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Preparation and characterization of Ni (II) transition metal mixed ligand complexes

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Abstract
A new mixed ligand complex has been prepared by reaction of chloride salts of Mn (II) with Shiff base 2- ((E)-4- (E)-phenal-diazenyl) phenol [PDIMP= (HL_1)] and (Z)-2- ((P-Tolylimino) methyl) phenol [TIMP= (HL_2)]. Both the ligand/complexes were characterized on the basis of microanalysis, melting point, conductivity, solubitity, determinating of the metal contact M%, IR, and UV/VIS spectral studies. The schiff bases acts as a monobasic bidentate ligands, coordinating through deprotonated phenolic oxygen and azomethine nitrogen atoms. The complexes are non-electrolytic in DMSO. From the analytical and spectral data the stoichiometry of these complexe has been found to be [M (L_1) (L_2) (H_2O)$_2$]. Where M=Ni (II). The electronic absorption spectra and magnetic susceptibility measurements suggest the square planer geometry for the Ni (II) ion. The in vitro antibacterial activities of the complex was test using number of bacteria species such as Escherichia coli, Salmonella typhi, Pseudomonas aeruginosa, Streptocuccus pyogenes, and Basillus subtilis. The Schiff bases acts as a monobasic bidentate ligands, coordinated phenolic oxygen and azomethine nitrogen atoms.

Keywords: synthesis, characterization, Ni (II), preparation, schiff base, mixed ligand complex, antimicrobial studies

Introduction
The synthesis of Schiff base ligand and their metal complex has been extensively studies because of their interesting biological activities [1-4]. The condensation product of an amine and a ketone or aldehyde with general formula of R2C=NR are well known Schiff base compound. They have been reported to be useful in medicine, catalyst, as antibiotics, antifungal and have applications in various field [7-10].
In the present work, We have prepared two Schiff base compounds, (PDIMP) and (TIMP). The structure of these compound was studies by elements analysis, IR, andUV-Vis spectra. Then, their Ni (II) mixed ligand complex was prepared and the spectral, magnetic and molar conductances properties of these complex was studies.

Experimental Section
All the chemicals and solvents used were of analar (AR) grade and were used with-out further purification. They are 4-aminoazobenzene was prepared as reported procedure. Melting point of compound was determined using griffin melting points apperatus. The melting point were determined by the open capillary method and are reported in ^{0}C by using a stuart melting point s m p 10.

Preparation of Schiff base ligand
A) Preparation of Schiff base ligand (HL_1)
The Schiff base ligand (HL) was prepared as discrived by Raman et al. 2004 (15). This was done by the hot solution (50^{0}C) of salisaldehyde (2. 442g, 20mmol0 was mixed with hot solution (50^{0}c) of p-aminoazobenzene (3. 944g, 20mmol) in (200ml) ethanol and two drops of glacial acetic acid. The mixture was then refluxed for 1hour. The product obtained was filtered, washed in distilled was filtered, washed in distilled water, dried, and preserved in a disiccator contained $CaCl_2$.

B) Preparation of Schiff base ligand (HL_2)
An ethanolic 100ml warm solution (40^{0}C) of salisaldehyde (4. 2884g, 40mmol) and warm solution (40^{0}C) p-toluidine (4. 286g, 40mmol) was added drop wise to 100ml of the metal (II) salts. The reaction mixture was refluxed for 1hour and formed shine yellow crystals product was separated by filterated, purified by recrystallized from hot ethanol, and then dried in a vacuum over $CaCl_2$.

Preparation of mixed ligand complex
The mixed ligand complex was prepared by adding the appropriate amount of the metal salrt (1mmol), name $NiCl_2$. $4H_2O$ dissolved in (4ml) of distilled water slowly with constant stirring to a solution of (0. 3013g, 1mmol). From 2-((E)-4- (E)-phenyl-di azenyl) phenylimino) methyl phenol (HL_1) and (0. 02113g, 1mmol) from (Z)-2- ((p-tolyiminio) methyl) phenol (HL_2) in (50ml) ethanol. The resulting mixture was refluxes for 1hour. The metal complex thus formed were filtered out, washed with (1:3) aqueous alcohols mixture followed by recycrystalised from hot ethanol, and dried over anhydrous $CaCl_2$.

Results and Discussion
The analytical data along with some physical properties are summarized in Table1. Thje Schiff bases ligands (PDIMP) and (TIMP) were red and shine yellow crystals respectively, but the complex of these ligands very in colour depended of metal ion. All the complexes are an stable and have sharp melting points (118-119^{0}C) except the ligands (L) which melted above 300^{0}C). Elemental analysis gave satisfactory

agreement betbeen observed and calculated values for Carbon, hydrogen, nitrogen and metal ions compositions in the complexes. The results show that the compounds formed in ratio (1:1:1) of M (II) ion to (PDIMP) to (TIMP), corresponding to the molecular formula proposed for the all mixed ligand complexes.

Table 1: Physical charecteristics and analytical data for the Schiff base ligand and the metal (II) complex

Compound	Molecular formula	Colour	Melting Point (ºC)	Yield (%)
[Ni (L_1) (L_2) (H_2O)$_2$]	$C_{33}H_{30}N_4O_4Ni$	red	144	69

Table 2: The microanalysis and metal estimation data of the Schiff base ligands and their metal complexes

Compound	Molecular Formula	Microanalysis, % found (Calc)			
		C	H	N	M
[Ni (L_1) (L_2) (H_2O)$_2$]	$C_{33}H_{30}N_4O_4Ni$	63. 46 (63. 29)	2. 27 (3. 44)	9. 61 (9. 82)	5. 77 (5. 68)

Microanalysis

The microanalysis of the ligands and their metal (II) complexes are presented in Table2. The revealed that the % C, H and N are in good agreement with the proposed structures. From the data obtained, it appears that the compound analyzed as [Mn (L_1) (L_2) (H_2O)$_2$] indicating a 1:2 mole ratio (M:L).

IR Spectra

The selected vibrational frequencies exhibited by the (PDIMP) and (TIMP) shiff base ligangs and thiir mixed ligand complexes are presented in Table 3. The infrared spectra of the free ligands (HL_1) and (HL_2) show a broad weak intensity band centered at around 3424 and 3402cm^{-1}respectively, due to the intra moleculer hydrogen bond O---H__N=C$^{(21)}$. This band disappeared in the spectra of the complex indicating probably the coordinating througg phenolic oxygen moiety. Avery weak intensity bands had been observed at 3020-3082 and 2980-2914cm^{-1} in the spectra of both Schiff bases and metal complex are due to N (C-N) aromatic and aliphatic respectively. Another strong bands appeared at 1620-1618cm^{-1} in the spectra of (HL_1) and (HL_2) ligands respectivilyy, due to (C=N) group. IR spectra of free azo-azomethine ligand (PDIMP) shows a medium absorption band at 1480cm-1 due to v (-N=N-) group. The new bands at 543-501cm^{-1} and 461-426cm^{-1} have been assigned to v (M-O) and v (M-N), respectively. Abroad medium intensity band at 3444cm^{-1} suggests the presence of coordinated water in Ni (II). A broad medium intensity band 3430cm^{-1}suggests the presence of coordinated water in Ni (II) complexes.

Electronic Spectra

The UV-VIS spectral of Ni (II) complex exhibited three transitions bands at the 13333, 16000 and 21277 cm^{-1} due to the $^3A_{2g}\rightarrow{}^3T_{2g}$ (F), $^3A_{2g}\rightarrow{}^3T_{1g}$ (F) and $^3A_{2g}\rightarrow{}^3T_{1g}$ (P) transitions respectively with in octahedral spatial configuration. This complex exhibit magnetic moment of (3. 21BM), which can be a normal value for octahedral high-spin Ni (II) cpmplexes.

Moler conductance studies

The conductance of the complexes in DMSO (10^{3-}M) at room temperature are shown in Table4. The molar conductance values lying in the range (14. 35-16. 88) scm^{2}mol^{-1} confirmed the monionic behavior of the complexe.

Conclusion

The spectral, molar conductance and magnetic studies of the prepared mixed ligands complex of (PDIMP) and (TIMP) Schiff base ligands reveals that Ni (II) metal complex is having octahedral geometry.

Table 3: Charecteristic IR frequencies (in cm-1) of Schiff bases and their mixed ligand complexes

Compound	v (O-H)	v (C-H) Ar	v (C-H) Alph	v (C=N)	v (N=N)	v (M-O)	V (M-N)
[Ni (L_1) (L_2) (H_2O)$_2$]	3430w	3020w	2919w	1597s	1471w	520w	426w

Table 4: Electronic spectra, conduvtivity and magnetic moment of mixed ligand complexes

Metal complexes	Absorption bands (cm^{-1})	Transition	Conductivity S. cm^2. mol^{-1}	μ_{eff} (B. M)
[Ni (L_1) (L_2) (H_2O)$_2$]	13333 16000 21277	$^3A_{2g}$ (F)$\rightarrow{}^3T_{2g}$ (F)V_1 $^3A_{2g}$ (F)$\rightarrow{}^3T_{1g}$ (F)V_2 $^3A_{2g}$ (F)$\rightarrow{}^3T_{1g}$ (P)V_3	16. 88	3. 21

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