Synthesis, characterization and antimicrobial activity of Mn(II), Fe(III), Co(III), Ni(II), Cu(II) and Zn(II) complexes of a new (O,N) type donor Schiff base ligand derived from Homoveratrylamine and *β*-Resorcinaldehyde

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Abstract. A monoionic bidentate Schiff base ligand of (O, N) type, **LH_2** and their complexes, **1-6** were synthesized from the reaction between homoveratrylamine and β-resorcinaldehyde followed by using respective metal salt. The synthesized **LH_2** and **1-6** have been characterized by spectroscopic and elemental analytical methods. The characterization data revealed that the coordination of **LH_2** to the central metal ion through monoionic bidentate (N,O^-) type donor. The antimicrobial activity of **LH_2** and **1-6** were evaluated. The metal complexes (**1-6**) were shows comparatively greater antimicrobial activity than ligand (**LH_2**)

Keywords: Schiff base, transition metal, complex, antimicrobial

1 Introduction

Schiff bases containing (O, N) donor atoms can be easily synthesized from simple amines and 2-hydroxyl aldehydes or ketones. Generally,the(O, N) type of ligands are stabilized by characteristic intra-molecular hydrogen bonding between the azomethine nitrogen and phenolic hydrogen and most of them are crystalline solids and have been structurally well characterized. These ligands act as good chelating agents and easily form complexes at room temperature with most of the metal ions intransition elements. This is because the increased nucleophilicity nitrogen in the azomethine group (>C=N-) and phenolateoxygen (PhO^-) to form stable six-membered metal-chelating rings on coordination with metals ions. A number of (O, N) type ligands and their complexes have proven prospective therapeutics and good biological activities viz., antibacterial, antidepressant, antifungal, antiglycation, anti-inflammatory, antimalarial, antioxidant, analgesic, anticonvulsant, antihypertensive

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antitumour, antiviral and lipid-lowering[1-10]. The metal complexes of (O, N) type ligands also have shown significant catalytic [11] and biological activities [8-14].

By the consideration of above facts, hereby reported the synthesis of (O,N) type ligand, **LH_2** and their complexes (**1-6**), [M(**LH**)$_2$], where M=Mn(II), Ni(II), Cu(II), Zn(II) and [M(**LH**)$_2$Cl], (where M=Fe(III), Co(III). Further, the antimicrobial activity of all the synthesized **LH_2** and **1-6** was carried out. The structures of the synthesized ligand, **LH_2** and its complexes, **1**-6 were given in **Chart 1.**

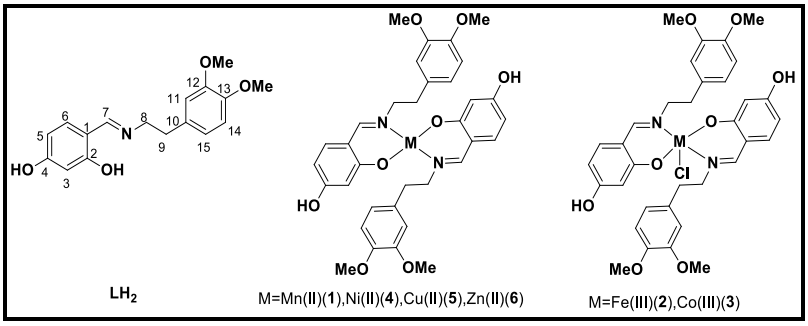


Chart 1 Structure of **LH_2** and **1-6**

2 Experimental

2.1 Reagents and Materials

2,4-dihydroxybenzaldehyde (*β*-resorcynaldehyde), 2-(3,4-dimethoxyphenyl)ethan-1-amine, metal salts were purchased from Sigma India Pvt Ltd. The microorganisms viz., Escherichia coli (E. coli) [NCIM-5051], Klebsiellaaerogenes(k. aerogenes) [NCIM-2098], Pseudomonas desmolyticum [NCIM-2028] and Staphylococcus aureus [NCIM-5022], Candida albicans [NCIM-3100] and Aspergillusflavus (A. flavus) [NCIM-544] have been purchased from NCL, Pune, India and these stains have been maintained at 4°C. The standard antibiotic and antifungal drugs were received from Hi-Media, Mumbai, India.

2.2 Analytical methods

Melting point of **LH_2** and **1-6** were determined by using melting point and are reported uncorrected. LECO–CHNSO–9320 elemental analyser was used for CHN elemental analysis. The UNI-CAM-UV 2-100 spectrophotometer is used to record UV–visible spectra. FT-IR data were recorded on Agilent spectrophotometer. The Bruker spectrometer (400MHz) with tetramethylsilane (TMS) used as an internal standard to record the $^{13}C\{^1H\}$ and ^1H NMR spectrum of **LH_2** and **6**.

2.3 Synthesis of ligand (LH2)

The solutions of 2-(2,4-dimethoxyphenyl)ethanamine in absolute ethanol (20 mL) and 2.4-dihydroxybenzaldehyde (1 mmol) in absolute ethanol were mixed along with stirring and is continued for 2h. The solid was separated out. The solvent was removed by using rotary evaporator to gave **LH_2** as yellow solid.

LH_2: Yield: 95%; M.P.:63-65°C; CHN Analysis for $C_{17}H_{19}NO_4$: Calcd.(Found) %: C 67.76(67.73); H 6.36(6.32); N 4.65(4.67); FT-IR (ATR, ν in cm^{-1}): 3402, 2998, 2935, 1638,

1513, 1465,1261, 1237, 1024, 845, 797, 603 and 551; UV-vis (λ_{max} in nm): 387, 307, 280 and 259; ^{1}H NMR (Solvent: $(CH_3)_2SO$-d_6, δ in ppm): 13.81 (s, 1H, 2-OH), 9.95 (s, 1H, 4-OH), 8.23 (s, 1H, >CH=N-), 7.088-7.108 (d, 1H, 6-H), 6.816-6.838 (d, 2H, 14-H, 15-H), 6.712-6.732 (d, 1H, 11-H), 6.202-6.220 (d, 1H, 5-H), 6.123 (s,1H, 3-H), 3.683 (s, 3H, methoxy), 3.627 (s, 3H, methoxy), 3.501-3.611 (t, 2H, 8-H), 2.750-2.830 (t, 2H, 9-H); $^{13}C\{^{1}H\}$ NMR (Solvent: $CDCl_3$, δ ppm): 36.62 (C-9), 55.78 (methoxy), 55.86 (methoxy), 55.93 (C-8), 111.56 (C-5), 112.15 (C-11), 112.54 (C-14), 120.79 (C-15), 120.91 (C-10), 147.77 (C-3), 130.82 (C-1), 134.28 (C-6), 148.94 (C-13), 149.07 (C-12), 163.35 (C-4), 165.18 (C-2), 170.58 (C-7).

2.4 Synthesis of [M(LH)$_2$] (1,4-6), where M=Mn(II) (1), Ni(II) (4), Cu(II)(5), Zn(II) (6)

A respective metal salt solution (0.29 mmol) in methanol, was added to a stirred solution of **LH$_2$** (0.58 mmol) in methanol followed by triethyl amine (0.58 mmol) at RT for 6-7 h. The consumption of ligand was monitored by Thin Layer Chromatography. The solvent in a reaction mixture was evaporated to dryness using rotavapor to gave respective complex, [M(**LH**)$_2$] (**1,4-6**).

[Mn(**LH**)$_2$] (**1**): Brown colour solid. Yield: 80%; M.P.: 246-250^{o}C (decomp.); CHN Analysis for $C_{34}H_{36}N_2MnO_8$ Calcd.(Found) %: C, 62.29(62.31) ; H, 5.53(5.55); N, 4.20(4.19); FT-IR (ν in cm^{-1}): 3582, 3517, 2920, 1598, 1514, 1235, 1134, 1024, 844, 763, 639; UV-Vis.: λ_{max} in nm: 282,305,347.

[Ni(**LH**)$_2$] (**4**): Green colour solid; Yield: 80%; M.P: 247-250°C (decomp.); CHN Analysis for $C_{34}H_{36}N_2NiO_8$ Calcd.(Found) %: C, 61.93(61.90); H, 5.50(5.49); N, 4.25(4.28); FT-IR (ν in cm^{-1}): 3432, 3064, 3009, 2925, 1622, 1541, 1513, 1417, 1342, 1278, 1235, 1173, 1113, 1022, 845, 823, 797, 629 and 498; UV-Visible (λ_{max} in nm): 253, 270,330, 404.

[Cu(**LH**)$_2$] (**5**): Dark violet crystalline solid; Yield: 73%; M.P.: 138-145°C (decomp.); CHN Analysis for $C_{34}H_{36}N_2CuO_8$ Calcd.(Found) %: C, 61.48(61.45); H, 5.46(5.45); N, 4.22(4.25); FT-IR (ν cm^{-1}): 3433, 3212, 2939, 2675, 1619, 1538, 1451, 1342, 1286, 1259, 1231, 1133, 1023, 845, 761, 740, 707, 679, 615, 520 and 498; UV-Visible (λ_{max} nm): 258, 290, 350.

[Zn(**LH**)$_2$] (**6**): White solid: Yield: 68%; M.P.:169-171°C (decomp); CHN Analysis for $C_{34}H_{36}N_2ZnO_8$ Calcd. (Found) %: C, 61.31(61.30); H, 5.45(5.44); N, 4.21(4.23); UV-Visible (λ_{max}nm): 272, 370; FT-IR: (ν cm^{-1}): 3401, 3010, 2980, 2862, 1586, 1481, 1205, 1099, 843, 744, 639, 482 and 450; ^{1}H NMR (Solvent: $(CH_3)_2SO$,δ ppm): 8.310 (s, 1H, CH=N), 9.834 (s, 1H, OH), 7.011-7.021 (dd, 1H, H6), 6.876-6.897 (d, 1H, H-3), 6.682- 6.705 (d, 1H, H-5), 6.542-6.590(dd, 3H, H-11, H-15, H-14), 3.603 (s, 3H, -O-CH_3), 3.474 (s, 3H, -O-CH_3), 3.136-3.291 (t, 2H, -N-CH_2), 2.181-2.292 (t, 2H, -CH_2-Ar). $^{13}C\{^{1}H\}$ NMR (Solvent: $CDCl_3$, δ in ppm): 36.60 (C-9), 55.73 (-O-CH_3), 55.87 (-O-CH_3), 62.64 (C8), 111.30 (C5), 112.18(C11), 114.62 (C14), 117.91 (C10), 123.14 (C15), 130.48 (C1), 135.03 (C6), 135.70 (C3), 147.65 (C13), 148.92 (C12), 164.35 (C4), 170.53 (C2), 171.38 (C7).

2.5 Synthesis of M(III) complexes, [M(LH)$_2$Cl] (2-3), where M= Fe(III)(2), Co(III)(3)

A respective metal salt solution (0.165 mmol) prepared in methanol added slowly to a solution of **LH$_2$** (0.33mmol) in methanol followed by triethyl amine (0.033 mmol) with stirring at RT and continued for 6-7 h. The consumption of ligand was monitored by TLC. The solvent in a reaction mixture was evaporated to dryness using rotavapor to gave respective complex, [M(**LH**)$_2$Cl] (**2-3**).

[Fe(**LH**)$_2$Cl](**2**): Dark green colour solid. Yield: 75%; M.P.:340-345^{o}C(dec.); Element. Anal. Calcd. (Found) $C_{34}H_{36}ClN_2CoO_8$: C, 58.75(58.71); H, 5.22; N,4.03(4.04); FT-IR (ν, cm^{-1}): 3627, 3545, 3408, 2142, 1941, 1616, 1514, 1232, 1024, 801, 659; UV-Vis.: λ_{max} in nm: 280, 315.

[Co(**LH**)$_2$Cl](**3**): Dark violet colour solid. Yield: 79%; M.P.:330-332^{o}C(dec.); Element. Anal. Calcd. (Found) $C_{34}H_{36}ClN_2FeO_8$: C, 59.02(59.04); H, 5.24(5.24); N, 4.05(4.03); FT-IR (ν, cm^{-1}): 3427, 2935, 1578, 1474, 1304, 1232, 1122, 1025, 849, 805, 642; UV-Vis.: λ_{max} in nm: 278, 316.

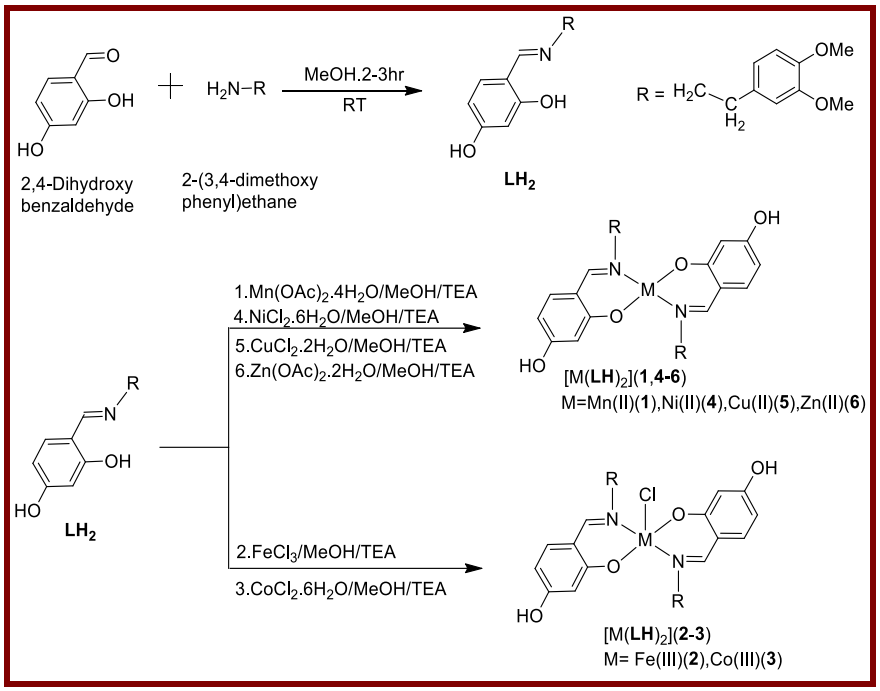
2.6 Antimicrobial activity

The antimicrobial activity of **LH$_2$** and complexes, **1-6** have been investigated as per the procedure reported [15-17]. The sample solutions of each **LH$_2$** and **1-6** prepared in sterile water by dissolving 0.5mg /50μL and and 1mg/100μL for antibacterial activity. A standard solution of Ciprofloxacin was prepared by dissolving 0.005mg/50μL. For antifungal activity, the solutions of **LH$_2$** and complexes (**1-6)** were made by dissolving 0.25mg/25 μL, 0.5mg/50μL and 0.75mg/75μL. A standard antifungal drug solution of Fluconazole (as a positive control) was made by dissolving 0.2mg/50 μL in sterile water. The region of inhibition of each sample was measured after 72 hours of the incubation period. For each compounds, duplicates were maintained and antimicrobial activity results were reported.

3. Results and Discussion

3.1 Synthesis

The (O, N) type ligand **LH$_2$** was synthesized by the reaction of 2,4-dihydroxy benzaldehyde and 2-(3,4-dimethoxyphenyl)ethan-1-amine [16,17]. The metal complexes, [M(**LH**)$_2$] (**1-6**), where M=Mn(II), Ni(II), Cu(II), Zn(II) and [M(**LH**)$_2$Cl], (where M=Fe(III), Co(III) were synthesized by the reaction of **LH$_2$** with respective metal salts in 2:1 ratio as given in **Scheme 1**.



Scheme 1 Synthesis of **LH_2** and **1-6**

LH_2 was freely dissolved in $CHCl_3$, CH_2Cl_2, CH_3OH, C_2H_5OH, DMF, DMSO, and insoluble in benzene, toluene and n-hexane. All the complexes, **1-6** were dissolved in $CHCl_3$, CH_2Cl_2, DMSO and DMF.

UV-visible spectroscopy

The UV-visible data of **LH_2** and **1-6** were take down by using DMSO and reported in **Fig. 1**. Within the UV–visible spectrum of **LH_2**, three strongly intense absorption bands watched at λ_{max}, 280, 307 and 387 nm. The primary two absorption bands obtained at λ_{max}, 280 and 307 nm were due to the π- π* transitions of the aromatic ring. The band showed at λ_{max}, 387 nm because of the transitions within the ligand charge transfer (ILCT) inside the delocalized π framework. Within the UV-visible spectra of **1-6** the groups watched between λ_{max}, 250-260 nm show $\pi \rightarrow \pi^*$ transition with two shoulders in interims of λ_{max}, 270-274 nm and 278-282 nm which indicate to another $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ excitations. In all the complexes these bands found blue shift of about 10-15 nm relative to **LH_2**. The bear of band watched around λ_{max}, ~390 to 400 nm in all complexes because of charge transfer transition between **LH_2** and central metal ion in complexes (LMCT) [16,17].

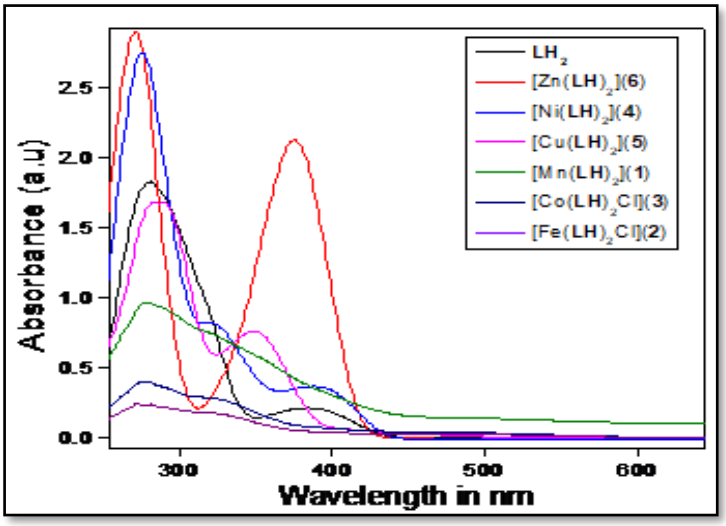


Fig.1 UV-Visible spectra of **LH_2** and **1-6**

IR spectroscopy

The band at ν, 3402 cm^{-1} appeared due to O-H strectching for **LH_2**. The >C=N streching frequency appeared at 1638cm^{-1} and was resulted red shift of about 20-50 cm^{-1} in **1-6** complexes, indicated a strong coordination between imine nitrogen and the central metal ion. Owing to the chelation ligand through the phenoxide (O^-) gather and the band moved to higher wave numbers in **1-6**. The bands between ν, 1578 and 1500 cm^{-1} in **LH_2** and **1-6** appeared due to >C=C< vibration in ary ring [17]. The IR spectra of **LH_2** and **1-6** complexes are as in **Fig.2** to **8**.

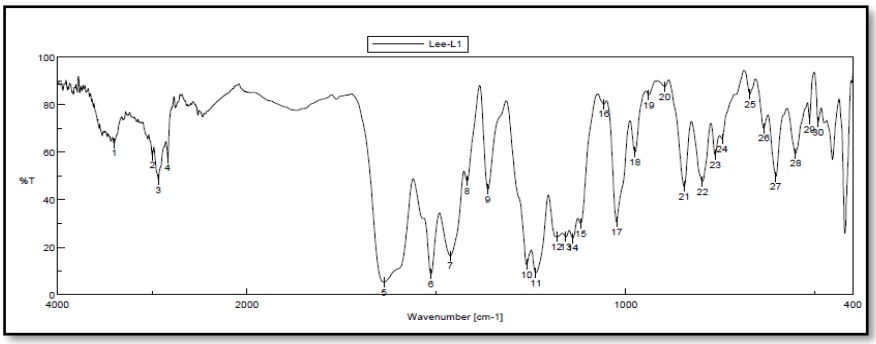


Fig.2 FT-IR spectrum of **LH_2**

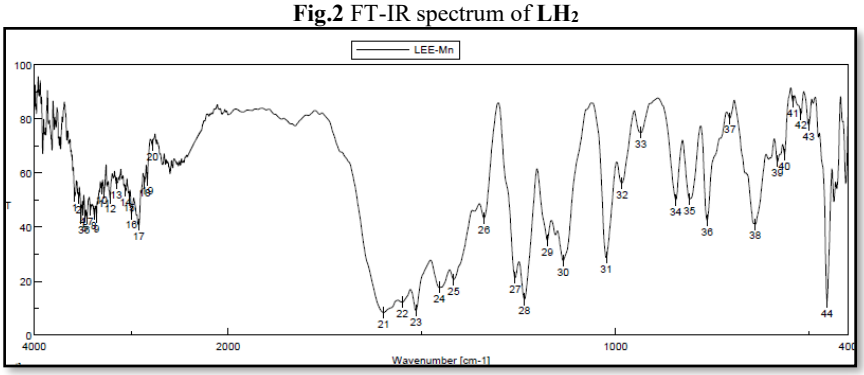


Fig.3 FT-IR spectrum of [Mn(**LH**)$_2$](**1**)

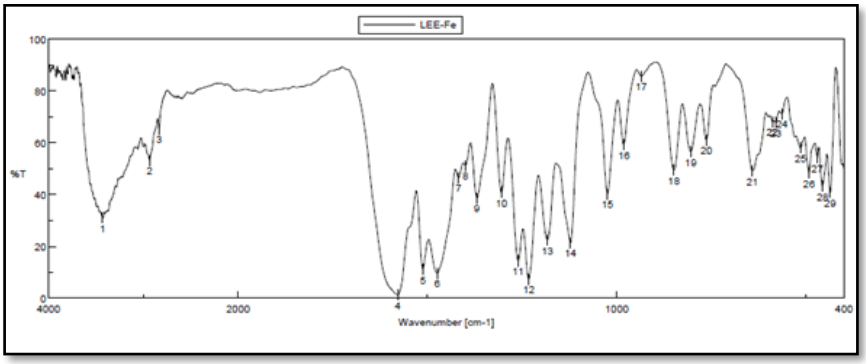


Fig.4 FT-IR spectrum of [Fe(**LH**)$_2$Cl](**2**)

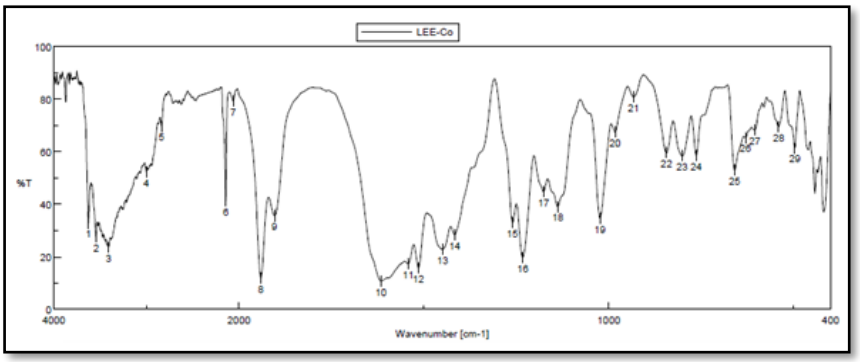


Fig.5 FT-IR spectrum of [Co(**LH**)$_2$Cl](**3**)

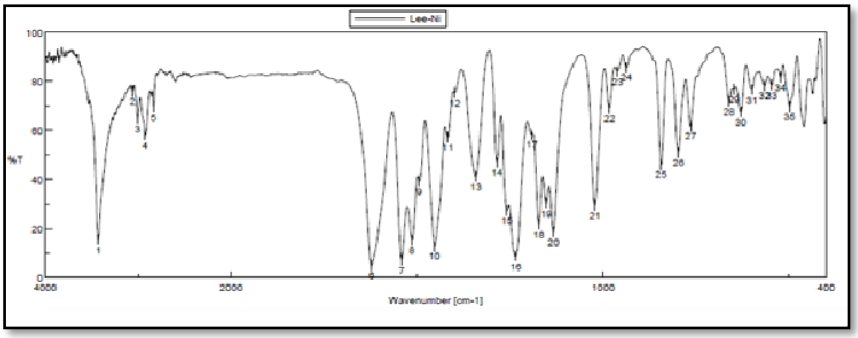


Fig.6 FT-IR spectrum of [Ni(**LH**)$_2$](**4**)

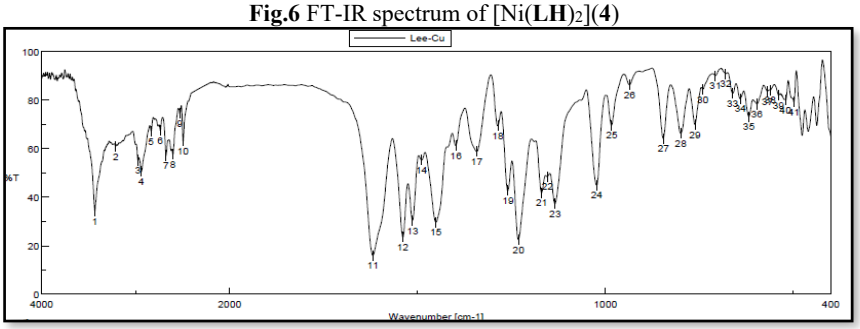


Fig.7 FT-IR spectrum of [Cu(**LH**)$_2$](**5**)

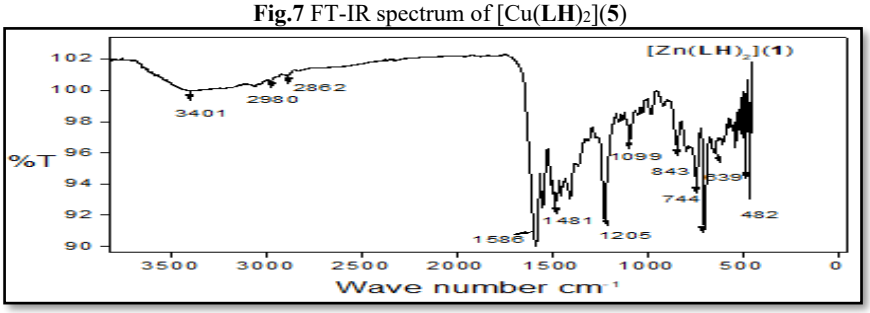


Fig.8 FT-IR spectrum of [Zn(**LH**)$_2$](**6**)

[1]H NMR spectroscopy

The [1]H NMR spectrum of **LH_2** and complex, **6** were shown in **Fig. 9 to 10**. In **LH_2**, the Ar-CH_2- and -CH_2-N protons appeared at δ, 2.750-2.830 and 3.501-3.611 ppm respectively as triplets. The two –OCH_3 froups gave two independent singlets at δ, 3.68 and 3.62 ppm. The -OH proton *ortho to C=N group* appeared as singlet in **LH_2** at δ, 13.81 ppm, and found highly deshielded due to intra-molecular H-bonding between O-H···N where as the signal for OH proton *para to C=N group* was appeared at δ, 9.95 ppm as singlet. The proton of CH=N in **LH_2** was observed at δ, 8.23 ppm as a singlet.

In complex **6**, the CH=N proton showed a downfield shift of 0.09 ppm when compared to that of the ligand **LH_2**. The absence of -OH proton signal in the proton NMR spectrum of zinc complex **6** indicate the coordination of (**LH_2**) to the metal through the *ortho*- PhO^-. In complex **6** the NCH_2 (0.4 ppm) and CH_2Ar (0.6 ppm) protons have been found to be shielded than ones protons in ligand **LH_2**[16,17].

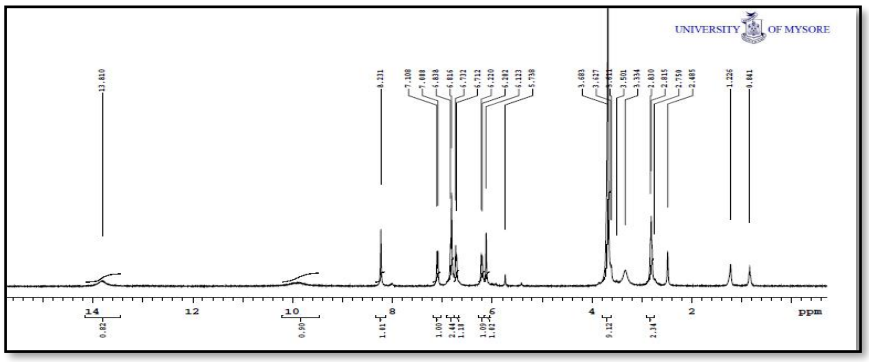


Fig.9 [1]H NMR spectrum of ligand (**LH_2**)

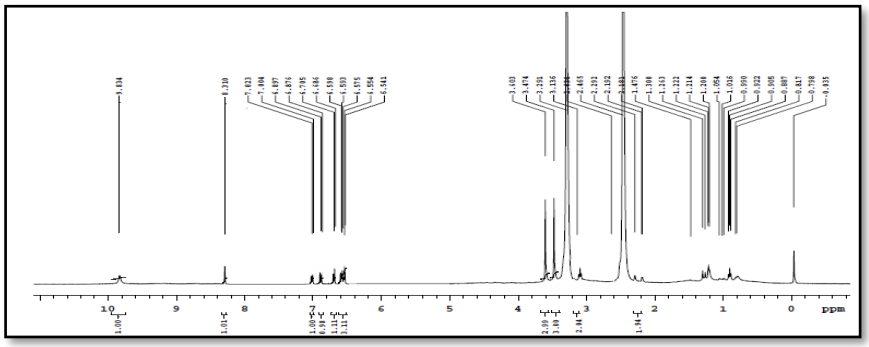


Fig.10 [1]HNMR spectrum of [Zn(**L**H)$_2$]**(6)**

[13]C NMR spectroscopy

In the [13]C-NMR spectrum of **LH_2** (**Fig.11**), the azomethine (>C=N-), C2 (*ortho* to PhC-O) and C4 (*meta* to $PhCO^-$) carbon signals appeared at δ, 170.58, 165.18 and 163.35 ppm respectively. The peaks for OCH_3 carbons appeared at δ, 55.7 and 55.8 ppm.

In the ^{13}C-NMR spectrum of complex **6** (**Fig.12**), the peaks for azomethine (>C=N-), C2 (*ortho* to PhC-O), and C4 (*meta* to $PhCO^-$) carbons found deshielded by about 2-6 ppm relative to the respective carbon signals in **LH_2**.

The NMR spectra of **LH_2** and **6** revealed that the **LH_2** coordinated to Zn(II) ion as monoanionic bidentate (N,O^-) type[16,17].

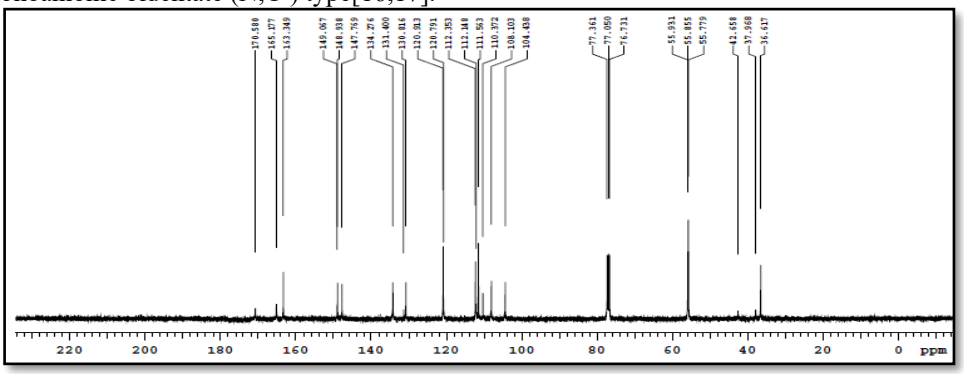


Fig.11 ^{13}C{^{1}H} NMR spectrum of **LH_2**.

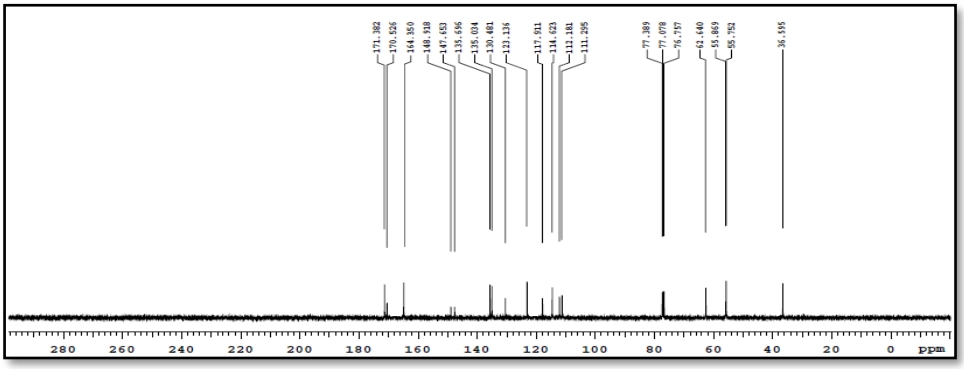


Fig.12 ^{13}C{^{1}H} NMR spectrum of [Zn(**LH**)$_2$](**6**)

Antimicrobial activity studies of LH_2 and 1-6 complexes

The antimicrobial properties of **LH_2** type (O,N) and metal complexes (**1-6**) as given within the **Fig.13** and **14**. The exploratory points of interest and region of restraint comes about in regard of both antimicrobial movement of **LH_2** and **1-6** completely different concentrations against the microbial strains as given in **Table 1** and **Table 2** individually. The results revealed that all the complexes were way better antimicrobial specialists than the ligand. The antimicrobial action increments with expanding concentration. Among the complexes tried copper and zinc complex appeared superior antimicrobial action[16,17].

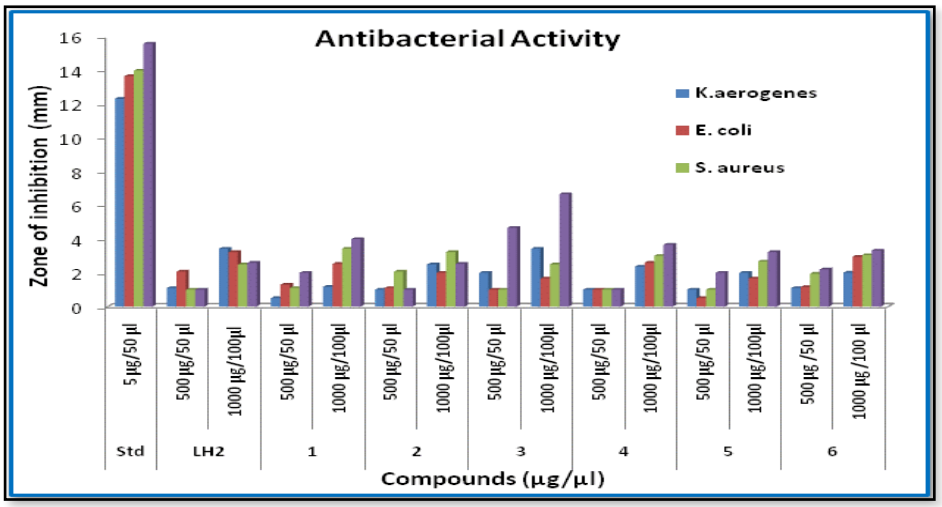


Fig.13 Antibacterial activity of **LH_2** and **1-6**

Table 1 Antibacterial activity of new compounds **LH_2**and **1-6** and a standard Ciprofloxacin

Compound	**Treatment**	***E. coli*** **(Mean ± SE)**	***K.aerogenes*** **(Mean ± SE)**	***S. aureus*** **(Mean ± SE)**	***P. desmolyticum*** **(Mean ± SE)**
Ciprofloxacin	0.005 mg/50 µl	13.67 ± 0.03	12.33 ± 0.03	14.00 ±0.00	15.60 ± 0.03
LH_2	0.5 mg/50 µl 1 mg/100µl	2.07 ± 0.07 3.23 ± 0.07	1.10 ± 0.06 3.43 ± 0.12	1.00 ± 0.00 2.50 ± 0.07	1.00 ± 0.00 2.60 ± 0.06
[Mn(**LH**)$_2$](**1**)	0.5 mg/50 µl 1 mg/100µl	1.30 ± 0.06 2.53 ± 0.03	0.50 ± 0.00 1.17 ± 0.17	1.10 ± 0.06 3.43 ± 0.12	2.00 ± 0.00 4.00 ± 0.00
[Fe(**LH**)$_2$Cl] (**2**)	0.5 mg/50 µl 1 mg/100µl	1.10 ± 0.06 2.00 ± 0.00	1.00 ± 0.00 2.50 ± 0.07	2.07 ± 0.07 3.23 ± 0.07	1.00 ± 0.00 2.53 ± 0.03
[Co(**LH**)$_2$Cl] (3)	0.5 mg/50 µl 1 mg/100µl	1.00 ± 0.00 1.67 ± 0.17	2.00 ± 0.00 3.43 ± 0.23	1.00 ± 0.00 2.50 ± 0.07	4.67 ± 0.17 6.67 ± 0.17
[Ni(**LH**)$_2$](**4**)	0.5 mg/50 µl 1 mg/100µl	1.00 ± 0.00 2.60 ± 0.06	1.00 ± 0.00 2.37 ± 0.09	1.00 ± 0.00 3.00 ± 0.00	1.00 ± 0.00 3.67 ± 0.17
[Cu(**LH**)$_2$](**5**)	0.5 mg/50 µl 1 mg/100µl	0.50 ± 0.00 1.67 ± 0.17	1.00 ± 0.00 2.00 ± 0.00	1.00 ± 0.00 2.67 ± 0.17	2.00 ± 0.00 3.23 ± 0.07
[Zn(**LH**)$_2$](**6**)	0.5 mg/50 µl 1 mg/100µl	1.15 ± 0.02 2.95 ± 0.01	1.10 ± 0.02 2.01 ± 0.01	1.95 ± 0.00 3.05 ± 0.01	2.20 ± 0.02 3.32 ± 0.00

Mean ± SE of zone of inhibition in mm

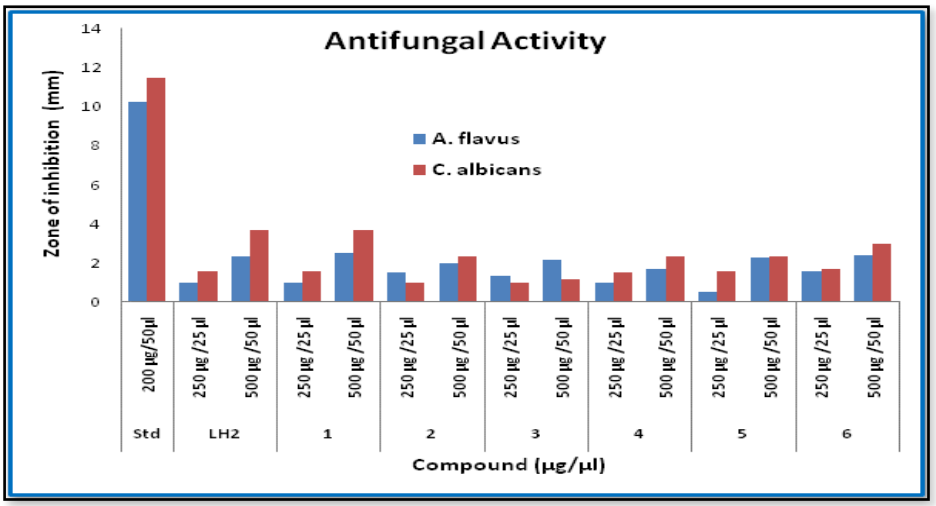


Fig.14 Antifungal activity of **LH_2** and**1-6**

Table 2 Antifungal activity of standard Fluconazole and new compounds **LH_2**and **1-6**.

Compound	**Treatment**	**C. albicans (Mean ± SE)**	**A. flavus (Mean ± SE)**
Fluconazole	0.2 mg/50µl	11.50 ± 0.06	10.27 ± 0.03
LH_2	0.25 mg /25 µl 0.5 mg /50 µl	1.57 ± 0.03 3.67 ± 0.03	1.00 ± 0.00 2.33 ± 0.33
[Mn(**LH**)$_2$](**1**)	0.25 mg /25 µl 0.5 mg /50 µl	1.57 ± 0.03 3.67 ± 0.03	1.00 ± 0.00 2.50 ± 0.06
[Fe(**LH**)$_2$Cl] (**2**)	0.25 mg /25 µl 0.5 mg /50 µl	1.00 ± 0.00 2.33 ± 0.33	1.50 ± 0.06 2.00 ± 0.00
[Co(**LH**)$_2$Cl] (3)	0.25 mg /25 µl 0.5 mg /50 µl	1.00 ± 0.00 1.20 ± 0.00	1.37 ± 0.03 2.17 ± 0.03
[Ni(**LH**)$_2$](**4**)	0.25 mg /25 µl 0.5 mg /50 µl	1.50 ± 0.06 2.33 ± 0.03	1.00 ± 0.00 1.67 ± 0.03
[Cu(**LH**)$_2$](**5**)	0.25 mg /25 µl 0.5 mg /50 µl	1.60 ± 0.03 2.33 ± 0.03	0.55 ± 0.00 2.27 ± 0.03
[Zn(**LH**)$_2$](**6**)	0.25 mg /25 µl 0.5 mg /50 µl	1.82 ± 0.01 3.01 ± 0.02	1.60 ± 0.00 2.41 ± 0.02

Mean ± SE of zone of inhibition in mm

4. Conclusions

A new ligand of Schiff's base **LH_2** has been synthesized by 2-(3,4-dimethoxyphenyl) ethan-1-amine condensed with 2,4-dihydroxy benzaldehyde and their metal complexes, **1-6** were synthesized. The **LH_2** and **1-6** were well known from CHN analysis and spectroscopic investigation. The antimicrobial and antibacterial activities of **LH_2** and **1-6** were assessed and

results were noteworthy exercises. The microbial development restraint act of ligand has been expanded on complexation.

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