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Synthesis, Spectral, Thermal and Antimicrobial Studies of Ni (II) Complexes with Oxygen Donor Ligands: Part-1

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Abstract: ***The mixed ligand complexes of Ni (II) derived from the condensation of 5-nitrosalicylaldehyde (HL_1) as primary ligand and secondary ligands (HL_2) like salicylaldehyde, substituted salicylaldehyde or 2-hydroxyaryl ketones were synthesized in 1:1:1 molar ratios and characterized by elemental analysis,TGA , magnetic measurements, conductometery, IR and UV-visible spectroscopy and amicrobial study. These O-donor ligands are act as bidentate monobasic and coordinating through deprotonated phenolic oxygen and carbonyl oxygen atoms. The presence of the two coordinated water molecules in these complexes was indicated by IR spectra and TGA of the complexes The studies revealed octahedral geometry of synthesized complexes.***

Keywords: Conductometery, TGA,IR Spectra, Mixed ligand complexes, Antimicrobial study.

1.Introduction

The synthesis and study of coordination compounds containing biologically important ligands have been paid much attention in recent years because of their wide applicability in biological, environmental and other system[1].In fact many naturally occurring metals complexes containing two or more different ligands[2] or if the ligand is a single macromolecule having two or more different kinds of donor atoms[3].Mixed ligand complexes are generally found to be more active biologically than the ligand itself[4].

This paper reports the synthesis of mixed ligand complexes of nickel using 5-nitrosalicylaldehyde (HL) as primary ligand and salicylaldehyde, substituted salicylaldehyde or 2-hydroxyarylketones as secondary ligands (HL’).The activity of ligands and the complexes against gram - negative *E. coli* and gram-positive *Entrococii, S. aureus* and *Straptococcus pneumoniae* were also studied.

2.Materials and methods

2.1Materials

$NiCl_2.6H_2O$ (Merck) was used of A.R. grade. The ligands 5-bromosalicylaldehyde Himedia), 5-chlorosalicylaldehyde (Himedia) and 5-nitrosalicylaldehyde (Aldrich) were purified by recrystallization from hot ethanol prior to use. Salicylaldehyde (Merck),2-hydroxyacetophenone(Aldrich),2-hydroxypropio phenone (Fluka) and ethanol were purified by distillation.

2.2 Analytical methods and Physical Measurements

Nickel was estimated volumetrically by EDTA using Eriochrome black-T as an indicator. Nitrogen was determined by Kjeldahl’s method. Carbon and hydrogen analysis were carried out on a Heraeus Carlo Erba 1108 instrument. Molar conductances were measured at room temperature in DMSO by a digital conductivity meter-NDC-736. Magnetic measurements were carried out using MSB MK1 Magnetic Susceptibility Balance, Sherwood Scientific. Electronic spectra were recorded in DMSO in the range of 200nm-800nm on a EC Double Beam UV-VIS Spectrophotometer using distilled water as a reference. Infrared spectra of the complexes were recorded in the region 4000-400cm^{-1} on a SHIMADZU-JAPAN 8400S FTIR spectrophotometer using KBr pellets. The ESI mass spectra were recorded on a MICROMASS QUATTRO II triple ionization mass spectrometer. Thermo gravimetric analysis were performed on Mettler Toledo star SW 701 instrument in the range 57.4-689°C with the heating rate 10°C per minute.

2.3 Antibacterial Assay: Paper Disc Diffusion Method:

This method was used to study the antibacterial activity of the complexes and their corresponding ligands against E.coli, Entrococii, Staphylococii and Straptococii pathogenic bacteria. In this method, 0.1 mL of inoculums of the test organism was spread uniformly on the surface of the agar medium in a petri plate by using a spreader. The sterilized Whatmann filter paper discs of 5 mm diameter were dipped into the 200 ppm solution of the complexes in DMSO and then were placed on the surface of the agar. Up to four discs in each plate were used. The plates were incubated at 37°C for 24 hours. During incubation, the complex diffuses from the filter paper into agar. The activity of the complexes was assessed by measuring the diameter of the inhibited zone in millimeters (mm) carefully. The results were compared against those of control standard antibiotic drug Ciprofloxacin which was screened simultaneously. Ciprofloxacin has a definite value for each strain but the zone of inhibition may vary according to the experimental conditions as temperature, pH of the media and other factors. So it becomes essential to incorporate a positive control with each sample and each micro-organism. This is the reason, why a corresponding plate is set for ciprofloxacin for each sample with each bacterial strain. Solvent DMSO, used as blank, was also run to know its activity.

2.4 Preparation of the complexes

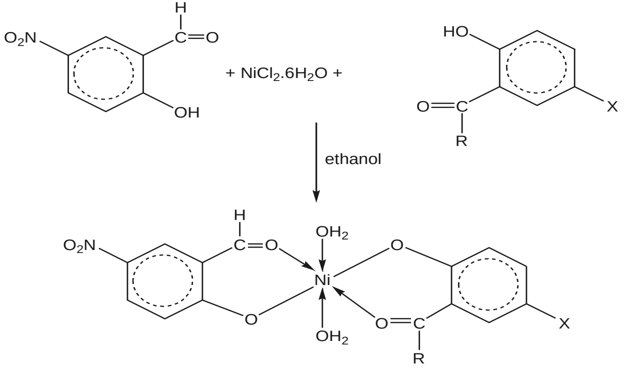
The metal complexes were prepared by mixing of an ethanolic solution of $NiCl_2.6H_2O$ (0.500gm in ~10ml ethanol), an ethanolic solution of primary ligand (HL) 5-nitro salicylaldehyde (0.3515gm in ~10ml ethanol) and secondary ligand (HL') salicylaldehyde (0.2568gm dissolved in ~10ml ethanol in 1:1:1 molar ratio with constant stirring. The reaction mixture was magnetically 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 hrs and then kept in the refrigerator for about 1-2 days. The settled solid was filtered, washed with ethanol and dried properly under reduced pressure.

A similar method was adopted to synthesize mixed ligand complexes of Ni(II) with 5-nitrosalicylaldehyde and 5-bromo-salicylaldehyde,5-chlorosalicylaldehyde, 2-hydroxyacetophenone or 2-hydroxypropiophenone. The analyses and characteristics of the complexes are mentioned in Table 1.

Table 1: Analytical data of the mixed ligand complexes $[Ni(L)(L')(H_2O)_2]$

Complex, Mol. Wt.	Analysis% found (Calculated)			
	C	H	N	Ni
$[Ni(5\text{-}NO_2sal)(sal)(H_2O)_2]$ 381.88	43.89 (44.03)	3.03 (3.43)	3.57 (3.66)	15.00 (15.35)
$[Ni(5\text{-}NO_2sal)(5\text{-}Brsal)(H_2O)_2]$ 460.63	36.09 (36.47)	2.45 (2.62)	2.89 (3.04)	12.32 (12.72)
$[Ni(5\text{-}NO_2sal)(5\text{-}Clsal)(H_2O)_2]$ 416.40	39.40 (40.38)	2.74 (2.90)	3.12 (3.36)	13.57 (14.07)
$[Ni(5\text{-}NO_2sal)(2\text{-}hap)(H_2O)_2]$ 395.91	44.05 (45.50)	3.31 (3.81)	3.11 (3.53)	14.08 (14.80)
$[Ni(5\text{-}NO_2sal)(2\text{-}hpp)(H_2O)_2]$ 409.94	45.99 (46.11)	4.05 (4.11)	3.07 (3.33)	15.21 (15.69)

The general equation for the formation of the complexes is shown below (scheme-1)



Scheme 1. Synthesis of mixed ligand complexes of Ni(II) with $5\text{-}NO_2sal$ and sal, 5-Brsal, 5-Clsal, hap or hpp

R= -H, X= -H; R= -H, X= -Br; R= -H, X= -Cl; R= $-CH_3$, X= -H; R= $-C_2H_5$, X= -H;

3.Results and Discussion

All resulting complexes are moisture stable amorphous solid. From the elemental analysis data the tentative molar ratio of mixed ligand complexes, is Metal: L:L' comes out to be 1:1:1 where HL = Primary ligand and HL' = various Secondary ligands respectively. It has been found that the theoretical values are in good agreement with the found values. They are insoluble in water and common organic solvents like methanol, benzene, chloroform and carbon tetrachloride but soluble in DMSO and DMF. Analytical data of the mixed ligand complexes $[Ni(L)(L')(H_2O)_2]$ have been given in Table-1. All the resulting complexes are pale yellow. On heating, these complexes decompose at high temperature without melting. Molar conductivity of 10^{-3} M solutions of the complexes in Dimethyl sulphoxide (Table-2) show that they have non- electrolytic in nature as the values are observed in the range of 12.08-18.51 Ω^{-1} cm^2 mol^{-1}. Quyoom[5] has found conductivity of mixed ligand complexes of nickel and zinc in the range 11-12 Ω^{-1} cm^{2}mol^{-1} indicating that they all are non electrolytic in nature. At room temperature all the complexes of nickel under study show the magnetic moment in the range of 2.83-2.99 BM (Table-2) which is well within the expected range for Ni(II) complexes with octahedral stereochemistry (2.83-4.0 BM).This indicates that the complexes are high spin paramagnetic and the ground state term is $^3A_{1g}$.

Table 2: Physicochemical characteristics of the mixed ligand complexes $[Ni(L)(L')(H_2O)_2]$

Complex Colour, (yield %)	Decom. Temp (°C)	Cond. Ω^{-1} cm^2 mol^{-1}	Mag. moment (μ_{eff})
$[Ni(5\text{-}O_2sal)(Sal))(H_2O)_2]$ Pale green (70.97)	230	15.15	2.98
$[Ni(5\text{-}NO_2sal)(5\text{-}Brsal)(H_2O)_2]$ Yellow brown (42.41)	256	17.06	2.84
$[Ni(5\text{-}NO_2sal)(5\text{-}Clsal)(H_2O)_2]$ Dirty-Yellow (52.56)	258	18.10	2.93
$[Ni(5\text{-}NO_2sal)(2\text{-}hap)(H_2O)_2]$ Dull-yellow (58.46)	285	18.10	2.93
$[Ni(5\text{-}NO_2sal)(2\text{-}hpp)(H_2O)_2]$ Dull- yellow (93.94)	310	17.45	2.83

3.1 Infrared Spectra

The IR spectra of ligands show a broad band centered at around 2950-3090 cm^{-1} due to free phenolic hydroxyl group present in free ligands which disappeared in spectra of their metal complexes indicating the coordination of ligands to metal ion in complexes through phenolic oxygen moiety after deprotonation as shown in scheme-1.Tripathi and coworkers[6] have been also found disappearance of band at 3080 cm^{-1} in metal complexes, originally present in free pyrazolines due to υ(O-H). The ligands show a medium intensity band at around 1150-1290cm^{-1} due to phenolic =C-O group; is shifted to higher reigon or downfield at around 1320-1400cm^{-1} in complexes further indicating the coordination in complexes through phenolic oxygen moiety. Neelkantan et al [7] have observed similar results for =C-O vibration (phenolic) in [NiL'B] mixed ligand complexes at 1207 cm^{-1} (where L'= o-vanillidene-2-aminobenzthiazole and B=1, 10-phenanthroline), whereas in free o-vanillidene-2-aminobenzthiazole =C-O vibration (phenolic) found at 1269 cm^{-1} towards upfield.

Table 3: IR absorption bands of mixed ligand complexes $[Ni(L)(L')(H_2O)_2]$ in cm^{-1}

Complex	$\nu_{C=O}$	$\nu_{(=C-O)}$	$\nu_{(O-H)}$ (H_2O Coordinated)	ν_{M-O}
$[Ni(5\text{-}NO_2sal)(sal)(H_2O)_2]$	1650	1320	3200- 3600	530, 420
$[Ni(5\text{-}NO_2sal)(5\text{-}Brsal)(H_2O)_2]$	1640	1320	3200- 3640	570, 440
$[Ni(5\text{-}NO_2sal)(5\text{-}Clsal)(H_2O)_2]$	1645	1330	3240- 3580	550, 492
$[Ni(5\text{-}NO_2sal)(2\text{-}hap)(H_2O)_2]$	1590	1400	3200- 3600	570, 440
$[Ni(5\text{-}NO_2sal)(2\text{-}hpp)(H_2O)_2]$	1600	1400	3200- 3580	520, 420

The IR bands in ligands are practically unchanged in the complexes but all complexes show new broad diffused band in the region at ~ 3100-3600 cm^{-1} and ~ 420-570cm^{-1} due to ν(OH) of the coordinated water molecules and due to M-O bonds respectively which are absent in the ligands. M.Sunitha et al[8] have been reported broad band in the range 3354-3423cm^{-1} in $[M(BMCP)(H_2O)_2Cl_2]$ where M= Co(II) & Ni(II), indicating the presence of coordinated water molecules. In present work the IR spectra of the complexes exhibit strong absorption bands in the region of 1590-1660cm^{-1} and 1450-1570cm^{-1} which may be assigned to coordinated ν(C=O) and ν(C=C) respectively, whereas the ν(C=O) bands in ligands show at 1640-1806cm^{-1} and υ(C=C) bands present in the region 1440-1555. The ν(C=O) bands of the complexes are observed in the lower wave number side as compared to free ligands confirming the coordination of ν(C=O) group to the metal atom. The IR absorption bands of Ni(II) mixed ligand complexes are recorded (table-3) and the spectra of $[Ni(5\text{-}NO_2sal)(Sal)(H_2O)_2]$ are reproduced (fig.1).Such a shifting of ν(C=O) to lower wave number side (at 1599-1591$cm^{-1)}$ have been reported by Suresh and Prakash[9] in the spectra of chelates of schiffs base derived from vanillin and anthranilic acid with $Cr^{3+,}$ $Mn^{2+,}$ $Co^{2+,}$ $Ni^{2+,}$ $Cu^{2+,}$ Zn^{2+} and Cd^{2+}. The ν(OH) of the coordinated water is absent in the spectra of the ligand but present in the complex in the range of 3443-3274cm^{-1}. This is supported by the presence of ν(M-O) noticed in the complex at 716-707 cm^{-1}.

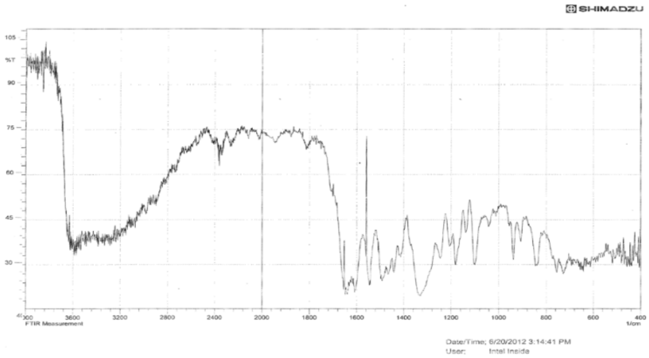


Figure 1: IR spectrum of $[Ni(5\text{-}NO_2sal)(2\text{-}hap)(H_2O)_2]$

Hence, the ligand coordinatrd with the metal ions as monodeprotonated bidentate and the coordination occours via the carbonyl and phenolic oxygen.

3.2 Electronic spectra

The electronic spectral data are given in table-4 and shown in fig. 2.The data is of relevance for the proposed structure of the complexes (scheme-1).The electronic spectra in the ultraviolet and visible region (200-800nm) of free ligands in DMF solution revealed maximum absorption band at 41322-40984 cm^{-1} (1.475 absorbance) in all free ligands which is attributed to aromatic chromophore O=C-C=C-OH of the ring in salicylaldehyde and substituted salicylaldehyde ligands.

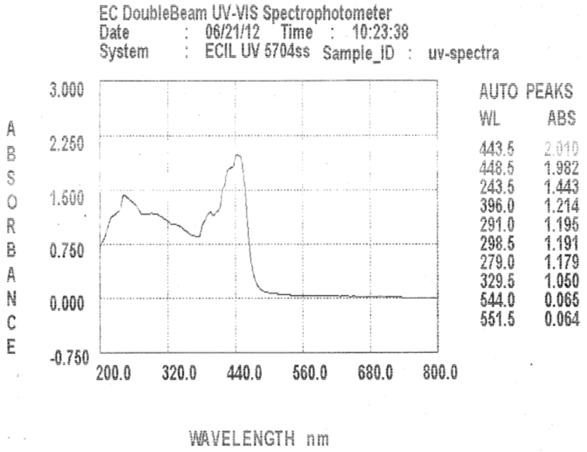


Figure 2: Electronic spectrum of $[Ni(5\text{-}NO_2sal)(sal)(H_2O)_2]$

The band observed for all the complexes of nickel in the range 40,984-40,152 cm^{-1} are assigned to the π-π* transition of the aromatic chromophore, which is lowered values than ligands indicating that chromophore modified by the interaction with metallic ion. In addition, the band in the range 40,000-33,333 cm^{-1} can be attributed to the n-π* transition. The electronic absorption spectra in the visible reigon of the Ni (II) complexes in DMF solution show absorption bands around 26,401-25,000 cm^{-1} and 23,670-20,408 cm^{-1} are attributed to d-d transitions (Table -4).Such type of bands in electronic spectra of nickel complexes have been found by Sanap and coworkers[10].

Table 4: Electronic spectral data of the mixed ligand complexes $[Ni(L)(L')(H_2O)_2]$

Complex	Intraligand π-π*	intraligand n-π*	d-d transition
$[Ni(5\text{-}NO_2sal)(sal)(H_2O)_2]$	40816	33333	25252 22321
$[Ni(5\text{-}NO_2sal)(5\text{-}Brsal)(H_2O)_2]$	40485	33557	26401 23584
$[Ni(5\text{-}NO_2sal)(5\text{-}Clsal)(H_2O)_2]$	40650	34013	26000 22321
$[Ni(5\text{-}NO_2sal)(2\text{-}hap)(H_2O)_2]$	40152	33670	25641 21691
$[Ni(5\text{-}NO_2sal)(2\text{-}hpp)(H_2O)_2]$	40322	34364	25575 23523

The electronic spectra of these complexes show π-π* and n-π* transition in the range 47,619-39,682 cm^{-1} and 29,850-28,985 cm^{-1} respectively and also show two bands in the region showed 24,271-16393 cm^{-1} due to d-d transitions by high spin nickel(II) metal. Same two bands in the range

26385-16722 cm^{-1} for d-d transition have been reported by Quyoom[6] in octahedral ternary nickel complexes of N-Acetyl Cysteine. M. Sunitha et al[8] have also found similar d-d transitions in the range of 22,222-27,397cm^{-1} for [Ni(BMCP)$(H_2O)_2$ Cl_2] mixed ligand complex.

3.3 Thermo gravimetric analysis

The results of thermal analysis of mixed ligand complex [Ni(5-NO_2sal) (Sal)$(H_2O)_2$] shown in table-5. The TG curve has been recorded for [Ni(5-NO_2sal)(sai)$(H_2O)_2$] The results showed that the complex was thermally decomposed in three successive decomposition steps within the temperature range 130-800°C can be represented as follows-

$[M(L)(L')(H_2O)_2] \rightarrow [M(L)(L')] \rightarrow [M(L)]$ or $[M(L')] \rightarrow MO$

The TG curves of these complexes indicated the presences of coordinated water molecules and the final product were the metal oxides. The first decomposition step for all three complexes within the temperature range130-230°C which is reasonably accounted for the removal of two coordinated water molecules, which confirms the presence of two coordinated water molecules in the complex. The perusal of thermogram shows the presence of water molecules in the complexes which further corroborates the observation made on the basis of infrared spectrum studies and is in the good agreement with the elemental analysis presented in Table-1.

El-Wahed et al[11] have also reported the loss of coordinated water molecules in the temperature range 150-300°C in a sulfasalazine Mn(II) complex of the type [Mn(SuzH)$(H_2O)_4$].$2H_2O$.

The second step is found between the temperature range 200-512°C may be attributed to the liberation of salicylaldehyde ligand moiety in first complex while 5-nitrosalicylaldehyde ligand moiety in remaining both complexes. The decomposition of the rest of the complex molecule ended with the formation of NiO in the third step within the temperature range 350-870°C.

Table 5: Thermal data of Ni mixed ligand complexes

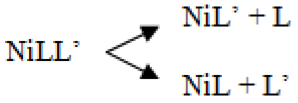
Complex	Step	Temp. range (°C)	Mass Loss %		Assignments (Mass)
		T_i-T_f	Cal.	Obs.	
[Ni(5-NO_2sal)(sal)$(H_2O)_2$]	1	168- 200	8.33	8.30	Loss of $2H_2O$
	2	200- 300	10.63	10.33	Loss of Nitro
	3	300-550	26.38	26.12	Loss of Sal
	4	550-800	39.35	39.09	Loss of sal moiety

Patil and coworkers[13] have been found the decomposition range 160-600°C for the complex [Ni$(PMINAP)_2$.$2H_2O$], in first step (162-250) loss in weight corresponding to decomposition of two water molecules accompanied with second step the decomposition of the rest of the complex molecule ended with the formation of NiO, which supports our result, as we have also found NiO in last step.

3.4 ESI Mass Spectral Studies

Electrospray ionization mass spectrum of [Ni(5-NO_2sal)(Sal)] has been recorded. The mass spectrum of the complex shows a large number of peaks corresponding to the successive degradation of the molecule

Complex exhibit molecular ion $[M]^+$ peaks at m/z 347 (35.1%) and a base peak m/z 453 due to $Ni_2(5\text{-}NO_2sal)_2$ dimeric species.A peak at m/z 301(14.04%) due to elimination of nitro group from molecular ion $[M\text{-}NO_2]^+$.Complexes exhibit peaks due to NiL^+ and NiL'^+ as a result of loss of any one ligand moiety from molecular ion NiLL' for eg. at m/z 225 due to $[Ni(5\text{-}NO_2sal)]^+$ (I 9.36%) and m/z 180 (24.36%) due to $[Ni(sal)]^+$.

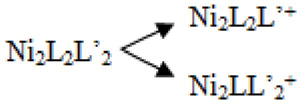


$$NiLL' \rightarrow NiL' + L \qquad NiLL' \rightarrow NiL + L'$$

The complex shows peak at m/z 392 (23.4%) due to $[Ni(5\text{-}NO_2sal)_2]^+$ and m/z 302 (18.7%) due to $[Ni(sal)_2]^+$ due to redistribution reaction.

$$2\ NiLL' \rightarrow NiL_2 + NiL'_2$$

Peaks due to dimeric species exhibit by complex m/z 701 (63.96%) show much higher intense peak for dimeric species



$$Ni_2L_2L'_2 \rightarrow Ni_2L_2L'^+ \qquad Ni_2L_2L'_2 \rightarrow Ni_2LL'^+_2$$

The spectrum of complex exhibit peaks due to $Ni_2L_2L'^+$ & $Ni_2LL'^+_2$ ions which might have been formed by the loss of either of any ligand moiety from dimeric species.

Further fragmentations of above species can also be explain by following equations-

$$Ni_2L_2L'^+ \rightarrow Ni_2L'^+$$

$$Ni_2LL'^+_2 \rightarrow Ni_2L^+$$

A peak can be observed at m/z 265 (14.04%) due to loss of water molecule from Ni_2L^+.

4.*In vitro* Antibacterial Screening

The ligands and the complexes were screened against the pathogenic bacterial strains gram - negative *E. coli* and gram-positive *Entrococii, S. aureus* and *Straptococcus pneumoniae*. The solvent DMSO was used as a negative control whereas Ciprofloxacin, a standard antibiotic, was used as a positive control. The antibacterial screening data have shown in Table-6. The results showed that ligands like salicylaldehyde and substituted salicylaldehyde exhibit moderate activity against all tested bacteria. It is important to note that the metal chelates exhibit more inhibitory effects towards bacterial strain than the parent ligands. Similar results have been previously reported by Jain et al[13] for some Mn(II) complexes derived from different sulpha drugs and heterocyclic ketones. Neelakantan et al[7] have also reported the similar observations for mixed ligand complexes of the type ML'B, HL'=o-vanillidene-2-aminobenzothiazole, B = phen and HL" = o-vanillidene-2-

amino-N-(2-pyridyl)-benzene sulfonamide). ML_2" and M_2L", (where M(II)= Mn(II), Co(II), Ni(II), Cu(II) and Zn(II).

Increased activity of the metal chelates as compared to ligands can be explained on the basis of Tweedy’s chelation theory[14]. According to chelation theory, chelation tends to make the ligand act as more powerful and potent bactericidal agents, thus killing more of the bacteria than the ligand. It is observed that in a complex, the positive charge of the metal is partially shared with the donor atoms present in the ligands and there may be π-electron delocalization over the whole chelating ring. This increases the lipophilic character of the metal chelate and favors its permeation through the lipoid layer of the bacterial membranes and blocks the metal bonding sites on the enzymes of micro-organism. These complexes also disturb the respiratory processes of the cell and thus block the synthesis of protein, which restricts further growth of the organism. There are some other factors like solubility, conductivity and bond length between the metal and the ligand, which also increase the activity.

Moreover, overtone concept[15] of cell permeability is also important in this contrast. According to this concept, the lipid membrane that surrounds the cell, favors the passage of only lipid-soluble material, due to which liposolubility is also an important factor that controls the antibacterial activity of the compound. Interestingly, it has been also observed from the study of antibacterial zone of inhibition data that the complexes are much potent bactericides than the standard control ciprofloxacin against *S. aureus* and *E. coli*.

Table 6: Zone of inhibition of mixed ligand complexes [Ni (L)(L')$(H_2O)_2$ in mm

HL/Complex	*E. coli*	*Entro cocci*	*S. aureus*	*S. pneumonia*
5-nitrosalicylaldehyde	9	6	8	6
[Ni(5-NO_2sal) (Sal)$(H_2O)_2$]	13	12	9	9
[Ni(5-NO_2sal) (5-Brsal) $(H_2O)_2$]	14	10	10	15
[Ni(5-NO_2sal) (2-hap)$(H_2O)_2$]	10	9	11	8
[Ni(5-NO_2sal) (2-hpp)$(H_2O)_2$]	11	9	10	7
Ciprofloxacin	24	22	20	20
DMSO	0	0	0	0

5.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 ν(C=O) to lower wave number 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 activities as compared to their parent ligands, hence further study of these complexes could lead to interesting results.

6.Acknowledgement

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