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Mixed Schiff Bases Chelates: Synthesis and Spectroscopic Investigation

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ABSTRACT: Co(II), Ni(II), Cu(II), Zn(II) and Fe(III) mixed Schiff base chelates with Schiff bases were investigated, firstly, Schiff base formed from the condensation of 2-hydroxyacetophenone and tyrosine as primary ligand (L^1) and the second one is derived from the reaction of 4-dimethylaminobenzaldehyde and 2-aminophenol as secondary ligand (L^2) were synthesized. The Schiff bases and their mixed Schiff base chelates under investigation were characterized by using several physiochemical techniques; in terms, CHN elemental analyses, molar conductivity, infrared, proton nuclear resonance, electronic, mass and electron paramagnetic resonance spectra. The CHN elemental analysis results exhibited the formation of the Schiff bases and their chelates in 1:1:1[M:L¹:L²] ratio. The molar conductivity values displayed that Co(II), Ni(II), Cu(II) and Zn(II) mixed Schiff base chelates found to be electrolytes due to the presence of sodium ions outside the frame of the coordination units of the chelates, however, except the iron(III) mixed Schiff base chelate found to be neutral, confirming the presence of non-electrolytic nature. The infrared spectral data of the mixed Schiff base chelates revealed the chelation behavior between the metal ions and the Schiff bases through the active groups presented in the Schiff bases such as nitrogen atom of the azomethine and oxygen atom of the hydroxyl group. The signals which obtained from the ¹H-NMR spectra of the Schiff bases and their Zn(II) chelate confirmed the chelate formation between the Zn(II) ion and the Schiff bases. The mass spectral fragmentations of the Schiff bases were reported and confirming the formation of the Schiff bases, as well as also the mass spectral fragmentation of the iron(III) mixed Schiff base chelate was done. The electron paramagnetic resonance spectral data confirmed the results which obtained from the electronic study. Meanwhile, the UV spectral results of the Schiff bases exhibited the $\pi \rightarrow \pi^*$ (Phenyl reing) and $n \rightarrow \pi^*$ (C=N and –OH). All transitions and the electronic spectral data of the mixed Schiff base chelates suggested the geometrical structure of octahedral structures.

Keywords: Metal ions; Schiff bases; Mixed Schiff base chelates and Physical techniques.

INTRODUCTION: The coordination compounds including chelates, complexes and mixed ligand complexes have been studied till date for their major important applications¹. Mixed ligand complexes have a good character which is not present in the ordinary complexes or chelates². Two series of Cu(II) and Ni(II) complexes with two Schiff bases; 2-((2,4-dimethylphenylimino)methyl)-6-methoxy-4-

nitrophenol and 2-((3,4-diflourophenylimino) methyl)-6-methoxy-4-nitrophenol have been prepared and characterized by using several physical techniques. A square planar structure was suggested for all the complexes³. The Schiff bases and their complexes were tested for both pathogenic bacteria and fungi using dilution method. For mixed ligand chelates formation of Co(II), Ni(II), Cu(II) and Zn(II) ions with Schiff base as main ligand and amino acid as coligand have been synthesized and investigated by some physical tools. All the obtained data exhibited an octahedral structure for all the chelates⁴.

The main aim of this study is to prepare and characterize the geometrical structures of the Schiff bases formed from the refluxing of 2-hydroxyacetophenone and tyrosine as primary ligand (L^1) . In addition, the other Schiff base derived from the condensation of 4dimethylaminobenzaldehyde with 2-aminophenol as secondary ligand (L^2) and their mixed Schiff base chelates with Co(II), Ni(II), Cu(II), Zn(II) and Fe(III) ions.

MATERIALS AND METHODS: All chemicals used in this study are of pure grade (BDH or Aldrich) 2-hydroxyacetophenone, tyrosine, include; 4dimethylaminobenzaldehyde, 2-aminophenol, DMSO, DMF, NaOH, methanol, ethanol, CoCl₂·6H₂O, NiCl₂·6H₂O, CuCl₂·2H₂O, ZnCl₂, FeCl₃·6H₂O and distilled water. The Schiff bases and their mixed Schiff base chelates were investigated by using several techniques; such as, CHN elemental analysis using 2400-CHN elemental analyzer. The molar conductivity of the mixed Schiff base chelates was measured in DMF solvent using digital conductivity meter CMD-650, at chemistry department, Benghazi University, Benghazi, Libya. The infrared spectra were carried out applying KBr disc technique using IFS-25 DPUS/IR spectrometer. Proton nuclear magnetic resonance spectra of the Schiff bases and their Zn(II) mixed Schiff base chelate were recorded on Varian Gemini 200-200MHz spectrometer using TMS as internal standard and d⁶-DMSO as a solvent. The electronic spectra were measured in DMF solvent by using a Perkin-Elmer lambda-4 β spectrophotometer. The mass spectra also carried out by using Shimadzu QP-2010 Plus. The electron paramagnetic resonance spectrum was recorded by using EMX ESR spectrometer (Bruker) 1998Y. All previous tools and analysis were done at micro-analytical centre, Assiut University, Assiut, Egypt.

Synthesis of the Schiff base (L¹): This Schiff base was synthesized as follows: NaOH (0.01mol; 0.4g) was dissolved in methanol (30 cm³) then the solution of amino acid (tyrosine) (0.01mol; 1.81g) was mixed together. The mixture was stirred magnetically at room temperature. When the mixture becomes homogeneous, 2-hydroxyacetophenone (1.36 g) was added. After 2 minutes, the mixture was evaporated upto 20% of its original volume and then 1cm³ of acetic acid was added immediately. After 2 hours with stirring, yellow crystals formed. The crystals were filtered, washed, dried and recrystallized from hot methanol to give pure crystals in excellent yield of 80.30%.

Synthesis of Schiff base (L²): This Schiff base was synthesized by dissolving 4-dimethylaminobenzaldehyde (0 .01mol; 1.49g) and same moles of 2aminophenol in ethanolic solution (30cm³). The obtained mixture was heated and stirred magnetically for 2 hours. Yellow crystals were formed then filtered, washed, dried and recrystallized from hot methanol to give pure crystals in excellent yield of 85.40%.

Synthesis of mixed Schiff base chelates: The mixed Schiff base chelates of Schiff bases under investigation with metal ions of Co(II), Ni(II), Cu(II), Zn(II) and Fe(III) were synthesized by adding 0.01 mol; 3.29g of the Schiff base (L^1) and the same ratio of the Schiff base (L^2) in 25cm³ of the absolute ethanol to 0.01mole; 2.38, 2.37, 1.70, 1.36 and 2.70g) of the desired metal salts, respectively. Few drops of sodium hydroxide solution were added slowly to adjust the pH value at 8 until the chelates separated, and then the mixtures were stirred and refluxed for 3 hours. The formed precipitates were filtered and washed several time with hot ethanol until the filtrates become clear. The resulted mixed Schiff base chelates were dried in desiccator under calcium chloride.

RESULTS AND DISCUSSION: The refluxing of 2hydroxyacetophenone with tyrosine (L^1) and 4dimethylaminobenzaldehyde (L^2) with 2-aminophenol gives two Schiff bases presented in Figure 1:





Figure 1: Structure of Schiff bases L¹ and L².

Physical properties, CHN elemental analyses and molar conductivity: The Schiff bases and their mixed Schiff base chelates were obtained in powder form with high melting points (Table 1). The Schiff bases are soluble in DMF, DMSO solvents or in alcohols, but, the mixed Schiff base chelates are not soluble in alcohols, but they are partially soluble in DMSO and DMF solvents. All Schiff bases and their mixed Schiff base chelates are stable in air. The CHN elemental analysis data of the Schiff bases and their mixed Schiff base chelates agree with the proposed compositions. This means that the Schiff bases were formed (Figure-1) and their mixed Schiff base chelates were also formed in 1:1:1[M:L¹:L²] ratio. The molar conductivity values reveal the presence of electrolytic nature for the divalent [Co(II), Ni(II), Cu(II) and Zn(II)] mixed Schiff base chelates, because attributed to the existence of sodium ion outside the coordination sphere, whereas, the molar conductivity value for Fe(III) mixed Schiff base chelate shows nonelectrolytic nature, this means that there is no negative or positive ions outside the coordination sphere chelates⁵.

Schiff bases/Chelates	M. wt.	Colour	Yield %	C% Calc.	C% Exp.	H% Calc.	H% Exp.	N% Calc.	N% Exp.	^*
$C_{18}H_{21}NO_{5}$. (L ¹)	331.00	Pale yellow	90.32	68.22	69.0 1	5.69	6.20	4.68	5.45	-
C ₁₅ H ₁₆ N ₂ O (L ²)	240.00	Yellow	85.40	75.00	75.8 8	6.67	7.59	11.67	12.6 3	-
$Na[Co(L^{1})(L^{2})(H_{2}O)].2H_{2}O$	703.90	Pale brown	75.51	56.25	57.2 2	5.68	6.21	5.96	7.10	137
$Na[Ni(L^1)(L^2)(H_2O)].H_2O$	685.70	Light yellow	86.88	57.75	57.0 3	5.54	5.91	6.12	6.65	156
$Na[Cu(L^1)(L^2)(H_2O)].H_2O$	690.50	Dark brown	70.34	57.35	58.03	5.50	6.10	6.08	7.59	149
$Na[Zn(L^1)(L^2)(H_2O)].H_2O$	692.40	Pale green	75.34	57.19	58.15	5.48	6.22	6.06	7.20	155
[[Fe(L ¹)(L ²)(H ₂ O)].2H ₂ O	679.00	Light brown	80.12	58.32	59.0 5	5. 89	5.53	6.18	7.27	37

 Table 1: Some physical properties and CHN elemental analyses of Schiff bases and their mixed Schiff base chelates.

^*= molar conductivity

Table 2: Infrared spectral data (cm⁻¹) and electronic spectral data (nm, cm⁻¹) ofmixed Schiff base chelates.

Schiff bases/ Chelates	v OH	U	v RC=N	U	U	v	nm (cm ⁻¹)	
	$(\mathbf{H}_2\mathbf{O})$	(COO)	L1, (L2)	С-ОН	M-O	M-N	(()	
$C_{18}H_{21}NO_{51}(L^1)$	3422	1591	1609	1158	-	-	258 (38759)	
-1621- (- 5) (-)	0.22	1071	100)	1100			270 (37037)	
$C_{15}H_{16}N_2O(L^2)$	3330	-	1587	1235	-	-	374 (26738)	
$Na[Co(L^{1})(L^{2})(H_{2}O)].2H_{2}O$	3419	1450	1591,(1514)	1042	649	493	733 (13643	
$Na[Ni(L^1)(L^2)(H_2O)].H_2O$	3416	1452	1608,(1590)	1042	648	528	734 (13624)	
$\mathbf{N}_{0}[\mathbf{C}_{1}(\mathbf{I}^{1})(\mathbf{I}^{2})(\mathbf{H}^{2})] \mathbf{H}^{2}$	2221	1456	1580 (1517)	1041	648	401	626 (15974	
	5521	1450	1389,(1317)	1041	040	491	733 (13643	
$N_{2}[7_{2}(I^{1})(I^{2})(H^{2})]H^{2}$	3/15	1454	1611,(1589)	1041	648	495	626 (15974	
	5415						733 (13643	
							734 (13624)	
$[[Fe(L^{1})(L^{2})(H_{2}O)].2H_{2}O$	3403	1453	1612,(1588)	1040	647	490	761 (13141)	
							775 (12903)	

 $R = CH_3$ for L1, (R = H for L2)

Table 3: ¹HNMR spectral data of the Schiff bases and their Zn(II) mixed Schiff base chelate.

Schiff bases/ mixed Schiff base chelate	∂СООН	δОН	δDMSO	HC=N	$\delta C_6 H_4$	δCH ₃
$C_{18}H_{21}NO_{5}$. (L ¹)	3.50	7.06	2.00	-	6.60-7.00	2.51
$C_{15}H_{16}N_2O(L^2)$	-	7.83	2.50	8.63	6.80-7.16	3.02
$Na[Zn(L^1)(L^2)(H_2O)].H_2O$	3.35	-	2.51	-	6.98-6.67	3.19

Infrared spectra: The infrared spectra for the Schiff bases and their mixed Schiff base chelates under investigation were taken in the 4000–400 cm⁻¹ range and helped to indicate regions of absorption due to the respective vibrations. The infrared spectral band assignments for the Schiff bases and their mixed Schiff base chelates are given in (Table 2). In this investigation, the interaction between the metal ions [Co(II), Ni(II), Cu(II), Zn(II) and Fe(III)] with Schiff bases (L¹) and (L²) gives stoichiometric ratio of 1:1:1[M:L¹:L²] mixed Schiff base chelates. The infrared spectra of the synthesized mixed Schiff base chelates exhibit bands in the range of 3321-3422 cm⁻¹ due

to the existence of water molecules as hydrated and coordinated ⁶. Meanwhile, the same spectra exhibit bands of the azomethine group in the range of 1514-1612cm⁻¹, the shifting of this group compared with the position of azomethine groups in the free Schiff bases (1609cm⁻¹ for L¹ and 1587cm⁻¹ for L²) suggesting the formation of chelates through nitrogen atom of the azomethine group^{7, 8}. The position of -OH group of Schiff bases L¹ and L² is overlapped with the position of water molecules in the spectra of the mixed Schiff base chelates indicating the involvement of this group in chelation with the metal ions through oxygen atom. The infrared spectral results of the mixed Schiff base

chelates display band 1450 to 1456 cm⁻¹ attributed to COO⁻ group⁹ and the appearance of new bands in the range of 490-528 and 647-649 cm⁻¹ due to v(M-N) and v(M-O) vibrations, supporting the participation of the azomethine and hydroxyl groups through nitrogen and oxygen atoms in coordination with metal ions^{10,11}.

¹**H-NMR spectrum:** The ¹H-NMR spectrum of Na[Zn^{II}L¹L² (H₂O)].H₂O chelate shows the absence of the OH/COOH protons indicating their replacement by the metal ion during complexation formation. The azomethine proton signal of L² is shifted slightly to lower field during the chelation with Zn(II) ion confirming the bonding of this group through azomethine nitrogen. The integrated intensities of all the signals (Table 3) agree well with the (Figures 2 to 4).



Figure 2: ¹H-NMR spectrum of Schiff base (L¹).



Figure 3: ¹H-NMR spectrum of Schiff base (L²).



Figure 4: ¹H-NMR spectrum of Zn(II) mixed Schiff base chelate.

Electronic spectra: The UV spectra of Schiff base compounds were recorded at room temperature in DMF $(10^{-3}M)$ and their data were recorded in (Table 2). The spectrum of Schiff base L^1 exhibits two bands at 258 nm (38759cm⁻¹) and 270 nm (37037cm⁻¹) corresponding to $\pi \rightarrow \pi^*$ (Phenyl ring) and $n \rightarrow \pi^*$ transitions, respectively. The electronic spectrum of Schiff base L^2 exhibits a high intensity band appeared at 374nm (26738cm⁻¹) confirming the presence of $n \rightarrow \pi^*$ transition. The electronic absorption spectrum of Co(II) chelate exhibits a band at 733 nm (13643 cm⁻¹) due to ${}^{4}T_{1}g(F) \rightarrow {}^{4}A_{2}g(F)$ transition. The intensity of the band supports the existence of an octahedral geometry around Co(II) ion^{12,13}. Whereas, the electronic spectrum of the Ni(II) chelate of the type Na[Ni (L1)(L2)(H₂O)] H₂O reveals a band at 734 nm (13624 cm⁻¹) which can be assigned to ${}^{3}A_{2}g(F) \rightarrow$ ${}^{3}T_{2}g(F)$ transition, the nature of the band confirms the existence of an octahedral structure for this chelate ^{14,15}. Also, the spectrum of the Cu(II) chelate shows two bands (Table-2) attributed to ${}^{2}Eg \rightarrow {}^{2}T_{2}g$ transition confirming the presence of an octahedral geometry around Cu(II) ion ¹⁶. The electronic spectral data of the Zn(II) chelate show two bands at 626nm (15974 cm^{-1}) and 733nm (13643 cm^{-1}) due to charge transfer transition and an octahedral geometry was suggested for this chelate¹⁷. The electronic spectrum of the Fe(III) mixed Schiff base chelate with L^1 and L^2 shows three bands at 733 $nm(13643cm^{-1})$, 761nm (13141 cm⁻¹) and 775 nm (12903cm⁻¹) assigned to d-d transition, indicating presence of an octahedral structure around Fe(III) ion¹⁸.

Mass spectra: The mass spectral fragmentations of the Schiff base (L1) are shown in (Table 4, Figure 5 and Scheme 1). The peak at $m/e^+ = 329$ analogous to $C_{18}H_{19}NO_5$. Meanwhile, another peak at m/e⁺=315 is equal to the formula of $C_{17}H_{17}NO_5$. The structure $C_{16}H_{13}NO_3$ is analogous to peak at m/e⁺=267. The same spectrum shows a peak at $m/e^+=207$ corresponding to the formula of $C_{14}H_{10}NO$, the peak of $m/e^+ = 107$ is attributing to C_7H_7O . The last peak at $m/e^+ = 77$ is attributing to $C_6H_5^+$. The mass spectral fragmentations of the Schiff base (L^2) , are shown in (Table 4, Figure 6 and Scheme 2). The base peak at $m/e^+ = 240$ is corresponding to the original molecular weight of the Schiff base (L^2) , whereas, the peak at $m/e^+=223$ due to the loss of carbon atom, five hydrogen atoms. The structure of C₁₂H₈O is analogous to the peak at $m/e^+ = 168$. The final peak appeared at $m/e^+= 65$ is assigned to C_5H_5 . The mass spectrum fragmentation of the Ni(II) mixed Schiff base chelate (Figure 7 and Scheme 3) shows a molecular ion peak at $m/e^+=$ 658 which is corresponding to [NiC₃₃H₃₃N₃O₇]. Meanwhile, the peak at $m/e^+=599$ is analogous to [NiC₃₀H₂₄N₂O₇]. The peak at m/e⁺ = 535 due to $[NiC_{29}H_{19}N_1O_6]$. Whereas, the peak at m/e⁺= 107 is assigned to $[C_8H_{10}]$. The final peak appeared at $m/e^+ = 88$ analogues to C_6H_{10} .



Figure 5: Mass spectrum of Schiff base (L¹).



Figure 6: Mass spectrum of Schiff base (L²).



Figure 7: Mass spectrum of Ni(II) mixed Schiff base chelate.

Table 4: Mass spectral fragmentations of the Schiff bases and their Ni(II) mixed Schiff base chelate.

Compound	Fragmented ion	m/e+ values
C ₁₈ H ₂₁ NO ₅ . (L ¹)	$\begin{array}{c} C_{18}H_{21}NO_5\\ C_{18}H_{19}NO_5\\ C_{17}H_{17}NO_5\\ C_{16}H_{13}NO_3\\ C_{14}H_{10}NO\\ C_7H_7O\\ C_6H_5+ \end{array}$	331 329 315 267 207 107 77
C ₁₅ H ₁₆ N ₂ O (L ²)	$\begin{array}{c} C_{15}H_{16}N_2O\\ C_{14}H_{11}N_2O\\ C_{12}H_8O\\ C_5H_5 \end{array}$	240 223 168 65
Na[Ni(L ¹)(L ²)(H ₂ O)].H ₂ O	NiC ₃₃ H ₃₃ N ₃ O ₇ NiC ₃₀ H ₂₄ N ₂ O ₇ NiC ₂₉ H ₁₉ N ₁ O ₆ C ₆ H ₈	658 599 535 107 80



Scheme 1: Mass spectral fragmentations of Schiff base (L¹).



Scheme 2: Mass spectral fragmentations of Schiff base (L²).



Scheme 3: Mass spectral fragmentations of the Ni(II)L1L2 chelate.

Electron paramagnetic resonance spectra: The electron paramagnetic resonance spectra of the chelates under investigation (Figure-8) show g_{eff} values in the range of 2.063-2.130 (Table-5). The deviation of these values from the ideal value of the free electron (2.0023) confirming the presence of partial ionic character of the covalent bond between the metal ions and the free Schiff bases (L¹ and L²). These values support

the existence of an octahedral geometry around the metal ions $^{19}\!\!$

Table 5: E.P.R. spectral data of the mixed Schiff

base chelates.					
Complexes	g _{eff} value				
$Na[Co(L^{1})(L^{2})(H_{2}O)].2H_{2}O$	2.130				
$Na[Ni(L^{1})(L^{2})(H_{2}O)].H_{2}O$	2.063				
$Na[Cu(L^{1})(L^{2})(H_{2}O)].H_{2}O$	2.109				
$[Fe(L^{1})(L^{2})(H_{2}O)].2H_{2}O$	2.110				



Figure 8: E.P.R spectra of the mixed Schiff base chelates $L^1 \& L^2$.

CONCLUSION: All the obtained physiochemical results display the existence of an octahedral structure for all the synthesized mixed Schiff base chelates, the general structure is shown below:



M=Co(II),Ni(II),Cu(II)and Zn(II) ions, n= 1 for Ni(II), Cu(II) and Zn(II) chelates n= 2 for Co(II) chelate



M= Fe(III) ion

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