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Modern spectroscopic techniques in the characterization of Schiff base macrocyclic ligand and its complexes with transition metals

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Abstract

Mn(II), Co(II), Ni(II), and Cu(II) complexes with a new azamacrocyclic tetradentate [N_4] ligand i.e. 2,3,9,10-tetraphenyl;l,4,8,11-tetraazacyclotetradeca;1,3,8,10-tetraene (L) have been synthesized and characterized by elemental analysis, molar conductance measurements, magnetic susceptibility measurements, mass, ^{1}HNMR, IR, electronic and EPR spectral studies. On the basis of their non-electrolytic nature, the probable formula of the complexes is proposed to be [$M(L)X_2$], where M = Mn(II), Co(II), Ni(II), and Cu(II), X = Cl^- and $NO_3{}^-$, in dimethylformamide (DMF). All the complexes are of high-spin type and found to have six coordinated, octahedral geometry for Mn(II), Co(II), and Ni(II) complexes, and tetragonal for Cu(II) complexes. Macrocyclic ligand and its complexes have also been screened against pathogenic bacteria and fungi in vitro as growth inhibiting agent.

Keywords: Spectroscopic technique; Schiff base macrocyclic ligand; Transition metals

1. Introduction

Synthetic macrocycles are a growing class of compounds with varying chemistry a wide range of different molecular topologies and sets of donor atoms [1–5]. The chemical properties of macrocyclic complexes can be tuned to force metal ions to adopt unusual coordination geometry. Transition metal macrocyclic complexes have received much attention as a active part of metalloenzymes [6] as biomimic model compounds [7] due to their resemblance with natural proteins like hemerythrin and enzymes. In view of the above, in the present paper, we report the synthesis and characterization of macrocyclic Mn(II), Co(II), Ni(II), and Cu(II) complexes of a 14-membered tetradentate (N_4) macrocyclic ligand viz. 2,3,9,10-tetraphenyl;1,4,8,11-tetraazacyclotetradeca;l,3,8,10-tetraene (L) Fig. 1. These complexes are characterized by elemental analysis, molar conductance, magnetic susceptibility measurements, mass, ^{1}HNMR, IR, UV/visible and EPR spectral studies.

2. Experimental

All the fine chemicals used were of AnalaR grade, and procured from Fluka. Metal salts were purchased from E. Merck and were used as received. All solvents were purified before use according to standard procedures.

2.1. Synthesis of ligand

Hot ethanolic solution (20 mL) of benzil (9.30 g, 0.05 mol), and a hot ethanolic solution (20 mL) of 1,3-diaminopropane (3.70 g, 0.05 mol) were mixed slowly with constant stirring. This mixture was refluxed at 80 °C for 6 h in the presence of few drops of concentrated hydrochloric acid. On cooling, white colored precipitate was formed. It was filtered, washed with cold EtOH, and dried under vacuum over P_4O_{10}. Yield 84%, m.p. 237 °C. Elemental analysis found % C 82.27; H 6.48; N 11.25. Calculated for $C_{34}H_{32}N_4$ (atomic mass calculated 496) was C 82.26; H 6.45 and N 11.29%.

2.2. Synthesis of complexes

Hot ethanolic solution (20 mL) of the ligand (0.992 g, 0.002 mol) and hot ethanolic solution (20 mL) of a given

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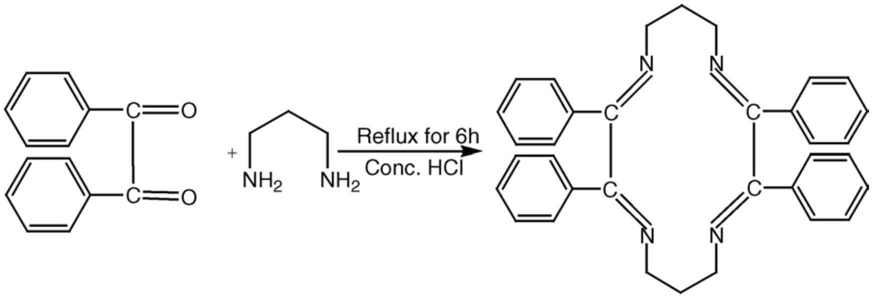

Fig. 1. Structure of the ligand (L).

metal salt (0.001 mol) were mixed together with constant stirring. The mixture was refluxed for 6–9 h at 79–80 °C. On cooling, colored complex precipitated out. It was filtered, washed with cold EtOH and dried under vacuum over P_4O_{10}.

2.3. Physical measurements

The C, H and N were analysed on a Carlo-Erba 1106 elemental analyzer. Molar conductance was measured on a ELICO (CM82T) conductivity bridge. Magnetic susceptibility was measured at room temperature on a Gouy balance using $CuSO_4 \cdot 5H_2O$ as a callibrant. Electron impact mass spectra were recorded on JEOL, JMS, DX-303 mass spectrometer. ^{1}HNMR spectra were recorded on Hitachi FT-NMR, model R-600 spectrometer, using DMF (spectroscopic grade) as solvent. Chemical shifts are given in ppm relative to tetramethylsilane. IR spectra (KBr) were recorded on a Perkin Elmer FTIR Spectrum BX-II spectrophotometer. The electronic spectra were recorded in DMF on a Shimadzu UV mini-1240 spectrophotometer. EPR spectra of the complexes were recorded as polycrystalline sample and in the solution of DMF, at liquid nitrogen temperature for Co(II) and at room temperature for Mn(II) and Cu(II) complexes on E_4-EPR spectrometer using the DPPH as the *g*-marker.

3. Results and discussion

3.1. Ligand

The electron impact mass spectrum of the ligand (L) confirm the probable formula by showing a peak (Fig. 2) at 495 amu, corresponding to the macrocyclic moiety $(C_{34}H_{32}N_4)^+$. In the ^{1}HNMR spectrum of the ligand, the presence of the middle CH_2 protons (4H) is confirmed by the appearance of a signal at δ 1.9–2.2 [8], an expected quintet. The presence of a multiplet in the region δ 7.38 may be assigned for the benzenoid hydrogen (20H). For $-CH_2-N=$ (8H), the signal as a multiplet at δ 7.00–7.19 ppm was observed. In the IR spectrum of ligand (L) the absence of bands around ~3400 cm^{-1} characteristic for hydroxyl group and at ~1720 cm^{-1} for free carbonyl group suggest complete condensation of the two reactants and elimination of water molecule. The appearance of bands at 1375 and 1428 cm^{-1} indicate the $C-CH_3$ and $-CH_2-$ groups present in the ligand. The characteristic IR band due to the phenyl rings is present in the ligand and complexes in the region ~700–770 cm^{-1}. The band at 1589 cm^{-1} may be assigned to ν (C=N). The shifting in the band of ν (C=N) toward the lower side in the complexes indicates that the coordination takes place through the nitrogen of the ν (C=N) group, thus implying that the ligand (L) is tetradentate.

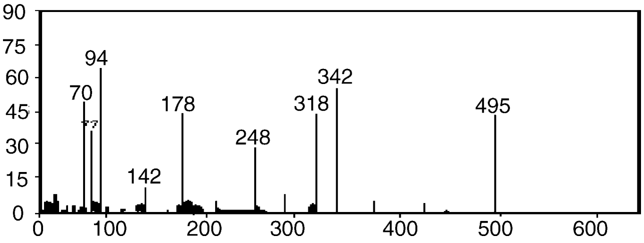

Fig. 2. Electron impact mass spectrum of the ligand (L).

3.2. Complexes

On the basis of elemental analysis the complexes were assigned to possess the composition listed in Table 1. The molar conductance measurements of the complexes in DMF correspond to be nonelectrolytic nature of the complexes. Thus, the complexes may be formulated as $[M(L)X_2]$ where M = Mn(II), Co(II), Ni(II) and Cu(II) and L is 2,3,9,10-tetraphenyl;l,4,8,11-tetraazacyclotetradeca;l,3,8,10-tetraene and X = Cl^- and $NO_3{}^-$.

3.3. Infra-red spectral bands due to anion

IR spectra of the nitrato complexes Fig. 3(a–c), display three, medium intensity bands, due to (N–O) stretching in the region ~1418–1427 cm^{-1} (ν_5), ~1305–1311 cm^{-1} (ν_1) and ~1003–1012 cm^{-1} (ν_2). The difference between ν_5 and ν_1, which is 107–119 [9], suggesting that both the nitrate groups are coordinated to the central metal ion.

4. Manganese(II) complexes

The Mn(II) complexes show magnetic moments corresponding to five unpaired electrons (5.87–5.94 B.M.)

Table 1
Molar conductance and elemental analysis data of the complexes

Compound	Molar conductance Ω^{-1} cm^2 mol^{-1} (±2%)	Colour	M.P. (°C)	Yield (%)	Elemental analysis data found (calculated) (%)			
					Metal	C	H	N
$C_{34}H_{32}N_4$ (ligand)	–	Off white	237	84	–	82.27 (82.26)	6.48 (6.45)	11.25 (11.29)
[Mn(L)Cl_2] $C_{34}H_{32}MnN_4Cl_2$	14.63	Light pink	292	69	8.65(8.78)	65.73 (65.78)	5.12 (5.15)	8.93 (8.95)
[Mn(L)$(NO_3)_2$] $C_{34}H_{32}MnN_6O_6$	15.78	Dark brown	248	71	8.83 (8.87)	65.78 (65.81)	5.19 (5.16)	13.51 (13.55)
[Co(L)Cl_2] $C_{34}H_{32}CoN_4Cl_2$	18.30	Mauve pink	270	75	9.39 (9.35)	65.33 (65.36)	5.17 (5.12)	8.93 (8.89)
[Co(L)$(NO_3)_2$] $C_{34}H_{32}CoN_6O_6$	14.60	Blue	240	68	8.61 (8.63)	60.23 (60.29)	4.69 (4.72)	12.76 (12.71)
[Ni(L)Cl_2] $C_{34}H_{32}NiN_4Cl_2$	9.80	Brown	295	69	9.25 (9.32)	65.44 (65.39)	5.07 (5.12)	8.83 (8.90)
[Ni(L$XNO_3)_2$] $C_{34}H_{32}NiN_6O_6$	10.4	Light brown	288	77	8.53 (8.60)	60.27 (60.31)	4.77 (4.73)	12.36 (12.31)
[Cu(L)Cl_2] $C_{34}H_{32}CuN_4Cl_2$	9.50	Green	285	78	9.93 (10.01)	64.85 (64.89)	5.03 (5.08)	8.79 (8.83)
[Cu(L$XNO_3)_2$] $C_{34}H_{32}CuN_6O_6$	10.70	Dark brown	>300	83	9.32 (9.24)	59.83 (59.89)	4.63 (4.69)	12.17 (12.22)

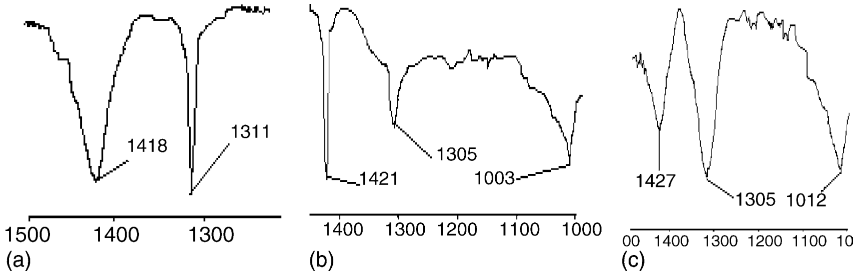

Fig. 3. IR spectral bands of anions (a) [Co(L)$(NO_3)_2$]; (b) [Ni(L)$(NO_3)_2$]; (c) [Cu(L)$(NO_3)_2$].

at room temperature (Table 2). These values are close to the spin only value of 5.92 B.M. Electronic spectra of the Mn(II) complexes display four weak absorption bands ($\varepsilon = 43–121$ L mol^{-1} cm^{-1}) (Table 2) characteristic of the octahedral geometry [10]. In these complexes, these bands may be assigned to the transitions: $^6A_{1g} \rightarrow {}^4T_{1g}$ (^{4}G), $^6A_{1g} \rightarrow {}^4E_g$, $^4A_{1g}$ (^{4}G) ($10B + 5C$), $^6A_{1g} \rightarrow {}^4E_g$ (^{4}D) ($17B + 5C$) and $^6A_{1g} \rightarrow {}^4T_{1g}$ (^{4}P) ($7B + 7C$), respectively.

The calculated values of parameters B, C, Dq and β are listed in Table 3. The values of B and C were calculated by using the ν_2 and ν_3 transitions. This is due to the fact the energies of these two transitions are independent of the crystal field splitting energy and depend only on B and C parameters.

The EPR spectra of the complexes have been recorded as polycrystalline sample and in DMF solution. Polycrystalline

Table 2
Magnetic moment and electronic spectral data of the complexes

Complex	μ_{eff} B.M. (±2%)	λ_{max} (cm^{-1})	ε (L mol^{-1} cm^{-1})
[Mn(L)Cl_2]	5.87	17698, 25090, 29279, 32421	43, 53, 67, 121
[Mn(L)$(NO_3)_2$]	5.97	18109, 24345, 28974, 32198	46, 49, 61, 115
[Co(L)Cl_2]	4.98	8970, 14410, 20110	67, 89, 94
[Co(L)$(NO_3)_2$]	5.01	9722, 14278, 20456	69, 83, 98
[Ni(L)Cl_2]	2.98	10065, 16065, 24513	33, 87, 126
[Ni(L)$(NO_3)_2$]	2.96	10238, 15792, 24284	37, 79, 121
[Cu(L)Cl_2]	1.97	14224, 20484, 22697	74, 93, 159
[Cu(L)$(NO_3)_2$]	2.01	14480, 20596, 23144	78, 96, 162

Table 3
Ligand field parameters of the complexes

Complex	Dq (cm^{-1})	B (cm^{-1})	C (cm^{-1})	β	F_4	F_2	h_x	LFSE (kJ mol^{-1})
[Mn(L)Cl_2]	1769.80	598	3822	0.76	109	1143	3.43	–
[Mn(L)$(NO_3)_2$]	1810.90	661	3547	0.84	101	1166	2.29	–
[Co(L)Cl_2]	1050.14	1093.90	–	0.87	–	–	–	10.37
[Co(L)$(NO_3)_2$]	1014.44	1056.71	–	0.84	–	–	–	96.96
[Ni(L)Cl_2]	1006.50	692.20	–	0.66	–	–	–	144.30
[Ni(L)$(NO_3)_2$]	1023.80	624.10	–	0.60	–	–	–	146.70

Table 4
EPR spectral data of the complexes

Complexes	Polycrystalline sample				In DMF solution			
	$g_{\|}$	$g_{\perp}$	g_{iso}	G	$S_{\|}$	$g_{\perp}$	g_{iso}	G
$[Mn(L)Cl_2]$[a]	–	–	2.03	–	–	–	2.02	–
$[Mn(LXNO_3)_2]$[a]	–	–	2.08	–	–	–	2.06	–
$[Co(L)Cl_2]$[b]	2.17	2.11	2.13	1.54	2.13	1.92	1.99	1.62
$[Co(L)(NO_3)_2]$[b]	2.13	2.09	2.10	1.44	2.10	1.93	1.99	1.43
$[Cu(L)Cl_2]$[a]	2.18	2.05	2.09	3.60	2.14	2.05	2.06	2.80
$[Cu(L)(NO_3)_2]$[a]	2.16	2.12	2.13	1.33	2.12	2.04	2.05	3.00

[a] At room temperature.

[b] At liquid N_2 temperature.

samples gives one broad isotropic signal centered at approximately the free electron 'g' value i.e. 2.0023 (Table 4) [10].

In DMF solution, Mn(II) complexes give EPR spectra Fig. 4, containing the six lines arising due to the hyperfme interaction between the unpaired electron with the ^{55}Mn nuclear (l = 5/2). The nucler magnetic quantum number M_1, corresponding to these lines are −5/2, −3/2, −1/2, +1/2, +3/2 and +5/2 from low to the high field.

5. Cobalt(II) complexes

The magnetic moment measurements of the cobalt(II) complexes at room temperature lie in the range 4.98–5.01 B.M.[11]. The electronic spectra of the Co^{2+} complexes are given in Fig. 5(a) and (b) and show three bands, characteristic of an octahedral geometry. These bands may be assigned to $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ (ν_1), $^4T_{lg} \rightarrow {}^4A_{2g}$ (ν_2) and $^4T_{lg}(F) \rightarrow {}^4T_{1g}(P)$ (ν_3) transitions, respectively [12,13]. Various ligand field parameters have been calculated [14,15] for the cobalt(II) complexes on the basis of electronic spectra and are listed in the Table 3.

The $g_{\|}$ values of the Co(II) complexes, EPR spectra recorded at liquid N_2 temperature as polycrystalline and in DMF solution, lie in the range 2.10–2.17 and the $g_{\perp}$ values are in the range 1.92–2.11 (Table 4). The deviation of the 'g' values from the spin only value (g = 2.0023) is due to the angular momentum contribution.

6. Nickel(II) complexes

The magnetic moment of the Ni(II) complexes at room temperature lie in the range 2.96–2.98 B.M. These values are in tune with a high spin configuration [16,17] and show the presence of an octahedral environment around the Ni(II) ion in all the complexes reported in this paper.

These complexes display three electronic spectral bands Fig. 5(c) and (d), at ∼10065–10238 (ν_1), 15792–16065 (ν_2) and 24284–24513 cm^{-1} (ν_3). These may be assigned to the three spin allowed transitions $^3A_{2g}(F) \rightarrow {}^3T_{2g}$ (F) (ν_1), $^3A_{2g}(F) \rightarrow {}^3T_{1g}$ (F) (ν_2), $^3A_{2g}(F) \rightarrow {}^3T_{1g}$ (P) (ν_3), respectively. These bands indicate that the complexes have an octahedral geometry and might possess D_{4h} symmetry. The ν_2/ν_1 ratios lie in the range 1.47–1.66.

7. Copper(II) complexes

Magnetic moment of all the Cu(II) complexes at room temperature lie in the range 1.97–2.01 B.M. corresponding to one unpaired electron [18,19].

Three spin allowed transitions may be expected in the visible region and the E_g and T_{2g} levels of ^2D free ion will split into B_{1g}, A_{1g}, B_{2g} and E_g levels, respectively.

Electronic spectra of six coordinated copper complexes display bands in the range 14224–14480 (ν_1), 20484–20596 (ν_2) and 22697–23149 cm^{-1} (ν_3), correspond-

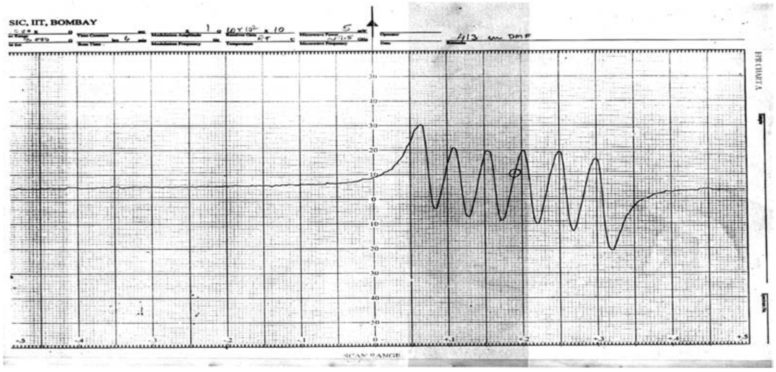

Fig. 4. EPR spectra of $[Mn(L)Cl_2]$ recorded at room tempemture/DMF.

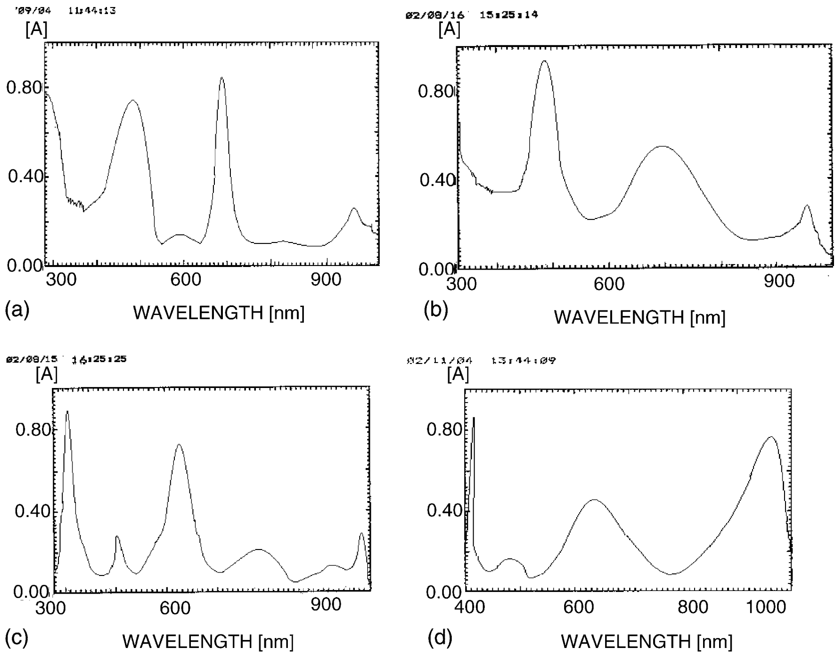

Fig. 5. Electronic spectra of the complexes (a) [Co(L)Cl$_2$]; (b) [Co(L)(NO$_3$)$_2$]; (c) [Ni(L)Cl$_2$]; (d) [Ni(L)(NO$_3$)$_2$].

ing to the following transitions $^2B_{1g} \rightarrow {}^2A_{1g}$ ($d_{x^2-y^2} \rightarrow d_{z^2}$) ν_1, $^2B_{1g} \rightarrow {}^2B_{2g}$ ($d_{x^2-y^2} \rightarrow d_{zy}$) ν_2 and $^2B_{1g} \rightarrow {}^2E_g$ ($d_{x^2-y^2} \rightarrow d_{zy}, d_{yz}$) ν_3 (Table 2).

The EPR spectra of Cu(II) complexes were recorded on X-Band at frequency 9.5 GHz under the magnetic field strength 3200 band scan rate 2000, recorded at room temperature. The EPR spectra (room temperature/DMF) of the complexes exhibit a single anisotropic broad signal, while splitting occurs in the solution phase, Fig. 6(a) and (b). The absence of the band for the $\Delta Ms = \pm 2$ transition can explained by proposing the interaction between two paramagnetic centers are negligible [20,21]. The spectra give $g_{\|}$ value in the range 2.12–2.18 and $g_{\|}$ values in the range 2.04–2.12. The observed $g_{\|}$ value for the Cu(II) complexes are less than 2.3, in agreement with the covalent character of the M–L bond. The ratio $g_{\|} > g_{\perp} > 2.0023$ calculated for Cu(II) complexes, suggest that the unpaired electron is localized in $d_{x^2-y^2}$ orbital and the spectral features are characteristic of axial geometry and tetragonally elongated geometry.

8. Ligand field parameters

Various ligand field parameters were calculated for the complexes and are listed in Table 3. The values of Dq in Co(II) complexes were calculated from transition energy ratio diagram using the ν_3/ν_2 ratio. Our results are in agreement with the complexes reported earlier [5,15]. The nephelauxetic parameter β is readily obtained by using the relation:

$$\beta = \frac{B(\text{complex})}{B(\text{free ion})}$$

where B (free ion) for Mn(II) is 786, for Ni(II) 1041 and for Co(II) 1120 cm^{-1} [10,11]. The β values lie in the range of 0.60–0.87. These values indicate the appreciable covalent character of metal ligand ‘σ’ bond.

9. Biological screening

The macrocyclic ligand and its transition metal complexes have been screened against a number of pathogenic bacte-

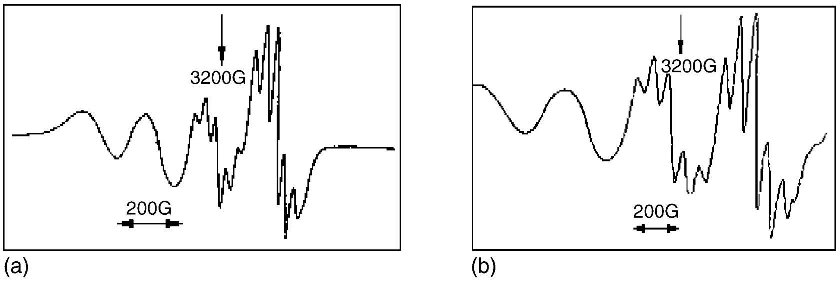

Fig. 6. EPR spectra recorded at R.T. in the solution of DMF. (a) [Cu(L)Cl$_2$]; (b) [Cu(L)(NO$_3$)$_2$].

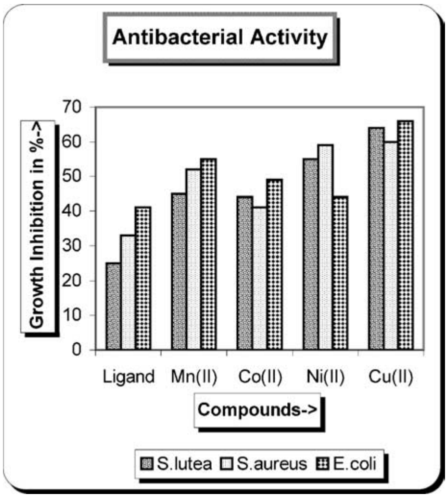

Plate 1. Antibacterial screening.

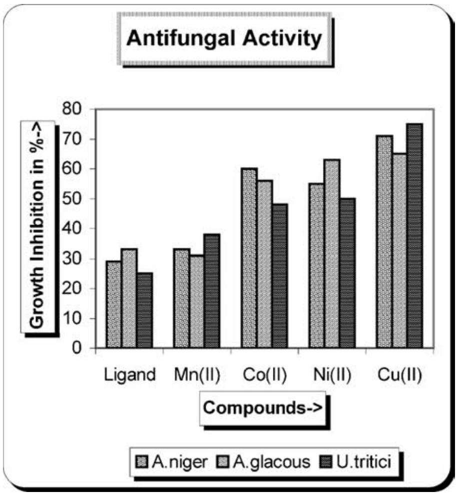

Plate 2. Antifungal screening.

ria and plant pathogenic fiingi. For the study of antibacterial activity the disc diffusion technique [22] was adopted on the bacterial species, i.e. *Sarcina lutea* (gram positive), *Staphylococcus aureus* and *Escherchia coli* (gram negative), in vitro.

The results of the antibacterial study shown in Graph 1, suggest the maximum inhibition capacity of the copper(II) complexes, although all complexes are more efficient to inhibit the bacterial growth as compare to the metal free ligand.

Antifungal screening of the compounds was done in vitro on *Aspergilus niger, Aspergilus glaucus* and *Ustilago tritici* by using the agar plate technique [22,23]. The results of the antifungal activity has been compared with the results reported earlier [24,25]. Maximum fungal growth inhibition was shown by the copper(II) complexes (Graph 2) and the minimum by the manganese(II) complexes.

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