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SYNTHESIS, SPECTROSCOPIC INVESTIGATION AND BIOLOGICAL ACTIVITY OF Ni (II), Zn (II) COMPLEXES DERIVED FROM TETRADENTATE SCHIFF BASE LIGAND

K. Deepa[1] and Rangaswamy Venkatesh [1*]

[1] PG & Research Department of chemistry, Muthurangam Government Arts College (Autonomous) Vellore-2, Tamilnadu, India.

***Corresponding Author Email: venkateshr64@yahoo.in**

ABSTRACT

Two different unsymmetrical chloro substituted nickel (II), zinc (II) complexes were synthesized by the reaction of corresponding precursor with Tetradentate Schiff base ligand. The ligand was characterised by UV-Visible, FT-IR, H^1NMR Spectral studies. The synthesized complexes were characterised by molar conductance, UV- Visible, FT-IR spectral studies. The ligand and its complexes screened for antibacterial activity.

KEY WORDS

Tetra-dentate Schiff base ligand, Nickel (II) complexes, Zinc (II) complexes antibacterial activity.

INTRODUCTION:

Schiff base ligands which are able to form binuclear transition metal (II) complexes have been interest for several years[1-7]. There are only a few reports regarding the synthesis of unsymmetrical Schiff bases deived from equimolar condensation of a diamine and different aldehydes/ketones which is more difficult to obtain. This paper reports on the synthesis and characterization of Nickel (II)complexes using tetradentate Schiff base ligand obtained from 5-chloro-2-hydroxyacetophenone, 5-nitro-o-Phenylenediamine and Salicylaldehyde.

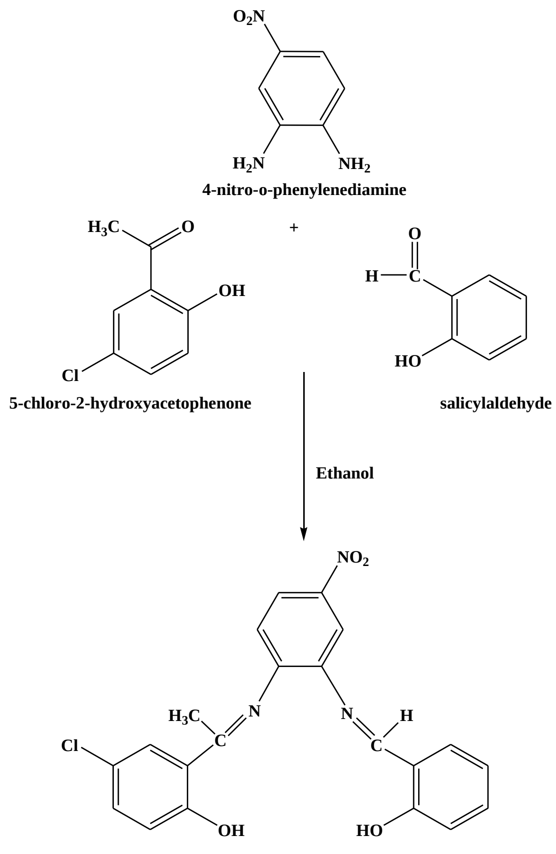
EXPERIMENTAL:

Materials and methods:

All chemicals and solvents used were of AR grade. IR spectra were recorded on a IR- Affinity-I Spectrometer using KBr pellets in the range 4000-400 cm^{-1}. Molar conductance of the ligand and complexes were measured in ethanol (10^{-3}M) solutions using a digital conductivity meter. UV-Visible spectra were recorded using Systronics spectrophotometer operating in the range of 200-800 nm with quartz. Antibacterial activities were done at Micro Labs, Vellore, Tamilnadu, India.

SYNTHESIS OF SCHIFF BASE LIGAND:

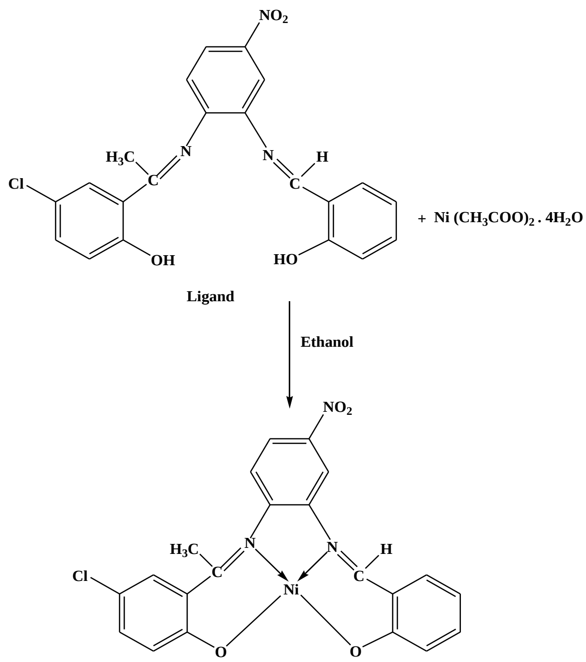
The Schiff base ligand were prepared by initially 5-chloro-2-hydroxyacetophenone (7 mmol) in ethanol was kept under magnetic stirring. To this ethanolic solution of 4-nitro-o-Phenylenediamine (7 mmol) and Salicylaldehyde (7 mmol) was added in the reaction mixture. The mixture was refluxed for 8 hours. The precipitate was filtered and washed with hot water and ethanol. The purified product was kept under oven at 60°C for 2 hours.



Ligand

SYNTHESIS OF NICKEL (II) COMPLEX:

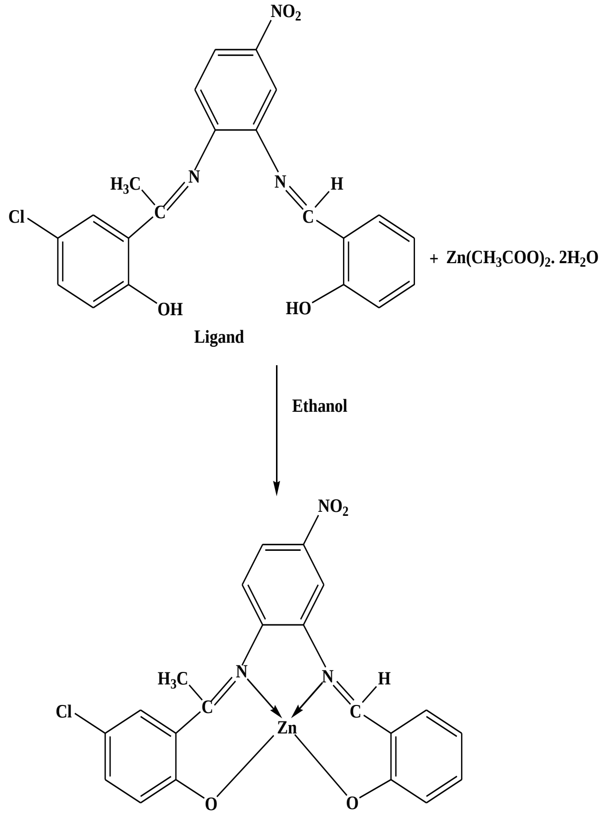
The schiff base ligand (0.5 mmol) dissolved in ethanol was kept under magnetic stirring. To this ethanolic solution of Ni (II) acetate tetrahydrate (0.5 mmol) was added in the reaction mixture. The mixture was refluxed for 8 hours. The precipitate was filtered and washed with hot water and ethanol. The purified product was kept under oven at 60°C for 2 hours.



Complex -1

SYNTHESIS OF ZINC (II) COMPLEX:

The synthesis of Schiff base ligand (0.5 mmol) dissolved in ethanol was kept under magnetic Stirring. Then added to ethanolic solution of Zn (II) acetate dihydrate (0.5 mmol) in the reaction mixture. The mixture was refluxed for 8 hours. The precipitate was filtered and washed with hot water and ethanol. The product was kept under oven at 60°C for 2 hours.



RESULTS AND DISCUSSION:

CONDUCTANCE MEASUREMENTS

The molar conductance of Ligand and metal (II) complexes are listed in the Table-1. The molar conductance values show that the complexes are non-electrolyte in nature[8]. The solvent used in molar conductance is ethanol.

Table 1: Physical properties of metal (II) complexes:

S.NO	Compound	Molecular formula	Molecular weight	Molar conductance ($cm^2\ ohm^{-1}\ mol$)
1	**Ligand**	$C_{21}H_{16}N_3O_4Cl$	409.69	-
2	**Complex-I**	$C_{21}H_{14}N_3O_4ClNi$	466.37	48
3	**Complex-II**	$C_{21}H_{14}N_3O_4ClZn$	473.08	50

UV-VISIBLE SPECTROSCOPIC ANALYSIS

UV-Visible spectra of Ligand and all the complexes were recorded using ethanol is illustrated in Figure: 1-3 and the spectral data are listed in Table-2. The absorption band observed in the region of 248 nm was due to π-π* transition of the benzene ring present in the ligand. The band at 364 nm was due to π-π* transition of the azomethine group (-HC=N) present in the ligand. The band at 420 nm was due to n –π* transition. The band observed in the region of 243-245 nm was due to π-π* transition of the benzene ring present in the complexes. The band at 352-358 nm was due to π-π* transition of the azomethine group (-HC=N) present in the complexes[9]. The band at 417-418 nm was due to n –π* transition. A new characteristic band observed in the region of 566- 568 nm was assigned to d-d transition of the complexes.

Table-2: UV-Visible Spectral data of Ligand and metal (II) complexes:

Compounds	Wavelength (nm)			
	π -π*(nm) (benzene)	π -π*(nm) (-HC=N)	n – π*(nm)	d-d(nm)
Ligand	248	364	420	-
Complex-I	243	352	417	566
Complex-II	245	358	418	568

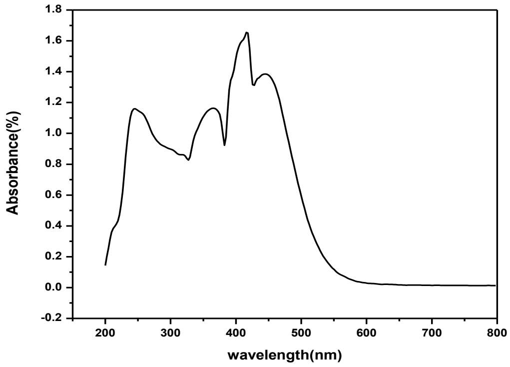


Figure 1: UV-Visible spectrum of Schiff base Ligand

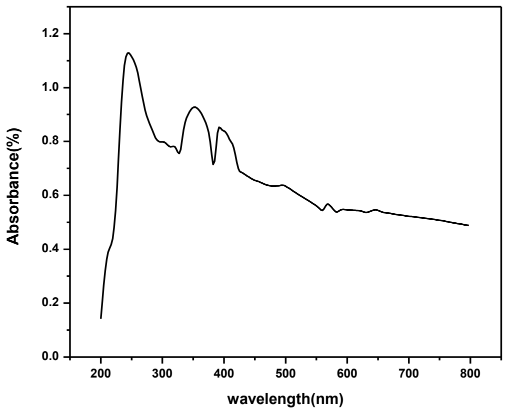


Figure 2: UV-Visible spectrum of Complex –I

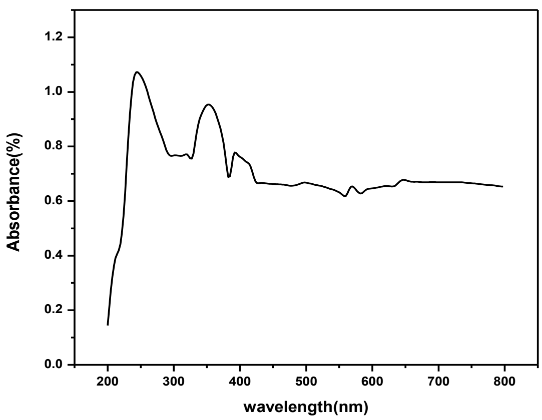


Figure 3: UV-Visible spectrum of Complex –II

FT-IR SPECTROSCOPIC ANALYSIS

The FT-IR spectrum of Ligand and metal (II) complexes have shown in Figure 4-5. The peak at 1620 cm^{-1} was due to the stretching frequency of the azomethine group (-HC=N) in the ligand. The peak appeared at 1332 cm^{-1} were assigned to C-O stretching vibration of the ligand. The peak at 3377 cm^{-1} have been assigned to the -OH stretching vibration of the ligand. The peak at 1616-1618 cm^{-1} was observed due to the stretching frequency of the azomethine group (-HC=N) in the complexes. The peak 1620cm^{-1} was due to azomethine group of the ligand and it was shifted to lower frequency after complexation[10]. The peak appeared at 1253-1259 cm^{-1} were assigned to C-O stretching vibration. The bands presence at 3329-3444 cm^{-1} have been assigned to the -OH stretching vibrations. The peak at 455-462 cm^{-1} was observed due to M-O stretching vibrations. The peak at 545-557 cm^{-1} was observed due to M-N stretching vibrations.

Table-3: FT-IR Spectral data of metal (II) complexes:

Compounds	υ(-C=N) cm^{-1}	υ(-C-O) cm^{-1}	υ(-OH) cm^{-1}	υ(M-O) cm^{-1}	υ(M-N) cm^{-1}
Ligand	1620	1332	3377	_	_
Complex-I	1616	1259	3444	462	557
Complex-II	1618	1253	3329	455	545

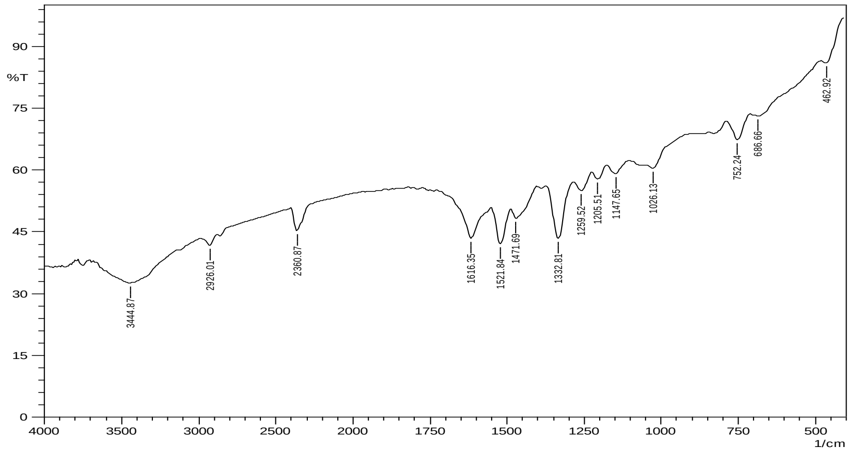


Figure 4: FT-IR Spectrum of Ni(II) Complex

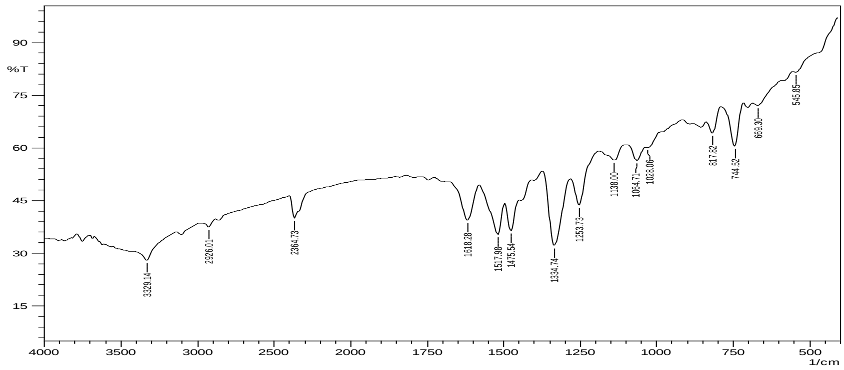


Figure 5: FT-IR Spectrum of Zn (II) Complex

^{1}H NMR SPECTROSCOPIC ANALYSIS OF LIGAND

The ^{1}H NMR spectra of Schiff base ligand are recorded using $CDCl_3$ as solvent, tetramethylsilane (TMS) as internal standard. The ^{1}H- NMR spectral data of Schiff base ligand and its spectrum is shown in Figure 6. The signal appeared at 8.00- 8.72 ppm was due to the protons of the azomethine group (-HC=N).The multiplet signal observed at 7.26 -7.88 ppm was due to the aromatic protons. The signal observed at 6.74-6.96 ppm was due to the aromatic hydrocarbon. The signal observed at 5.40-5.68 ppm is due to protons of aromatic alcohol. The signal observed at 2.63 ppm is due to methyl group.

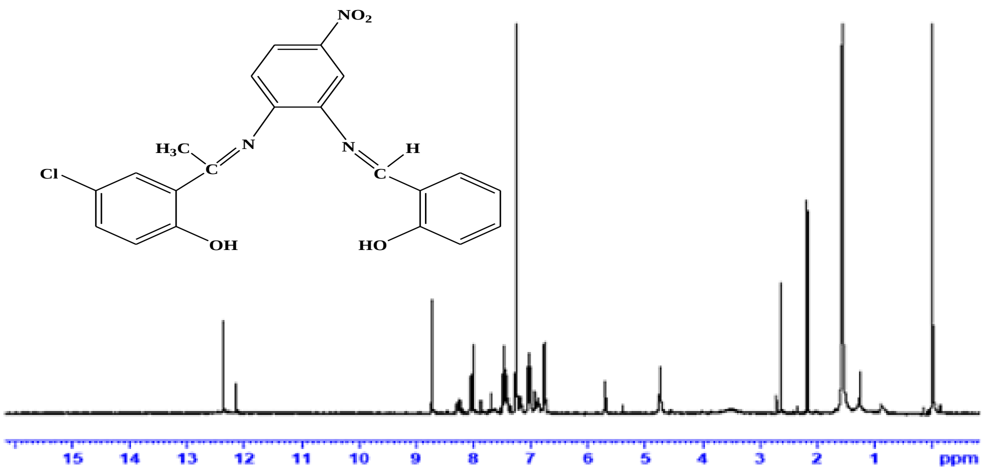


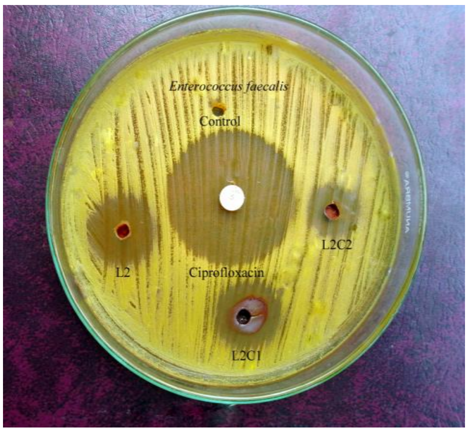
Figure 6: ^{1}H NMR Spectrum of Schiff Base Ligand

BIOLOGICAL ACTIVITY

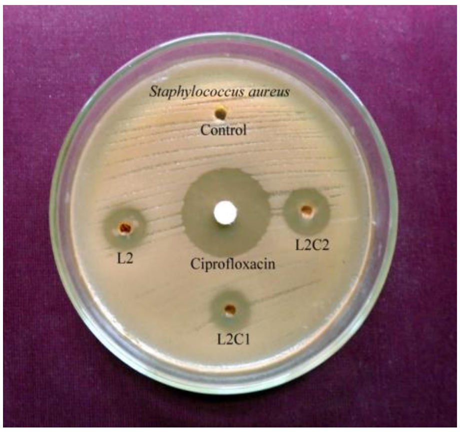
The ligand and the complexes were screened for *in-vitro* antibacterial activity against *Enterococcus faecalis, Staphylococcus aureus, Salmonella typhi, Escherichia coli*. Ciprofloxacin was used as standard drug and DMSO as control. The antibacterial data summarized in table 4:

Table 4: Zone of inhibition of Ligand and Metal (II) complexes:

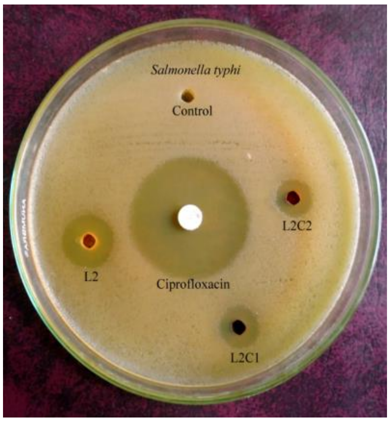
S.No	Microorganisms	L2	L2C1[Ni (II)]	L2C2[Zn (II)]	Ciprofloxacin
1	**Enterococcus faecalis**	15	16	12	30
2	**Staphylococcus aureus**	15	15	17	32
3	**Salmonella typhi**	15	11	10	33
4	**Escherichia coli**	7	5	9	20



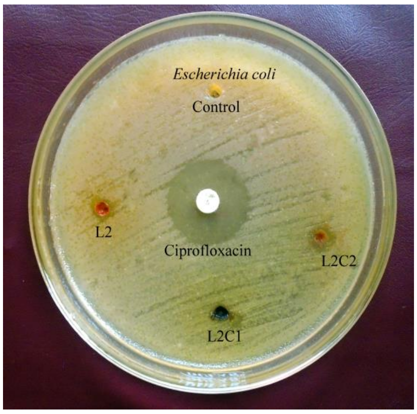
Zone inhibition of against Enterococcus faecalis



Zone inhibition of against Staphylococcus aureus



Zone inhibition of against Salmonella typhi



Zone inhibition of against Escherichia coli

The Schiff base complex L2C1 shows higher zone of inhibition value then other complex against Enterococcus faecalis. The complex L2C2 shows higher zone of inhibition value then other complex against Staphylococcus aureus.

CONCLUSION:

Tetradentate Schiff base ligand were synthesized using 4-nitro-o-phenylenediamine, 5-chloro-2-hydroxyacetophenone and salicylaldehyde. The Synthesized transition metal [Ni(II), Zn(II)] complexes were characterized by using various analytical techniques Such as UV-Visible, FT-IR, Conductivity measurement. The Synthesized transition metal [Ni (II), Zn(II)] complexes were Screened for their antibacterial activity against *Staphylococcus aureus, Enterococcus faecalis, Salmonella typhi, Escherichia coli*. The ligand and complexes were found to good inhibition to all selected micro organisms.

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