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Synthesis and Characterization of New Substituted 4,4'-Thiodianiline Containing Schiff Base Compounds as Corrosion Inhibitors

Nour Abd Alrazzak[1,*], Mohanad Mousa Kareem[2], Sadiq Abdul Hussain Karim[3]

[1]Department of Chemistry, College of Science for Women, University of Babylon, Babylon, Hilla, Iraq.
[2]University of Babylon, College of Science, Department of Chemistry, P.O.BOX 51002 Hilla, Babylon, Iraq
[3]Department of Chemistry, College of Education for Pure Science, University of Babylon, Iraq.

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Abstract

A new Schiff base derivative were prepared from diamine derivatives (4,4'-thiodianiline) and various aromatic aldehydes (2-chrobenzaldehyde,3,5-dichlorobenzaldehyde, 4-hydroxy-3-methoxybenzaldehyde, 2-hydroxy benzaldehyde, 4-hydroxy benzaldehyde and indol-3-carbadehyde) by condensation reaction using acid catalyzed under reflux to yield final products which acted as good corrosion inhibitor against 1M hydrochloric acid at different temperatures (293, 303 and 313 °C). These compounds showed excellent protection for carbon steel 45. Based on the results obtained from the present study, the IE% inhibition efficiency was observed for the synthesized compounds [N1-N6], ranging from 87% to 96%. The final reaction was investigated using thin-layer chromatography. All structures of new synthesis compounds were proven using FTIR, ^{1}H-NMR, ^{13}C-NMR, and mass spectroscopy.

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*Corresponding author: wsc.noorabd@uobabylon.edu.iq

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1. Introduction

The Schiff base was named after German scientist Hugo Schiff (1864-1915), who discovered the bases [1]. These compounds are an important type of organic compound, prepared through the condensation reaction of a primary amine with aldehydes or ketones [2-5].The mechanism of synthesis of these compounds includes a nucleophilic substitution reaction whereby the primary amine, behaves as a nucleophile and carbonyl compounds as an electrophile to produce carbinol amine, an intermediate compound with hydrolysis using acid or base catalysis and an azeotroping agent (if necessary) by losing a water molecule [5-10]. This reaction within non-aqueous organic solvents takes 8-12 hours. Since the reaction is highly reversible, it is advantageous to remove the generated water from the process to maximize the yield of Schiff bases [11, 12]. The general formula (R-C=N-R-) contains (-C=N-) azomethine group and substituents that may be aromatic, aliphatic, heterogeneous, homogeneous, and other different substituents [13-18]. Schiff base derivatives are important to researchers due to their wide range of applications and metal-binding characteristics [19-22]. These Schiff base ligands play a crucial role in the development of coordination chemistry [23,24], which is widely used in syntheses [25]. These compounds can act as bi-, tri- and tetr-adentate chelates with metals to form stable complexes [26]. Schiff base from anisaldehyde and its derivative contain hydroxyl group as a good chelating agent have thermochromic and photochromic characteristic. The Schiff base synthesis from salicylaldehyde and its derivatives is considered a good ligand that binds with metals to form highly stable complexes [27]. The presence of imine group in Schiff compound makes these compounds a good compound in myriad field, such as analytical, inorganic [28], and a wide range of biological activities, such as antioxidant [29,30], antiviral [31,32], antitumor [33], anti-inflammatory [34], antimicrobial [35-37], anticancer [38-42] and, analgesic activities [43]. It also has various applications in different fields, such as corrosion inhibitors [44], analytical chemistry as chemical sensors [45], and dyes [46, 47]. Imines are important

intermediate and starting material to synthesize different compounds like Mannich bases [48], and heterocyclic compounds like pyrimidine [49] and indole [50, 51]. The corrosion effect occurs in various industries, including the gas and oil sector. The corrosion chemically and electrically erodes metals, caused by corrosive elements in the air and affected by temperature and time [52-54]. Corrosion inhibitors reduce or prevent the hydrogen evolution reaction, which is the predominant process in acidic media. Two electrochemical processes are involved in corrosion: oxidation at the anodic site and reduction at the cathodic site [55-57]. This research aimed to synthesize derivatives of Schiff base compounds as anti-corrosive agents, and FTIR, ^{1}H NMR, ^{13}C NMR, and mass spectrometry characterized these compounds.

2. Experimental Work

2.1. Chemicals

All used compounds were supplier from BDH and sigma, the characterization device were as follows (FTIR 8400s, Shimadzu (Japan); 1H-NMR, Bruker (400 MHz) and ^{13}C NMR, Bruker (~100 MHz) and Mass spectrometer UFLC LC-AD20 system -3200 QTRAP , Shimadzu (Japan).

2.2. Methods

2.2.1. Synthesis of bisamine derivatives [N1-N6]:

0.5 g (0.0023 mole) 4-(4-aminophenyl) sulfanyl aniline reacted with 0.647, 0.805, 0.699, 0.565, 0.565, 0.635 g (0.004 mole) (2-chrobenzaldehyde-3,5-dichlorobenzaldehyde, 4-hydroxy-3-methoxy benzaldehyde, 2-hydroxy benzaldehyde, 4-hydroxy benzaldehyde and indol-3-carbadehyde) respectively in absolute EtOH using dropwise of glacial acetic acid under reflux for 3 hrs. Then, the solvent was evaporated, and the final product was recrystallized [58].

Compound N,N'-(thiobis(4,1-phenylene)) bis(1-(2-chlorophenyl)methanimine) [N1]: IR (v, cm^{-1}); 3057 (C-H)arm., 1616 C=N, 1597 C=C, 632 C-Cl; ^{1}HNMR (ppm, DMSO-*d6*) 7.350-8.650 (m, 8 H, C-H aromatic), 8.927 (s, H, CH=N); ^{13}CNMR (ppm, DMSO-*d6*) 124.31-133.58 (8C, Carm), 133.78 (C, C-Cl), 151.01 (C,C-N), 159.41 (C, CH=N); MS (EI, m/z): Found 462, Calculated 461.40, base peak 217.1, Chemical formula; $C_{26}H_{18}Cl_2N_2S$, molecular weight: 461.40, color: Pale green, melting point: 191-193, yield: 79%, TLC: (solvent: Hexane: ethylacetate, ratio: 2:3, R_f: 0.7).

Compound N,N'-(thiobis(4,1-phenylene)) bis (1-(3,5-dichlorophenyl)methanimine) [N2]: IR (v, cm^{-1}); 3072 (C-H)arm., 1614 C=N, 1583 C=C, 624 C-Cl; ^{1}HNMR (ppm, DMSO-d6) 6.868-7.584 (m, 7 H, C-H aromatic), 8.952(s, H, CH=N); ^{13}CNMR (ppm, DMSO-*d6*) 122.43- 132.74(7C, Carm), 133.06 (C, C-Cl), 150.69 (C,C-N), 161.42 (C, CH=N); MS (EI, m/z): Found 531, Calculated 530.29, base peak 96.7, Chemical formula; $C_{26}H_{16}Cl_4N_2S$, molecular weight: 530.29, color: Pale orange, melting point: 183-185, yield: 77%, TLC: (solvent: Hexane: ethylacetate, ratio: 2:3, R_f: 0.5).

Compound 4,4'-(((thiobis(4,1-phenylene)) bis (azaneylylidene))bis(methaneylylidene)) bis(2-methoxyphenol) [N3]: IR (v, cm^{-1}); 3292 O-H, 3049 CH=N, 3100 (C-H)arm., 2966-2850 (C-H) aliph., 1651C=N, 1595 C=C, 1030 C-O-C; ^{1}HNMR (ppm, DMSO-*d6*) 3.849 (s, 3 H, C-H aliphatic), 7.021-7.590 (m, 7 H, C-H aromatic), 8.590 (s, H, CH=N), 9.784 (s, H, OH); ^{13}CNMR (ppm, DMSO-d6) 56.01 (C, O-CH_3), 124.31-132.51 (7C, C arm.), 149.15(C,C-O-C), 149.91 (C,C-N), 153.57 (C,C-OH), 163.37 (C, CH=N); MS (EI, m/z): Found 486.7, Calculated 485.57, base peak 217.2, Chemical formula; $C_{28}H_{24}N_2O_4S$, molecular weight: 484.57, color: Dark orange, melting point: 122-124, yield: 84%, TLC: (solvent: Hexane: ethylacetate, ratio: 2:3, R_f: 0.6).

Compound 2,2'-(((thiobis(4,1-phenylene)) bis (azaneylylidene))bis(methaneylylidene)) diphenol [N4]:IR (v, cm^{-1}); 3186 O-H, 3022 (C-H)arm., 1668 C=N, 1649 C=C; ^{1}HNMR (ppm, DMSO-*d6*) 6.734-7.961(m, 8 H, C-H aromatic), 8.447 (s, H, CH=N), 10.557 (s, H, OH); ^{13}CNMR (ppm, DMSO-*d6*) 122.11-132.08 (8 C, Carm), 151.63(C,C-N), 160.73 (C,C-OH), 161.30(C, CH=N); MS (EI, m/z): Found 425.1, Calculated 424.52 , base peak 321.1 , Chemical formula; $C_{26}H_{20}N_2O_2S$, molecular weight: 424.52, color: orange, melting point: 146-148, yield: 82%, TLC: (solvent: Hexane: ethylacetate, ratio: 2:3, R_f: 0.7).

Compound 4,4'-(((thiobis(4,1-phenylene))bis (azaneylylidene))bis(methaneylylidene))diphenol [N5]:IR (v, cm^{-1}); 3412 O-H, 3034 (C-H)arm., 1626 C=N, 1599 C=C; ^{1}HNMR (ppm, DMSO-*d6*); 7.071-7.627 (m, 8 H, C-H aromatic), 8.535 (s, H, CH=N), 9.790 (s, H, OH). ^{13}CNMR (ppm, DMSO-d6) 122.30-132.52 (6 C, Carm),
150.39 (C,C-N), 160.38 (C,C-OH), 160.52 (C, CH=N); MS (EI, m/z): Found 425, Calculated 424.52 , base peak 85.2, Chemical formula; $C_{26}H_{20}N_2O_2S$,

molecular weight: 424.52, color: Pale green, melting point: 228-230, yield: 83%, TLC: (solvent: Hexane: ethylacetate, ratio: 2:3, R_f: 0.7).

Compound N,N'-(thiobis(4,1-phenylene))bis (1-(1H-indol-2-yl)methanimine) [N6]:IR (v, cm^{-1}); 3151N-H, 3034 (C-H)arm., 1647 C=N, 1616 C=C; ^{1}HNMR (ppm, DMSO-d6) 7.506-7.583 (m, 9 H, C-H aromatic), 8.305 (s, H, CH=N), 11.045 (s, H, NH); ^{13}CNMR (ppm, DMSO-*d6*) 117.04-124.43 (11 C, Carm), 149.81(C,C-N), 154.52 (C, CH=N); MS (EI, m/z): Found 471.3, Calculated 470.59, base peak 217.3, Chemical formula; $C_{30}H_{22}N_4S$, molecular weight: 470.59, color: yellow, melting point: 179-181, yield: 86%, TLC: (solvent: Hexane: ethylacetate, ratio: 2:3, Rf: 0.3).

2.3. Anti-corrosion study for synthesized compounds

Procedure as shown in literature [59, 60], using 1 M hydrochloric acid at different temperatures C45 293, 303, and 313 °C, and the composition of metal (carbon steel) as shown below in Table 1.

Table 1 : Chemical composition of carbon steel

Metal	C%	Si%	Mn%	S%	P%
Carbon Steel 45	0.36-0.42	0.15-0.30	1.00-1.40	0.05	0.05
Metal	Cu%	Ni%	Cr%	Fe%	
Carbon Steel45	0.50	0.20	0.20	96.88-97.49	

3. Results and Discussion

New compounds from [N1-N6] were synthesized by condensation reaction of primary bis amine (4-(4-aminophenyl) sulfanylaniline) with different substituted aromatic aldehyde compounds (2-chrobenzaldehyde,3,5-dichlorobenzaldehyde, 4-hydroxy-3-methoxybenzaldehyde, 2-hydroxy benzaldehyde, 4-hydroxy benzaldehyde and indol-3-carbadehyde) containing electron with donating group that gave good yield and excess reaction. These reactions occurred using 1:2 molar ratio of amine to aldehyde. The aldehyde preferred the ketone due to its electronic and steric effects, that the two aryl or alkyl substituents less the nucleophilicity of carbonyl carbon in ketone more than in aldehyde which contain one alkyl or aryl group, also, the substituents in ketone make hindered to approach the nucleophiles from electrophilic center. The mechanism of synthesized compounds is a nucleophilic addition elimination reaction involve addition of nucleophile to primary amine in the first step and formation of imine group and elimination of water in second step. Compounds [N1-N6] were fully soluble in dimethyl sulfoxide, ethanol, and acetone, while partially soluble in toluene, and soluble in hexane and water, as shown in Table 2. The structures of all synthesized compounds were observed using FTIR, ^{1}H-NMR, ^{13}C-NMR spectra and mass spectroscopy. FTIR showed that the carbonyl of aldehyde and amine of starting material disappearance and show new peaks of CH (aromatic), imine and C=C (aromatic) at 3022 -3072, 1614 -1668, 1595-1649 respectively [61]. Characterization of compounds by ^{1}H-NMR spectroscopy using DMSO-*d6* as solvent that appeared at 2.5 and the other signals which improved the structure of synthesis compounds were shown in 6.734-8.650, 8.305-8.927, for (m, H, C-H aromatic) and (s, H, CH=N), moreover, ^{13}CNMR spectroscopy using DMSO-*d6* which appeared at 40 and the other characteristics peak were shown in 122.11-133.58, 149.81-151.63 and 154.52 -163.37 for (8C, Carm),(C,C-N), (C, CH=N) respectively [62-65]. The basic concept of mass spectrometry is to ionize a material using the ionization method: separate the ions according to their mass/charge ratio using the ion separation method, and then record the number of ions that correspond to each mass/charge unit as a spectrum. The produced compounds were structurally verified by mass spectroscopy using molecular ions (m/z) and the remaining peaks showed fragments of ions. All Schiff base derivatives [N1-N6] contained parent ion and base peaks in the same value, indicating the compounds stability. Using Thin Layer Chromatography (TLC), which displays new spots for target chemicals, the reaction completion time was tracked. Various solvents were used to check the final products' purity based on factors like polarity and the retention factor started at 0.3 to 0.7. The yield of synthesis compounds ranged from 77 to 86, depending on the groups linked to aromatic aldehydes. The corrosion parameters are evaluated and displayed in Tables 3 and the polarization curves for the test's compounds are shown in figures 1-6.

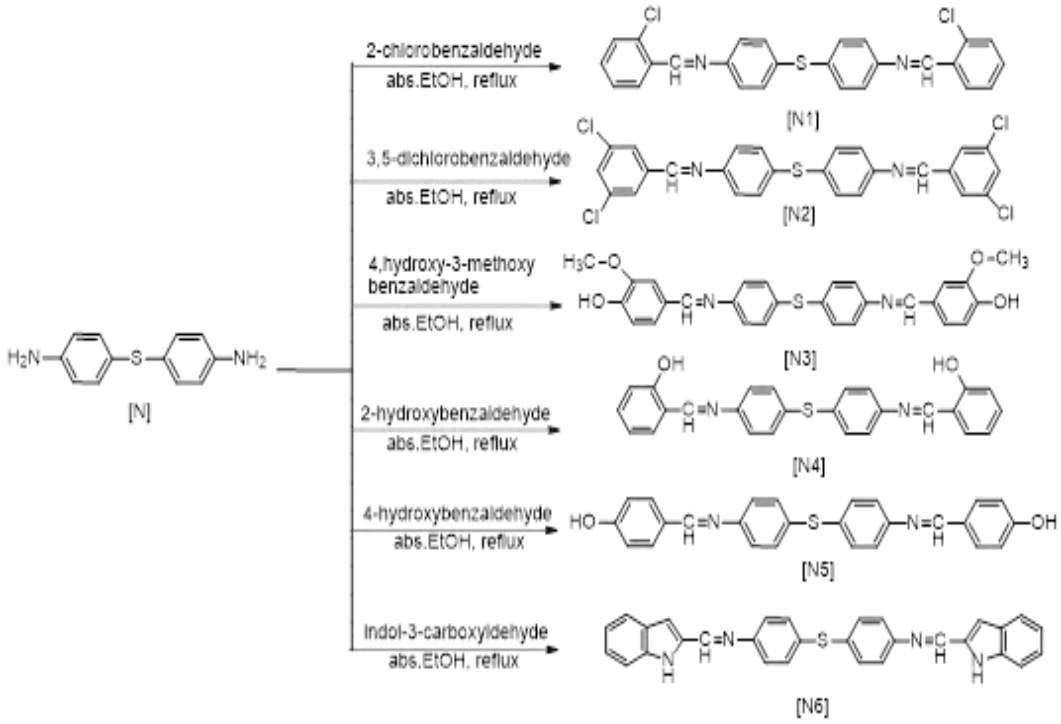
Table 2: solubility of compounds [N1-N6]:

Compounds	Solvents					
	dimethyl sulfoxide	acetone	hexane	water	benzene	toluene
[N1]	+	+	-	-	-	δ
[N2]	+	+	-	-	-	δ
[N3]	+	+	-	-	-	δ
[N4]	+	+	-	-	-	δ
[N5]	+	+	-	-	-	δ
[N6]	+	+	-	-	-	δ
*soluble (+), partially soluble (δ), insoluble (-)						

The anodic and cathodic Tafel curves were extrapolated to determine corrosion potential (Ecorr) and corrosion current density (Icorr). Using [1] M hydrochloric acid solution, polarization curves were examined in an acidic media. When inhibitor [N1-N6] was added to acidic solution as the temperature increased, the anodic reaction slowed down and the cathodic reaction of hydrogen gas generation was prolonged. These observations demonstrated the inhibitor characteristic of synthesized compounds. The corrosion current density also increased as shown in table 7. Using the following formula, the (IE) inhibition efficiency was determined [66]:

$$\% IE = \frac{(I\ corr)o - (I\ corr)}{(I\ corr)o} \times 100 \quad \ldots (1)$$

Where (I corr)o and (I corr) represented corrosion current density with and without inhibitor. The Schiff-base compounds showed excellent ability against corrosion according to the obtained results, due to the lone pair of electrons on hetero atom. These compounds made a barrier layer between corrosive metal and carbon steel [67-79]. Electron-donating functional groups such as ethers, hydroxyls, and amines are typically used for corrosion inhibition. These groups strengthen the metal's resistance to oxidation and corrosion by increasing the electron density on its surface. In contrast to inorganic inhibitors, organic inhibitors those containing functional groups like ethers, hydroxyls, and amines can provide superior protection in environments that include both carbon and chloride. Consequently, compounds [N2 and N3] showed the best anti-corrosion action at different temperatures (293, 303 and 313) ^{o}C. The highest inhibition efficiency at low temperature was 96% (N2) and 95% (N3) and at highest temperature 95% (N2) and 94% (N3) respectively. Compounds [N1, N6, N4 and N5] exhibited comparable corrosion resistance, probably due to the similarity of their structural and functional groups, but they can still be considered a very good percentage (87-90%) when compared to other organic compounds.



Scheme1: synthesis compounds [N1-N6]

Table 3: Corrosion parameters for blank and compounds [N1-N6] in HCl solutions.

Comp.	Temp.	-Ecorr. (mV)	icorr (µA/cm2)	I corr./ r (A/cm²)	Resis. (Ω)	-Bc (mV/Dec)	Ba (mV/Dec)	Corr. rate, (mm/y)	IE%
Blank	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	-
[N1]	293	-0.681	50.19	1.004E-4	1271	0.239	0.383	0.493	90
	303	-0.686	55.75	1.115E-4	1220	0.262	0.391	0.547	90
	313	-0.772	63.01	1.260E-4	1056	0.393	0.251	0.618	90
[N2]	293	-0.752	19.99	3.998E-5	2438	0.237	0.214	0.196	96
	303	-0.740	28.04	5.608E-5	1868	0.256	0.228	0.275	95
	313	-0.707	30.00	6.000E-5	1897	0.217	0.331	0.294	95
[N3]	293	-0.770	17.66	3.532E-5	2544	0.226	0.191	0.173	96
	303	-0.692	34.40	6.879E-5	1379	0.189	0.260	0.338	94
	313	-0.745	40.11	8.022E-5	1434	0.280	0.252	0.394	94
[N4]	293	-0.718	53.99	1.080E-4	1290	0.256	0.429	0.530	89
	303	-0.736	63.30	1.266E-4	1366	0.325	0.514	0.621	88
	313	-0.783	70.00	1.400E-4	1023	0.382	0.290	0.687	88
[N5]	293	-0.804	64.98	1.300E-4	2166	0.619	0.681	0.638	87
	303	-0.797	69.78	1.396E-4	1818	0.611	0.560	0.685	88
	313	-0.775	73.44	1.469E-4	1787	0.607	0.602	0.721	88
[N6]	293	-0.652	49.96	9.992E-5	1780	0.375	0.451	0.490	89
	303	-0.666	55.89	1.118E-4	1701	0.434	0.442	0.549	90
	313	-0.683	64.05	1.281E-4	1959	0.554	0.604	0.629	89

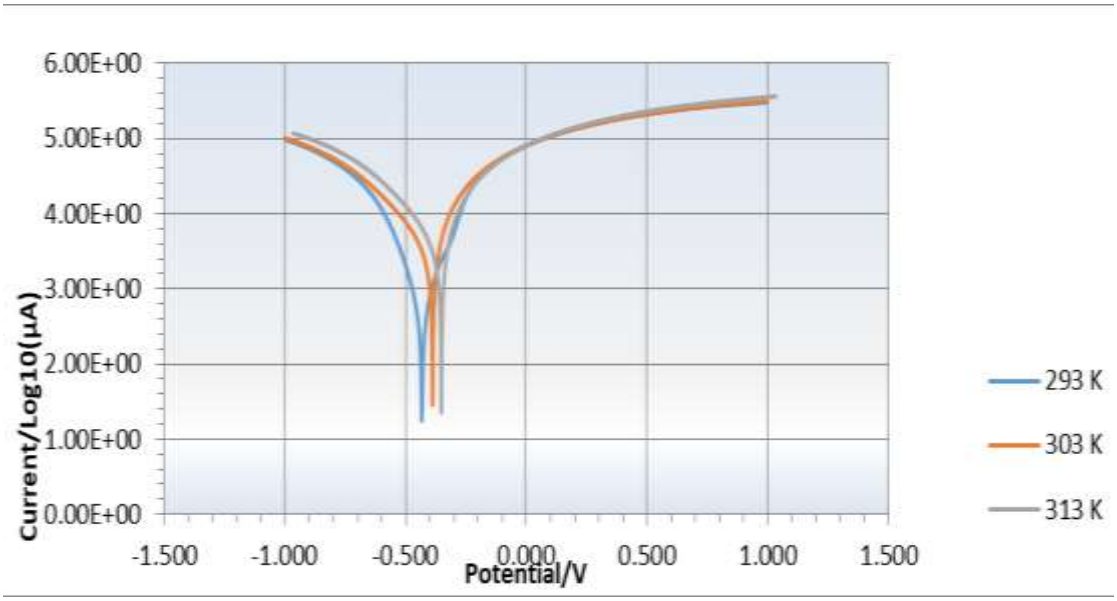


Figure 1. Polarization curves of corrosion for blank HCl

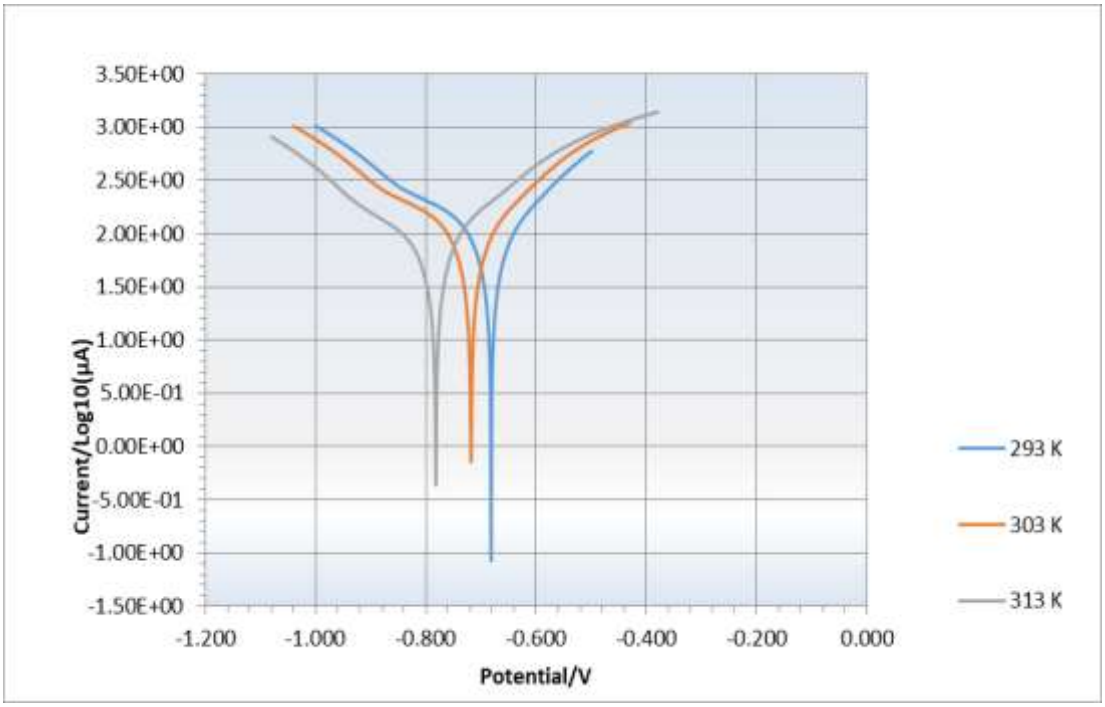


Figure 2. Polarization curves of anti-corrosion for compound [N1].

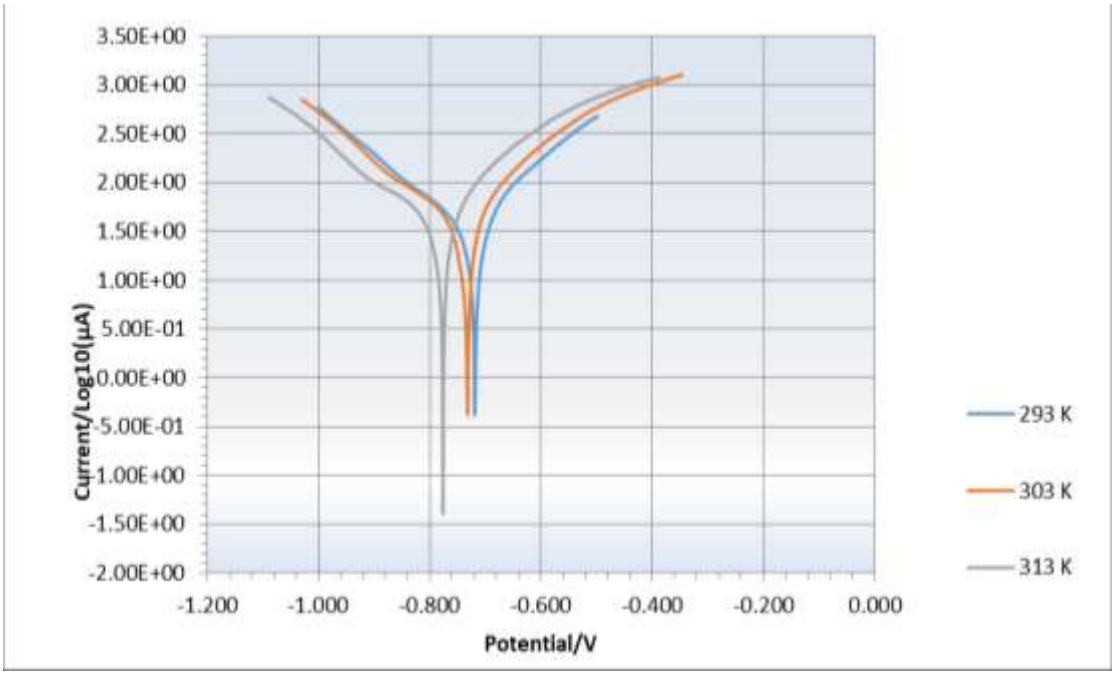


Figure 3. Polarization curves of anti-corrosion for compound [N2].

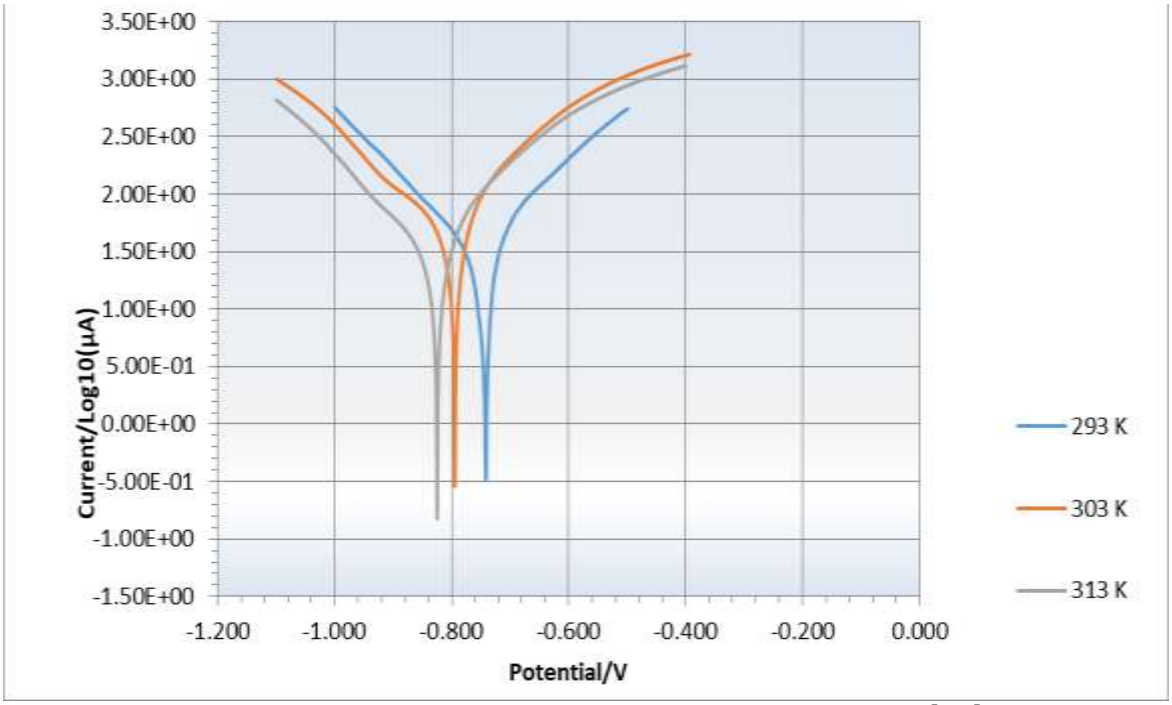


Figure 4. Polarization curves of anti-corrosion for compound [N3].

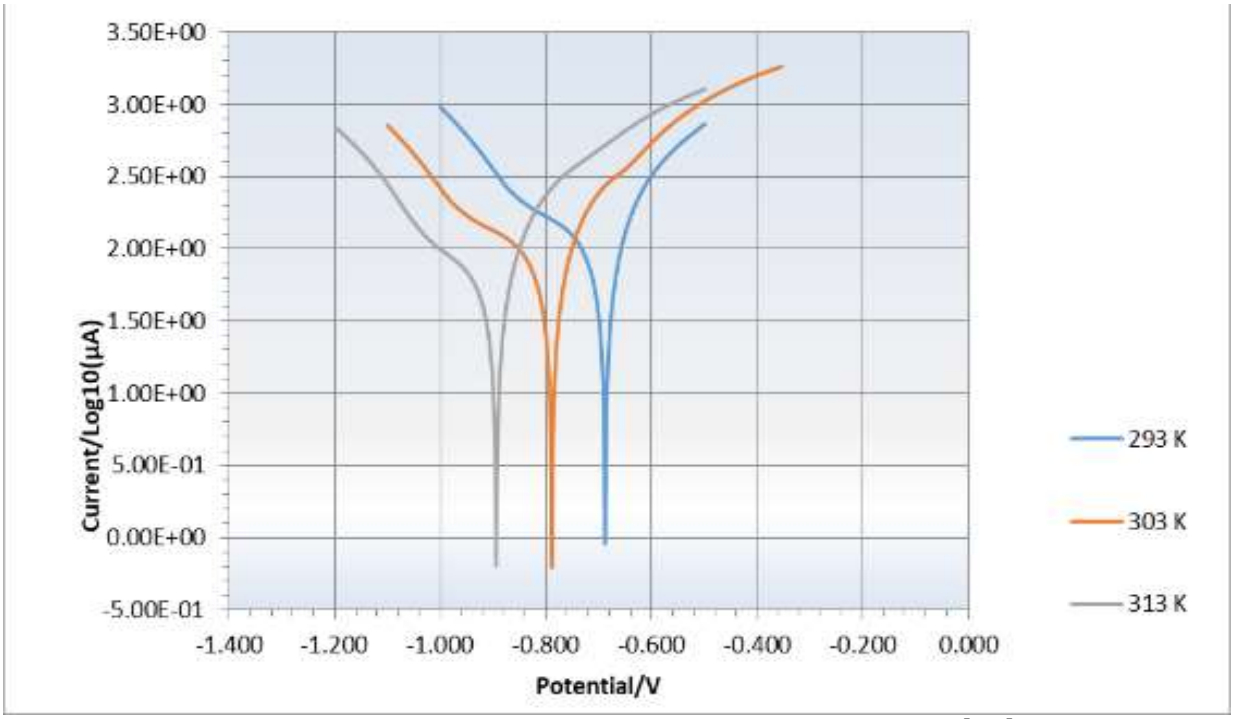


Figure 5. Polarization curves of anti-corrosion for compound [N4].

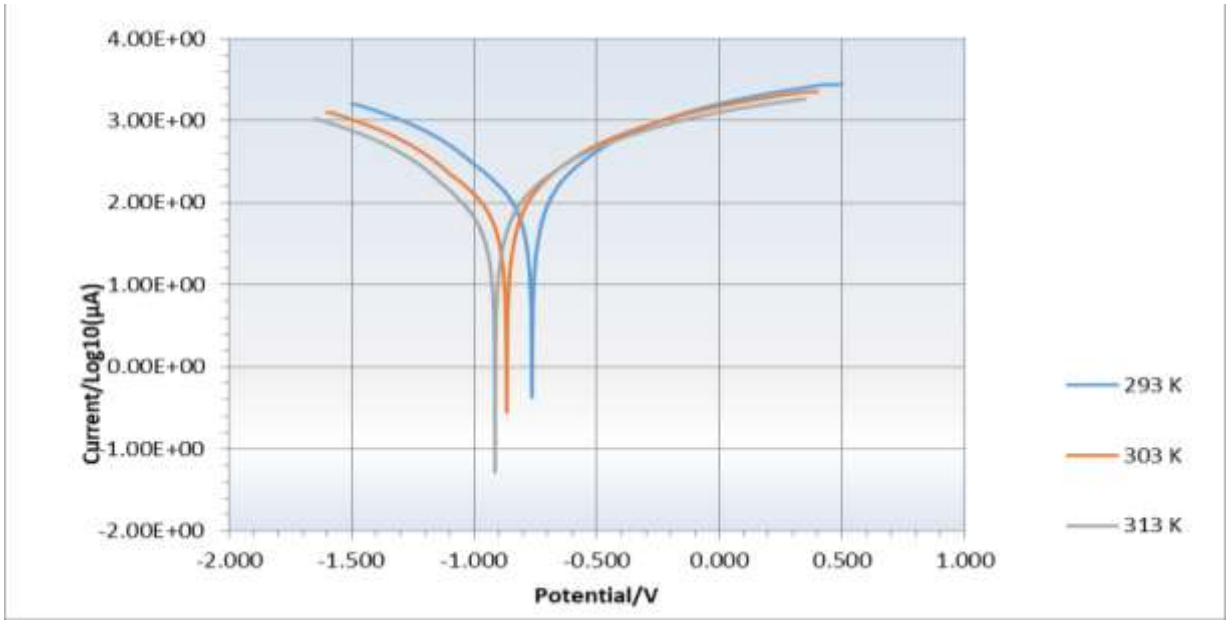


Figure 6. Polarization curves of anti-corrosion for compound [N5]

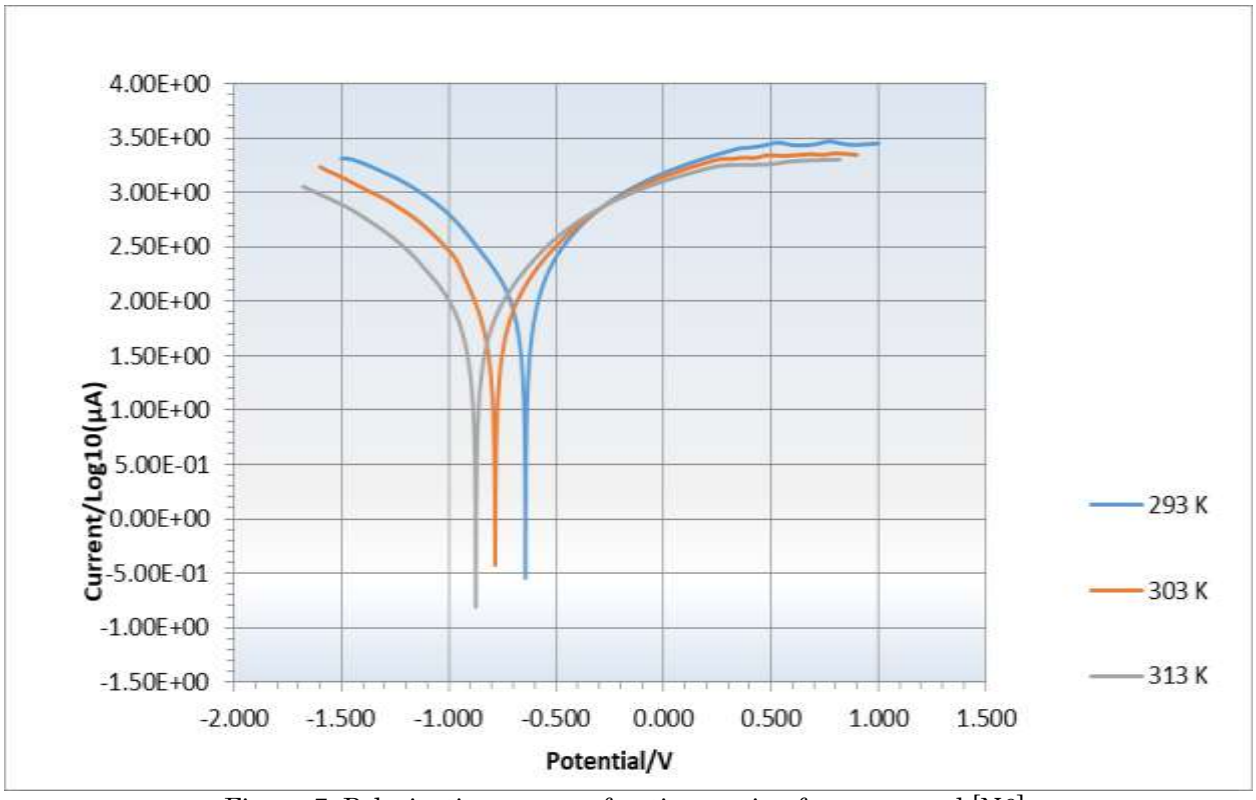


Figure 7. Polarization curves of anti-corrosion for compound [N6].

4. Conclusion

The bis-Schiff base compounds were stable at room temperature and colored because they had a chromophore represented by an imine group (C=N). These compounds were synthesized with good yield due to the presence of an electron-donating group in the aromatic structure. The end of the reaction and the purity of the synthesized compounds were indicated using TLC, and the structure of the compounds was observed using spectroscopic techniques, such as mass spectroscopy, proton and carbon nuclear magnetic spectroscopy, and Fourier transform infrared spectroscopy. All these compounds exhibited good corrosion-inhibiting properties by forming a protective layer between carbon steel 45 and the corrosive substance, thereby protecting the metal. The high % IE (inhibition efficiency) was 96% and low IE% was 87 % for all synthesized compounds at different temperatures (293, 303 and 313 °C) especially compound [N2] which was considered the best inhibitor.

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Conflicts of Interest: The authors declare no conflict of interest.

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