

Synthesis, Composition, Spectral, Geometry and Antibacterial Applications of Mn^{II} , Ni^{II} , Co^{II} , Cu^{II} and Hg^{II} complexes of N_2O_2 -mixed donor Schiff Base with 1,10-phenanthroline

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Abstract: $Mn(II)$, $Ni(II)$, $Co(II)$, $Cu(II)$ and $Hg(II)$ mixed complexes of Schiff base ligand with 1,10-phenanthroline were synthesised. The structural characterization of Schiff base derived from ethylene diamine and 2-benzoyl benzoic acid and its mixed ligand complexes, structural characterization of Schiff base and its mixed complexes were done on the basis of their melting point, magnetic sensitivity, solubility, conductivity techniques, UV-Visible, FTIR spectroscopy, and elemental analyses studies. These measurements suggest the following molecular formulas for the metal complexes are octahedral configuration with the formula; $[M(PHN)(L)]$ where $M=Mn(II)$, $Ni(II)$, $Co(II)$, $Cu(II)$ and $Hg(II)$; $PHN=1,10$ -phenanthroline and $L=$ Schiff base ligand. The Schiff base and mixed ligand complexes were preliminarily scanned against different strains of bacteria and fungi to study their biological effect.

Keywords: Mixed complexes, ethylene diamine, composition and biological effect.

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I. Introduction

Compounds including (-CH=N-) an azomethine group, recognised as ligand called (Schiff bases) are established by the condensation of a carbonyl with compounds such as a primary amine.¹ Schiff bases of aromatic aldehydes are very stable due to they have an active conjugation framework while those of aliphatic aldehydes, comparatively readily polymerizable and are unstable.² Schiff bases have a numerous of uses such as identification, preparative application, discovery and measurement of ketones or aldehydes, purity of amino or carbonyl compounds, the safeguard of these groups during sensitive or complex reactions.³⁻⁵ As well as they compose basic modules in some dyes and used as liquid crystals. In general, Schiff bases are bi-, tri- or multidentate ligands capable of making very stable complexes with transition metals.⁶⁻⁷ Synthesis of inorganic compounds is beneficial reactions of Schiff base to make bonds between nitrogen and carbon.⁸ Schiff bases show to be an essential intermediate in many of enzymatic reactions including the interaction of a carbonyl or an amino group of the substrate with an enzyme.⁸⁻¹⁰ The most important fundamental kind of catalytic technicality is the biochemical operation that includes the condensation of a carbonyl group of the substrate with a primary amine in an enzyme to form Schiff base or an imine.¹¹ The condensation of an aldehyde and an amine to give an azomethine group is recognised to be double-faced; the disposal of water to get a perfect major reaction and good produce.¹² Manufacturing production and utilisation of Ni, Fe, Zn, Cu, Co, Pb and Cd elements may purpose environmental pollution.^{13,14} Furthermore, some of these metals ions are existent as fundamental elements in tracing amounts for the biological frame and them in bioinorganic chemistry have very importance through structural studies in a biological system.^{15,16} The oxygen, sulphur and nitrogen donor atoms supply a multitude of binding possibilities.¹⁷ Both the ligand and their metal complexes show a broad scope of biological activity.^{18,19} The target of this study is to illustrate properties and the arrangement structures of 1,10-phenanthroline and the Schiff base derived from 2-benzoylbenzoic acid and ethylene diamine in coordinating with divalent transition metal ions Copper, Manganese, Cobalt, Mercury, and Nickel.

II. Experimental

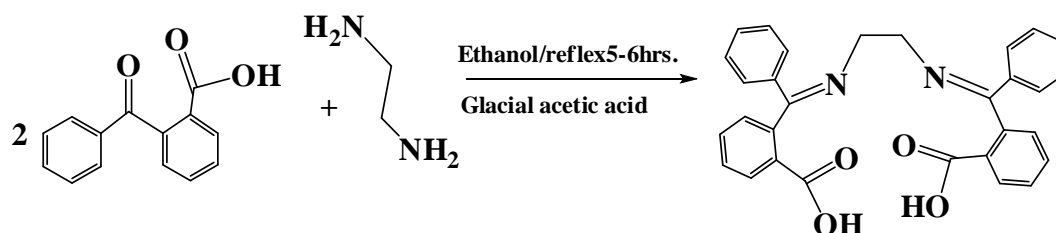
2.1 Chemicals

$C_6H_5COC_6H_4CO_2H$ (BDH), $(CH_3)_2CO$ (BDH), $NH_2CH_2CH_2NH_2$ (BDH), KOH (Fluka), $CH_3)_2SO$ (Fluka), CH_3COOH (BDH), CH_3OH (BDH), $C_{12}H_8N_2$ (Merck), $(CH_3)_2NC(O)H$ (Fluka), $CoCl_2 \cdot 6H_2O$ (Merck), $NiCl_2 \cdot 2H_2O$ (BDH), $HgCl_2$ (BDH), $CuCl_2 \cdot 2H_2O$ (BDH) and $MnCl_2 \cdot 4H_2O$ (BDH).

2.2 Methods: Micro elemental analysis (C, H and N %) of the synthesised compounds was carried out utilising a CHN Analyzer on Perkin Elmer 2400 series II. 1H & ^{13}C -NMR spectra of the compounds were registered utilising {Bruker spectros pinultrashield magnets 300 MHz} instruments utilising as a solvent was DMSO- d_6 . Magnetic susceptibility measurements were obtained utilising Bruker BM6 at 298°K. An FT-IR spectrum was registered on {SHIMADZU FTIR-8400 spectrophotometers as KBr disc}. Electronic spectra were registered utilising {U.V-Vis. spectrophotometer type CECIL, England}, in a $10^{-3}M$ solution of ethanol in range (200-1000) nm at room temperature. The suggested molecular frames of the compounds were drawing by utilising { chem. Office prog.3DX (2006)}.

2.3 Synthesis of Ligand/ H_2L /(S1)

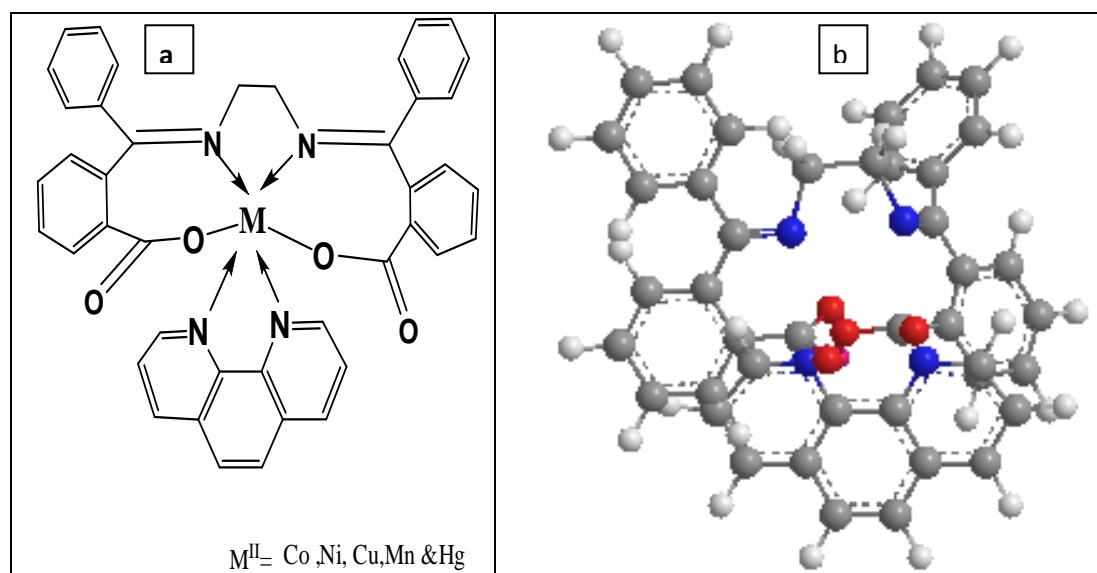
A solution of (0.02 mole) $C_6H_5COC_6H_4CO_2H$ in (10 ml) C_2H_5OH with 5 drops from CH_3COOH was appended dropwise of (0.01 mole) $NH_2CH_2CH_2NH_2$ in (10 ml) C_2H_5OH . The reaction mixture was heated at 70°C under reflux for 5hrs. and then to 25°C. The product was cooled, washed with hot C_2H_5OH and purified by recrystallization from $C_2H_5OC_2H_5$ (1:2), as shown in Scheme (1).



Scheme (1) Synthetic route of the (S1)

2.4 Synthesis of the mixed-Schiff base ligand (S1) and 1,10-phenanthroline (S2) complexes with some metal ions

To a solution of (MCl_2) metal salt (0.01mmol) in 10ml C_2H_5OH , was added a solution of (S1) (0.01mmole) in (12) ml C_2H_5OH . This solution was adjusted to pH = 7 utilising (10%) C_2H_5OH solution of KOH. Finally, a solution of (0.01 mmole) (S1) in (10) ml C_2H_5OH was also added, the resulting mixture was heated under reflux for (1.5) hrs. Then the medley was filtered and the precipitate was washed with a more quantity of C_2H_5OH and recrystallized from ($CH_3OH:C_2H_5OC_2H_5$) (1:2) solvents. (Scheme 2).



Fig(1):Molecular structure of (a)(H_2L)of the metal chelate complexes

III. Results and Discussion

Generally, the preparation (Scheme 1) includes the condensation of $C_6H_5COC_6H_4CO_2H$ with $NH_2CH_2CH_2NH_2$ in C_2H_5OH . The reaction of metallic salts with (S1) and (S2) in C_2H_5OH as solvent at room temperature produced the regarded mixed complexes. The mixed complexes were synthesized by reacting the metal chlorides with the two ligands using 1:1:1 mole ratio, i.e. one mole of metal chloride: one mole of (S1) and

one mole of (S2) complexes were studied in various solvents which showed that all complexes are soluble in (CH₃)₂SO and (CH₃)₂NC(O)H while they are insoluble in H₂O and common solvents. The experimental and calculated values of elements and metal percentage in the mixed complex are in fair agreement can be found in Table (1). The test for chloride ion with AgNO₃ solution was negative (Nil %) indicating that there is no chloride ion outside the coordination sphere of the central metal.³ Molar conductance's (Λ_m) in 10⁻³M DMSO solutions of the mixed complexes lie in range (10-17) ohm⁻¹cm²mol⁻¹ that very low range supporting their non-electrolytic behaviour.⁴

3.1 NMR spectrum for Schiff base ligand (S1): The ¹H and ¹³C NMR, spectra of Schiff base ligand (S1) are consistent with structure. In the ¹H NMR spectrum, shown in Fig.(2), in Table (2) the singlet signal was observed at ($\delta=2.50ppm$) is attributed to the proton of DMSO. The singlet signal at ($\delta=6.02ppm$) refers to the CH₂proton.⁵ The multiple signals were observed at ($\delta=6.87-7.67ppm$) are assigned to protons of aromatic rings. The spectrum also showed the singlet signal at ($\delta=12.67ppm$) which can be assigned to (COOH) proton of the carboxylic group.⁶

In the ¹³C NMR spectra, shown in Fig.(3), in Table (3) the singlet signal was observed at ($\delta=40.31ppm$) is assigned to the carbon of DMSO. The multiple signals were observed at the range ($\delta=114.64-129.27ppm$) are assigned to carbons of aromatic rings.⁷ The signal at ($\delta=143.50ppm$) refers to the N=C carbon of azomethine group.⁸ The signal was observed at ($\delta=47.41-55.78ppm$) is assigned to the carbon of CH₂ group in NH₂CH₂CH₂NH₂. The spectrum also showed the singlet signal at ($\delta=161.07ppm$) which can be assigned to (COOH) carbon of carboxylic group.⁸

3.2 The IR spectrum studies

The IR spectrum of (S2) fig.(5) appeared the bands at (1620)cm⁻¹ and (1549)cm⁻¹ attributed to stretching vibrations of $\nu(C=N)$ and $\nu(C=C)$, respectively.⁹ The IR spectra of the ligand fig.(4) exhibited broadband at (3337)cm⁻¹, which was attributed to the stretching vibration of $\nu(OH)$.¹⁰ In the spectra of all mixed complexes, this absorption was disappeared, that expressed deprotonation and participation of the enol oxygen in coordination.¹¹ The (FT-IR) spectrum for (S1) exhibited band at (1627) cm⁻¹ is due to stretching vibration of azomethine group $\nu(C=N)$. This band has been shifted to lower frequencies (1610), (1608), (1603), (1601) and (1598) cm⁻¹ in the spectra of the (1), (2), (3), (4) and (5) complexes respectively.¹² This shift can be attributed to the coordination of nitrogen atom of the azomethine group to the metal atom. The spectrum of (S2) exhibit band at (1620)cm⁻¹ due to the stretching vibration of azomethine group $\nu(C=N)$ which has been shifted to lower frequencies (1600), (1597), (1589), (1587) and (1581)cm⁻¹ for (1),(2),(3),(4) and (5) complexes respectively, indicating the coordination through nitrogen atom of $\nu(C=N)$ group.¹³ The new bands at (497,468), (486,443), (545, 462), (531,478) and (527, 441)cm⁻¹ were assigned to $\nu(M-N)$ and $\nu(M-O)$ for complexes (1), (2), (3), (4) and (5) respectively, indicating that the nitrogen of azomethine group and the oxygen of ligand (S1) are involved in chelation with metal ions together with nitrogen of (S2).¹⁴ Additional bands and their assignments can be found in Table (4)

3.3 The Electronic spectrum

The electronic spectrum for (S1), exhibits two peaks at (265nm) and (332nm) which are attributed to ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) transitions, respectively.¹⁵ (1) complex, exhibits 5 peaks, the first high intense peak at (260nm) is due to the (L.F), while the second peak at (341nm) is due to the (C.T). The third weak peak at $\nu_3 =$ (498 nm) which assigned to ${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$. The fourth peak at $\nu_2 =$ (703 nm) which assigned to ${}^4T_{1g(F)} \rightarrow {}^4A_{2g(F)}$ transition. The fifth peak at $\nu_1 =$ (867nm) which assigned to ${}^4T_{1g(F)} \rightarrow {}^4T_{2g(F)}$ transition. The room temperature magnetic moment ($\mu_{eff} = 4.86B.M$) corresponded to a high spin octahedral symmetry.¹⁶ (2) complex, exhibits four peaks, the first and second high intense peak at (268 nm) and (335nm) is due to the (L.F) and (C.T) transitions, while the third peak at (662 nm) which assigned to ${}^3A_{2g(F)} \rightarrow {}^3T_{1g(F)}(\nu_3)$ (d-d) transition. The fourth peak at (789nm) which assigned to $({}^3A_{2g(F)} \rightarrow {}^3T_{1g(F)}(\nu_2))$ transition, in an octahedral geometry.¹⁷ The complex exhibited a value of $\mu_{eff} = 2.73B.M$, which suggests an octahedral geometry around the central Ni ion.¹⁸ (3) complex, exhibits three peaks, the first and second high intense peaks at (271 nm) and (338 nm) are due to the (L.F) and (C.T) transitions. The third weak peak at (686nm) which assigned to $(Eg(F) \rightarrow {}^2T_{2g(F)})$ transition. Hence (3) complex showed an octahedral geometry. The (3) complex displayed a value of ($\mu_{eff} = 1.76B.M$) (4) complex, exhibits three peaks, the first and second high peaks at (269nm) and (336 nm) are due to (L.F) and (C.T) transitions. The third weak peak at (781nm) can be assigned to the ${}^6A_{1g(F)} \rightarrow {}^4Eg(D)(\nu_3)$ transition.¹⁹ Magnetic moment $\mu_{eff} = 5.41 B.M$ at room temperature, this low data the magnetic moments suggest high spin a coordination number of 6 for the central manganese (II) ion and attaining

[an octahedral geometry].²⁰ (5) complex exhibits two high peak at (260 nm) is due to the (L.F), while second and third peaks at (352 nm) and (396 nm) is attributed to the (C.T), in an octahedral geometry. There is no ligand field stabilization effect in Hg (II) ions because of its completed (d¹⁰) shell. This metal ion is diamagnetic and does not possess any d-d transition.²¹

3.4 Biological activities

The ligand (S1) and its mixed complexes have been examined for in vitro growth inhibitory efficiency versus (*Staphylococcus aureus*, *Bacillus*, *Pseudomonas* and *Escherichia Coli*) by utilising {well-diffusion} method. {The minimum inhibitory concentration} (MIC) data of the checked compounds are listed in Table (6), the observed MIC values signal that (1), (2), (3) (4) and (5) mixed complexes have higher antimicrobial and antifungal effectiveness than ligands (S1) and (S2). The growing in biological efficacy is the result of the faster spread of its mixed complexes as a whole [22, 23]. The increased efficacy of metal complexes must be explicated on the foundation of the chelation theory and over tone concept. Approving to this concept of cell permeability, the lipid overlay that perimeter the cell prefers the transit of only {Lipid-soluble materials} in that lipid solubility are a substantial agent that controls the biological efficacy.

IV. Conclusion

A tetradentate Schiff base ligand (H₂L) formed from the condensation of 2-benzoyl benzoic acid with ethylene diamine was synthesised and composited. The metal complexes with Cu^(II), Ni^(II), Hg^(II), Co^(II), and Mn^(II) ions with Schiff base (S1) and (S2) were synthesised and composited. The bonding of the ligand in the metal complexes and the thorough geometry has been concluded on the basis of different spectroscopic mechanics. The relative in vitro antimicrobial and antifungal results suggested that all complexes display a significant antimicrobial and antifungal activity as compared to ligand, (S1) and its mixed (1), (2), (3), (4) and (5) complexes with (S1).

Supplementary Information

All additional information pertaining to characterization of the complexes using ¹H & ¹³C-NMR spectra (figures S2, S3), IR spectra (figures S4, S5), and inhibitory concentrations (MIC) technique (figures 6,7) and (tables S6) are given in the supporting information available at www.ias.ac.in/chemsci.

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Table (1):Elemental analysis and physical properties of (S1) and its mixed complexes

Comp.	Formula	Molecular Weight gm/mol	Colour	Weight gm/mol Yield %	M.P. °C	%Elemental Analysis Found % (Calculated)			
						C	H	N	M
(S1)	$C_{30}H_{24}N_2O_4$	476.52	Pink	0.20g 86%	121	64.98 (75.61)	4.09 (4.54)	12.120 (12.60)	-
(1)	$C_{42}H_{30}CoN_4O_4$	713.65	Brown	0.21g, 66%	222	70.28 (70.69)	4.12(4.24)	7.54 (7.85)	8.11 (8.26)
(2)	$C_{42}H_{30}NiN_4O_4$	713.41	Greenish brown	0.35g, 80%	210	70.43 (70.71)	4.17 (4.24)	7.14 (7.85)	8.14 (8.23)
(3)	$C_{42}H_{30}CuN_4O_4$	70.23	Reddish brown	0.30g, 69%	235	63.12 (63.73)	3.88 (4.10)	12.07 (12.39)	8.67 (8.85)
(4)	$C_{42}H_{30}MnN_4O_4$	709.65	Light brown	0.29g, 75%	217	70.77 (71.08)	4.13(4.26)	7.54 (7.89)	7.35 (7.74)
(5)	$C_{42}H_{30}HgN_4O_4$	855.30	Off-White	0.38g, 81%	226	58.36 (58.98)	3.66 (3.54)	6.05(6.55)	23.15 (23.45)

Table2: 1H -NMR chemical shifts for ligand(S1)(ppm in DMSO)

DMSO	CH ₂	HC=CH	COOH
2.46	6.02	6.68-6.96	12.67

Table3: ^{13}C -NMR chemical shifts for ligand(S1)(ppm in DMSO)

DMSO	CH ₂	HC=CH	C=N	COOH
40.30	64.54	142.69-145.22	166.07	169.69

Table4: Infrared spectral values (wavenumber) cm^{-1} for all the compounds

Compound	$\nu(OH)$	$\nu(C-H)_{arom.}$	$\nu(C-H)_{aliph.}$	$\nu(C=O)_{carboxyl}$	$\nu(HC=N)_{imine}$	$\nu(C=O)_{asy}$	$\nu(C=O)_{sym}$	$\Delta\nu\ cm^{-1}$	M-N M-O
(S1)	3337	3020	2993	1695	1627	-	-	-	-
(S2)	-	-	-	-	1620	-	-	-	-
(1) [Ni(PHN)(L)]	-	3056	2964	-	1610 1600	1556	1458	98	497 468
(2) [Mn(PHN)(L)]	-	3073	2924	-	1608 1597	1543	1451	92	486 443
(3) H2L	-	3041	2973	-	1603 1589	1563	1442	121	545 462
(4) [Ni(PHN)(L)]	-	3052	2910	-	1601 1587	1558	1453	105	531 478
(5)	-	3062	2962	-	1598 1581	1548	1463	85	527 441

Table5: UV-Vis spectral data of all the compounds

Compound	λ_m ohm.cm ² mole ⁻¹	μ_{eff}	λ_{nm}	ν wavenumber cm^{-1}	Assignments	Proposed structure
(S1)	-	-	265	37735	$\pi \rightarrow \pi^*$	-
			332	30120	$n \rightarrow \pi^*$	
(S2)	-	-	202	49504	789	-
			228	43859	1992	
			264	37878	1345	
(1)	17	4.86	260	38461	L.F	Octahedral

			341	29325	C.T	
			498	20080	$4T_{1g}(F) \rightarrow 4T_{1g}(P)$	
			703	14224	$4T_{1g}(F) \rightarrow 4A_{2g}(F)$	
			867	11534	$4T_{1g}(F) \rightarrow 4T_{2g}(F)$	
(2)	10	2.73	268	37313	L.F	Octahedral
			335	29850	C.T	
			662	15105	$3A_{2g}(F) \rightarrow 3T_{1g}(F)$	
			789	12674	$3A_{2g}(F) \rightarrow 3T_{2g}(F)$	
(3)	15	1.76	271	36900	L.F	Octahedral
			338	29585	C.T	
			686	14577	$E_g(F) \rightarrow 2T_{2g}(F)$	
(4)	12	5.41	269	37174	L.F	Octahedral
			336	29761	C.T	
			781	12804	$6A_{1g}(F) \rightarrow 4E_g(D)$	
(5)	13	-	260	38461	L.F	Octahedral
			352	28409	C.T	
			396	25252	C.T	

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Table 6: Outcomes of antibacterial bioassay (concentration utilized 100 μg /mL of DMSO). { (a) *E. coli*, (b) *S. aureus* (c) *B. subtilis* (d) *P. aeruginosa*, antifungal bioassay (concentration utilized 200 μg /mL). (a) *A. niger* (b) *A. flavus* (c) *R. stolonifer* and (d) *C. albicans* .10 <: weak; >10: moderate; >16: significant }.

Compounds	Bacteria				Organism			
	(a)	(b)	(c)	(d)	(a)	(b)	(c)	(d)
(S1)	4	8	7	6	15	17	21	22
(1)	9	13	15	12	18	21	25	26
(2)	13	19	16	14	24	25	29	24
(3)	17	18	19	18	26	24	30	27
(4)	15	16	16	16	31	27	28	29
(5)	17	15	14	17	30	29	31	31

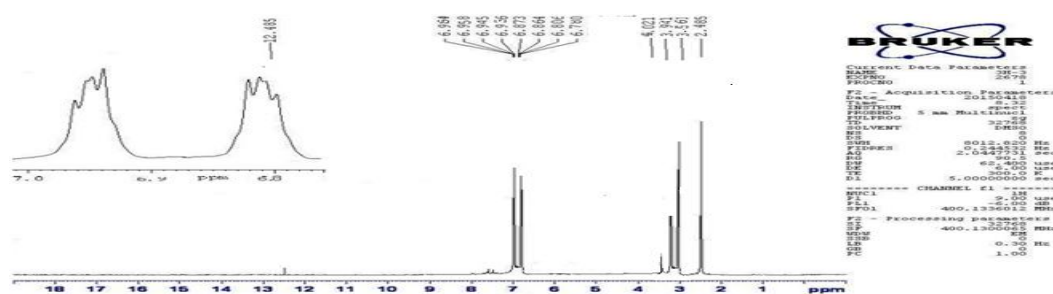
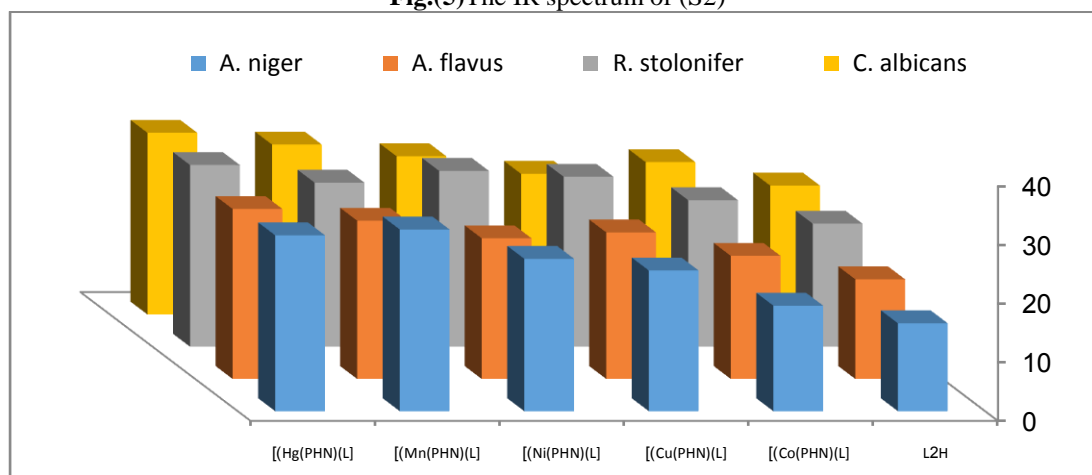
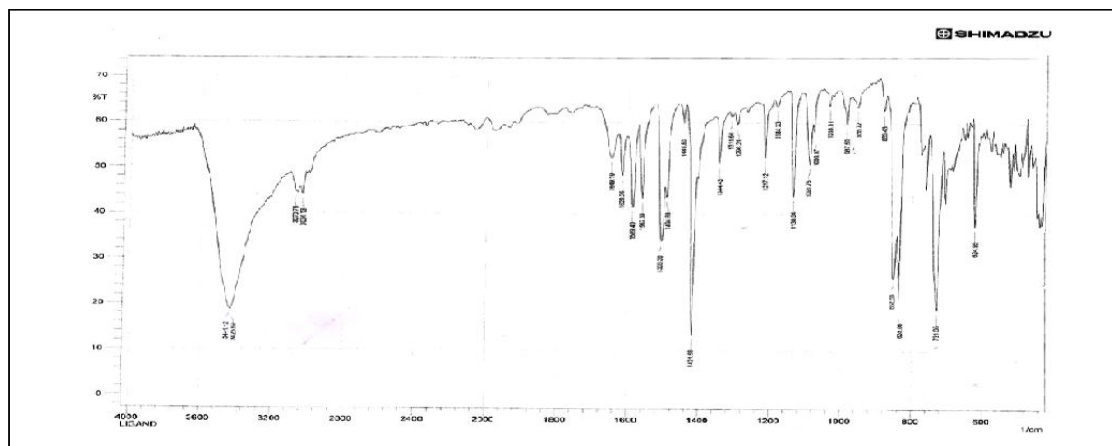
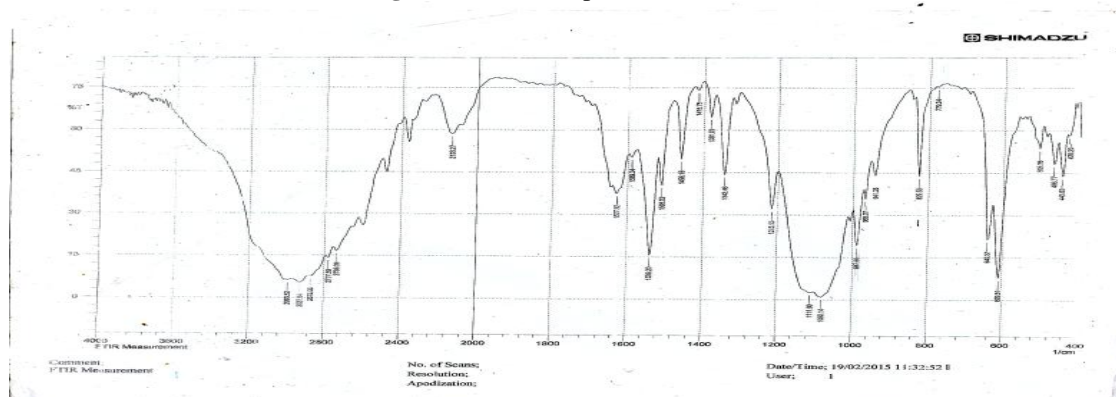
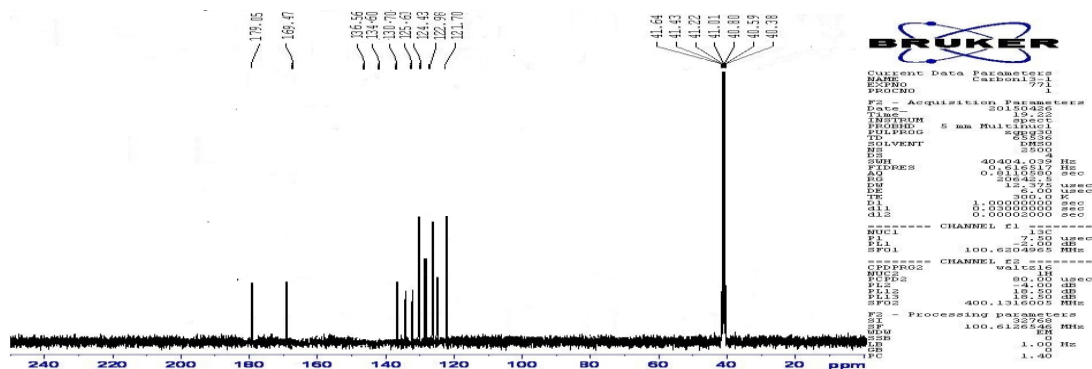


Fig.(2) : $^1\text{H-NMR}$ spectrum of (S1)



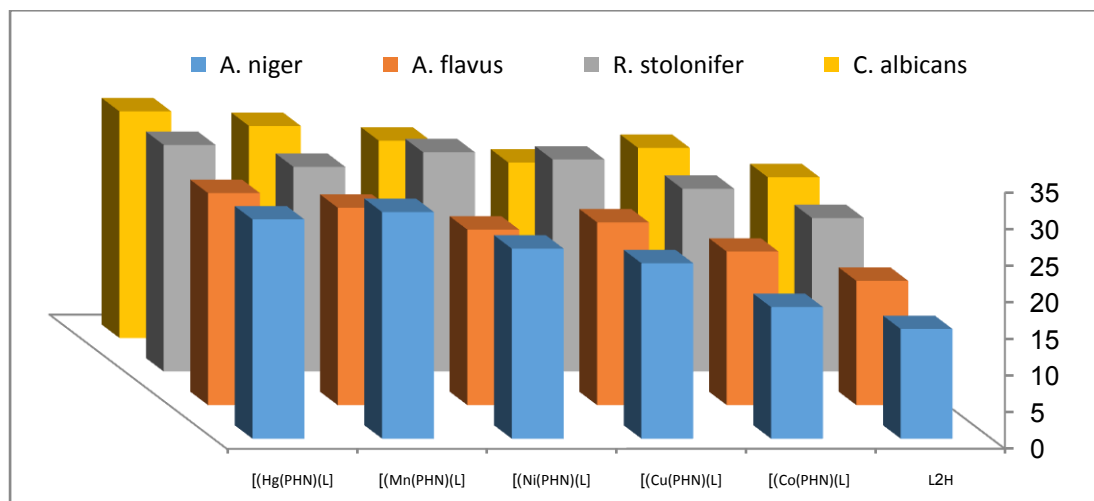


Fig.(7)Divergence between the antifungaleffectiveness of (S1)& its mixed complexes

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