C, the product yield also decreased significantly. In conclusion, it can be said that one of the most important factors after temperature is definitely the mole ratio of the initial substances. The

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yield of the obtained product was 89.4 percent. The resulting compound is well soluble in water, ethanol and dimethylformamide at a temperature above 40 ^{0}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[12,13].



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

In Figure 1, the IR-(BRUKER) spectrometer results of (1,4-phenylenebis(azanedyl)) dimethanol show that in the IR-(BRUKER) 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.

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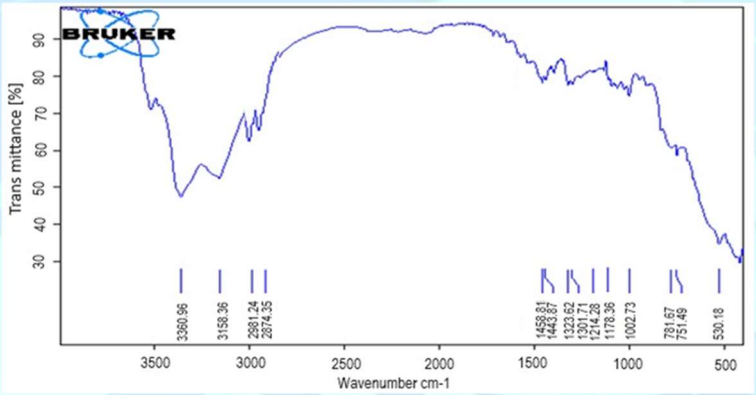


Figure 1. IR-(BRUKER) spectrum of (1,4-phenylenebis(azanedyl)).

Figure-2 IR-(BRUKER) spectrometer results of PFA show that the IR-(BRUKER) spectrum of X 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}.

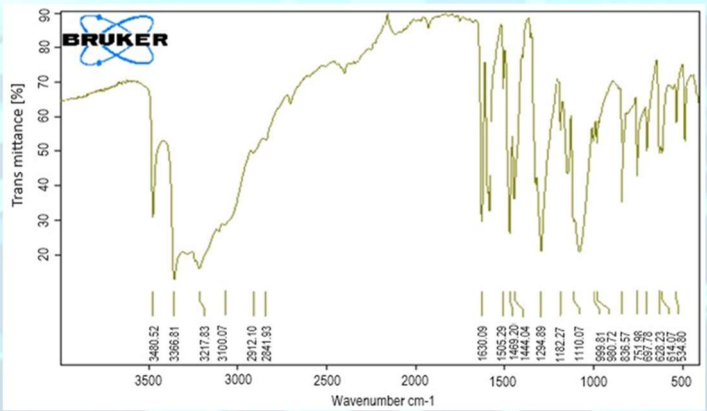
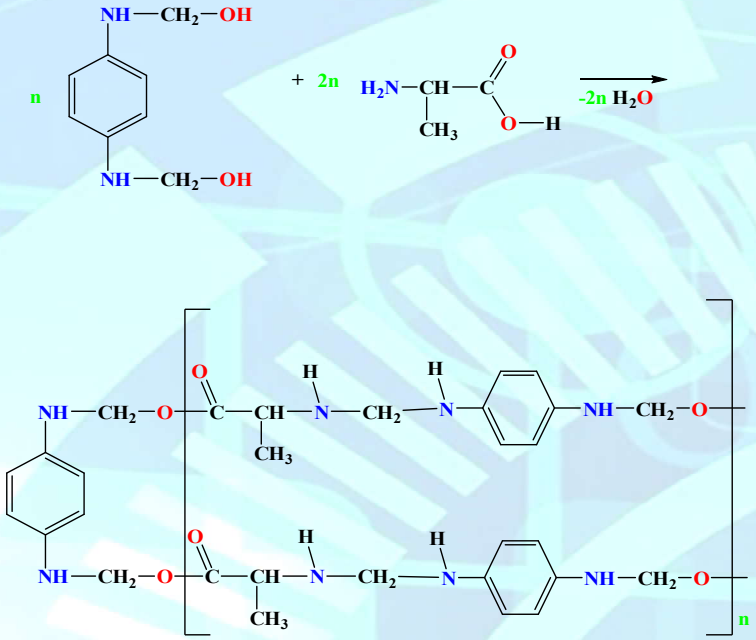


Figure 2. IR-(BRUKER) spectrum of PFA brand corrosion inhibitor.

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Scheme 2. Reaction of PFA brand corrosion inhibitor.

Quantum chemical analysis of the reactivity of PFA brand corrosion inhibitor

Quantum chemical calculation of the reactivity of the inhibitor molecule by semi-empirical (UHF) method constrained in Avogadro, Hyper Chem 8.01, Asselrys MS Modeling 3.0.1, with semi-empirical AM1, MNDO, PM3, RM1 and MNDO3 methods using SCF-MO Calculations were performed on an Intel Pro Pentium 1.40 GHz computer. Molecular geometry optimization was done using the Polak-Ribiere (Conjugate gradient) algorithm. These methods make it possible to determine the total energy of the molecule and electron densities of molecular orbitals, as well as the geometric optimization of the studied molecule. One of the important electronic characteristics is the Mulliken effective charges on atoms (CHARGES) and the total energy of the system (TOTAL ENERGY) (Table 3). AM1, MNDO, PM3, RM1 and MNDO3 based on the results of quantum-chemical calculations using semi-empirical methods, it can be concluded that the high values of the negative effective charge in the PFA molecule are on the oxygen atoms of the C=O and C-O-C groups, and the nitrogen atoms of the primary and secondary amine N-H groups. indicates that these atoms can form complexes by connecting with metal through a coordination bond[14,15].

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Table 2.

Effective charge distribution on donor atoms of PFA molecules

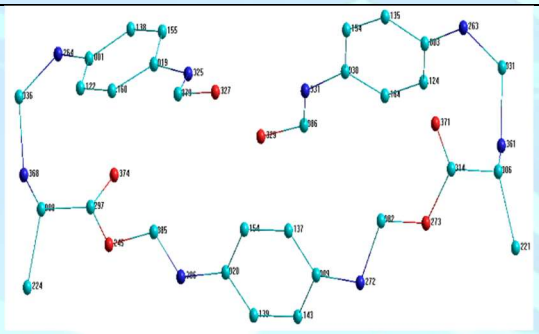
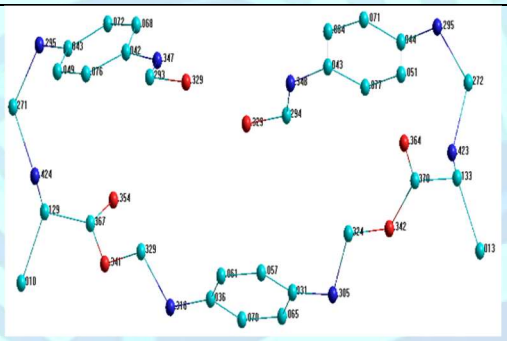
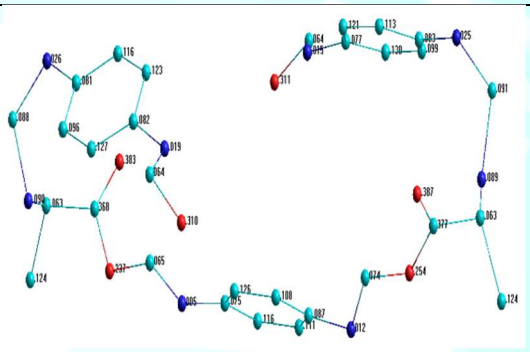
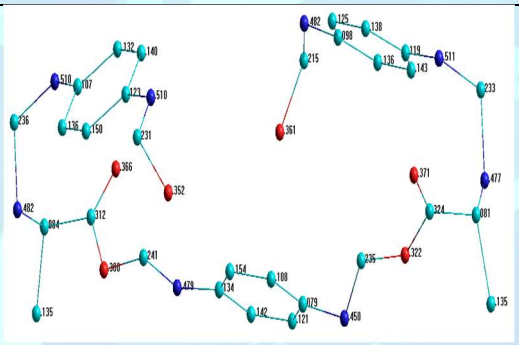
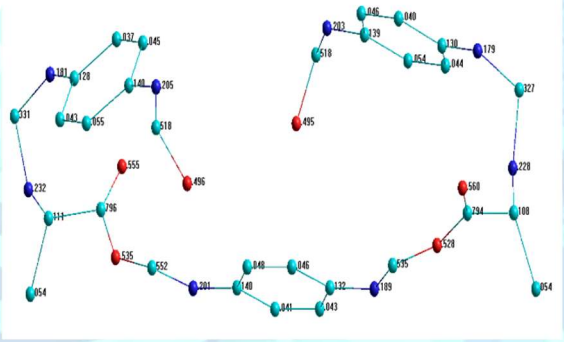
Calculation method	PFA
	
AM1	MNDO
	
PM3	RM1
	
PM3	

Table 3

Effective charge values of donor atoms in 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

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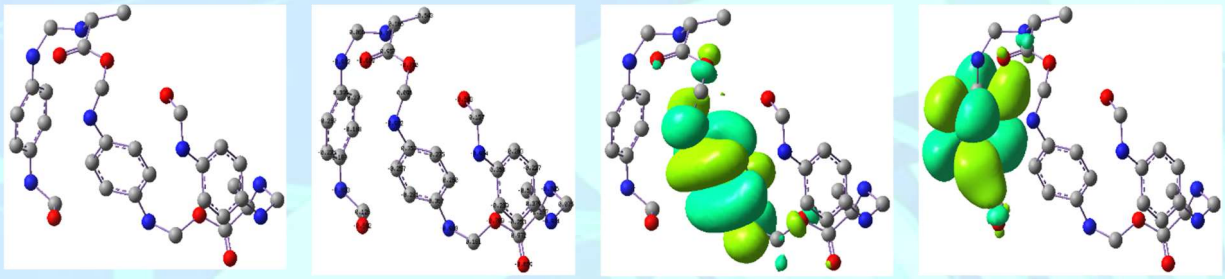
$\delta_q O^1_{(C\text{-}O\text{-}C)}$	-0,273	-0,342	-0,254	-0,322	-0,528
$\delta_q N^4_{(NH)}$	-0,272	-0,305	-0,012	-0.,450	-0,189
$\delta_q N^5_{(NH)}$	-0,306	-0,310	-0,005	-0,479	-0,201
$\delta_q O^2_{(C\text{-}O\text{-}C)}$	-0,245	-0,341	-0,237	-0,300	-0,535
$\delta_q O^2_{(C=O)}$	-0,374	-0,354	-0,383	-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)

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.

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[16].

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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.

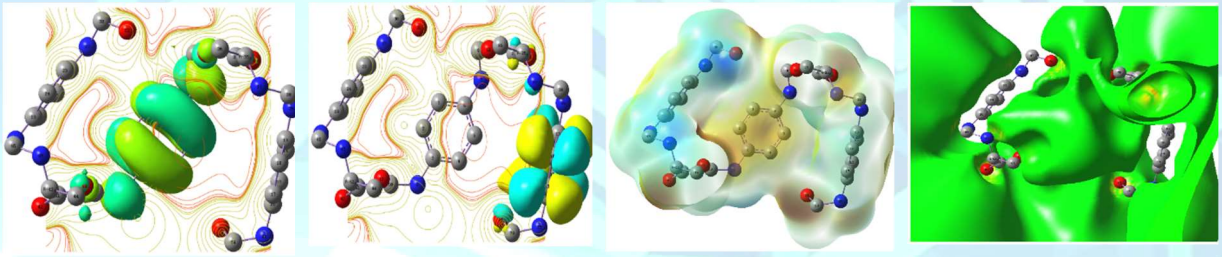
Calculated quantum chemical parameters for PFA

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
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

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Electrophilic index, $\omega=\mu_p^2/2\eta$ (eV)	0,23
Dipole moment, μ (Debay)	3,82

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[17]

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