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Research Article

Eco-Friendly Synthesis, Characterization and Biological Significance of Some Transition Metal Complexes

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ABSTRACT

This work details the synthesis and comprehensive structural analysis of VO(IV) and Co(II) derived from 2-pyridine carboxaldehyde with isoniazide (PCI) have been synthesized by conventional as well as microwave methods. Elemental analysis, FTIR, FAB-mass, molar conductance, electronic spectra and magnetic susceptibility have characterized these compounds. The complexes are coloured and stable in air. Analytical data revealed that all the complexes exhibited a 1:1 (metal: ligand) ratio with a coordination number of 4 or 6. FAB-mass data show a degradation pattern of the complexes. The Schiff base and metal complexes show a 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 base. Chelation tends to make the ligand act as a more powerful and potent bactericidal agent. Further chelation can help in MDR problems.

Keywords: Microwave synthesis, FAB-mass, IR, UV-vis, Biological activities.

INTRODUCTION

A large number of Schiff bases and their metal complexes have been found to possess important biological, catalytic activity inorganic, analytical and medicinal chemistry because of their flexibility when coordinated with different transition metal ions. Due to their diverse uses and chemical activity, such metal complexes containing Schiff bases have been widely researched in recent years. Coordination chemistry, or the study of Schiff base ligands interacting with metal ions, has remained one of the most active research topics in inorganic chemistry. Organic and inorganic molecules are linked via coordination compounds. Researchers have been paying close attention to Schiff bases as nitrogen, sulfur, oxygen donor ligands in coordination with a variety of transition metal ions because of their notable biological applications in anticancer, antitumor, analgesic and antiinflammatory, antibacterial and antifungal activity, etc [1-4].

The present-day industrialization has led to immense environmental deterioration. The increasing environmental consciousness throughout the world has put a pressing need to develop an alternate synthetic approach for biologically and synthetically important compounds. This requires a new approach, which will reduce the material and energy intensity of chemical processes and products, minimize or eliminate the dispersion of harmful chemicals in the environment in a way that enhances the industrially benign approach and meets the challenges of green chemistry. Over the past two decades, the green approach to the synthesis of Schiff base ligands and their metal complexes has gained significant attention due to its sustainability and environmental benefits. Microwave-assisted reactions offer reduced pollution, low cost and high yield together with simplicity in processing and handling.[5-8]

In this paper, we have described the synthesis, physicochemical characterization, and biological significances of VO(IV) and Co(II) complexes with a ligand derived from 2-pyridine carboxaldehyde with isoniazide (PCI) (Fig. 1). The reaction was carried out by both conventional and microwave methods.

EXPERIMENTAL

MATERIALS AND METHODS

All the chemicals and solvents were of analytical grade. All the reagents used for the preparation of the Schiff base were obtained from HI media. Metal salts were purchased from CDH Chemie. The progress of the reaction was monitored by silica gel-G coated TLC plates using a MeOH: $CHCl_3$ system (1:9). The spot was visualized by exposing the dry plate to an iodine vapour chamber. Elemental analyses were performed on the Heroes elemental analyzer, SAIF, CDRI, Lucknow. Molar conductance measurements were conducted using 10^{-3} M solutions of the complexes in methanol on Elico-CM 82 Conductivity Bridge at room temperature. Magnetic susceptibility measurements were carried out on a Gouy balance at room temperature using $CuSO_4.5H_2O$ as the calibrant. Diamagnetic corrections were applied in compliance with Pascal's constant. The

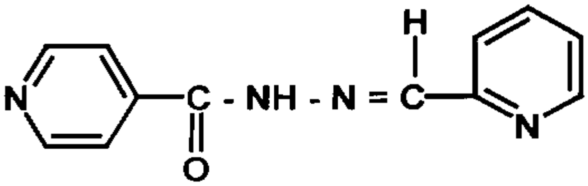

Fig 1: Structure of Schiff base ligand (PCI)

FAB mass spectra were recorded at room temperature on a JEOL SX 102/DA-6000 mass spectrometry/data system using argon/xenon (6 kV, 10 mA) as the FAB gas (accelerating voltage 10 kV) at SAIF. CDRI, Lucknow. FTIR spectra were recorded in KBR medium on a Perkin Elmer RX1 spectrophotometer, SAIF, CDRI Lucknow and SAIF Panjab University, Chandigarh in ware wavenumber region 4000-400 cm^{-1}. Electronic Spectra (in MeOH) were recorded on a Perkin-Elmer Lambda-2- 2B Spectrophotometer (range 200–700 nm) at the Department of Chemistry, Dr. Harisingh Gour University, Sagar (M.P.). Microwave-assisted synthesis was carried out in an open glass vessel on a modified microwave oven model 2001 ETB with a rotating tray and a power source 230 V, microwave energy output of 800W and microwave frequency 2450 MHz. A thermocouple device was used to monitor the temperature inside the vessel of the microwave. The microwave reactions were performed using on/off cycling to control the temperature.

Antimicrobial Activity

The *in-vitro* biological activity of the investigated Schiff base and its metal complexes was evaluated using the disk diffusion method to test their effectiveness against the gram-positive bacteria, *Staphylococcus aureus* and gram-negative bacteria, *Escherichia coli,* using nutrient agar as medium and streptomycin as a control. The antifungal activities of the compounds were also tested by the well diffusion method against the fungi *Aspergillus niger* and *Candida albicans*, on potato dextrose agar as the medium and Nystatin as a control. The stock solution was prepared by dissolving the compounds in DMSO. In a typical procedure, a well was made on agar medium inoculated with a microorganism. The well was filled with the test solution using a micropipette and the plate was incubated for 24 hours for bacteria at 37°C and 72 hours for fungi at 30°C. During this period, the test solution diffused and the growth of the inoculated microorganism was affected.

Conventional synthesis of Schiff base

The Schiff base PCI has been synthesized by adding the methanolic solution of 2-Pyridinecarboxaldehyde to the methanolic solution of isoniazide in an equimolar ratio. The reaction mixture was then refluxed on a water bath for about 4.5 hours. The condensation product was filtered, thoroughly washed with ethanol and ether, recrystallized and dried in vacuo. The purity of the synthesized compound was monitored by TLC using silica gel G (Yield: 75.5%).

Microwave method for the synthesis of Schiff base

The equimolar (1:1) ratio of pyridinecarboxaldehyde and isoniazide (PCI) was mixed thoroughly in a grinder. The microwave oven then irradiated the reaction mixture by taking 3 to 4 mL of solvent. The reaction was completed in a short time (5.5 minutes) with higher yields. The resulting product was then recrystallized with ethanol and finally dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. The progress of the reaction and purity of the product were monitored by TLC using silica gel G (yield: 85%).

Conventional synthesis of metal complexes

The metal complexes have been prepared by mixing (50 mL) methanolic solution of $VOSO_4.5H_2O/CoCl_2.6H_2O$ with the (50 mL) methanolic solution of Schiff base (PCI) in 1:1 (metal:ligand) ratio. The resulting mixture was refluxed on a water bath for 7 to 9 hours. A coloured product appeared on standing and cooling the above solution. The precipitated complex was filtered, washed with ether and recrystallized with ethanol several times and dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. It was further dried in an electric oven at 50 to 70°C (yield: 63–68%).

Microwave method for the synthesis of metal complexes

The ligand and the metal salts were mixed in 1:1 (metal:ligand) ratio in a grinder. The microwave oven then irradiated the reaction mixture by taking 3 to 5 mL of solvent. The reaction was completed in a short time (7–8 minutes) 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 were monitored by TLC using silica gel G (yield: 76–82%).

RESULTS AND DISCUSSION

Analytical and Physical Data

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 the reaction mixture was increased by the rotation of the reaction platform tray. The repeating of the synthesis process also checked the confirming of the results.

All the metal complexes are colored, solid and stable towards air and moisture at room temperature. They decompose at high temperatures on heating. The comparative results of conventional and microwave methods and analytical data of the compounds, together with their physical properties, are consistent with the proposed molecular formula and magnetic moment values are given in Table 1. Elemental analysis of the complexes indicates the stoichiometry to be 1:1 metal:ligand (Schiff base). The molar conductance in methanol (10^3M) of the complexes is 151.5 and 80.6 $\Omega^{-1}cm^2mol^{-1}$, respectively, for Co(II) and VO(IV) complexes. This indicates the uni-bivalent electrolytic nature [9,10].

FAB-mass spectra

The FAB mass spectra of the ligand (PCI) and its cobalt complex $[Co(C_{12}H_{10}N_4O)H_2O].Cl_2.H_2O$ was recorded and they are used to compare their stoichiometry composition. The Schiff base shows a molecular ion peak at m/z 230. The $[Co(C_{12}H_{10}N_4O)H_2O].Cl_2.H_2O$ complex showed a molecular ion peak at m/z 395 confirm the stoichiometry of metal complexes as ML type. It is good agreement with the microanalytical data [11,12].

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

Molcular formula/Mol.Wt./(Colour)	Reaction period		Yield (%)		Elemental analysis, found (Calcd.) %			*Λ_m
	CM (h.)	MM (min.)	CM	MM	C	H	N	
$C_{12}H_{10}N_4O$ (PCI) 226.0 (White)	4.5	5.5	75	85	63.9 (63.7)	4.3 (4.4)	24.8 (24.7)	-
$[VO(C_{12}H_{10}N_4O)H_2O].SO_4.3H_2O$ 442.9 (Dark Brown)	7.2	7.2	68	82	32.2 (32.5)	4.2 (4.0)	11.5 (12.6)	80.6
$[Co(C_{12}H_{10}N_4O)H_2O].Cl_2.H_2O$ 391.9 (Dark Green)	9.0	7.9	63	76	36.5 (36.7)	3.9 (3.9)	11.5 (11.2)	151.5

*$\Lambda_m = (\Omega^{-1}cm^2mol^{-1})$

IR spectra

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

In the IR spectrum of the ligand (PCI) amide I band ν(C—O) appears at 1666 cm^{-1}, which has shifted towards the lower frequency region 1639 ± 10 cm^{-1} in the spectra of the complexes, indicating a decrease in the stretching force constant of the C=O bond as a consequence of coordination through the carbonyl oxygen atom of the free base. The band due to N-H stretching (assy. and sym.) in the free ligand occurs at 3449 and 3293 cm^{-1} and remains unaffected after complexation. This precludes the possibility of coordination through the imine nitrogen atom. Another important band, which occurs at 1630 cm^{-1}, is attributed to ν(C=N) azomethine mode. This band shifts down by 15 to 20 cm^{-1} in the complexes, indicating the involvement of the azomethine group in coordination through the nitrogen atom. The spectrum of the complexes exhibits a symmetric shift in the position of the band in the region 1544 to 1358 cm^{-1} and 1065 to 1000 cm^{-1} due to ν(C=C) and ν(C=N) vibration modes and their mixing patterns are different from those present in ligand spectra. These observations suggest the participation of pyridinic nitrogen in complexation. The IR spectra of complexes exhibited a broad band at about 3490 ± 16 cm^{-1} (stretching) and new bands at 518 ± 2 and 435 ± 10 cm^{-1} in the complexes have tentatively been assigned to ν(M-O) and ν(M-N) bond, respectively.

A very sharp peak at 983 cm^{-1} suggests the presence of cm^{-1} V=O bond in VO(IV) complex. The presence of an ionic sulphate group in VO(IV) complex has been confirmed by the appearance of three bands at 1119(ν_3), 905(ν_1) and 613(ν_4) cm^{-1}. The absence of the ν_2 band and the non-splitting of ν_3 band indicate that T_d symmetry is still held [13-17].

Magnetic Moments and Electronic Spectra

The electronic spectral data of the metal complexes in MeOH solution are given in Table 2. The nature of the ligand field around the metal ion has been deduced from the electronic spectra.

The electronic spectrum of Co-PCI complex shows two bands of appreciable intensity at 12376 and 20408 cm^{-1}, which have tentatively been assigned to $^4A_2$-$^4T_1$ (F) (ν_2) and $^4A_2$-$^4T_1$ (P) (ν_3) transition. The magnetic moment is 4.15 B.M. Thus, the tetrahedral geometry has been suggested for this complex.

Table 2: Electronic spectral and magnetic moment values of complexes

S.No.	Complexes	Transitions	Bands(cm^{-1})	Magnetic moment (B.M.)
1.	VO(IV)-PCI	$^2B_2$-$^2E(\nu_1)$	12312	1.79
		$^2B_2$-$^2B_1(\nu_2)$	20408	
		$^2B_2$-$^2A_1(\nu_3)$		
2.	Co(II)-PCI	$^4A_2$-$^4T_1(F)(\nu_2)$	12376	4.15
		$^4A_2$-$^4T_1(P)(\nu_3)$	20408	

VO(IV)-PCI complex exhibits two bands at 12312 and 20408 cm^{-1}, which have tentatively been assigned to $^2B_2$-^2E (ν_1) and $^2B_2$-$^2B_1(\nu_2)$ transition. The magnetic moment is 1.79 B.M. This data suggests the trigonal bipyramidal/square pyramidal geometry for VO(II) complex [18-22].

Antimicrobial activities

The *in-vitro* Antimicrobial activity of the synthesized Schiff base ligands and their corresponding metal complexes (Fig. 2) on selected bacteria, *E. coli* and *S. aureus* and two fungi, *A. niger* and *C. albicans,* was carried out. All of the tested compounds showed good biological activity against microorganisms. On comparing the biological activity of the Schiff base and its metal complexes with the standard bactericide and fungicide, it is shown that some metal complexes have good activity as compared to the standard, but all the complexes are more active than their respective ligands. The higher inhibition zone of metal complexes than that of the ligands can be explained on the basis of Overton's concept and the Chelation theory. On chelation, the polarity of the metal ion will be reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of the positive charge of the metal ion with donor groups. Further, it increases the delocalization of π-electrons over the whole chelating ring and enhances the penetration of the complexes into lipid membranes and the blocking of the metal binding sites in the enzymes of microorganisms. There are other factors that also increase the activity, such as solubility, conductivity and bond length between the metal and ligand [23-28].

The antibacterial and antifungal data are listed in Table 3. The investigation of antibacterial data revealed that the Co(II) and VO(IV) complex displayed highly activity against bactericides and fungicides.

Table 3: *In-vitro* antibacterial activity of compounds and their inhibition zone (%)

Compound	*Diameter of inhibition zone (mm), concentration in ppm*											
	Antibacterial screening data						*Antifungal screening data*					
	E. coli			*S. aureus*			*A. niger*			*C. albicans*		
	25	**50**	**100**	**25**	**50**	**100**	**25**	**50**	**100**	**25**	**50**	**100**
PCI	15	18	24	16	18	22	12	14	16	13	16	20
VO(IV)-PCI	21	23	27	19	22	26	20	24	30	20	24	28
Co(II)-PCI	20	22	25	16	20	22	16	22	26	17	20	25
Streptomycin	22	24	28	18	22	26	-	-	-	-	-	-
Nystatin	-	-	-	-	-	-	22	26	30	22	24	28

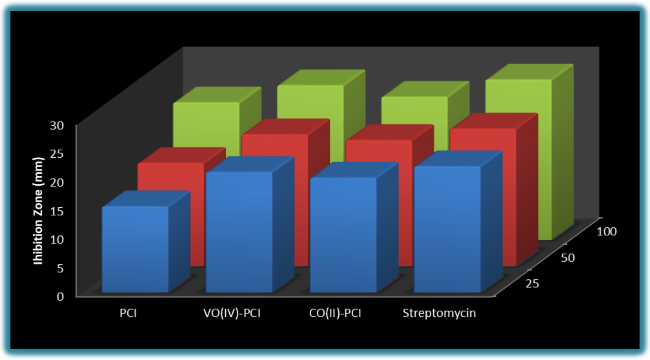

Fig. A: Antibacterial activity of PCI Schiff base and its metal complexes against *E. coli*

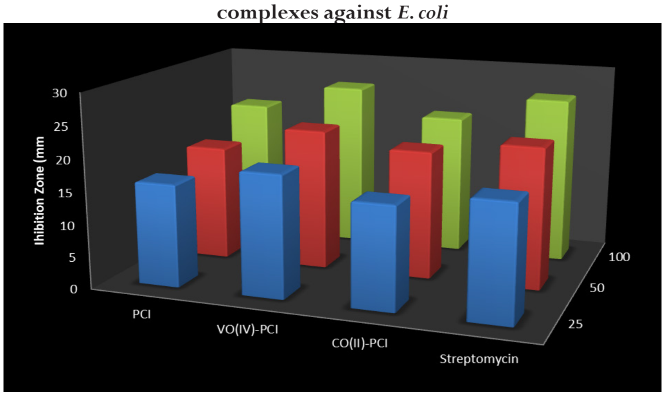

Fig. B: Antibacterial activity of PCI Schiff base and its metal complexes against *S. aureus*

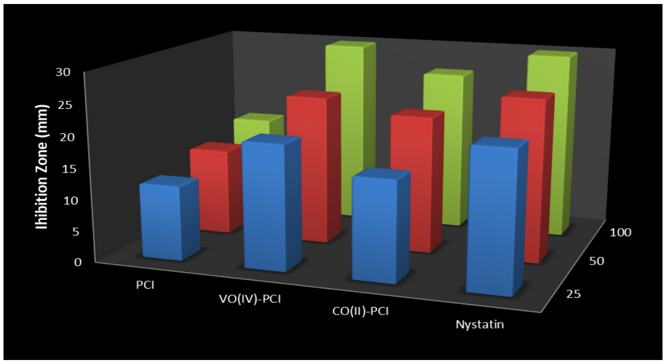

Fig. C: Antifungal activity of PCI Schiff base and its metal complexes against *A. niger*

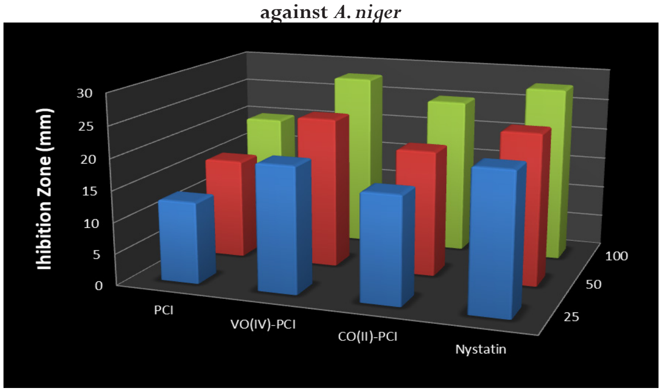

Fig. D: Antifungal activity of PCI Schiff base and its metal complexes against *C. albicans*

Fig. 2 (A-D): Biological activity of Schiff base and its metal complexes

CONCLUSION

In the present research studies, our efforts are synthesized from some newly discovered compounds from the conventional as well as microwave methods. Various physicochemical and spectral analyses characterize these synthesized compounds. As a result of microwave synthesis, it has been observed that the reaction time decreased from hours to minutes and the availability of the product within better yields compared to the conventional methods. FAB-mass shows the degradation pattern of the complexes. The antimicrobial data show that the metal complexes are more biological active compared to the parent Schiff base ligand against all pathogenic species. The compounds also inhibit the growth of fungi and bacteria to a greater extent as the concentration is increased.

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