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Synthesis and characterization of vanillin schiff base with Ni (II) complexes

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Abstract
A new complex of vanillin Schiff base of was used to mononuclear Ni (II) co-ordination compound. The complexes were synthesized and characterized by elemental analysis, magnetic properties, color, UV, Visible and IR spectral are discussed below. The purity and composition of the Schiff base and the metal (II) complexes were established by elemental analysis which suggests a metal: ligand ratio of 1:2. The IR spectra revealed that the complexes coordinated though azomethine nitrogen and methoxy oxygen of the ligands. futher conclusive evidence of the metal ions was shown by the appearance of new bands due to v(M-N)and v(M-O) in the metal complexes. Bbased on the electronic spectral transition, an octahedral structure has been assigned. The ligand its metal complexes were screened for antibacterial activity against Klebsiella pneumonia, Bacillus cereus and Pseudomonas aeruginosa and fungicidal activity against Aspergillus nigar, Candida albicans and Candida Kefyr.

Keywords: Synthesis, characterization, Ni (II) Schiff base, transition metal complexes, vanillin.

Introduction
Schiff base have a chelating structure and are in demand because they are straight for word to prepare and are moderate electron donor with easily-tunable eletronic and steric effect. The synthesis and application of Schiff base and their co-ordination compounds have been highly considered in inorganic and bioinorganic fields, since their structural properties similar to some of the biological system (1-4). Many Schiff base and their complexes have been widely studied because of their industrial, antifungal antibacterial, anticancer and herbicidal applications 8-13.Schiff base compounds (-CH=N-) are usually formed by the condensation of a primary amines with ketones or aldehydes. The crosslinking agents can also be derived from metal complexes with N, S or O ligands.

Experimental:
All the chemical and solvents used were of A R grade and were used without further purification.

Preparation of Schiff base legend (L):
The Schiff base ligand (L) was prepared by the following general method. This was done by the condensation of 20ml of vanillin (0.03g, 10mmol) with 2-aminophenol (0.022g, 10mmol) in ethanol (1:1molar ratio). The mixture was stirred for 4h.

Preparation of the Schiff base metal (II) complexes:
An ethanolic (10ml) solution of Schiff base ligand (20mmol, 0.055g) was to 10ml of the metal (II) salts (10mmol, 0.024g of $NiCl_2.6H_2O$ in boiling ethanol (78.3^0C).The reactions took place in 1:2 mole ratio of metal (II): L. The reaction mixture was refluxed for 4h on a water bath and the volume of the solution was reduced to half of the initial volume.

Antimicrobial test:
The in vitro antimicrobial properties of the schiff base ligands and metal complexes were assayed with the following becteria: Klebsiella pneumoniae, Bacillus Cereus and Pseudomonas aeruginosa and fungal species Aspergillus nigar, Candida Kefyr and Candida Albicans using disc diffusion method .Agar diffusion assay was carried out to evaluate the antimicrbial activite of some synthesized compounds. The plaates were incubated at 37^0C for 24hours during which activity was evidenced by the presence of a zone of inhibition surrounding the well and antibacterial and antifungal activity was expressed as mean of diameter of inhibition zones(mm) produced by the synthesised compounds when compared to controls.

Results and discussion:

The analytical data along with some physical properties are summarized in Table1. The Schiff base ligand (L) on interaction with Ni(II) formed complexes with moderate yields (18-24%).All the complexes are air stable and have sharp melting points (140-180^{0}C) except the ligand(L) which melted above 350^{0}C. The sharp melting points indicates that the complexes are probably pure.

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

Compound	Molecular formula	Molar mass	Colour	Melting Point (ºC)	Yield
Ni(L-L)X_2	$Ni(C_{14}H_{13}NO_3)_2$	545.21	Brown	177	20

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

Compound	Molecular Formula	Molar Mass	Microanalysis, % found (calc)			
			C	H	N	M
Ni(L-L)X_2	$Ni(C_{14}H_{13}NO_3)_2$	545.21	40.49 (61.68)	4.05 (4.13)	5.79 (5.14)	08.65 (09.81)

Micro analysis:

The micro analysis of the ligands and their metal (II) complexes are presented in Table2.The results revealed that the %C,H and N are in good agreement with the proposed structures From the data obtained, it appears that the compounds analyzed as (M(L-L)X_2) indicating a 1:2 mole ratio(M:L).

IR specta of Schiff base ligand (L):

The selected vibrational frequencies for the Schiff base ligand and its metal complexes are presented in Table 3.very strong band at 1569CM^{-1} characteristics of the azomethine nitrogen present in the Schiff base ligand (L)[8]. This was shifted to 1624-1638 CM^{-1} in the complexes, which indicates the co-ordination of the metal to the azomethine nitrogen. The metal complexes showed broad band 2600-2800 CM^{-1} which is characterisitic of v (OH).This indicates that the phenolic-OH group does not participate in bond formation with the metals. The infrared spectrum of the Schiff base ligand showed strong band at 1492, which was assigned to v(C-N) stretching. This was shifted to 1432-1596 CM^{-1} region in all the complexes. The spectral bands of the complexes at 1286-1291 were assigned to v(C-O) which did not show considerable shift from the region 1290 CM^{-1} of the ligand. Thus it is suggested that the oxygen atoms of terminal methoxy and hydroxyl group are not co-ordinated to the metal ions v (M-N) and sv(N-O) were observed in the far infrared region. These bands are absent in the spectra of the ligand.

Electronic spectra:

UV-Visible spectra of the Ni (II) complexes were recorded at 200-600nm using methanol as a solvent. The absorption regions band assignment and the proposed geometries of the complexes are given in Table 4. The orgal diagram for d^5 configuration for Mn (II) shows at 22935, 23901 and 2823CM^{-1} assigned for $^6A_{1g} \rightarrow {}^4T_{1g}$, $^6A_{1g} \rightarrow {}^4T_{2g}$, and $^6A_{1g} \rightarrow {}^4A_{2g}$.

Table3: Relevant infrared frequencies (CM^{-1}) of the Schiff base ligands and their metal (II) complexes.

Compound	v (OH) Phenolic	v (C-O)	v (C-N)	v (C=N)	v (O-CH_3)	v (M-N)	v (M-O)
Ni(L-L)X_2	3316sh	1290vs	1492vs	1595vs	3258sh	596m	503m

Table4: Electronic absorption spectral data for the metal complexes.

Compound band	Absorption (cm^{-1})	Assignment	Geometry
Ni(L-L)X_2	22883	$^3A_2g(F) \rightarrow {}^3T_1g(P)$	Octahedral

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