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Synthesis, spectral characterization and biological evaluation of Mn^{II}, Ni^{II} and Cu^{II} complexes with new N_2S_2 Schiff base ligand

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Schiff base ligand of isophthalidene-bis-(methylhydrazinylcarbothiamide) abbreviated as (IMHC) was synthesized by the condensation reaction of isophthaldehyde and *N*-methylhydrazinylcarbothiamide. Metal complexes were synthesized by using metal ions, Mn^{II}, Ni^{II}, Cu^{II} and the ligand, IMHC which were characterized by UV-Visible, IR, and ^1H NMR spectroscopy and further confirmed by mass analysis. The IMHC and the metal complexes were screened for *in vitro* antimicrobial activity against *Salmonella typhimurium, Shigella flexneri* and *Micrococcus leuteus* by disc diffusion method and found that Mn^{II} and Cu^{II} complexes showed higher inhibitory activity compared to Ni^{II} complex.

Keywords: Schiff base, isophthalidene-bis-(*N*-methylhydrazinylcarbothiamide), biological activity, antimicrobial agents, tetradendate ligand.

Introduction

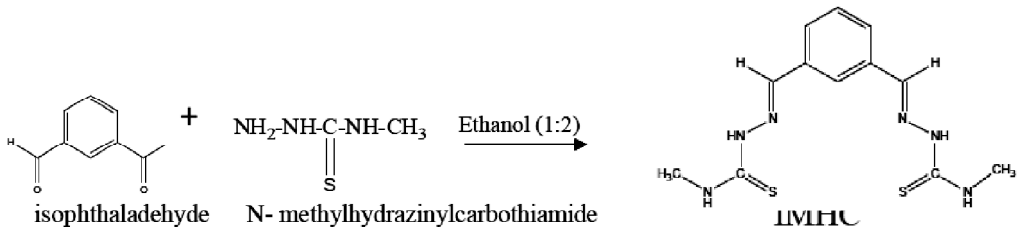
Schiff base ligand and their complexes of transition metals have been investigated in biological, clinical and pharmacological areas[1]. Usually metals coordinate the Schiff base through imine nitrogen which are known for their biological activity[2,3]. The serious medical problem of microbial resistance to drug and the rate at which it develops have led to increasing level of research in finding new molecules as substitute for classical antibiotics. Binding of a drug with functionalized metal enhance the drug delivery system by its unique activity possessing more biological activity than the parent drug[4].

Antimicrobial activity by the Schiff bases derived by the condensation of salicyaldehyde and different heterocyclic compounds with amino group were reported[5]. 1,2,4-Triazole derivatives as Schiff bases and their complexes possessing activity as equivalent to standard antibiotic drug[4]. Metal complexes are known for bio-activity which can be explored to solve the multidrug résistance problem. The synthesis of Schiff base derived from isophthaldehyde and *N*-methyl hydrazinyl carbothiamide are not yet reported. In the present study, importance is given to focus on the synthesis of IMHC and their Mn^{II}, Ni^{II} and Cu^{II} complexes, characterization and their microbial activity.

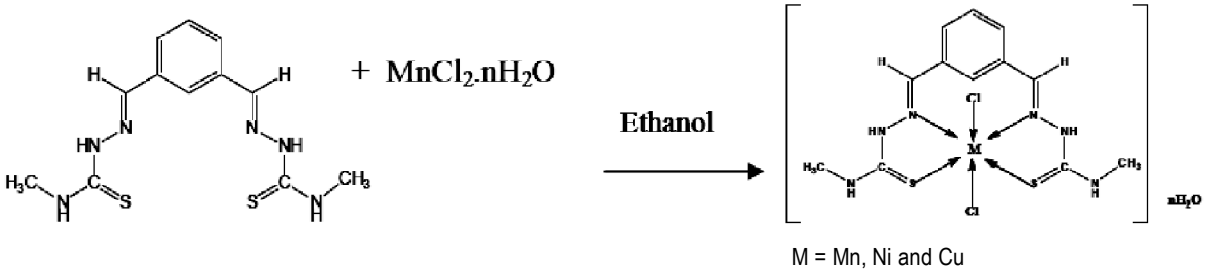
Experimental

Chemicals were purchased commercially. The FTIR - Schimadzu, UV - Jasco V-730, ^1H NMR - Jeol 400 MHz, and Micromass UK PLATFORM II LC-MS spectrometer were used for characterisation.

Isophthaldehyde and *N*-methylhydrazinylcarbothiamide in 1:2 molar ratio are condensed in ethanolic medium, to yield IMHC (Scheme 1). The metal ions [$MnCl_2.4H_2O$, $NiCl_2.6H_2O$ and $CuCl_2.2H_2O$] and IMHC in 1:1 molar ratio are condensed in ethanolic medium (Scheme 2). The *in vitro* biological screening effects of the investigated compounds were tested against the bacteria: *Salmonella typhimurium, Shigella flexneri* and *Micrococcus leuteus* by the disc diffusion method using standard procedure. DMSO was used as control and streptomycin as a standard drug to determine the inhibition zone.



Scheme. 1. General synthetic scheme for Schiff base ligand.



Scheme. 2. General synthetic scheme for complexes.

Results and discussion

The molar conductivity of complexes using DMSO solvent indicated that the complexes as neutral and non-electrolytic behavior (Table 1). The UV-Visible absorption spectrum of synthesized complexes were recorded in 10^{-6} *M* DMSO (Table 2). The IR spectrum of the ligand showed the peaks around 1650–1530 cm^{-1} and 1200–1100 cm^{-1} due to -C=N and -C=S group respectively which are shifted in the spectra of all the complexes indicating the bond formation to the metal ion (Table 3). ^1H NMR spectral data of the ligands recorded in $CDCl_3$ is given in Fig. 1. *In vitro* antimicrobial activity of the complexes were tested with the observed zone of inhibition in Table 4 and found that Mn^{II} and Cu^{II} complexes showed higher inhibitory activity compared to the Ni^{II} complex.

Table 1. Physical parameters of the synthesized IMHC and metal complexes

Compound	Colour	Yield (%)	Melting point (°C)	Mol. conductivity (Ω^{1} cm^2 mol^{1})	Molecular weight
IMHC (L)	Brown	88	112	–	–
$[Mn(IMHC)(Cl)_2]$	Flesh colour	77	>250	0.15	434
$[Ni(IMHC)(Cl)_2].2H_2O$	Pale yellow	76	>250	0.2	440
$[Cu(IMHC)(Cl)_2]$	Green	72	>250	0.10–0.25	473

Table 2. UV-Visible spectral data of Schiff base complex

Compound	Wavelength (nm)	d-d transition	Band assignment	Geometry
IMHC (L)	326		Intra ligand transition	
$[Mn(L)Cl_2]$	312	425	$^6A_{1g} \rightarrow {}^4T_{1g}$	Octahedral
		560	$^6A_{1g} \rightarrow {}^4E_g$	
$[Ni(L)Cl_2].2H_2O$	319	400	$^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$	Octahedral
		540	$^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$	
		780	$^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$	
$[Cu(L)Cl_2]$	310	375–600	$^2T_{2g} \rightarrow {}^2E_g$	Distorted octahedral

Table 3. IR spectral data of ligands and complexes

Band assignment	IMHC (L)	$[Mn(IMHC)Cl_2]$	$[Ni(IMHC)Cl_2].2H_2O$	$[Cu(IMHC)Cl_2]$
N-H	3276	3285	3283	3254
C-H	3250–2985	3245	3254	3246–3142
C=N	1640	1600	1652	1549
C=C	1541	1100	1522	1524
C=S	1253	1276	1257	1239
C-N	1105–1060	740	740	725–691
M-N	–	513–524	520–538	542
M-S	–	500	500	500

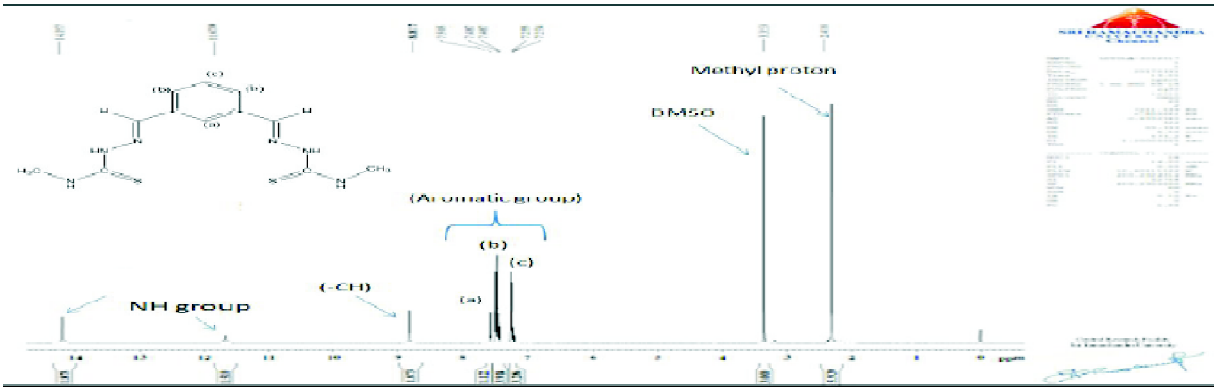


Fig. 1. ^1H NMR signal for the synthesized IHMC ligand.

Table 4. Zone of inhibition of bacterial growth for synthesized complexes

Organism	MTCC No.	Zone of inhibition (mm)			
		$[MnLCl_2]$	$[NiLCl_2]$	$[CuLCl_2]$	Control
Salmonella typhimurium	3224	–	–	–	–
Shigella flexneri	1457	14	–	11	–
Micrococcus leuteus	106	16	–	12	10

Conclusions

In this report, IMHC with coordination sites as N_2S_2 system, and their Mn^{II}, Ni^{II} and Cu^{II} complexes were synthesized and confirmed by spectral analysis. *In vitro* antimicrobial activity, showed remarkable inhibition of the bacterial growth and found that Mn^{II} and Cu^{II} complexes showed higher inhibitory activity compared to the Ni^{II} complex.

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