Applied Chemical Engineering (2025) Volume 8 Issue 4 ISSN: 2578-2010 (O)
doi: 10.59429/ace.v8i4.5784

ORIGINAL RESEARCH ARTICLE

Synthesis, Structural Characterization, and Anti-corrosion Study of a New Fluorinated Bis-Schiff Base

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ABSTRACT

A new series of bis-Schiff base compounds were synthesized. The fluorinated ketone (4-Biphenylyl trifluoromethyl ketone) was prepared by Friedel Crafts acylation of biphenyl as aromatic compound with trifluoroacetic anhydride in present (AlCl3) as a Lewis acid with very good yield. A bis-amine [1,1-bis(3-methyl-4-aminophenyl)-1-(4-biphenylyl)-2,2,2-trifluoroethane] was synthesized by refluxed of mixed fluorinated ketone, o-toluidine and o-toluidinehydrochloride to afforded the required bis-amine with a good yeild. The bis-Schiff bases were synthesized used bis-amine with several aldehydes (2-chlorobenzaldehyde, 4-chlorobenzaldehyde, 3-salicyldehyde, 5-nitrosalicyldehyde) into ethanol as a solvent and few drops of hydrochloric acid as catalyst with high yield. All compounds were confirmed by FTIR, 1HNMR, 13CNMR, Mass techniques. The compounds A3, A4, A5, and A6 exhibited high inhibition efficiency against steel corrosion in HCl solution, with values ranging from 87% to 94%. Compound A24 showed the highest performance, followed by A19, indicating the formation of a stable protective film that significantly reduces the corrosion rate.

Keywords: Fluorinated Schiff base; aromatic aldehyde; bisamine, fluorinated ketone; anti-corrosion.

ARTICLE INFO

Received: 25 September 2025
Accepted: 24 October 2025
Available online: 13 November 2025

1. Introduction

Schiff bases are formed through a condensation reaction between an aldehyde or a ketone and a primary amine[1-3]. The characteristic functional group of Schiff bases is the azomethine group (–C=N–)[4-6], and their general structure is represented as R–CH=N–R′[7]. The attached R groups may be aliphatic, aromatic, or contain other distinctive substituents with various chemical properties [8-10] nitrogen atoms in these compounds carry lone electron pairs, which facilitate coordination with metal ions. [11,12] The chemical stability of these compounds is primarily attributed to the basic character of the nitrogen atom present in the azomethine (–C=N–) moiety. [13-15]. Fluorine-containing Schiff bases are considered important compounds in the fields of medicine and catalysis. They exhibit a strong ability to adsorb onto metal surfaces, forming an effective protective layer that helps reduce corrosion. These compounds are particularly effective for protecting metals in acidic environments, such as hydrochloric acid (HCl). Their efficiency is attributed to the presence of electron-donating atoms like nitrogen and oxygen, which enable them to adsorb onto the metal surface and form a protective layer that reduces corrosion [16-21]. Schiff bases can also form coordination complexes with metal atoms, enhancing the surface's resistance to corrosion. Numerous studies have demonstrated their ability to reduce corrosion rates by over 94%, and they are often less toxic and more environmentally friendly compared to traditional inhibitors [22]. n this work, novel fluorinated bis-Schiff base compounds containing a biphenyl moiety were synthesized and characterized, and their anti-corrosion efficiency was investigated for the first time. The introduction of a trifluoromethyl bridge significantly enhanced the hydrophobicity and stability of the protective film compared to previously reported Schiff bases.” compounds contain at least one fluorine atom. The introduction of this atom enhances the importance and performance of the compounds, making them more effective.

2. Experimental part

2.1. Materials and methode

Experimental part Three companies CDH (Central drug house)and Merck and sigma supplied all the chemicals used in this study.

Fourier Transform Infrared (FT-IR) Spectroscopy: FT-IR results were obtained by infrared spectra using KBr pellets on a Shimadzu FT-IR-8400S spectrophotometer. This technology is used to detect functional groups contained within compounds.(NMR) Nuclear Magnetic Resonance: using an Oxford 400 Magnet and NMR Innova 5 Console spectrometer with deuterated dimethyl sulfoxide (DMSO *d6*) as the solvent, The NMR spectra of ^{1}H and ^{13}C were performed. These spectra provide detailed information about the high molecular structure of compounds and indicate the uniqueness of the environments of discrete hydrogen and carbon atoms.

2.2. Synthesis of compound

4-Biphenylyl trifluoromethyl ketone[A1][23]

This ketone was synthesized used anhydrous aluminum chloride$AlCl_3$ (6.65 g, 0.05mmol) in dichloromethane (DCM) (125mL), trifluoroacetic anhydride (TFAA) (10.5g, 0.113 mmol) in DCM (35 mL) and biphenyl (3.85g, 0.0265mmol) in DCM (25 mL). The mixture was stirred for 2 hours to afford 4-biphenylyl trifluoromethyl ketone (3.81 g, 75%, lit. 70%) as colorless crystals. Mp = 102–103 °C (lit. 102–103 °C); FTIR (solid, cm^{-1}) ν = 3065, 3032 (CH_{arm}), 1716 (C=O), 1558 (C=C),1139(CF_3); ^{1}HNMR (400MHz, DMSO-*d6*,ppm),7.4-8.3 (m, H, CH_{arm}), ^{13}CNMR (DMSO-*d6*,ppm), 179 (C,C=O), 128-145(C,C_{arm}), 117(C,CF_3).

Synthesis of 1,1-bis(3-methyl-4-aminophenyl)-1-(4-biphenylyl)-2,2,2- trifluoroethane [A2].[24]

The bis amine was synthesized using 4-biphenylyl trifluoromethyl ketone (10.00 g, 9.93 mmol), 2-methylaniline hydrochloride (25.25 g, 175.85 mmol) and 2-methylaniline (51.39 g, 479.58 mmol). The mixture was refluxed for 72 hours to afford 1,1-bis(3-methyl-4-aminophenyl)-1-(4-biphenylyl)-2,2,2-trifluoroethane (A2) (22.50 g, 63%) as a white powder. Mp = 192–193 °C; FTIR (solid, cm^{-1}) v = 3491 & 3390 (NH_2), 1576 (C=C), 1132= (CF_3); ^{1}H NMR (400MHz, DMSO-*d6*,ppm) 5.9-6.6 (m, H, CH_{arm}), 2.1(s, H,CH_3), 7.5(s, H, NH_2). ^{13}CNMR DMSO-*d6*,ppm),145(C,C-N),125 (C,CF3) ,132-135 (C, C_{arm}).

Synthesis of Schiff base

General procedure:

An equivalent molar of the bis amine (4,4'-(1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methylaniline)(A2) (2.23mmole, 0.5g) and equivalent amount of aldehydes(2-chlorobenzaldehyde ,4-chlorobenzaldehyde, salicyldehyde, 5-nitrosaildehyed were dissolved in (10-15 mL) ethanol after completely dissolved, then, 3 drops of HCl was added to mixture. This mixture is placed in round bottom flask and reflexed for (4hours). After that, the mixture was completely dried and recrystallized by methanol,

1/ N,N'-((1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(1-(2-chlorophenyl)methanimine)[A3]: the general method was followed, bis-amine (0.5g, 1.11mmol) with 2-chlorobenzaldyhyed (0.25mL, 1.11mmol) to afforded A3 (0.573g, 85%) as an orange powder, FTIR(v,cm-^{1}):

1622 (CH=N), 3003 (CHarm.), 1591 (C=C),1147(CF3),2910(CHal),787(C-Cl). (^{1}HNMR, 400MH, DMSO-*d6*,ppm), 8.7(s, H-C=N), 6.5-8.5 (m, H, CHarm), 2.3(s, CH$_3$). ^{13}CNMR (DMSO-*d6*,ppm), 155 (C=N), 133-135 (C, Carm),126 (C, CF$_3$),25(C,CH3), .Mass spectrum: m/z calculated 691.61 g/mol, m/z found 692.6 g/mol, base peak 448g/mol.

2/N,N'-((1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(1-(4-chlorophenyl)methanimine)[A4]: the general method was followed, bis-amine (0.5g, 1.11mmol) with (0.31g, 1.11mmol) 4-chlorobenzaldyhyed to afforded A4 (0.659g, 81%) as an yellow color, FTIR(v,cm-^{1}): 1628(C=N), 3023 (CH $_{\text{arm}}$.), 1593(C=C), 1149(C-F3),763 (C-Cl). ^{1}HNMR (400MH, DMSO-*d6*, ppm), 8.5(s, H-C=N), 6.5-7.9 (m, H, CH $_{\text{arm}}$), 2.4 (s, CH$_3$). ^{13}CNMR(DMSO-*d6*, ppm), 163(C=N),130-134 (C,C $_{\text{arm}}$),122 (C,CF3),19(C,CH3) Mass spectrum: m/z calculated 691.61 g/mol, m/z found 692.6 g/mol, base peak 448g/mol.

3/ 2,2'-((((1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(azaneylylidene))bis(methaneylylidene))diphenol[A5]: the general method was followed, bis-amine (0.5g, 1.11mmol) with (0.345 mL, 1.11mmol) salicyldehyde to afforded A5 (0.650g, 77%) as a yellow color, FTIR(v,cm-^{1}):1618(C=N), 3057(CHarm.),1599(C=C),3383(O-H),1118(CF3) 2918(CHal), . ^{1}HNMR(400MH, DMSO-*d6*,ppm), 8.6(s, H-C=N), 7.9-7(m, H, CHarm), 2.3(s, CH$_3$),13(s,2H,OH).^{13}CNMR(DMSO-*d6*), 165(C=N), 115-145 (C,Carm), 126 (C,CF3).19(CH$_3$)160 (C,OH).Mass spectrum: m/z calculated 654.72 g/mol, m/z found 655.1 g/mol, base peak 137 g/mol. As shown Figure(1,2,3,4)

4/2,2'-((((1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(azaneylylidene))bis(methaneylylidene))bis(4-nitrophenol) [A6]: the general method was followed, bis-amine (0.5g, 1.11mmol) with (0.25mL, 1.11mmol) to afforded A6(0.322g, 85%), as an orange color, FTIR (v,cm^1):1622(C=N),3045(CHarm.),1556(C=C),3402(OH),1147(CF3),2966(CHal)1583(NO2).^{1}HNMR(400MH, DMSO-*d6*), 8.5(s, H-C=N) 6.5-8.5, (m, H, CH $_{\text{arm}}$), 2.4 (s, CH$_3$),10(S.2H,OH) ,^{13}CNMR(DMSO-*d6*), 167 (C,C=N)110-145 (C, C $_{\text{arm}}$),125 (C,CF$_3$), 25 (C, CH3), 145(C,C-NO$_2$),162 (C,OH).Mass spectrum: m/z calculated 744.71 g/mol, m/z found744.7 g/mol, base peak 488.1 g/mol, As shown Figure(5,6,7,8)

2.3. Anticorrosion details

Electrochemical corrosion measurements were performed using a potentiostat/galvanostat (EmStat 4s, PalmSens, Holland) connected to a host computer. The system included a thermostat and magnetic stirrer to maintain uniform temperature and solution mixing during the tests.A 250 mL Pyrex corrosion cell equipped with three electrodes was used:

- Working electrode: carbon steel (C45) specimen, composition shown in Table
- Reference electrode: saturated calomel electrode (Hg/Hg$_2$Cl$_2$, sat. KCl).
- Counter electrode: platinum wire (10 cm length).

The working electrode was immersed in the test solution for 15 minutes to establish a steady open circuit potential (E_ocp). Measurements were then carried out within a potential range of ±200 mV vs. OCP.

Polarization curves were recorded in 1 M HCl solution in the absence and presence of different concentrations of the synthesized Schiff base inhibitors. The corrosion current density (i_corr) and corrosion potential (E_corr) were obtained by extrapolating the anodic and cathodic Tafel regions. The anodic (b_a) and cathodic (b_c) slopes were also calculated.

Table 1. Solubility and TLC of compounds (A1-A6)

*soluble (+), partially soluble (δ), insoluble (-)

Comp. no.	**solvent**						**TLC**		
	DMSO	**acetone**	**water**	**Hexane**	**Ethanol**	**benzene**	**Solvent**	**Ratio**	**Rf**
A1	+	+	-	-	□	-	Ethylacetate:hexane	3:2	0.5
A2	+	+	-	-	□	-	Ethylacetate:hexane	3:2	0.8
A3	+	+	-	-	□	-	Ethylacetate:hexane	3:2	0.7
A4	+	+	-	-	□	-	Ethylacetate:hexane	3:2	0.5
A5	+	+	-	-	□	-	Ethylacetate:hexane	3:2	0.6
A6	+	+	-	-	□	-	Ethylacetate:hexane	3:2	0.7

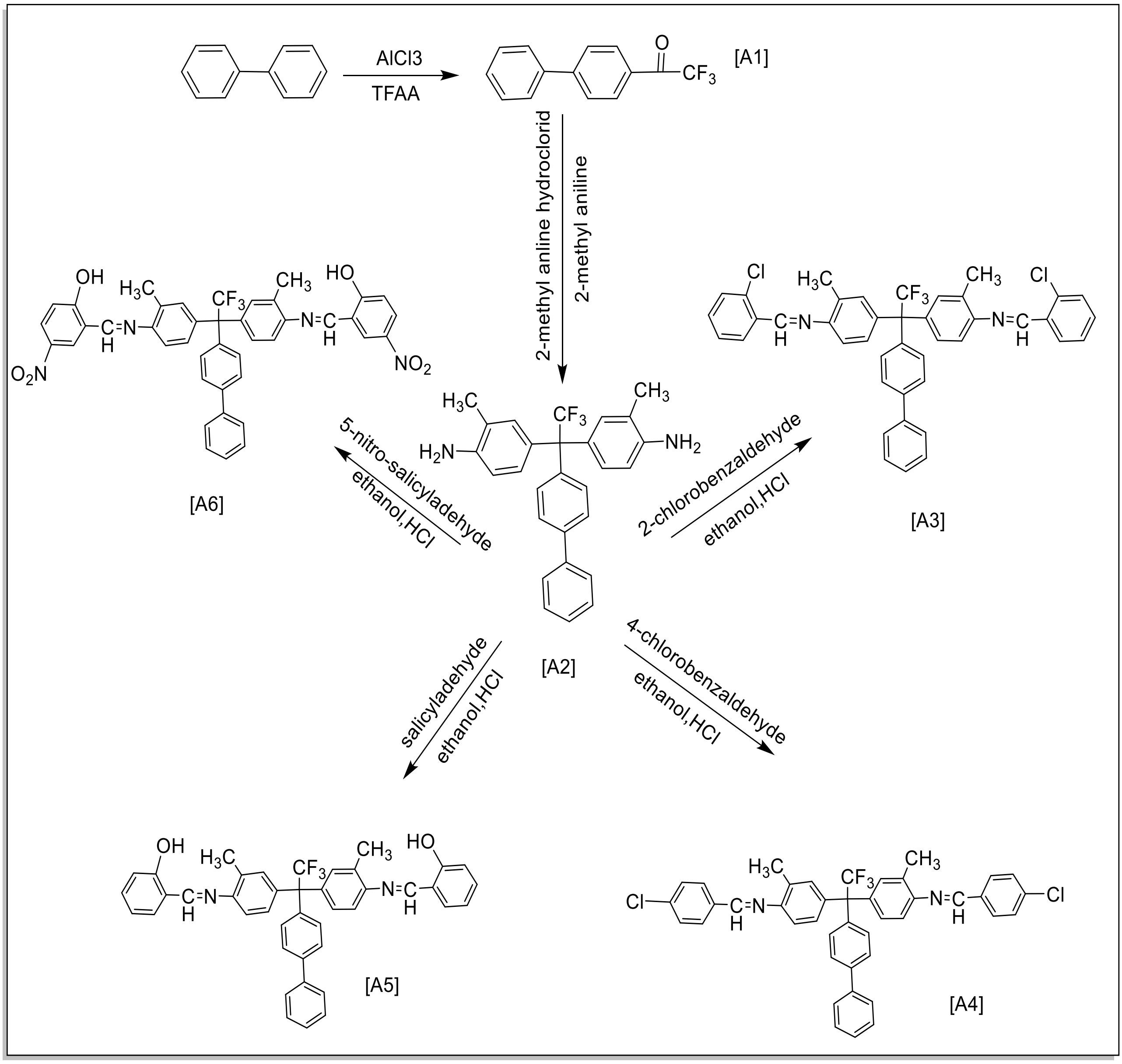


Figure 1. synthesis of compound from [A1-A6]

Table 2. Some of physical properties to the synthesized compounds[A1-A6].

comp.	name	color	M.P(°C)	yield
A1	4-Biphenylyl trifluoromethyl ketone	colorless crystals.	102-103	75%,
A2	1,1-bis(3-methyl-4-aminophenyl)-1-(4-biphenylyl)-2,2,2- trifluoroethane	white powder	192-193	63%
A3	N,N'-((1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(1-(2-chlorophenyl)methanimine	orange	80-82	85%
A4	N,N'-((1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(1-(4-chlorophenyl)methanimine	yellow	100-102	81%
A5	2,2'-((((1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(azaneylylidene))bis(methaneylylidene	yellow	172-174	77%
A6	2,2'-((((1-([1,1'-biphenyl]-4-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(azaneylylidene))bis(methaneylylidene))bis(4-nitrophenol	yellow	140-142	88%

3. Results and discussion

In this work, a new series of Schiff bases containing fluorine atoms were prepared and the fabrication of these compounds was confirmed by a several of techniques such as FTIR, ^{1}HNMR, ^{13}CNMR, and mass spectroscopy, where it was confirmed by the appearance of distinct spectra at 1600-1650 cm^{-1} for the C=N groupin FTIR, which indicates the successful preparation of Schiff bases and the absence of unreacted materials. Also, the appearance of additional peaks at (1137-1149) is due to the C-F bond, which confirms the presence of fluorine atoms, and the absence of aldehyde peaks confirms the success of the reaction. As for the ^{1}HNMR, the appearance of signals of 8-9 ppm is due to the CH=N proton, which enhances the formation of Schiff bases. In the ^{13}CNMR, clear signals appear at 160-170 ppm, which is due to the carbon atom bonded to C=N. The compounds were analyzed and the spectrum showed a clear molecular peak which is identical to the theoretical molecular weight of the prepared compound and confirms the chemical structure of the compound. The preparation method used showed high efficiency and good results and acceptable purity were obtained, which indicates the effectiveness of the method in preparing Schiff bases containing fluorine atoms. The method was characterized by speed and efficiency. The values are presented in the tables.3,4. Some physical measurements were also performed, such as melting point, color, and chromatography, as shown in Tables 1 and 2

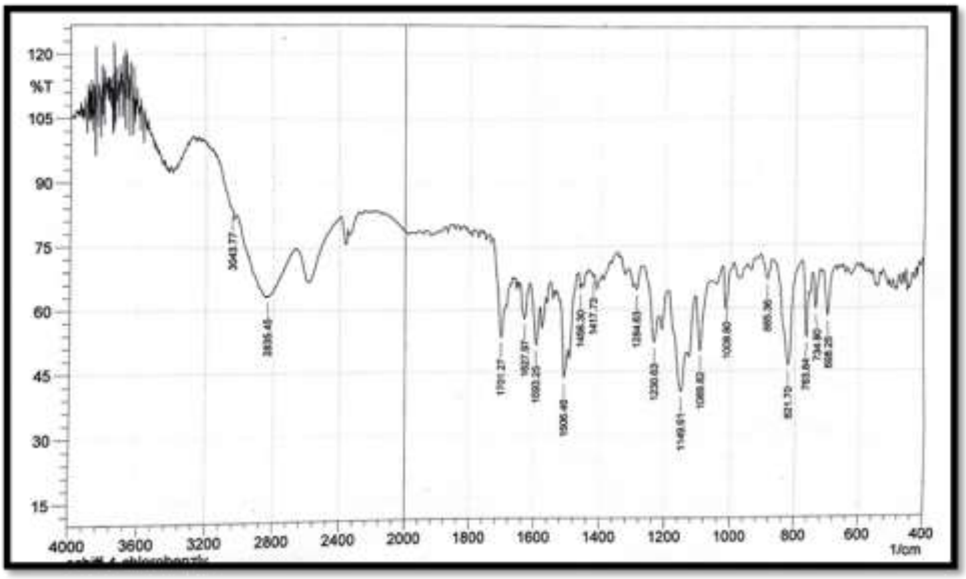


Figure 2. FTIR of compound [A3]

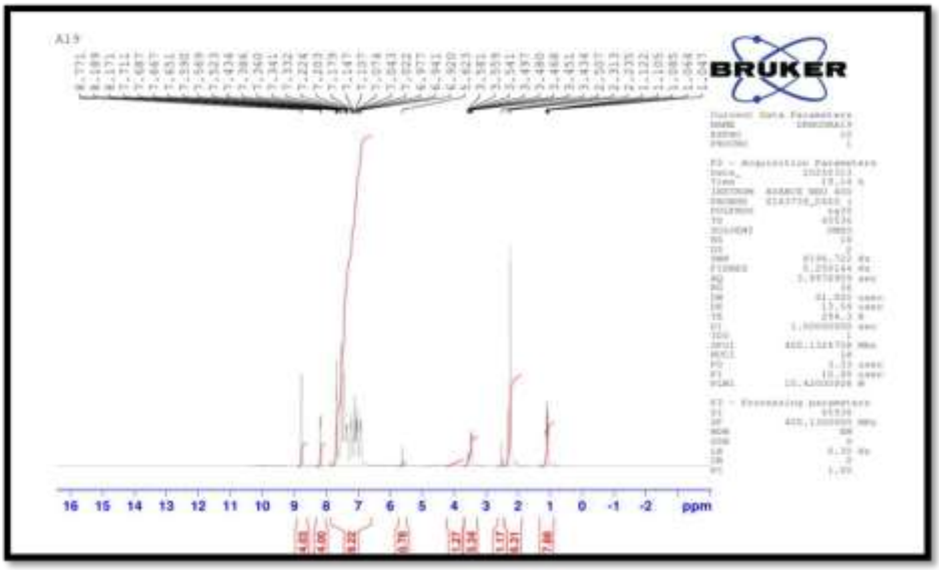


Figure 3. 1HNMRof compound [A3]

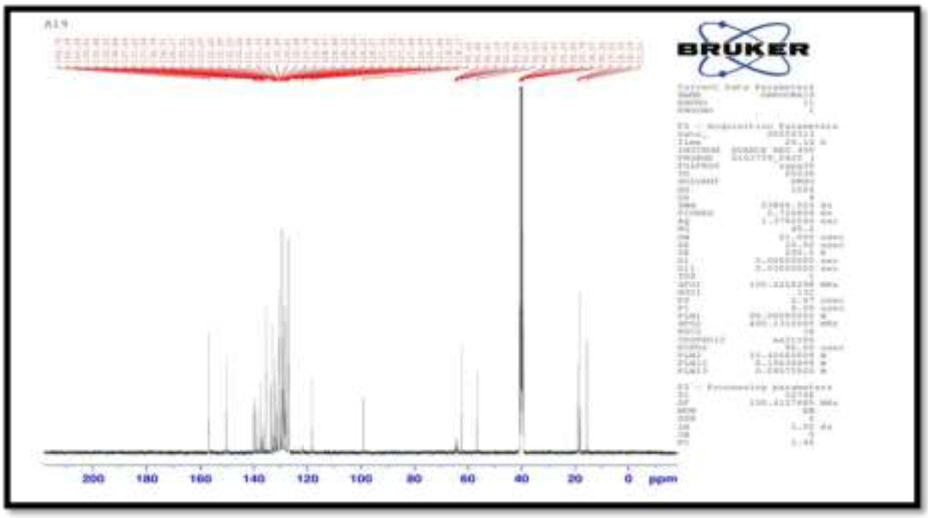


Figure 4. 13CNMRof compound [A3]

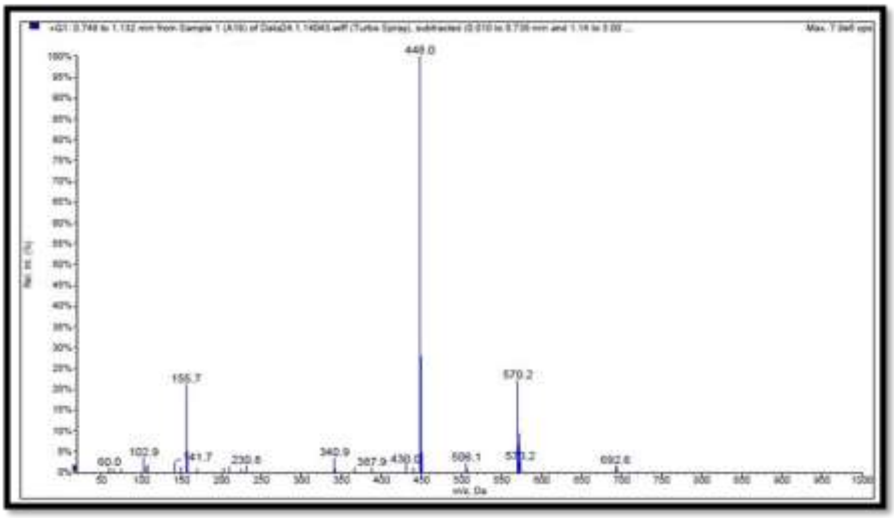


Figure 5. mass of compound [A3]

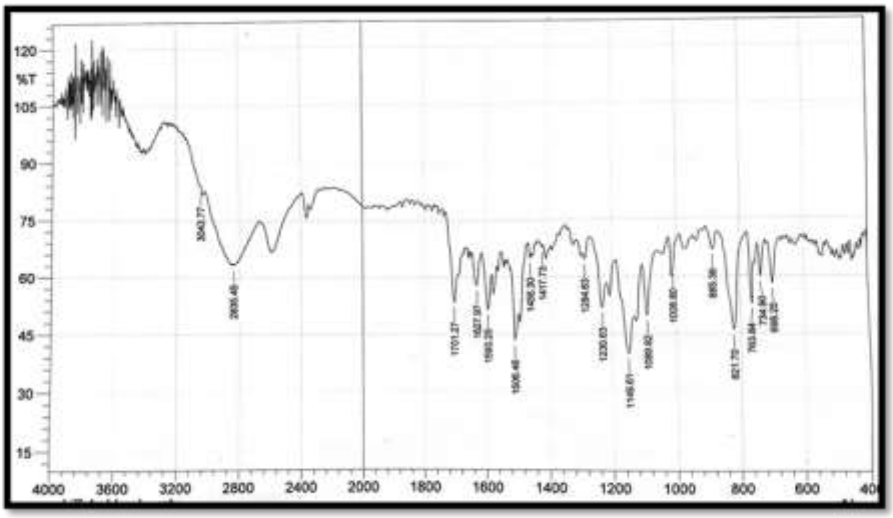


Figure 6. FTIR of compound [A4]

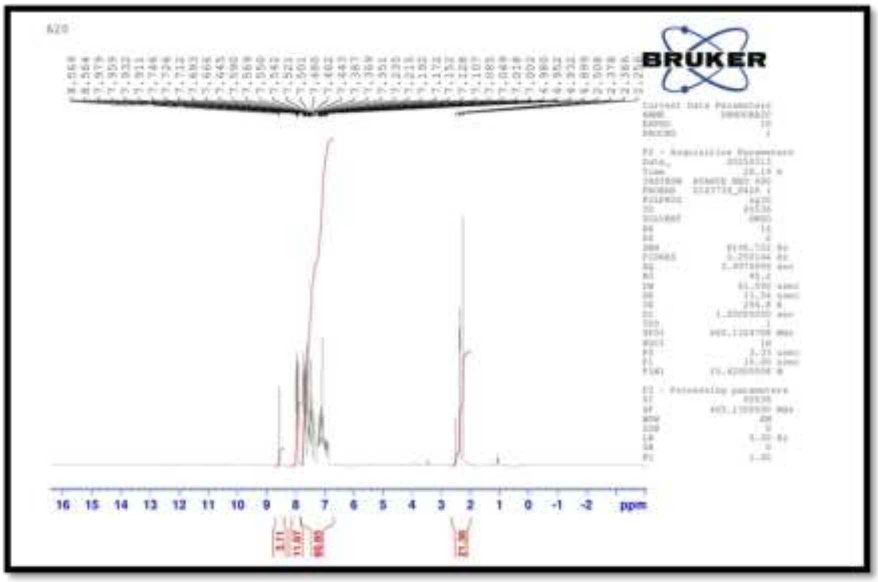


Figure 7. 1HNMRof compound [A4]

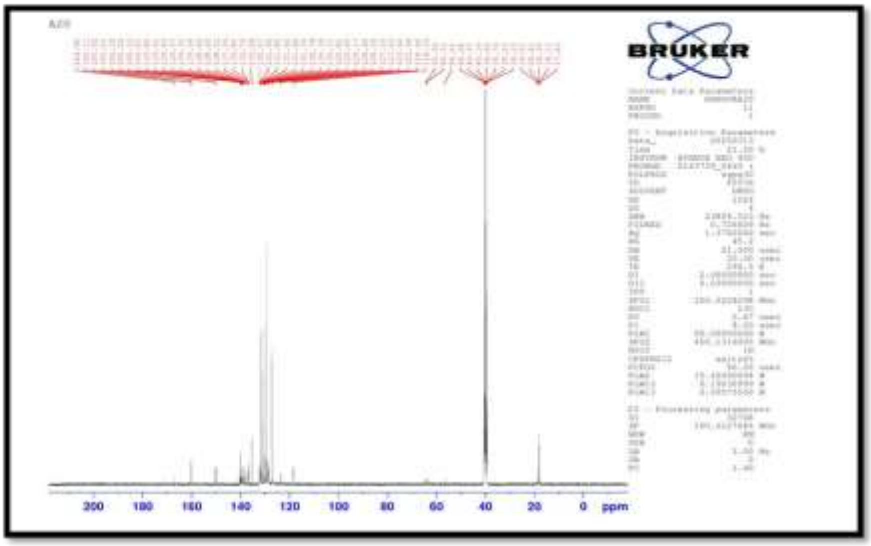


Figure 8. 13CNMRof compound [A4]

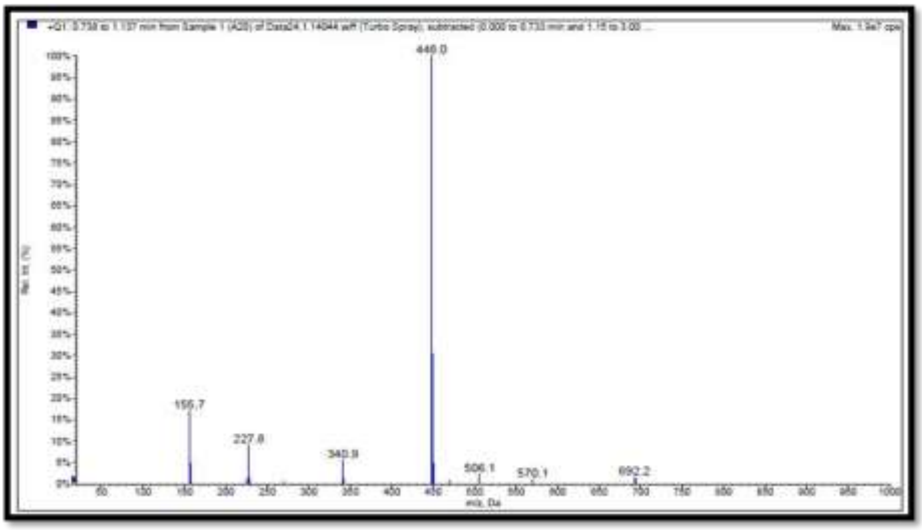


Figure 9. mass of compound [A4]

Table 3. Synthasis of compound [A1-A6], FTIR ,^{1}HNMR, ^{13}CNMR

COMP.	FTIR	^{1}HNMR	^{13}CNMR
A1	3065, 3032 (CH arm.), 1716 (C=O), 1558 (C=C), 1139(CF3)	7.4-8,3(H, CH arm)	179(C,C=O), 128-145(C,C)arm, 117(C,CF3).
A2	3491 & 3390 (NH_2), 1576 (C=C). 1132= (CF_3).	5.9-6.6(m,CH arm) 2.1(S,H,CH3) 7.5(S,H,)NH2	145(C,C-N) 125 (C,CF3) 132 (C, C arm)
A3	1622 (C=N) 3003(CH arm) 2910(CHal) 1591(C=C) 758(C-Cl) 1147 (CF3).	7.8(S,2H,(CH=N) 2.3(S,6H,(CH3.) 7.9-7(m.H,CH arm.)	133-135(C,C arm) 126 (C,CF3) 155 (C=N) 25(C,CH3)
A4	1628 (C=N) 1593 (C=C) 763 (C-Cl) 1150 (CF3) 3023(CH arm) 2910(CHal)	6.5-8.5 (H,CH arm 8.5(S,CH=N) 2.4 (S,CH3). 6,5-7.9(m,H,CH arm)	120-160 (C,C arm), 122 (C,CF3) 163(C=N) 130-134(C,C arm) 19(C,CH3)
A5	1618 (C=N) 1599 (C=C) 785 (C-Cl) 1118 (CF3) 3383(OH) 3057(CH arm) 2918(C Hal)	7.9-7.2 (m,H,CH arm) 8.6 (S,CH=N) 2.3 (S,CH_3) 14(S,2H,OH)	115-145 (C,C arm), 126 (C,CF3). 19(CH3) 165(C=N). 160 (OH)
A6	1622 (C=N) 1556 (C=C) 756 (C-Cl3) 1147 (CF3) 3402(OH) 3045(CH arm) 1583(NO2) 2966(CHal)	6.5-8.5 (m,H,CH arm), 8.5(CH=N), 2.4 (S,CH3) 10(S,2H,OH)	110-145 (C,C arm), 125 (C,CF3) 162 (C-OH) 145 (C-NO_2) 25(C,C arm) 167 (C,C=N)

Table 4. (A3,A4, A5, A6) in the Mass spectrum

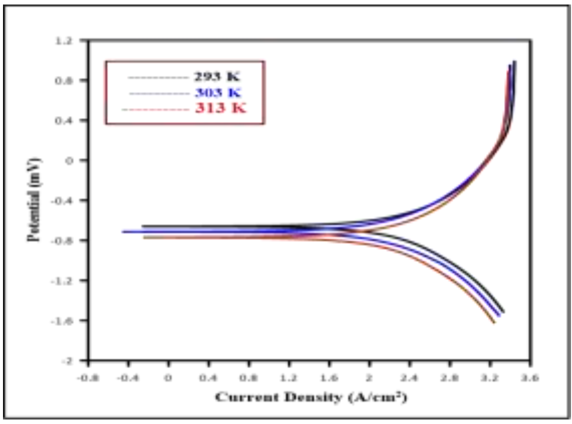
Compound	M.W	m/z Found	Base Peak	Last Fragment
A3	691	692.6	448.0	60.0
A4	691	692.2	448.0	155
A5	654	655.1	59.9	47.8
A6	744	744.7	448.1	108.9

Corrosion Parameters:

The electrochemical corrosion measurements summarized in Table 5 indicate that all tested Schiff base compounds (A3 to A6) exhibit significant inhibition efficiency (IE%) against the corrosion of metal in 1 M HCl solution at different temperatures (293 K, 303 K, and 313 K), compared to the blank sample.For the blank sample, the corrosion current density (icorr) increases with temperature, indicating enhanced corrosion rate under acidic conditions. The corrosion rate (mm/y) reaches its highest value of 6.091 mm/y at 313 K, highlighting the aggressive nature of the environment in the absence of inhibitors. Upon addition of Schiff base compounds, a notable decrease in icorr is observed across all temperatures. This is directly reflected in the significant increase in polarization resistance (Resis) and reduction in corrosion rate, demonstrating effective surface protection provided by the compounds. Among all tested compounds, compound A6 showed the highest inhibition efficiency, reaching 94% at 303 K, with a very low corrosion rate of 0.309 mm/y. This indicates strong adsorption and film-forming ability of the compound on the metal surface, likely due to the presence of electron-donating groups and heteroatoms (e.g., nitrogen, fluorine) that coordinate with the metal surface. As the temperature increases to 313 K, a slight decrease in inhibition efficiency is observed for most compounds, suggesting partial desorption of the inhibitor from the surface. However, A6 maintained a high efficiency of 93%, confirming its thermal stability and strong adsorption behaviour. Overall, the data suggest that the tested Schiff base compounds, especially A6, act as efficient corrosion inhibitors in acidic medium, and their performance is closely related to their molecular structure and ability to form a protective layer on the metal surface

Table 5. Corrosion parameters for blank and compound in HCl solutions and different compound as shown in figure (9,10,11)

Comp.	Temp.	-Ecorr(mV)	icorr (μA/cm^2)	Icorr.\r(A\cm^2)	Resis.	-Bc (mV/Dec)	Ba (mV/Dec)	Corr. rate, (mm/y)	%IE
Blake	293	-0.427	493.9	9.878E-4	70.31	0.156	0.164	4.848	-
	303	-0.385	563.2	0.001	28.96	0.055	0.120	5.529	-
	313	-0.425	620.6	0.001	57.49	0.166	0.163	6.091	-
A3	293	-0.695	40.77	8.153E-5	1938	0.421	0.320	0.400	92
	303	-0.668	45.90	9.181E-5	1636	0.369	0.326	0.451	92
	313	-0.645	51.23	1.025E1-4	1640	0.359	0.419	0.503	92
A4	293	-0.615	46.67	9.335E-5	1965	0.334	0.575	0.458	90
	303	-0.645	65.74	1.315E-4	1748	0.483	0.586	0.645	88
	313	-0.645	68.88	1.378E-4	1666	0.496	0.566	0.676	89
A5	293	-0.627	68.17	1.363E-4	1492	0.397	0.571	0.669	86
	313	-0.642	73.71	1.474E-4	1527	0.481	0.562	0.724	87
	313	-0.650	76.55	1.531E-4	1592	0.508	0.627	0.751	88
A6	293	-0.647	31.44	6.288E-5	2142	0.285	0.339	0.309	94
	303	-0.629	38.34	7.668E-5	1750	0.259	0.383	0.376	93
	313	-0.665	42.69	8.538E-5	2176	0.427	0.429	0.419	93



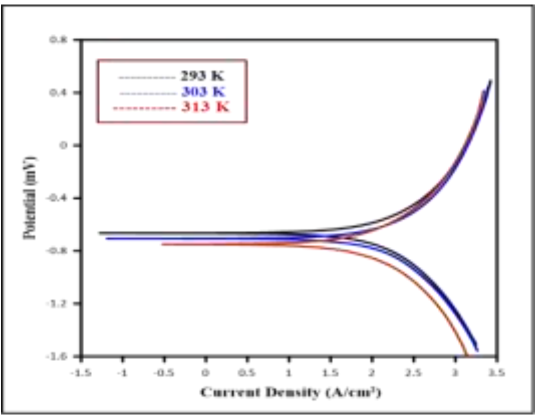


Figure 10. Polarization curves for corrosion[A3]   FIGURE10. Polarization curves for corrosion[A6]

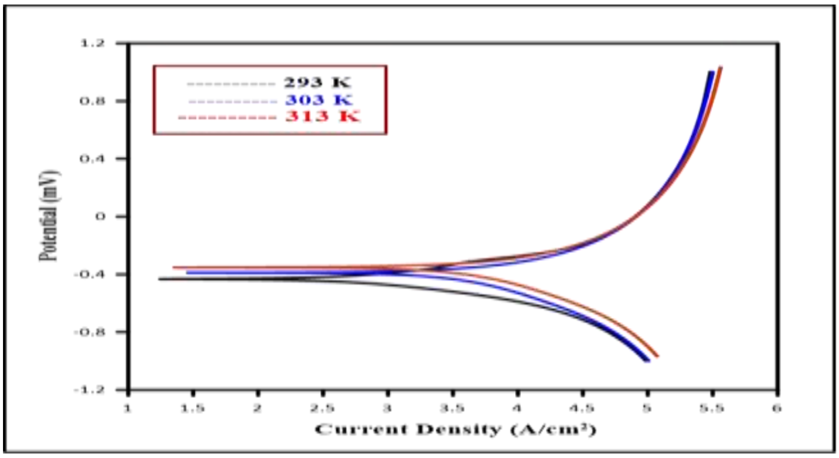


Figure 11. Polarization curves for corrosion of blank HCl

4. Conclusion

In this study, a new series of fluorinated Schiff base compounds was synthesized and characterized using FT-IR, UV–Vis, and NMR spectroscopy, confirming the proposed structures. Electrochemical measurements revealed that all compounds acted as effective corrosion inhibitors for carbon steel in acidic media. Compound A6 showed the highest inhibition efficiency (94%) and the lowest corrosion rate (0.3 mm/year), whereas compound A5 exhibited the lowest performance (86%).These results indicate that the presence of substituents, particularly the $–CF_3$ group, enhances adsorption and the formation of a protective film on the metal surface.The synthesized Schiff bases can be considered promising eco-friendly corrosion inhibitors, and further studies are recommended to explore the structure–activity relationship (SAR) and inhibition mechanism in more detail.

Conflict of interest

The authors declare no conflict of interest

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