Avances en Química
ISSN: 1856-5301
clarez@ula.ve
Universidad de los Andes
Venezuela

Mishra, A. P.; Sharma, Neha; Jain, Rajendra K.
Microwave synthesis, spectral, thermal and antimicrobial studies of some Ni(II) and Cu(II) Schiff base complexes
Avances en Química, vol. 7, núm. 1, enero-abril, 2012, pp. 77-85
Universidad de los Andes
Mérida, Venezuela

www.saber.ula.ve/avancesenquimica
Avances en Química, 7(1), 77-85 (2012)
Artículo científico

Microwave synthesis, spectral, thermal and antimicrobial studies of some Ni(II) and Cu(II) Schiff base complexes

A P Mishra*, Neha Sharma, Rajendra K Jain

Department of Chemistry, Synthetic Inorganic & Coordination Chemistry Laboratories,
Dr. H.S. Gour Central University, Sagar (M.P.)-470 003, India

(*) apm19@rediffmail.com

Recibido: 20/02/2012 **Revisado**: 02/04/2012 **Aceptado**: 04/04/2012

Resumen

Se sintetizaron bases de Schiff bidentadas y tridentadas (NO), (ONO) a través de la reacción de condensación entre la metil-isobutilcetona y el 2-amino-4clorofonol y 2-hidroxiacetofenona con la hidracina del ácido isonicotínico. Los complejos metálicos 1:1 o 1:2 han sido preparados mediante la interacción de estas bases de Schiff y los iones Ni(II) y Cu(II). La síntesis fue realizada empleando el método convencional y el de microondas, y los productos fueron caracterizados por análisis elemental, FT-IR, UV-Vis, RES, conductancia molar, análisis térmico y difracción de rayos X. Los complejos son coloreados y estables al aire y temperatura ambiente. El comportamiento térmico de los complejos metálicos hidratados muestran una primera etapa de pérdida de agua de hidratación, seguidos de la descomposición de las moléculas de ligando en etapas subsecuentes. El análisis de los cristales del complejo $[Ni(HINH)(H_2O)]Cl.3H_2O$ ***a*** = ***b*** = 13,9338Å, ***c*** = 34,7975Å, V= 6755, 96Å^3, Z = 12, D_{obs} = 1,2421g/cm^3, D_{cal} 1,2847g/cm^3, refleja una estructura cristalina en un sistema ortorómbico. La conductividad eléctrica de estado sólido de los complejos metálicos indica que éstos poseen una naturaleza semiconductora. Las bases de Shiff y los complejos metálicos muestran buena actividad contra bacterias Gram-positivas del tipo *Staphylococcus aureus*, bacterias Gram-negativas del tipo *Escherichia coli* y hongos del tipo *Aspergillus niger* y *Candida albicans*. Los complejos metálicos son mejores agentes antimicrobiales que las bases de Schiff.

Palabras clave: síntesis por microondas; hidracina del ácido isonicotínico; análisis térmico; actividad biológica

Abstract

Bidentate and tridentate (NO), (ONO) Schiff bases have been synthesized by condensing methyl isobutyl ketone with 2-amino-4-chlorophenol and 2-hydroxy acetophenone with isonicotinic acid hydrazide. The 1:1 or 1:2 metal complexes have been prepared by interacting these Schiff bases with metal ions *viz.* Ni(II), Cu(II). These compounds have been synthesized by conventional as well as microwave methods and characterized by elemental analysis, FT-IR, UV-Vis, ESR, molar conductance, thermal analysis and X-ray diffraction. The complexes are colored and stable in air at room temperature. The thermal behavior of metal complexes shows that the hydrated complexes loses water molecules of hydration in the first step; followed by decomposition of ligand molecules in the subsequent steps. Crystal data of $[Ni(HINH)(H_2O)]Cl.3H_2O$ complex ***a*** = ***b*** =13.9338Å, ***c*** = 34.7975Å, V = 6755.96Å^3, Z = 12, D_{obs} = 1.2421g/cm^3, D_{cal} 1.2847g/cm^3, reflect that this complex has crystallized in orthorhombic system. The solid state electrical conductivity of the metal complexes has also been measured. Solid state electrical conductivity studies reflect semiconducting nature of the complexes. The Schiff bases and metal complexes show good activity against the Gram-positive bacteria; *Staphylococcus aureus* and Gram-negative bacteria; *Escherichia coli* and fungi *Aspergillus niger* and *Candida albicans*. The antimicrobial results also indicate that the metal complexes are better antimicrobial agents as compared to the Schiff bases.

Keywords: Microwave synthesis; isonicotinic acid hydrazide; thermal study; biological activity.

Introduction

Schiff bases and their bio-active complexes have been studied extensively over the past decade. Schiff bases provide potential sites for bio-chemically active compounds. Various transition and inner-transition metal complexes with bi, tri- and tetradenate Schiff bases containing nitrogen and oxygen donor atoms play important role in biological systems. Interest in the stu of hydrazides and corresponding hydrazones arises the fact that hydrazides of organic acids and hydrazones can function as antituberculous. antituberculous activity was ascribed to their abilit form more or less stable chelates with the transition m

ions[1-3]. Hydrazones possessing an azometine -NHN=CH- proton, constitute an important class of compounds for new drug development. Coordination compounds derived from aroyl hydrazones have been reported to act as enzyme inhibitor and are useful due to their pharmacological applications. Many drugs inhibit modified toxicological and pharmacological properties when they are in the form of metal complexes. The most widely studied metal in this respect is copper (II) which has proved to be beneficial in diseases such as tuberculosis, gastric ulcers, rheumatoid arthritis and cancers. Metal chelation therapy can emerge to answer the problems of multidrug resistance (MDR) as against various viruses, bacteria, fungi and other pathogens[4-7].

Microwave-assisted synthesis is a branch of green chemistry. The application of microwave-assisted synthesis in organic, organometallic and coordination chemistry continues to develop at an astonishing pace. Microwave irradiated reactions under solvent free or less solvent conditions are attractive offering reduced pollution, low cost and offer high yields together with simplicity in processing and handling. The salient features of microwave approach are shorter reaction times, simple reaction conditions and enhancements in yields[8-12]. Reports on the synthesis of metal complexes by microwave methods have been comparatively less.

The present investigation aims at the conventional and microwave synthesis, physico-chemical characterization and bio-inorganic studies of Schiff bases involving methyl isobutyl ketone with 2-amino-4-chlorophenol and 2-hydroxy acetophenone with isonicotinic acid hydrazide and their metal chelates with Ni(II) and Cu(II).

Experimental

General experimental procedures:

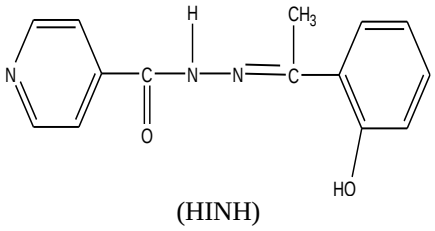
All the used chemicals and metal salts were of A.R. grade. Methyl isobutyl ketone and 2-hydroxy acetophenone were obtained from CDH and isonicotinic acid hydrazide has been purchased from Sigma-Aldrich. Metal salts were purchased from Loba Chemie. Elemental analyses were performed on an Elemental Vario EL III Carlo Erba 1108 analyzer. Electronic spectra (in DMSO) were recorded on Perkin Elmer Lambda-2B-spectrophotometer. Molar conductance measurements were conducted using 10^{-3} M solutions of the complexes in DMSO on Elico-CM 82 Conductivity Bridge at room temperature. Magnetic susceptibility measurements were carried out on a Gouy balance at room temperature using $Hg[Co(SCN)_4]$ as the calibrant. FT-IR spectra were recorded in KBr pellets on a Perkin Elmer RX1 spectrophotometer in wave number region 4000-400cm^{-1}. X-band EPR spectra were recorded on a Varian E-112 spectrometer at room temperature operating at the X-band region with 100kHz modulation frequency, 5mw microw power and 1 G modulation amplitude using TCNE as internal standard. Thermogravimetric (TG) analysis was at NIPER, Chandigarh under N_2 atmosphere with a hea rate of 20°Cmin^{-1}. Powder X-ray diffraction (XRD) pat were recorded on a RINT2000 wide angle goniometer. X diffractometer, operated at 40kV and 30mA generator u the CuKα line at 1.54056Å as the radiation sources. Sa was scanned between 5° to 60° (2θ) at 25°C. The solid electrical conductivity has been measured by imped spectroscopic method using HIOKI 3532-50 LCR Hitest fixed frequency 1 KHz in the temperature range of 377K. Microwave assisted synthesis were carried out in glass vessel on a modified microwave oven model 2001 with rotating tray and a power source 230V, microw energy output 800W and microwave frequency 2450MH thermocouple device was used to monitor the tempera inside the vessel of the microwave. The microwave reac were performed using on/off cycling to control temperature.

Biological activity:

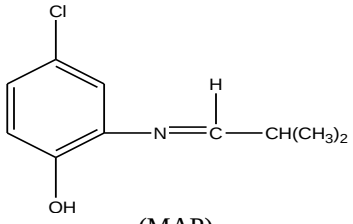
The *in-vitro* biological activity of the Schiff bases and complexes was tested against the bacteria *Escherichia* and *Staphylococcus aureus* by disc diffusion method u nutrient agar as medium and streptomycin as control. antifungal activities of the compounds were also tested b Well diffusion method against the fungi *Aspergillus niger* *Candida albicans*, on potato dextrose agar as the medium miconazole as control. Each of the compounds was disso in DMSO and solutions of the concentrations (25, 50 100ppm) were prepared separately. In a typical procedu well was made on agar medium inoculated microorganism. The well was filled with the test solu using a micropipette and the plate was incubated 24h bacteria at 37°C and 72h for fungi at 30°C. During period, the test solution diffused and the growth of inoculated microorganism was affected. The inhibition was developed, at which the concentration was noted.

Conventional method for the synthesis of Schiff bases:

HINH and MAP Schiff bases (Fig. 1) were synthesized b condensation of equimolar ratio of methyl isobutyl ke with 2-amino-4-chlorophenol and 2-hydroxy acetophe with isonicotinic acid hydrazide dissolved in ethanol. resulting reaction mixture was stirred well, refluxed for and then allowed to cool overnight. The coloured precipitate of Schiff base obtained was filtered, washed cold ethanol several times and dried in air at r temperature and finally stored under reduced pressure $CaCl_2$ desiccator. The purity of synthesized compounds checked by TLC using silica gel G (yield: 75-78%).



(HINH)



(MAP)

Fig. 1: Structure of Schiff base Ligands

Microwave method for the synthesis of Schiff bases:

The equimolar (1:1) ratio of methyl isobutyl ketone with 2-amino-4-chlorophenol and 2-hydroxy acetophenone with isonicotinic acid hydrazide were mixed thoroughly in a grinder. The reaction mixture was then irradiated by microwave oven by taking 3-4mL of dry ethanol as a sol The reaction was completed in a short time (4-5min) higher yields. The resulting product was then recrystal with ethanol, finally dried under reduced pressure anhydrous $CaCl_2$ in a desiccator. The progress of the reac purity of the product was monitored by TLC using silic G (yield: 87-88%).

Conventional method for the synthesis of metal complexe

The metal complexes (Figs. 2 and 3) were prepared by mixing of (50mL) ethanolic solution $NiCl_2.6H_2O/CuCl_2.2H_2O$ with the (50mL) ethanolic solu of Schiff bases (HINH/MAP) in 1:1 or 1:2 (metal:lig ratio. The resulting mixture was refluxed on water bath f 8h. A coloured product appeared on standing and coolin above solution. The precipitated complex was, fil washed with ether and recrystallized with ethanol se times and dried under the reduced pressure over anhyd $CaCl_2$ in a desiccator. It was further dried in electric ov 50-70°C (yield: 65-70%).

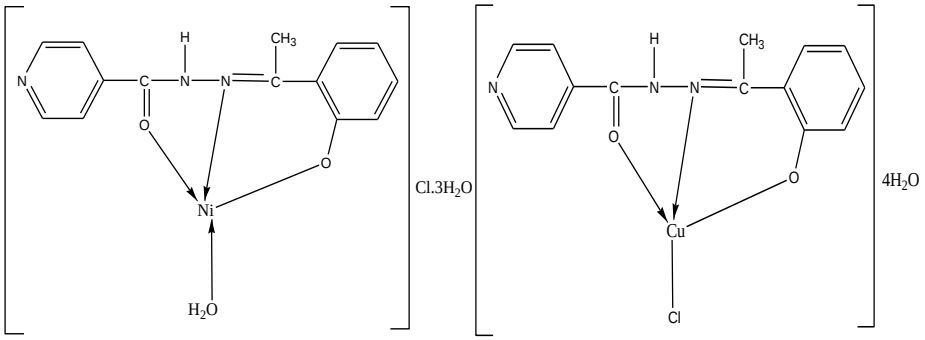


Fig. 2: proposed structure of metal complexes of HINH ligand.

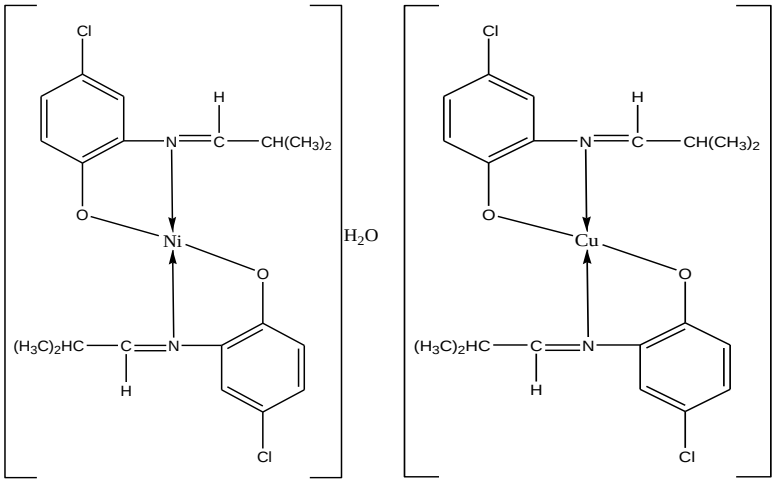


Fig. 3: proposed structure of metal complexes of MAP ligand

Microwave method for the synthesis of metal complexes:

The ligand and the metal salts were mixed in 1:1 or 1:2 (metal:ligand) ratio in a grinder. The reaction mixture was then irradiated by the microwave oven by taking 3-4mL of dry ethanol as a solvent. The reaction was completed in a short time (6-9min) with higher yields. The resulting product was then recrystallized with ethanol and ether and finally dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. The progress of the reaction and purity of the product was monitored by TLC using silica gel G (yield: 80-84%).

Results and Discussion

As a result of microwave-assisted synthesis, it was observed that the reaction was completed in a short time with higher yields compared to the conventional method. In the microwave method homogeneity of reaction mixture was increased by the rotating of reaction platform tray. The confirming of the results was also checked by the repeating of the synthesis process.

All the metal complexes are coloured, solid and s towards air and moisture at room temperature. T decompose on heating at high temperature, more or soluble in common organic solvents. The comparison s data of microwave and conventional methods, with analy and physical data of the compounds are given in the Tab Analytical date show that metal chelates have 1:1 or (metal:ligand) stoichiometry. The observed m conductance of the complexes in DMSO at room tempera is consistent with non-electrolytic nature of all the compl except Ni(II) complex of HINH.

IR Spectra:

The IR spectra of the complexes were compared with tho the free ligand in order to determine the involvemen coordination sites in chelation. Characteristic peaks in spectra of the ligand and complexes were considered compared.

Table 1: The comparative results of conventional and microwave methods, analytical, physical data and magnetic moment valu the compounds

Compounds molecular weight/Colour	Reaction period		Yield (%)		Elemental analysis, found (calcd.) %			$*\Lambda_m$	#
	CM (h.)	MM (min.)	CM	MM	C	H	N		
HINH 255.0/Cream	3.6	4.3	78	88	65.72 (65.88)	5.03 (5.09)	16.45 (16.47)	-	-
$[Ni(HINH)(H_2O)]Cl.3H_2O$ 421.2/Yellow	8.0	8.4	67	82	39.89 (39.88)	4.91 (4.98)	9.64 (9.97)	112.5	Dia
$[Cu(HINH)(Cl)]4H_2O$ 426.0/Light Brown	7.6	7.9	70	84	39.40 (39.43)	4.95 (4.92)	9.64 (9.85)	52.3	1.9
MAP 211.0/Light Coffee	3.5	4.5	75	87	62.32 (62.41)	6.55 (6.61)	6.21 (6.61)	-	-
$[Ni(MAP)_2]H_2O$ 499.7/Blackish Brown	7.5	8.2	65	80	52.75 (52.83)	6.10 (6.00)	6.07 (5.60)	20.5	Dia
$[Cu(MAP)_2]$ 486.5/Brown	6.5	6.8	67	83	54.30 (54.26)	5.82 (5.75)	6.07 (5.75)	15.7	1.8

CM = Conventional method, time in hours; MM = Microwave method, time in minutes
$*\Lambda_m = (\Omega^{-1}cm^2mol^{-1})$; $\#\mu_{eff}$ = B.M.

IR spectrum of HINH ligand shows the band at $3116cm^{-1}$ due to N-H (stretching) frequency. A strong band at about $1682cm^{-1}$ in the ligand ν(C=O) group has shifted to lower frequency $1651 \pm 10cm^{-1}$ in the complexes. This indicates the involvement of C=O group in coordination. Medium intensity band at about $1607cm^{-1}$ in the ligand due to ν(C=N) group shifts down by $10\text{-}15cm^{-1}$ in the complexes suggesting coordination through azomethine nitrogen, this can be explained by the donation of electrons from nitrogen to the empty d-orbital of the metal atom. A band in the ligand spectrum at $1373cm^{-1}$ due to phenolic group (OH-deformation) was found absent in complexes. This suggests deprotonation of phenolic OH, on chelation with metal io band at $1281cm^{-1}$ due to phenolic ν(C-O) in ligand spec shifts to higher side $1338 \pm 8cm^{-1}$ in the metal compl indicating its involvement in chelation. Practically, no sh the characteristic bands position of pyridine nitrogen (has been observed indicating the non-involvement of donor group in coordination. The appearance of a broad around $3420 \pm 10cm^{-1}$ in the spectra of complexes tentatively been assigned to ν_{stre} water molecules. A ban medium intensity at $708 \pm 5cm^{-1}$ (-OH rocking), sugges presence of coordinated water in Ni(II) complex. This ba absent in the Cu(II) complex. The new bands of

intensity observed at 556 ± 5 and 481 ± 5cm^{-1} in the spectra of metal complexes, are assignable to ν(M-O) and ν(M-N) mode, respectively[13-16]. The MAP ligand band at 1604cm^{-1} due to ν(C=N) azomethine group shifts down at 1590 ± 10cm^{-1}, suggesting participation of this group in chelation. The ligand spectrum shows band at 3080 and 1386cm^{-1} due to the stretching vibration and phenolic -OH deformation respectively. These remain absent in spectra of complexes. An intense ligand band observed at about 1209cm^{-1} (phenolic C-O) shift to higher frequency (1275 ± 10cm^{-1}) in the spectra of complexes. This suggests deprotonation of the phenolic OH group on its chelation with metal ion. The broad band around 3440 ± 10cm^{-1} in the spectra of Ni(II) complex has been assigned to ν(OH) water molecule. This band is absent in Cu(II) complexes. The new bands at 548 ± 5 and 475 ± 5cm^{-1} in the complexes have tentatively been assigned to ν(M-O) and ν(M-N) mode, respectively[13-16].

Electronic spectra:

The electronic spectral measurements were used for assigning the stereochemistry of metal ions in the complexes, which is based on the position, shape, intensity and number of d-d transition bands.

The electronic spectrum of Ni(II) complex of HINH exhibits two bands at 12465 and 24324cm^{-1}, which are assignable to $^1A_{1g}\rightarrow{}^1E_g$ and $^1A_{1g}\rightarrow{}^1B_{1g}$ transitions, respectively. It is a diamagnetic complex; therefore sq planar geometry has been suggested. Two absorp bands at 12553 and 20509cm^{-1} in the spectrum of C complex of HINH have tentatively been assigne $^2B_{1g}\rightarrow{}^2B_{2g}$ and $^2B_{1g}\rightarrow{}^2E_g$ transitions. It magnetic mo is 1.91B.M. This favors the square planar st arrangements[17-21].

The electronic spectrum of Ni(II) complex of M exhibits two bands at 14823 and 22351cm^{-1} which assignable to $^1A_{1g}\rightarrow{}^1E_g$ and $^1A_{1g}\rightarrow{}^1B_{1g}$ transit respectively. Since the complex is diamagnetic, so sq planar geometry has been suggested. Two absorp bands at 14841cm^{-1} and 20148cm^{-1} in the Cu(II) com have tentatively been assigned to $^2B_{1g}\rightarrow{}^2B_{2g}$ and $^2B_{1g}$– transitions. The magnetic moment value for this com is 1.87B.M. On the basis of findings the square pl geometry has been suggested for this complex[17-21].

ESR Spectra

The ESR spectra of Cu(II) provide information abou extent of the delocalization of unpaired electron. Th band ESR spectrum of Cu(II) complexes (Fig. 4) recorded in the solid state at room temperature, their g Δg, g_{av}, G have been calculated.

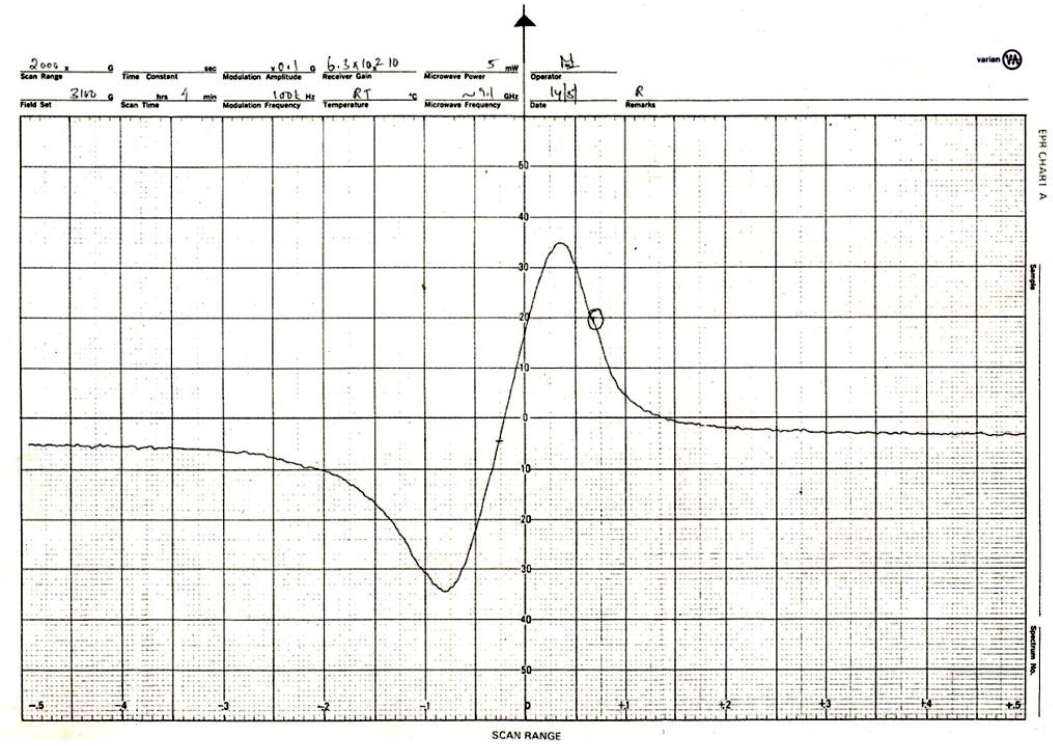


Fig. 4: ESR spectrum of Cu(II) complex of HINH ligand

The values of ESR parameters $g_{\parallel}$, $g_{\perp}$, g_{av}, Δg, G for Cu(II) complex of HINH are 2.2513, 2.0793, 2.1366, 0.1720, 3.2250, respectively.

ESR spectra of the complexes revealed two g values ($g_{\parallel}$ and $g_{\perp}$). Since the $g_{\parallel}$ and $g_{\perp}$ values are closer to 2 and $g_{\parallel} > g_{\perp}$ suggesting a tetragonal distortion around the Cu(II) ion. The trend $g_{\parallel} > g_{\perp} > g_e(2.0023)$ shows that the unpaired electron is localized in $d_{X^2-Y^2}$ orbital in the ground state of Cu(II), spectra are characteristic of axial symmetry. The $g_{\parallel} > 2.3$ is characteristic of an ionic environment and $g_{\parallel} < 2.3$ indicates a covalent environment in metal ligand bonding. The $g_{\parallel}$ values for these complexes are less than 2.3 suggesting the environment is covalent. The exchange coupling interaction between two Cu(II) ions is explained by Hathaway expression $G = (g_{\parallel}-2.0023)/ (g_{\perp}-2.0023)$. According to Hathaway, if the value G is greater than four ($G > 4.0$), the exchange interaction is negligible; whereas when the value of G is less than four ($G < 4.0$) a considerable exchange coupling is present in solid complex. The G values for these Cu(II) complexes are less than four indicate, considerable exchange interaction in the complexes[22, 23].

Thermal studies:

The thermal behavior of metal complexes shows that the hydrated complexes lose molecules of hydration first; followed by decomposition of ligand molecules in the subsequent steps.

Thermogram of Ni(II) complex of HINH shows weight between 60º-150ºC. This corresponds to three lattice w molecules. The complex does not show any loss in w between 150-230ºC. Elimination of one coordinated w molecule has been observed between the temperatures 260ºC (remaining wt% obs/cal 82/82.91). Above temperature, a weight loss has been observed in general u 360ºC, indicating the loss of major part of the ligand in step (temaining wt% obs/cal 49/42.5). The decompositio remaining intermediate moiety occurs between 360-50 After 500ºC, a horizontal curve has been obtained; suggests the formation of an ultimate pyrolysis produc metal oxide (remaining wt% obs/cal 21/19.9)[24,25].

X-Ray Studies

X-Ray powder diffractograms of Complex and were reco using CuKα (1.54056Å) as source in the range 5º-60 ºC X-ray crystal system has been worked out by trial and method, for finding the best fit between observed calculated $\sin^2\theta$ values. The crystal system, lattice param units cell have been determined with the help of diffra data. Crystal data for Ni(II) complex of HINH are as a = 13.9338Å, c = 34.7975Å, V = 6755. 96Å^3, Z = 12, D 1.2421g/cm^3, D_{cal} 1.2847g/cm^3, reflect that this complex crystallized in orthorhombic system. Crystal data for N complex (Fig. 5) of MAP are as: a = b = 20.5097Å. 34.4477Å, V = 14490.34Å^3, Z = 26, D_{obs} = 1.488g/cm^3, = 1.5176g/cm^3, this reflects that this complex has crystal in tetragonal system. The calculated values show agreement and are within experimental error limits[26, 27].

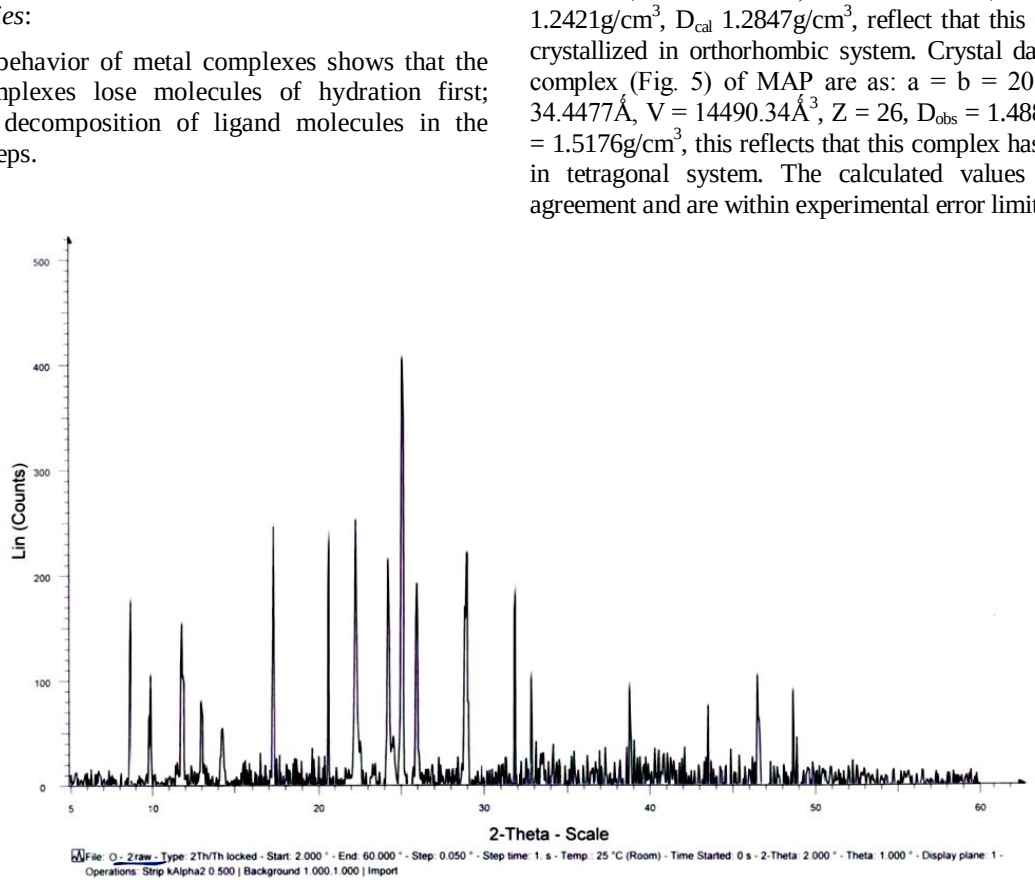


Fig. 5: XRD diffractogram of Ni(II) complex of MAP ligand

Electrical conductivity

The temperature dependence of the solid state conductivity (σ) of the compounds in their compressed pellet form have been measured at fixed frequency 1KHz in the temperature range 297-377K. the values of the solid state electrical conductivity of the Schiff base and its complexes increases with increasing temperature and decreases upon cooling over the studies temperature range indicating their semiconducting behavior. The general behavior of electrical conductivity follows the Arrhenius equation:

$$\sigma = \sigma_o \exp(-Ea/KT)$$

where Ea is the thermal activation energy of conduction, σ_o is the conductivity constant and K is the Boltzman constant.

The lots of σ vs 1000/T for all the compounds are found to be linear over a studies temperature range. The room temperature electrical conductivity of all the compo lies in the range 6.45×10^{-6}-2.15×10^{-7}ohm^{-1}cm^{-1}. T values show their semi-conducting nature. The elect conductivity at room temperature for the complexe HINH are Cu > Ni and for the metal complexes of M are Cu > Ni. The activation energy of the compound li the range 0.229-0.768eV[28]. The confirming of temperature dependence conductivity of the compo was also checked by the repeating of the conduct measurements.

Antimicrobial activities

The *in-vitro* Antimicrobial activity of the synthesized S base ligands and their corresponding metal complexe selected bacteria *E. coli* and *S. aureus* and two fungi *A. n* and *C. albicans* was carried out. All of the tested compo showed good biological activity against microorganism.

Table 2: Antibacterial screening data for the ligands and their complexes

Comp.	*E. coli*						*S. aureus*					
	Diameter of inhibition zone (mm)			% Activity index*			Diameter of inhibition zone (mm)			% Activity index*		
	25	**50**	**100**	**25**	**50**	**100**	**25**	**50**	**100**	**25**	**50**	**100**
HINH	11	14	17	50	58	61	13	16	18	72	73	75
Ni(II)	14	16	20	64	67	71	13	17	19	72	77	79
Cu(II)	16	19	23	73	79	82	14	18	20	78	82	83
MAP	12	14	16	55	58	57	12	15	18	67	68	75
Ni(II)	15	17	21	68	71	75	12	16	18	67	73	75
Cu(II)	17	20	24	77	83	86	14	18	20	78	82	83
Streptomycin (Standard)	22	24	28	100	100	100	18	22	24	100	100	100

$$^{*}\%\ \text{Activity Index} = \frac{\text{Zone of inhibition by test compound (diameter)}}{\text{zone of inhibition by standard (diameter)}} \times 100$$

Table 3: Antifungal screening data for the ligands and their complexes

Compound	Diameter of inhibition zone (mm); Concentration in ppm					
	A. niger			*C. albicans*		
	25	**50**	**100**	**25**	**50**	**100**
HINH	12	16	20	13	16	20
Ni(II)	15	19	23	17	19	24
Cu(II)	14	18	22	14	17	22
MAP	13	17	21	14	16	20
Ni(II)	15	20	24	15	17	22
Cu(II)	15	21	25	18	20	25
Miconazole (Standard)	20	24	30	22	24	29

On comparing the biological activity of the Schiff base its metal complexes with the standard bactericide fungicide, it is show that the some metal complexes good activity as compared to the standard but all complexes are more active than their respective liga The higher inhibition zone of metal complexes than t of the ligands can be explained on the basis of Overto concept and Chelation theory. On chelation, the polari the metal ion will be reduced to greater extent due to overlap of the ligand orbital and partial sharing of positive charge of the metal ion with donor gro Further, it increases the delocalization of π-electrons the whole chelating ring and enhances the penetratio the complexes into lipid membranes and blocking of metal binding sites in the enzymes of microorgani There are other factors which also increases the activity

solubility, conductivity and bond length between the metal and ligand[29-33].The bactericidal and fungicidal investigation data of the compounds are summarized in Tables 2 and 3. The results of the investigations account for the antipathogenic behavior of the compounds and this efficacy is positively modified on complexation.

Conclusion

In the present research studies, our successful efforts are synthesis of some newly compounds from the conventional as well as microwave methods. These synthesized compounds have been characterized by various physicochemical and spectral analyses. In the result of microwave-assisted synthesis, it has been observed that the reaction time decreased from hours to minutes and availability of the product within better yields compared to the classical method. Thermal data shows degradation pattern of the complexes. The XRD patterns indicate crystalline nature of the complexes. Electrical conductivity data suggest that all the complexes fall in the semiconducting range. The antimicrobial data show that the metal complexes to be more biological active compared to those parent Schiff base ligand against all phathogenic species. The compounds also inhibit the growth of fungi and bacteria to a greater extent as the concentration is increased. The Schiff base ligands were found to be biologically active and their metal complexes displayed enhanced antimicrobial activity against one or two strains. Chelation tends to make the ligand act as more powerful and potent bactericidal agent. Further chelation can help in MDR problems.

Acknowledegments

We are thankful to I.I.T. Mumbai for ESR analysis. We also acknowledge SAIF, CDRI Lucknow for micro analysis and spectral analyses. Thanks are also due to the Head, Department of Chemistry, Botany and Physics, Dr. Hari Singh Gour University, Sagar (M.P.) for providing Laboratory facilities.

References

1. S Malik, S Ghosh, L Mitu. Complexes of some 3d-metals with a Schiff base derived from 5-acetamido-1,3,4-thiadiazole-2-sulphonamide and their biological activity. **J. Serb. Chem. Soc., 76(10),** 1387–1394 (2011).
2. N Raman, S Raja, J Joseph, J Raja. Synthesis, spectral characterization and DNA cleavage study of heterocyclic Schiff base metal complexes. **J. Chilean Chem. Soc., 52,** 1138-1144 (2007).
3. Z Chohan. Ni(II), Cu(II) and Zn(II) metal chelates with some thiazole derived Schiff-bases: their synthesis, characterization and bactericidal properties. **Metal Based Drugs, 6,** 75-79 (1999).
4. G Bagihalli, S Patil, P Badami. Synthesis, physicoche investigation and biological studies of Zn(II) complexes 1,2,4-triazole Schiff bases. **J. Iran Chem. Soc., 6,** 25 (2009).
5. J Parekh, P Inamdhar, R Nair, S Baluja, S Chanda. Synt and antibacterial activity of some Schiff bases derived fr aminobenzoic acid. **J. Serb. Chem. Soc., 70(10),** 1155– (2005).
6. R Coombs, M Ringer, J Blacquiere, J Smith, J Neilse Yoon-Seo, J Gilbert, L Leger, H Zhang, A Irvin Wheaton, C Vogles, S Westcott, A Decken, F Baerlo Palladium(II) Schiff base complexes derived sulfanilamides and aminobenzothiazoles. **Transition M Chem., 30(4),** 411-418 (2005).
7. R Garg, M Saini, N Fahmi, R Singh. Spectroscopic biochemical studies of some manganese(II), oxovanadiu and dioxovanadium(VI) complexes S/O and N donor a synthesized under microwave conditions. **Transition M Chem., 31(3),** 362-371 (2006).
8. K Mahajan, M Swami, R Singh. Microwave synthesis, sp studies, antimicrobial approach and coordination behavi antimony(III) and bismuth(III) compounds with b thiazoline. **Russian J. Coord. Chem., 35(3),** 179-185 (200
9. K Mahajan, N Fahmi, R Singh. Synthesis, characteriz and antimicrobial studies of Sb(III) complexes of substi thioimines. **Indian J. Chem., A46(8),** 1221-1225 (2007)
10. K Sharma, R Singh, N Fahmi, R Singh. Microwave ass synthesis, characterization and biological evaluatio palladium and platinum complexes with azometh **Spectrochim. Acta, A75(1),** 422-427 (2010).
11. K Mohanan, B Kumari, G Rijulal. Microwave ass synthesis, spectroscopic, thermal and antifungal studi some lanthanide(III) complexes with a heteroc bishydrazone. **J. Rare Earths, 26,** 16-21 (2008).
12. B Garg, D Kumar. Spectral studies of complexes of nick with tetradentate Schiff bases having N_2O_2 donor gr **Spectrochim. Acta, A59(2),** 229-234 (2003).
13. R Dubey, U Dubey, C Mishra. Synthesis and physicoche characterization of some Schiff base complexes chromium(III). **Indian J. Chem., A47(8),** 1208-1212 (2008
14. K Nakamoto. Infrared and Raman Spectra of Inorganic Coordination Compounds, 5th ed Part A and B, New Y John Wiley & Sons (1998).
15. M Neelakantan, S Marriappan, J Dharmaraja, T Jeyakum Muthukumaran. Spectral, XRD, SEM and biological acti of transition metal complexes of polydentate ligands conta thiazole moiety. **Spectrochim. Acta, A71(2),** 628-635 (200
16. G Mohamed, M Omar, A Hindy. Metal complexes of S bases: preparation, characterization and biological act **Turk J. Chem., 30,** 361-382 (2006).
17. ABP Lever. Inorganic Electronic Spectroscopy, 2nd Ed. York; Elsevier (1984).

18. S Chandra, D Jain, A Sharma, P Sharma. Coordination modes of a Schiff base pentadentate derivative of 4-aminoantipyrine with cobalt(II), nickel(II) and copper(II) metal ions: synthesis, spectroscopic and antimicrobial studies. **Molecules, 14,** 174-190 (2009).

19. B. Cristovao. Spectral, thermal and magnetic properties of Cu(II) and Ni(II) complexes with Schiff base ligands. **J. Serb. Chem. Soc., 76(12),** 1639–1648 (2011).

20. R Dutta, A Syamal. Elements of Magneto Chemistry, 2nd Ed, New Delhi; Affiliated East West Press (1993).

21. B Hathaway. Comprehensive Coordination Chemistry, Pergamon Press (UK) 5, 534-540, (1987).

22. A Mishra, L Pandey, Synthesis, characterization and solid state structural studies of oxovanadium(IV)-O,N donor Schiff base chelates. **Indian J. Chem., A44(1),** 94-97 (2005).

23. A Al-Shihri, H Abdel-Fattah. Thermogravimetric and spectroscopic characterization of trivalent lanthanide chelates with some Schiff bases, **J. Therm. Anal. Cal., 71,** 643-649 (2003).

24. Y Wang, J Liu, H Xian, G ZhaoL. Synthesis, crystal structure and kinetics of thermal decomposition of the nickel(II) complex of the Schiff base 2-[(4-methylphenylimino)-methyl]-6-methoxyphenol. **Molecules, 14,** 2582-2593 (2009).

25. K Mohanan, C Athira, Y Sindhu, M Sujamol. Synthesis, spectroscopic characterization, electrochemical behavior and thermal decomposition studies of some transition metal complexes with an azo derivative. **Spectrochim. Acta, A75(1),** 106-112 (2010).

26. A Mishra, M Soni. Synthesis, structural and biological studies of some Schiff bases and their metal complexes. **Metal Based Drug,** doi:10.1115/2008/875410(2008).

27. J Makode, A Yaul, S Bhadange, A Aswar, Physicochemical characterization, thermal and electrical conductivity studies of some transition metal complexes of bis-chelating Schiff base. **Russian J. Inorg. Chem., 54,** 1372-1378 (2009).

28. G Mohamed, M Omar, A Ibrahim. Biological activity studies on metal complexes of novel tridentate Schiff base ligand Spectroscopic and thermal characterization. **Eur. J. Med. Chem., 44,** 4801-4812 (2009).

29. Z Chohan, A Munawar, C Supuran. Transition metal ion complexes of Schiff bases synthesis, characterization and antibacterial properties. **Metal Based Drugs, 8,** 137-143 (2001).

30. W Hanna, M Moawad. Synthesis, characterization and antimicrobial activity cobalt(II), nickel(II) and copper(II) complexes with new asymmetrical Schiff base lagands derived from 7-formyanil- substituted diamine-sulphoxine and acetylacetone. **Transition Metal Chem., 26(6),** 644-651 (2001).

31. J Iqbal, S Tirmizi, F Watto, M Imran. Biological Properties of Chloro-Salicylidene Aniline and its Complexes With Co(II) And Cu(II). **Turk J. Biol., 30,** 1-4 (2006).

32. V Singh, A Katiyar. Synthesis, characterization of some transition metal(II) complexes of acetone p-amino acetophenone salicyloyl hydrazone and their antimicr activity. **Bio Metals, 21(4),** 491-501 (2008).