The International Journal Of Engineering And Science (Ijes)
||Volume||2 ||Issue|| 2 ||Pages|| 166-168 ||2013||
Issn: 2319 – 1813 Isbn: 2319 – 1805

Microwave Assisted Synthesis Spectral and Antibacterial Investigations on Co (II) Complexes With Amide Ligands

[1,]Neha Yadav, [2,]N. Bhojak
[1,]*Department Of Chemistry, School Of Applied Sciences, Singhania University*
[2,]*GCRC, P.G. Department of Chemistry, Govt. Dungar College (A-Grade), University of Bikaner, Bikaner – 334003*

----------Abstract----------
The Present research work describes the synthesis, spectral and antibacterial studies on the complexes of few complexes of Cobalt (II) with amide group containing ligands. Synthesis of complexes has been carried out by conventional method as well as by microwave method. The complexes have been characterized on the basis of elemental analysis, infrared, electronic spectra and magnetic susceptibility studies.. The microwave method of synthesis of complexes have been found easier, convenient and ecofriendly.

Key Word: *Cobalt (II), microwave, amide ligands*

Date Of Submission: 12, February, 2013 Date Of Publication: 27, February 2013

I. Introduction

Cobalt in small amount is essential to many living organism, including humans. It is a central compound of the vitamin B12 (cobalamin), which has two major coenzyme functions in the body first of methylcobalamin promoters methionine synthesis, (Methionine supply ultimately influence DNA synthesis) and second Deoxyadenosylcobalmin performs a key role in the energy metabolism precursor of glucose in ruminants.Vitamin B12 is also necessary for myelin formation (an insulating layer found around nerves, to support red blood cells production) and it is also essential for the metabolism of fats, carbohydrates and the synthesis of proteins [1]. When cobalt particles are not bound to or sediment particles the uptake by plants and animals is higher and accumulation in plants and animals may occur. Cobalt is used to treat anaemia with pregnant woman, because it stimulates the production of red blood cells. The total daily intake of cobalt is variable and may be as much as 1 mg, but almost it will pass through the body unabsorbed, except that in vitamin B12. However too high concentration of cobalt may damage human health, when we breathe in too high concentration of cobalt through air we experience lungs effect, such as asthama and pneumonia. It mainly occurs with people that work with cobalt. When plants grow on contaminated soils they will accumulate very small particles of cobalt, especially in the parts of the plant we eat, such as fruits and seeds, can cause health effects such as vomiting and nausea, vision problems, heart problem and thyroid damage [2-3]. The present research work describes the synthesis, spectral and antibacterial studies on the complexes of Ni (II) with amide group containing ligands. The complexes have been characterized on the basis of elemental analysis, infrared, electronic spectra and magnetic susceptibility studies.

II. Experimental

All the chemicals and solvents used were of AR grade. Purity of synthesized compounds has been checked by thin layer chromatography. IR spectra are recorded on FT-IR Perkin –Elmer spectrophotometer RX1 (vmax in cm^{-1}) using KBr disc. ^1H NMR spectra are recorded in $CDCl_3$ on a Bruker DRX-300 MHz using TMS as internal standard. The chemical shifts are reported as parts per million (ppm). Magnetic susceptibility measurements were carried out on the vibrating sample magnetometer (VSM) model 155 at 5500 Gauss field strength. Microwave synthesis was carried out in domestic microwave oven model L.G. MS-194W, 230-50Hz, 800W. Beck Man DU-64 Spectrophotometer, with quartz cell of 10mm light path was used for absorption measurement.

2.1 Microwave Irradiation Synthesis Of Ligands

Four ligands i.e. N, N'-bis-(3-carboxy-1-oxopropanyl)-1,2-ethylenediamine(CPE), N,N'-bis-(3-carboxy-1-oxo-propanyl)-1,2-phenylenediamine (CPP), N,N'-bis-(2-carboxy-1-oxophenelenyl)-1,2-

phenylenediamine(CPPP) N,N'-bis-(3-carboxy-1-oxoprop-2-enyl)-1,2-phenylenediamine (CPP-2) were synthesized. In a typical preparation mixture of amine (1.0 mmol) and carboxylic acid (2.1 mmol) were taken in Erlen Meyer flask capped with a funnel placed in a microwave oven and irradiated at 200 watt for 2 minutes. The reaction was monitored by TLC. After completion the reaction, the reaction mixture was allowed to attain room temperature and solid separated was filtered. The crude product was recrystallized from redistilled ethanol.

2.2 Microwave Irradiation Synthesis Of Co (II) Complexes

For the preparation of various complexes, a slurry of ligand (i.e. CPE, CPP, CPPP or CPP-2) (1 mmol) was prepared in water or in water-ethanol mixture. In this solution of cobalt perchlorate (0.001 mole in 30 ml ethanol) was added. The resulting mixture was irradiated in a microwave oven for 2 to 6 minutes at medium power level (600W) maintaining the occasional shaking. The mixture was cooled to room temperature and poured into ice chilled methanol and dried in vacuum over P_2O_5. In order to synthesize complexes with CPPP prolonged irradiation and cooling was required.Complexes and ligands were also synthesized by conventional method and results were found satisfactory.

III. Results and Discussion

Ligands and complexes were identified on the basis of elemental analysis and spectral studies. Colour, yield and elemental analysis data are represented in Table 1.

Vibrational Spectra: Few diagnostic ir bands are given in Table 2. $\nu_{(C=O)}$ and $\nu_{(C-O)}$ stretching frequencies in the region 1595-1535 cm^{-1} and 1420-1400 cm^{-1} observed for free ligands and assigned to asymmetric and symmetric modes respectively are shifted in the complexes. These shifts consequently increase the difference between the frequencies of asymmetric and symmetric modes of carboxylate group known as $\Delta\nu$. An increase in the value of $\Delta\nu$ has been ascribed to coordination of carboxylate groups to central metal ion in unidentate fashion. The ir bands due to amide $\nu_{(N-H)}$ mode observed at 3397-3209 cm^{-1} for the free ligands are shifted to higher frequencies indicating non-participation of N of amide group in coordination. Amide I bands due to $\nu_{(C=O)}$ shift negatively opposite to that of $\nu_{(N-H)}$ in the complexes suggesting carbonyl oxygen coordination. Bands observed at 500-545 cm^{-1} assigned to $\nu_{(Co-O)}$

Magnetic Moments And Electronic Spectra: Room temperature magnetic moments of the Ni (II) complexes fall in the range 4.74-5.25 BM. These values are typical of distorted octahedral geometry coordinated around nickel which has three unpaired electrons. The visible spectra of these complexes have been measured in methanol are reported in Table 3. The observed values exhibits bands in the region 19040 and 20,725 cm^{-1} to 21,050 cm^{-1} assignable to $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ transitions. These are also typical of tetrahedral environment around the manganese[4-5].

Antibacterial Activity: The antibacterial activity of the compounds against *E.coli* and *S.aureus* were carried out using Muller Hinton Agar media (Hi media). The activity was carried out using paper disc method represented in Table 4. Among the various compounds CPPP and its Co (II) complexes has been found out to be most effective against these microbes showing maximum clarity of zones.

IV. Conclusions

Co (II) complexes were found to coordinate through amide oxygen and carboxylate oxygens as revealed by the i.r. spectroscopy. The magnetic moment for all the complexes recorded corresponds to two unpaired electron. Although we were unable to get single crystals for X-ray studies, magnetic, electronic and vibrational spectroscopic data showed the distorted octahedral geometry for all the complexes.

References

[1] Jarvis, AR and Bresson L, Esnouf S, Lamouroux C, Solari PL & Den Auwer C, XAS Investigation of biorelevant cobalt complexes in aqueous media, New J. Chem; 2006;30:416.

[2] Chohan Zahid H. and Sherazi Syed KA, "Biological Role of Cobalt(II), Copper(II) and Nickel(II) Metal Ions on the Antibacterial Properties of Some Nicotinoyl-Hydrazine Derived Compounds" Met. Based Drugs, 1997; 4: 69.

[3] Kaliman PA, Nikitchenko IV, Sokol QA and Strelchenko EV, Regulation of Heme Oxygenase Activity in Rat Liver during Oxidative Stress Induced by Cobalt Chloride and Mercury Chloride, Biochemistry (Moscow), 2001 ; 66(1) : 784.

[4] Rizzi Alberto C, Brondino Carios D, Calvo Rafael, Baggio Richardo, Garland Maria T & Rapp Raul ES, Structure and Magnetic Properties of Layered High-Spin Co(II)(L-threonine)$_2$(H_2O)$_2$, Inorganic Chemistry, 2003; 42 : 4409.

[5] Singh BK, Bhojak N and Prakash A, "Unsymmetrical Schiff base Functionalized as Monobasic Tridentate Ligand on Complexation with some transition metal ions", E-Journal of Chemistry, 2012 ; 9(2): 104.

Table 1. Physico-Chemical Data of Co (II) complexes (C.M. = conventional method; M.M. = Microwave method)

S.N.	Complex	Colour	Reaction Period		Yield %		Elemental Analysis Calcd(Found) %		
			C.M. h	M.M. min	C.M.	M.M.	C	H	N
1	Co(II)-CPE	Light Brown	3	2	40	55	37.25 (37.10)	5.15 (5.10)	5.0 (5.08)
2	Co(II)-CPP	Light pink	5	4	45	60	45.27 (45.29)	4.40 (4.41)	4.28 (4.41)
3	Co(II)-CPPP	Brown	3	3	35	35	54.52 (54.33)	3.31 (3.48)	3.51 (3.48)
4	Co(II)-CPP2	light Brown	4.5	2.5	40	45	45.6 (46.81)	3.4 (3.34)	3.38 (3.34)

Table 2. IR Spectral assignments of diagnostic bands of ligand and its Co (II) complexes

S.N.	Ligand and complexes	ν_{N-H}	$\nu_{C=O}$[a]	$(\nu_{C-N}+\delta_{N-H})$[b]	$(\nu_{N-H}+\delta_{C-N})$[c]	ν_{COO^-}(asym)	ν_{COO^-}(sym)	(ν_{Co-O})
1.	CPE	3303	1643	1428	1280	1553	1419	---
2.	Co(II)-CPE	3320	1612	1437	1310	1560	1425	513
3.	CPP	3377	1646	1461	1299	1587	1419	---
4.	Co(II)-CPP	3384	1614	1481	1312	1590	1412	544
5.	CPPP	3397	1635	1475	1301	1595	1400	---
6.	Co(II)-CPPP	3403	1624	1512	1322	1572	1323	540
7.	CPP-2	3209	1636	1480	1280	1535	1415	---
8.	Co(II)-CPP2	3320	1622	1492	1294	1540	1401	538

a = amide I band b = amide II band c = amide III band

Table 3. Magnetic moments and electronic spectral data of the Co(II) complexes

.

S.No.	Complex	μ_{eff} (BM)	Electronic Spectral bands $\lambda max(cm^{-1})$	Tentative assignments	Comment
1.	Co(II)-CPE	4.86	19047, 20725, 21053	$^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$	Distorted Octahedral Co(II) geometry
2.	Co(II)-CPP	4.70	19048, 19569, 20986	$^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$	Distorted Octahedral Co(II) geometry
3.	Co(II)- CPPP	5.20	19047, 20725, 21053	$^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$	Distorted Octahedral Co(II) geometry
4.	Co(II)-CPP-2	4.98	19029 20833	$^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$	Distorted Octahedral Co(II) geometry

Table 4. Antibacterial activity of synthesized compounds

S.N.	Compound (100 ppm)	*E.coli*	*S.aureus*
1	CPE	18	10
2	Co(II)-CPE	19	12
3	CPP	16	30
4	Co(II)-CPP	18	24
5	CPPP	20	30
6	Co(II)-CPPP	22	32
7	CPP-2	20	10
8	Co(II)-CPP-2	14	08
9	Chloramphenicol	22	20