

New Complexes with Triazole Schiff Bases Derivative Ligand Containing Aspirin: Synthesis, Structure, Conductivity and Magnetic Susceptibility

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ABSTRACT--- Complexes of Cu(II), Ni(II) and Co(II) of a new triazole Schiff-base of two different ligand, namely (E)-2-(4-((2-hydroxybenzylidene)amino)4H-1,2,4-triazol-3-yl)phenyl acetate and (E)-2-(4-((2-hydroxy-4-methoxybenzylidene)amino)-4H-1,2,4-triazol-3-yl)phenyl acetate were prepared and characterized by elemental analysis, ^1H , ^{13}C -NMR, FTIR and UV-Visible spectroscopic data. The complex is found to be an ionic in nature by conductivity measurements and have been found to possess 1:2(M:2L) stoichiometry. The magnetic moment values of 1.540–1.7295 B.M. for Cu(II) complexes without doubt recommend octahedral geometry with one unpaired electron for Cu(II) complexes. The magnetic moment values of 3.863–4.039 B.M. for Co(II) complexes that recommend high spin octahedral geometry with three unpaired electron for Co(II) complexes. The magnetic moment value of 2.351 B.M. for Ni(II) complex was matches to two unpaired electrons and octahedral geometry. Considerable variations in biological activity with structure and types are observed for these complexes.

Keywords--- Aspirin, Electrochemistry, Magnetic susceptibility, Triazole, Complexes

1. INTRODUCTION

Metal complexes of Schiff base have played a central role in the development of coordination chemistry [1]. Various Schiff base complexes have been widely studied because they have antimicrobial, anticancer, analgesic, anti-inflammatory, antifertility, and herbicidal applications [2, 3]. Chelating ligands containing N, N, and O donor atoms show broad biological activity and are of special interest because of the ways in which they are bonded to the metal ions [4]. It is known that existence of metal ions bonded to biologically active compounds may enhance their activities [5–7]. Even though many Schiff bases using salicylaldehyde and substituted salicylaldehyde and amines had been studied [8–11] as ligands, no work had been done with salicylaldehyde and aminotriazole as the basic nucleus of Schiff bases. Schiff base metal complexes had been a widely studied subject to their industrial and biological applications [12]. The synthesis, physicochemical characterization, and biological activity of 2-aminobenzimidazole complexes with different metal ions have been reported [13]. An efficient synthesis of Schiff bases containing triazole moiety catalyzed by transition metal nitrates had been done [14]. The synthesis and antimicrobial activity of Cu(II), Co(II), and Ni(II) complexes with 2-aminotriazole were available [15]. The present work aimed to synthesis and characterization of three Schiff base complexes derived from triazole of aspirin with salicylaldehyde and o-vanillin.

2. EXPERIMENTAL

Melting point apparatus and were uncorrected, Elemental analysis (CHN) were recorded in EA300 Euro-Vector in University of Tehran-Iran. FT-IR Spectra were recorded on Shimadzu FT-IR 8400 Fourier Transformer infrared as KBr disk. Ultraviolet spectra were recorded in spectro scan 80 in the wave length 200–800 nm. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker spectropin ultra shield magnets 400 MHz instrument using tetramethyl silane (TMS) as an internal standard and DMSO-d₆ as a solvent in university of Tabriz-Iran. Thin layer chromatography were performed on pre-coated sheets with 0.25 mm layer of Silica Gel GF254 of the Merck Company.

Synthesis of Compounds:

1- Synthesis of Triazole

Synthesis of Triazole equimolar amounts of thiocarbonylhydrazide and appropriate carboxylic acids (10 mmol of each) were mixed and heated at 180 °C for 45 min. Boiling water (20 ml) was added to the solid and the mixture was kept at room temperature for 24 h. The precipitate was filtered and recrystallized from ethanol.

2- *Synthesis of Schiff Base.*

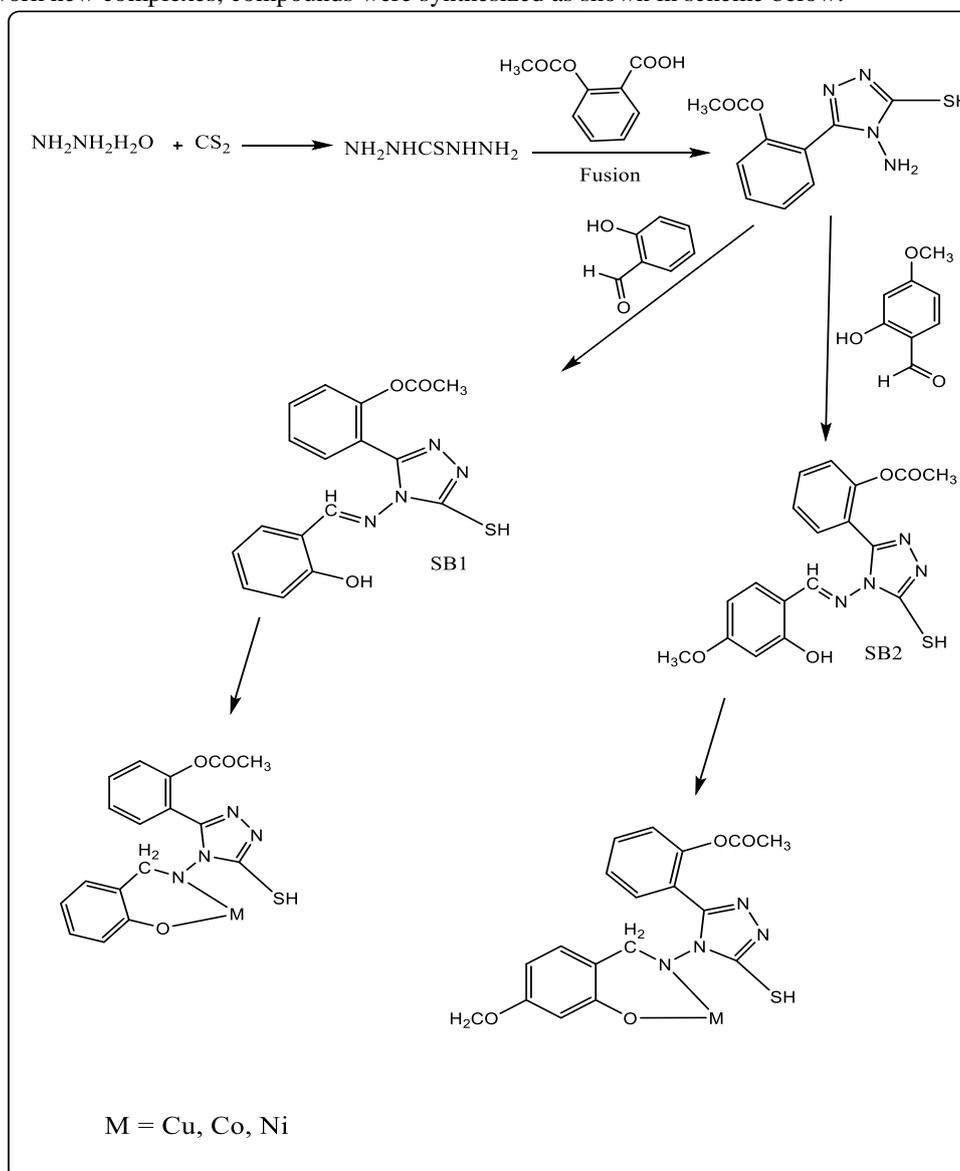
The Schiff base was prepared by mixing equimolar amounts (0.001mole) of amino triazole and the aromatic aldehyde in ethanol. The mixture was refluxed for about 4 hours. Concentration of the solution was done to reduce it to one-half of its original volume when deep yellow crystals were formed in the reaction mixture, which were filtered, washed thoroughly with the same solvent, and recrystallized with acetone. The yellow Schiff base product obtained (m.p. 138 & 125°C) produced in 65&75% respectively yield was subjected to analysis [16,17].

3- *Synthesis of complexes*

0.02Mole solution of SB and 0.02Mole metal salt solution were prepared separately in pure ethanol and mixed. Colour of the resulting solution was yellow green. The pH of the solution was raised to 7.5 by adding 10N NaOH solution. This solution was refluxed for 4 hours and kept for four days when pale green colored product was obtained. This product was then filtered, washed with the same solvent, dried over fused CaCl₂, weighed, and subjected to analysis [18].

3. RESULTS AND DISCUSSION

In the present work new complexes, compounds were synthesized as shown in scheme below:



Scheme(1): Synthesis of compounds and complexes.

The majority of the reactions between carbon disulfide and N-nucleophiles involve addition of carbon disulfide to N–H bonds. The products of these reactions, dithiocarbamate salts, can be transformed into dithiocarbamic acids and esters, which find application in the synthesis of a wide range of organosulfur compounds[19-21].

Elemental Analysis

The structure of the synthesized substituent chalcones and some heterocyclic compounds were confirmed by their elemental analysis. Found and calculated data, the calculated values of carbon, hydrogen, nitrogen and sulphur elements were situated within the range, which confirmed the validity of the suggested structure of the synthesized compounds.

Table 1. The elemental analysis of compounds.

Complex	Found			Calculated		
	%C	%H	%N	%C	%H	%N
Cu(C ₁₇ H ₁₃ N ₄ O ₃ S)Cl ₂	41.68	2.60	11.30	41.86	2.69	11.49
Co(C ₁₇ H ₁₃ N ₄ O ₃ S)Cl ₂	42.21	2.68	11.49	42.26	2.71	11.59
Ni(C ₁₇ H ₁₃ N ₄ O ₃ S)Cl ₂	42.16	2.71	11.55	42.28	2.71	11.60
Cu(C ₁₈ H ₁₅ N ₄ O ₄ S)Cl ₂	41.70	2.88	10.78	41.75	2.92	10.82
Co(C ₁₈ H ₁₅ N ₄ O ₄ S)Cl ₂	42.00	2.90	10.88	42.12	2.95	10.92

F.T.I.R. Spectral Study.

The comparative analysis of the IR spectra of the complex and of the free ligand (Table 2) revealed that the absorption band characteristic of the stretching vibrations of –C=N– (azomethane group) is shifted towards higher wave number from 1612 cm⁻¹ in the spectrum of the ligand to 1695 cm⁻¹ in the complex [8, 21]. This indicates the coordination of the nitrogen of this group with the metal [22]. A broadband appearing at ~3566 cm⁻¹ assigned to O–H in the Schiff base was no longer found in the spectra of the investigated metal complex [23] and, instead, appearance of a new band at 1272 cm⁻¹ due to (C–O) stretching vibrations indicated deprotonation and coordination of the hydroxyl oxygen to the metal [24]. In addition, the appearance of new band at 335-cm⁻¹ showing the involvement of M–Cl bond in complex formation, supporting the IR spectral study. Further conclusive evidence of coordination of the Schiff base with the metal ion was shown by the appearance of low frequency new band at 648 cm⁻¹ due to metal-oxygen (M–O) vibrations [25, 26] and was observed in the spectra of the thus confirming participation of the oxygen atom in the coordination. Further coordination of metal to azomethane nitrogen is confirmed by a new band at 508 cm⁻¹ with the involvement of the imidazole ring nitrogen in the coordination with metal ion [27] due to (M–N).

Table 2. IR Spectral data of the synthesized compounds.

Sy.of Comp.	ν HC=N cm ⁻¹	ν C-O cm ⁻¹	ν C=C cm ⁻¹	ν C=O cm ⁻¹	ν Specific bands cm ⁻¹
SB ₁	1595cm ⁻¹	1296 cm ⁻¹ 1261 cm ⁻¹	1485 cm ⁻¹	1665 cm ⁻¹	3066 cm ⁻¹ 3107 cm ⁻¹ C=C-H Ar
SB ₁ Ni	1610cm ⁻¹	1246 cm ⁻¹ 1296 cm ⁻¹	1483 cm ⁻¹	1660 cm ⁻¹	3234 cm ⁻¹ OH N-Ni 569, O-Ni 462
SB ₁ Co	1610cm ⁻¹	1246 cm ⁻¹ 1296 cm ⁻¹	1246 1296	1658 cm ⁻¹	Co-O 532 Co-N468
SB ₁ Cu	1604cm ⁻¹	1205 cm ⁻¹ 1246 cm ⁻¹	1444 cm ⁻¹	1666 cm ⁻¹	Cu-O 569 Cu-N460
SB ₂	1610cm ⁻¹	1296 cm ⁻¹ 1247 cm ⁻¹	1477 cm ⁻¹	1658	3059 cm ⁻¹ 3008 cm ⁻¹ C=C-H Ar 3240 OH
SB ₂ Co	1606cm ⁻¹	1251 cm ⁻¹ 1294 cm ⁻¹	1465 cm ⁻¹	1658	3257 OH, O-Co 578 N-Co 466
SB ₂ Cu	1612cm ⁻¹	1247 cm ⁻¹ 1211 cm ⁻¹	1481 cm ⁻¹	1658	3240 OH O-Cu532 N-Cu 460

¹H NMR Spectra.

¹H NMR spectrum of the ligand (SB₁) shows a signal at 10.1 ppm as singlet due to phenolic–OH [28]. And 10.2 ppm for the complex SB₂, the absence of any signal due to phenolic proton (–OH) in this region indicates the involvement of phenolic oxygen in coordination via deprotonation [29], thus further supporting the IR spectral observations. In the ligand, the signal at 9.63 ppm as singlet due to proton of azomethane group [30] shows an up field shifting of 1.18 ppm and appears at 10.81 ppm in the complex indicating the coordination of azomethane nitrogen with metal [31]. The signals for the ligand were obtained at 6.8–7.7 ppm [32] due to protons of aromatic rings (8H, Ar–H) which is shifted to higher field in the complexes due to shielding and appears as singlet and multiplet.

Table 3. ¹H NMR Spectra data of synthetic compounds.

Comp.	Chemical Shift ¹ H NMR δ (ppm)					
	O-H	S-H	Ar-H	DMSO	N-H	Specific
SB ₁	10.1	13.6	6.8-7.7	2.5	-	CH ₃ 2.0
SB ₂	10.2	13.6	6.8-7.7	2.5	-	O-CH ₃ 3.8 CH ₃ 2.3

Table 4. ¹³C NMR Spectra data of synthetic compounds.

Comp.	Chemical Shift ¹³ C NMR δ (ppm)					
	C=O	C-S-H	Ar-C	DMSO	N=C	Specific
SB ₁	161	172	116.6-160.4	39.52	148	161 C-OH
SB ₂	172	192	112.9-160	39.52	148	O-CH ₃ 56.07 C-CH ₃ 10.8

Mass Spectra.

The mass spectrum of A, B, C, D and E shows a molecular ion peak at m/z 487 and 517 due to [Cu(L)]⁺; 476 and 513 due to [Co(L)]⁺; 515 due to [Ni(L)]⁺ which suggests the monomeric nature of the complex. The intense peak at m/z 410.1 corresponds to loss of ligand molecule A⁺, that is, Cu(C₁₁H₁₀Cl₂CuN₄O₂S)⁺ from indicating molecular mass of the ligand. Other peaks of appreciable intensity observed at m/z values of 241 due to loss of (Cl)⁺ and other peaks appear in 223, 92, and 41 due to losing Cl, C₆H₇N and C₄H₄. The intense peak at m/z 517 corresponds to loss of ligand molecule B⁺, that is, (Cu(C₁₈H₁₅N₄O₄S)Cl₂)⁺ from indicating molecular mass of the ligand. Other peaks of appreciable intensity observed at m/z values of 368, 120, 92 and 41. The intense peak at m/z 410.1 corresponds to loss of ligand molecule C⁺, that is, Co(C₁₇H₁₃N₄O₃S)Cl₂ from indicating molecular mass of the ligand. Other peaks of appreciable intensity observed at m/z values of 368 due to loss of (Cl)⁺ and other peaks appear in 115, 92, and 69. The intense peak at m/z 410.1 corresponds to loss of ligand molecule D⁺, that is, Co(C₁₈H₁₅N₄O₄S)Cl₂ from indicating molecular mass of the ligand. Other peaks of appreciable intensity observed at m/z values of 341 due to loss of (Cl)⁺ and other peaks appear in 120, 92, and 41. The intense peak at m/z 410.1 corresponds to loss of ligand molecule E⁺, that is, Ni(C₁₇H₁₃N₄O₃S)Cl₂ from indicating molecular mass of the ligand. Other peaks of appreciable intensity observed at m/z values of 298 due to loss of (Cl)⁺ and other peaks appear in 120, 92, and 41. The relative intensities of these peaks give an assessment of the stabilities of the various fragments. Such type of fragmentation patterns has been reported by many workers [33–35].

Conductance, Magnetic Susceptibility Measurements and Biological activity.

The values of the molar conductance of metal complexes were calculated at room temperature taking DMSO as a solvent and the results were shown in Table (6). The complexes were found to be ionic in nature by conductivity measurements and have been found to possess 1:2 (M:2L) stoichiometry. The molar conductance values are commonly used to estimate the electrolytic or non-electrolytic nature of the metal complexes. The magnetic moment values of 1.540–1.7295 B.M. for Cu(II) complexes without doubt recommend octahedral geometry with one unpaired electron for Cu(II) complexes. The magnetic moment values of 3.863–4.039 B.M. for Co(II) complexes that recommend high spin octahedral geometry with three unpaired electrons for Co(II) complexes. The magnetic moment value of 2.351 B.M. for Ni(II) complex matches to two unpaired electrons and octahedral geometry [36–39].

The metal complexes show higher anti-bacterial and antifungal activity in comparison to free ligands. Also, metal complexes show higher antifungal activity against the *Aspergillus* species. The complexes show a significant increase in % inhibition zone for SB₁Cu (15) to SB₁Co (22). Similarly, the complexes SB₁Ni, SB₂Cu and SB₂Co show a significant increase in percent of inhibition zone. Co(II) complexes show the best anti-bacterial and antifungal activity, followed by Ni(II) complexes. SB₁Cu(II) complexes show the least activity as shown in Table (6). This inhibition behavior against bacteria and fungi might be due to azomethine (-HC=N-) linkage and metal atoms present in these compounds.

Table 5. magnetic susceptibility measurements and Conductance data of synthetic compounds.

Com.	Correc. Factor $D \times 10^{-4}$	Mass magnetic Suscepti $X_g \times 10^{-4}$	Molar magnetic Suscepti X_m	Atomic magnetic Suscepti X_A	Effective magnetic moment $\mu_{\text{eff(B.M)}}$	M.Wt (gm/mol)	Molar Conductivity Ohm^{-1}
SB ₁ Cu	-3.793	0.013	6.34×10^{-4}	1.013×10^{-3}	1.540	487.82	35.6
SB ₂ Cu	-3.957	0.017	8.8×10^{-4}	1.276×10^{-3}	1.729	517.85	50.6
SB ₁ Co	-3.793	0.124	5.991×10^{-3}	6.370×10^{-3}	3.863	483.21	44.6
SB ₂ Co	-3.957	0.128	6.569×10^{-3}	6.965×10^{-3}	4.039	513.24	62.6
SB ₁ Ni	-3.794	0.041	1.980×10^{-3}	2.359×10^{-3}	2.351	482.97	36.5

Table 6. Antibacterial and antifungal activite

No.	.Com	Inhibition zone			
		S.arureus	E.coli	Condida.	Asperillus
1	SB ₁ Cu	15	11	18	30
2	SB ₁ Co	22	18	18	15
3	SB ₁ Ni	20	18	20	25
4	SB ₂ Cu	22	15	15	15
5	SB ₂ Co	22	18	20	25

4. CONCLUSION

The study of the reaction between the transition metal and the derived Schiff base indicates its high stability. This encourages the synthesis and careful investigation of the nature of bonding between the Schiff base and the transition metal cation of important biological role, using physicochemical method of analyses. It is clear from above discussion that the fragmentation pattern and spectral studies of the complex confirm and illustrate the proposed geometry obtained by elemental analysis, IR, ¹HNMR, and mass spectra.

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