WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 7.523
Volume 7, Issue 8, 52-58. Conference Article ISSN 2277– 7105

SPECTROSCOPIC ANALYSIS AND ANTIBACTERIAL ACTIVITY OF SOME MIXED LIGAND COMPLEXES OF Ni(II)

S. Vijayakumar and Rangaswamy Venkatesh*

*P.G. & Research Department of Chemistry, Muthurangam Govt. Arts College,
(Autonomous), Vellore-2.

Article Received on
05 March 2018,
Revised on 25 March 2018,
Accepted on 15 April 2018
DOI: 10.20959/wjpr20188-10932

***Corresponding Author**
Rangaswamy Venkatesh
P.G. & Research
Department of Chemistry,
Muthurangam Govt. Arts
College, (Autonomous),
Vellore-2.

ABSTRACT

Mixed ligand complexes of Ni(II) with 5-methylsalicylaldehyde (HL) as primary ligand and various secondary ligands (HL') like salicylaldehyde, substituted salicylaldehyde and nickel(II)acetate tetrahydrate have been synthesized in 1:2 and 1:1:1 molar ratios. The resulting complexes have been characterized by various spectral studies. From the analytical and spectral data the stoichiometry of these complexes have been found to be [M (L) (L') $(H_2O)_2$]. It has been found that complexes exhibited octahedral geometry. At the same time, above mentioned complexes and ligands were studied for *in-vitro* antibacterial properties and found to be more potent bactericides than parent ligands.

KEYWORDS: Mixed ligand complexes, octahedral geometry, antibacterial properties.

INTRODUCTION

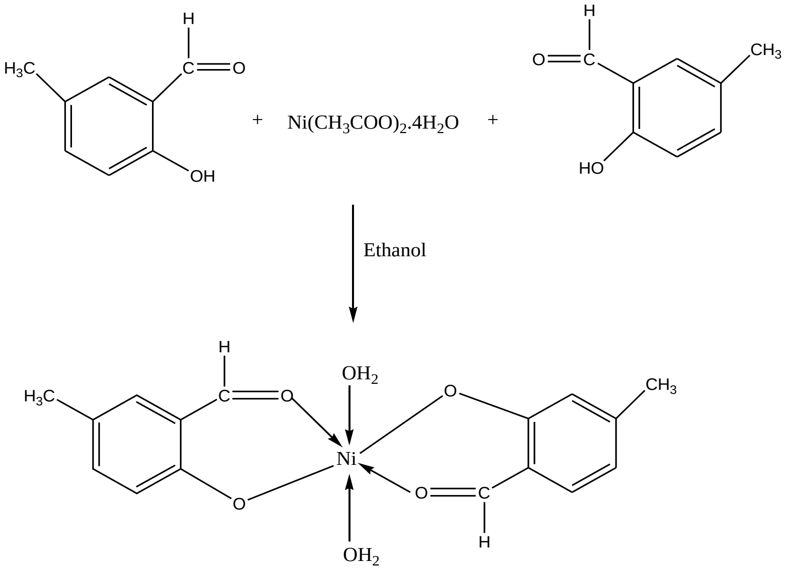
The preparation and study of inorganic compounds containing biologically important ligands is made easier, because certain metal ions are active in many biological processes; species of low molecular weight are, hence, sought that reproduce, as far as possible, the structural properties and the reactivity of naturally occurring complexes of these ions in such processes.[1] Over the last few years, the chemistry of β-diketones and hydroxyaryl aldehydes or ketones containing compounds has become an active area of research. The coordination compounds of transition metal (II) ions with ligand containing oxygen donor atoms have been the subject of greatest interest due to containing chelate groups and provide the extra stability of complexes.[2] we have to synthesized, structurally characterized and determined biological activities of mixed ligand complexes of Ni(II) with 5-methylsalicylaldehyde or ketones or β-diketones like [Ni(5-methylsal)(L')$(H_2O)_2$] and results have been reported.

MATERIALS AND METHODS

Solvents and metals salts were used analytical grade. IR spectra were recorded on IR AFFINITY Spectrometer using KBr pellets in the range 4000–400 cm^{-1}. UV-visible spectra were obtained in Ethanol. Molar conductance of the complexes were measured in DMF (10^{-3} M) solutions using a digital conductivity meter. The molecular weight of the complexes were recorded on LC-MS using Water-Q-T of Premier- HAB213 instrument model with the Electron spray ionisation method. Antibacterial activities were done at MICRO LABS, Vellore, Tamilnadu, India.

Synthesis of mixed ligand complexes of Ni(II)

The Nickel(II) complexes were synthesized using 5-methyl salicylaldehyde (10mmol) and Nickel(II)acetate tetrahydrate(5mmol) in the ratio of 1:2 using ethanol as a solvent and mixture was stirred for about 2 hours. The pH of the solution was raised up to ~ 6.5 to by drop wise addition of 5% aqueous solution of sodium hydroxide with constant stirring. The reaction mixture was stirred for 5-6 hours and then kept in the refrigerator for about 1- 2 days the settled solid was filtered washed with ethanol and dried property under reduced pressure. A similar method was adopted to synthesize mixed ligand complexes of Ni(II) with 5-methylsalicylaldehyde and salicylaldehyde in 1:1:1 molar ratio (Complex –II), 5-methyl salicylaldehyde and 5-nitrosalicylaldehyde in 1:1:1 molar ratio (Complex –III).



Scheme 1: Synthesis of mixed ligand complexes of Ni(II) with 5-methylsalicylaldehyde.

RESULTS AND DISSCUSION

The molar conductance of Ni(II) complexes are listed in the Table–1. The molar conductance values show that the complexes are non-electrolyte in nature. The solvent used in molar conductance is DMF.[3]

Table 1: Analytical data of Ni(II) mixed ligand complexes.

S.No	Compound	Molecular Formula	Molecular weight	Molar conductance ($cm^2\,ohm^{-1}mol$)
1	Complex- I	$C_{16}H_{18}O_6Ni$	366.28	12
2	Complex- II	$C_{15}H_{16}O_6Ni$	350.83	10
3	Complex- III	$C_{15}H_{15}NO_8Ni$	395.84	13

UV–VISIBLE SPECTRA

The electronic spectra of Ni(II) complexes in ethanol is illustrated in Figure: 1 and the spectral data are listed in Table-2. The absorption band observed in the region of 246 – 249 nm was due to π-π* transition of the benzene ring present in the complexes. The band observed in the region of 406– 409 nm was due to n-π* transition of the carbonyl group present in the complexes. A new characteristic band observed in the region of 550 – 552 nm was assigned to d-d transition of the complexes.

Table-2: UV-Visible spectral data of Ni(II) complexes.

Compound	π-π* (nm) (benzene)	n-π* (nm)	d-d (nm)
Complex I	246	409	550
Complex II	248	408	552
Complex III	249	406	550

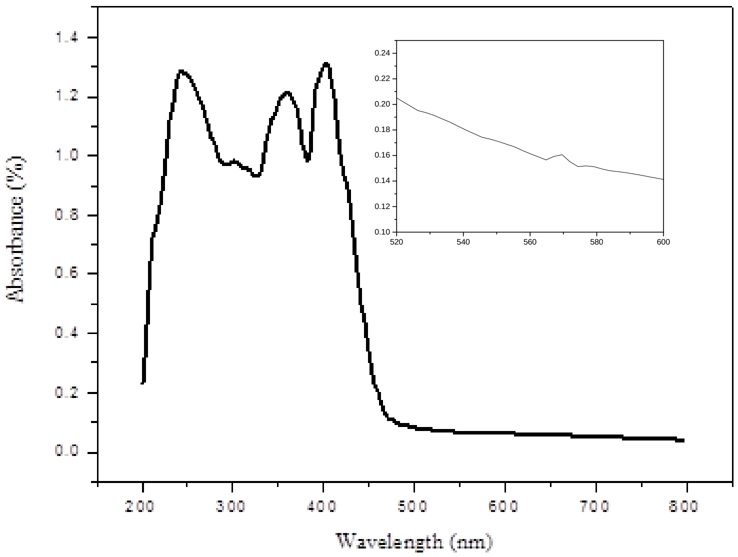


Figure 1: UV – Visible Spectrum of Complex – II

FTIR SPECTRA

The FTIR spectrum of metal(II) complexes have shown in Figure 2. The FTIR spectrum showed a number of absorption bands which were characteristic of the various group present in the metal complexes. Infrared spectra of ligands show a broad band centered at around 2950-3090 cm^{-1} due to free phenolic hydroxyl group in free ligands[4] which disappeared in spectra of their complexes indicating the coordination of ligands in complexes through phenolic oxygen moiety as shown in scheme1. The sharp peak at 1639 - 1651 cm^{-1} was found that C=O stretching vibration. The v(C=O) bands of the complexes are observed in the lower wave number side as compared to free ligands[5,6] confirming the coordination of v(C=O) group to the Nickel atom. The peak appeared at 1249 - 1257 cm^{-1} were assigned to C-O stretching vibration. The bands present at 3398 - 3446 cm^{-1} have been assigned to the O-H stretching vibrations. The peak at 432 - 451 cm^{-1} was observed due to M – O stretching vibrations. The band appeared at 1527–1541 cm^{-1} associated to M–OCO stretching vibaration.

Table-3: FTIR spectral data of Ni(II) complexes.

Compound	υ(-C=O) cm^{-1}	υ(-C-O) cm^{-1}	υ(-OH) cm^{-1}	υ(M-O) cm^{-1}	υ(M-OCO) cm^{-1}
Complex I	1639	1257	3446	451	1527
Complex II	1651	1255	3423	443	1529
Complex III	1645	1249	3398	432	1541

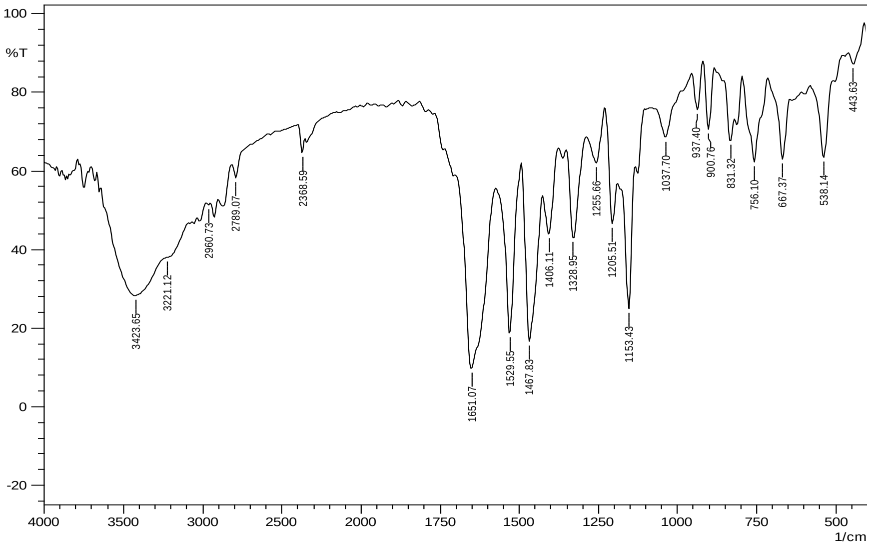
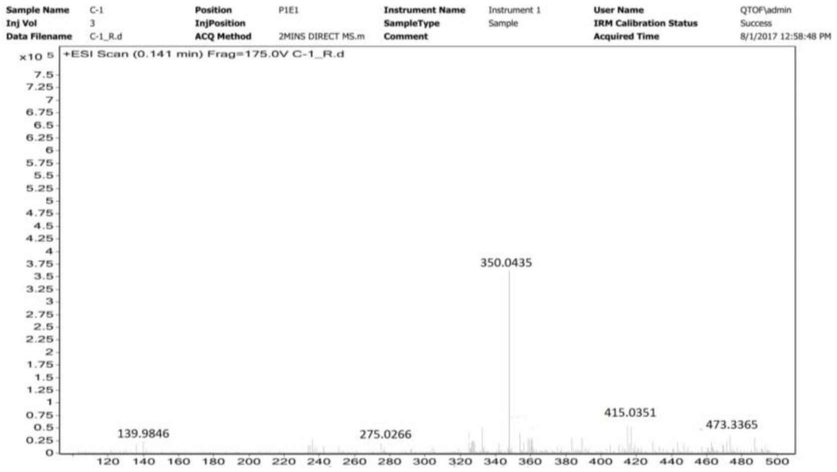


Figure 2: FTIR spectrum of Complex – II.

MASS SPECTRA

The mass spectrum of complex II exhibited peak at m/z 350.0435 This data is in good agreement with the proposed molecular formula of 350.83. The mass spectrum of complex II is given in Figure: 3.



Sample Name	C-1	**Position**	P1E1	**Instrument Name**	Instrument 1	**User Name**	QTOF\admin
Inj Vol	3	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	C-1_R.d	**ACQ Method**	2MINS DIRECT MS.m	**Comment**		**Acquired Time**	8/1/2017 12:58:48 PM

Figure 3: Mass Spectrum of Complex- II.

APPLICATION

IN VITRO ANTI BACTERIAL ACTIVITY

The Ni(II) complexes were screened for *in vitro* antibacterial activity against two gram positive bacteria and two gram negative bacteria. Ciprofloxacin was used as standard drug and DMSO as control. The antibacterial data summarized in Table-4.

Bacterial strain

- Staphylococcus aureus (Gram +ve)
- Streptococcus faecalis (Gram +ve)
- Klebsiella pneumoniae (Gram -ve)
- Escherichia coli (Gram - ve)

Table 4: Zone of inhibition of Ni(II) complexes.

S.NO	MICRO ORGANISMS	Complex I	Complex II	Complex III	CIPROFLOXACIN
1.	Staphylococcus aureus	10	07	15	21
2.	Streptococcus faecalis	11	05	13	19
3.	Klebsiella pneumoniae	05	05	05	15
4.	Escherichia coli	06	05	07	32

It is important to note that the metal chelates exhibit more inhibitory effects towards bacterial strain than the parent ligands. Increased activity of the metal chelates as compared to ligands can be explained on the basis of chelation theory.[7,8] The present series complex III with nitro substitutent exhibits higher activity against *Staphylococcus aureus, Streptococcus* faecalis and Escherichia coli. Complex I exhibits good zone of inhibition againt Staphylococcus aureus and Streptococcus faecalis.

CONCLUSION

In the light of the above discussion, an octahedral geometry for Ni(II) complexes is proposed. All the complexes are non-electrolyte and diamagnetic in nature. In the IR spectra of the complexes shifting of *v*(C=O) to lower wavenumber side supports the chelation of the ligand to the metal atom and also support the presence of coordinated water molecules in the complexes. Mass spectral study further confirms the proposed structure of the complexes. The complexes are biologically active and exhibit enhanced antibacterial activity.

REFERENCES

1. Albertin G, Bordignon E, Orio A Five-coordinate copper (II) complexes. Synthesis and properties of $[Cu(tren)L]^{2+}$ cations. Inorg Chem., 1975; 14: 1411-1413.
2. W. Rehman, F. Saman and I. Ahmad, *Russian. J. Coord. Chem*, 2008; 34(9): 678-682.
3. Geaery W.J., The use of conductivity measurements in organic solvents for the characterization of coordination compounds, *Coord. Chem. Rev.*, 1971; 7: 81-122.
4. A. K. Mapari and K. V. Mangaonkar, Int.J. Chem *Tech* Res., 2011; 3(2): 637-641.
5. M. S. Suresh, V. Prakash, Int. J. Phys. Sci., 2010; 5(9): 1443.

6. M. G. Abd El-Wahed, M.S. Refat, S.M. El-Megharbel, *Bull. Mater Sci*., 2009**;** 32(2): 205.
7. Y. S. Malghe, R.C. Prabhu, R.W. Raut, *Acta Pol. Pharm. Drug Res.*, 2009**;** 66(1): 45.
8. S. B. Kalia, G. Kaushal, M. Kumar, S.S. Cameotra, A. Sharma, M. L. Verma, S.S. Kanwar, Braz J Microbiol, 2009; 40: 916.