MICROWAVE SYNTHESIS, SPECTRAL AND ANTIMICROBIAL STUDIES OF SOME SCHIFF BASE METAL COMPLEXES

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Abstract

Microwave-assisted synthesis is a branch of green chemistry. The salient features of microwave approach are shorter reaction times, simple reaction conditions and enhancements in yields. Some new Schiff base complexes of VO(IV) and Co(II) derived from 4-dimethyl aminobenzaldehyde with 3, 4-dichloroaniline (DCA) have been synthesized by conventional as well as microwave methods and characterized by elemental analysis, FT-IR, FAB-mass, ESR, molar conductance, and thermal analysis. FAB mass and thermal data show degradation pattern of the complexes. 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. 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 base and metal complexes show a good activity against the bacteria; *E. coli, S. aureus, S. fecalis* and fungi *A. niger, T. polysporum*. The antimicrobial results also indicate that the metal complexes are better antimicrobial agents as compared to the Schiff bases.

Keywords: Microwave synthesis; Schiff base; 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. Because of increasing biological and catalytic significance of vanadium, intense attention has been focused on it, over the past two decades. Vanadium constitutes 0.015% of earth's crust which is nearer to abundance of zinc. Biochemical role of vanadium has now become a widely chosen topic of bioinorganic chemistry. The Schiff base ligands widely vary in their structure flexibility, electronic nature and the presence of additional donor atoms besides imino nitrogen. The central metal in these complexes act as active sites and thereby successfully catalyse chemical reactions. The Schiff base transition metal complexes are a family of attractive oxidation catalysts for a variety of organic substrates because of their cheap & easy synthesis and their chemical & thermal stability [1-6].

Microwave-assisted synthesis is a branch of green chemistry. Microwave irradiated reactions are offering reduced pollution, low cost and offer high yield together with simplicity in processing and handling. The basis of this technique of synthesis is much faster with high yields compared to conventional methods. The salient features of microwave approach are shorter reaction times, simple reaction conditions and enhancements in yields [7-13].

In this study we report the synthesis and physicochemical characterization of Co(II) and VO(IV) complexes with ligands derived from 4-dimethyl aminobenzylidine-3,4-dichloroaniline (DCA) (Figure 1). The metal complexes formed with new ligand may be used as precursors for the synthesis of new compounds. Some of them may exhibit interesting physical chemical and biological properties.

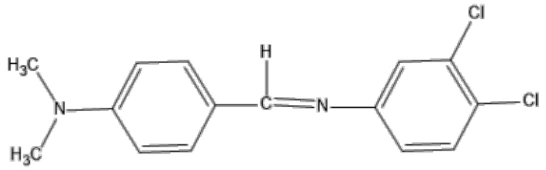

Figure 1. Structure of Schiff base Ligands

Experimental:-

All the used chemicals and solvents were of Anal R grade. All the reagents used for the preparation of the Schiff base were obtained from HI media. Metal salts were purchased from CDH Chemie. Elemental analyses were performed on Heroes elemental analyser SAIF, CDRI, Lucknow. Electronic spectra (in MeOH) were recorded on Perkin Elmer Lambda- 2B Spectrophotometer (range 200-700 nm) at Department of Chemistry, Dr. Harisingh Gour University, Sagar (M.P.). 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. FT-IR spectra were recorded in KBr medium on a Perkin Elmer RX1 spectrophotometer SAIF, CDRI Lucknow and SAIF Panjab University, Chandigarh in wave number region 4000-400 cm^{-1}. X-band EPR spectra were recorded on a Varian E-112 spectrometer at room temperature operating at the X-band region with 100 kHz modulation frequency, 5 mw microwave power and 1 G modulation amplitude using TCNE as the internal standard. Microwave assisted synthesis were carried out in open glass vessel on a modified microwave oven model 2001 ETB with rotating tray and a power source 230 V, microwave energy output 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.

Conventional synthesis of the Ligand:

DCA Schiff base was synthesized by the condensation of 1:1 ratio of 4-dimethyl aminobenzaldehyde with 3, 4-dichloroaniline dissolved in methanol. The resulting reaction mixture was refluxed for 4.5 hrs and then allowed to cool overnight. The coloured solid precipitate of Schiff base obtained was filtered, washed with cold ethanol and finally recrystallized from ethanol and ether and dried in air at room temperature and preserved in a $CaCl_2$ desiccator. The purity of synthesized compounds was checked by TLC using silica gel G (yield: 76.7%).

Microwave method for the Synthesis of Schiff bases:

The equimolar (1:1) ratio of 4-dimethyl aminobenzaldehyde and 3, 4-dichloroaniline with methanolic solution were mixed thoroughly in a grinder. The reaction mixture was then irradiated by the microwave oven by taking 3-4 mL of dry ethanol as a solvent. The reaction was completed in a short time (5 min) with higher yields. The resulting product was then recrystallized with ethanol, finally dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. The progress of the reaction, purity of the product was monitored by TLC using silica gel G (yield: 87%).

Conventional method for the Synthesis of metal complexes:

The metal complexes have been prepared by mixing the methanolic solution of $VOSO_4.5H_2O/CoCl_2.6H_2O$ (0.003 mole) to the methanolic solution of Schiff base (DCA)

(0.006 mole) in 1:2 molar ratio. The resulting mixture was then refluxed on water bath for about 8-10 hours. A coloured product appeared on standing and cooling the above solution. The complex was filtered, washed with ether and dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. It was further dried in an electric oven at 30-70°C.

Microwave method for the Synthesis of metal complexes:

The ligand and the metal salts were mixed in 1:2 (metal:ligand) ratio in a grinder. The reaction mixture was then irradiated by the microwave oven by taking 3-4 mL of dry ethanol as a solvent. The reaction was completed in a short time (6-9 min) 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: 80-82%).

Biological activity:

The *in-vitro* biological activity of the Schiff base and their complexes was tested against the bacteria *Escherichia coli*, *Staphylococcus aureus* and *S. feacalis* by disc diffusion method using nutrient agar as medium and gentamycin as control. The antifungal activities of the compounds were also tested by the Well diffusion method against the fungi *Aspergillus niger*, and *Trichoderma polysporum*, cultured on potato dextrose agar as medium. In a typical procedure, a well was created on the agar medium and nystatin as the control was inoculated with the fungi. Each of the compounds was dissolved in DMSO and solutions of the concentrations (25, 50 and 100 ppm) were prepared separately. In a typical procedure, a well was made on agar medium inoculated with microorganism. The well was filled with the test solution using a micropipette and the plate was incubated 24 hrs for bacteria at 37°C and 72 hs for fungi at 30°C. During this period, the test solution diffused and the growth of the inoculated microorganism was affected.

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 stable towards air and moisture at room temperature. They decompose on heating at high temperature, more or less soluble in common organic solvents. The comparison study data of microwave and conventional methods, with analytical and physical data of the compounds are given in the Table 1. Elemental analysis of the complexes indicates the stoichiometry to be l:2 metal:ligand (Schiff base). The molar conductance in methanol, of the complexes is 31.5 and 81.3 S cm^2 mole^{-1} this suggest the non-electrolytic nature of Co(II) complex and uni-bivalent electrolytic nature of VO(IV) complex [14,15].

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

Compound	Reaction period		Yield (%)		Elemental analysis, found (calcd.) %			$^*\Lambda_m$	#μ_{eff}
Mol. Wt. (Colour)	CM (hs)	MM (min.)	CM	MM	C	H	N		
$C_{15}H_{14}N_2Cl_2$ (DCA) 293 (Light Yellow)	4.0	5.0	76.7	87.0	61.6 (61.4)	4.2 (4.7)	9.7 (9.5)	-	-
$[VO(C_{15}H_{14}N_2Cl_2)_2(H_2O)_2].SO_4.H_2O$ 802.9 (Turmeric)	8.9	6.9	71.2	82.1	45.3 (45.8)	4.1 (4.0)	6.9 (6.9)	81.3	1.77
$[Co(C_{15}H_{14}N_2Cl_2)_2Cl_2].4H_2O$ 787.9 (Tobacco Green)	9.2	8.2	68.5	80.5	45.8 (45.6)	4.3 (4.5)	6.9 (7.1)	31.5	4.39

CM = Conventional method, time in hours; MM = Microwave method, time in minutes

$^*\Lambda_m$ = (Ω^{-1}cm^{2}mol^{-1}); #μ_{eff} = B.M.

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 DCA ligand medium intensity band at 1578 cm^{-1}due to *v*(C=N) azomethine group has shifted to lower wave numbers by 20-25 cm^{-1} in the complexes. It indicates that coordination takes place through the azomethine nitrogen. The appearance of broad bands at 3340 and 3380 cm^{-1} in the spectra of both the complexes have been assigned to associated water molecules. However, a medium intensity band at 669 cm^{-1} in VO(IV) complex is assignable to rocking mode of coordinated water molecule. The new bands at 461±17 cm^{-1} in both the complexes and a band at 562 cm^{-1} in VO(IV) complex have been assigned to (M-N) and (M-O) bonding respectively. A new band appears at 977 cm^{-1} has been assigned to ν(V=O) vibration [16-18].

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-DCA complex shows two bands of appreciable intensity at 12388 cm^{-1} and 20325. These transitions have tentatively been assigned to $^4A_2$-$^4T_1$ (F) (ν_2) and $^4A_2$-$^4T_1$ (P) (ν_3) respectively. The magnetic moment is 4.30 B.M. Thus the tetrahedral geometry has been suggested for this complex. Oxovanadium(IV)- DCA complex exhibit two bands at 12471 cm^{-1} and 21341 cm^{-1} which have tentatively been assigned to $^2B_2$-^2E (ν_1) and $^2B_2$-$^2B_1$(ν_2) transition. The magnetic moment is 1.77 B. M. This data suggest the trigonal bipyramidal/square pyramidal geometry for VO(IV) complex [19-24].

Table 2: **Electronic spectral and magnetic moment values of complexes**

S.No.	Complexes	Transitions	Bands(cm^{-1})	Magnetic Moment (B.M.)
1.	VO(IV)-DCA	$^2B_2$-^2E(ν_1)	12471	1.77
		$^2B_2$-$^2B_1$(ν_2)	22341	
		$^2B_2$-$^2A_1$(ν_3)		
2.	Co(II)-DCA	$^4A2$-$^4T_1$(F)(ν_2)	12388	4.30
		$^4A_2$-$^4T_1$(P)(ν_3)	20325	

ESR Spectra:

The spectra of VO(IV) complex have been recorded on X-band EPR spectra were recorded on a Varian E-112 spectrometer at room temperature operating at the X-band region with 100 kHz modulation frequency, 5 mw microwave power and 1 G modulation amplitude using TCNE as the internal standard. The values of ESR parameters of VO(IV) complex of DCA *viz.* g^{11}, $g^{\perp}$, g_{av}, and Δg are as 1.9198, 1.9604, 1.9468 and 0.0406 respectively. The parameter gav was obtained by equation $[(g_{av}) = 1/3(2g^{\perp} + g^{11})]$ [25, 26].

Antimicrobial activities:

The *in-vitro* Antimicrobial activity of the synthesized Schiff base ligands and their corresponding metal complexes on selected bacteria *E. coli*, *S. aureus* and *S. feacalis* and two fungi *A. niger* and *T. Polysporum* was carried out. All of the tested compounds showed good biological activity against microorganism. On comparing the biological activity of the Schiff base and its metal complexes with the standard bactericide and fungicide, it is show that the 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 those of the ligands can be explained on the basis of Overtone's concept and Chelation theory. On chelation, the polarity of the metal ion will be reduced to 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 blocking of the metal binding sites in the enzymes of microorganisms. There are other factors which also increases the activity are solubility, conductivity and bond length between the metal and ligand [27-31].

The bactericidal and fungicidal investigation data of the compounds are summarized in Tables 3 and 4. The results of the investigations account for the antipathogenic behavior of the compounds and this efficacy is positively modified on complexation.

Table 3. Antibacterial screening data for the ligands and their complexes

Comp.	Diameter of inhibition zone (mm)								
	E.Coli			***S.aureus***			***S.feacalis***		
	25	**50**	**100**	**25**	**50**	**100**	**25**	**50**	**100**
DCA	11	13	15	11	12	18	10	14	20
Co(II)	20	24	27	13	14	16	12	14	18
VO(IV)	14	16	18	12	12	15	12	13	17
Gentamycin (Standard)	22	24	28	100	100	100	18	22	24

Table 4. Antifungal screening data for the ligands and their complexes

Compound	Diameter of inhibition zone (mm); Concentration in ppm					
	A. niger			***T. Polysporum***		
	25	**50**	**100**	**25**	**50**	**100**
DCA	10	14	22	11	15	20
Co(II)	17	19	22	19	22	24
VO(IV)	16	18	20	18	20	22
Nystatin	20	22	24	23	25	27

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 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. 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.

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