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Studies on synthesis and characterization with antimicrobial activity of mixed ligand coordinating co (ii) Complexes with phthalic acid and heterocyclic amines

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Abstract

The synthesis and characterization of mixed ligand complexes of Cobalt(II) with phthalic and heterocyclic amines having composition $[Co(DPH)(Py)_4]$, $[Co(DPH)(Q)_4]$, $[Co(DPH)(IQ)_4]$, $[Co(DPH)(2\text{-}Pic)_4]$ were synthesized and characterized on the basis of elemental analysis, conductometric, magnetic measurements, UV-vis and IR spectral studies. Where, DPH=Deprotonated phthalic acid, Py=Pyridine, IQ=Iso-quinoline, Q=Quinoline and 2-Pic=2-Picoline. All these data supported the octahedral structure of Co(II) complexes. All the synthesized complexes showed moderate to strong antimicrobial activity.

Keywords: Transition metal complex, Electronic spectra, biological activity, Heterocyclic amines, phthalic acid

1. Introduction

Survey of the existing literature reveals that phthalic acid are used as centrifugal molding aids for concrete. [1] It can be used to make an antifreeze compound which showed good corrosion inhibiting properties for cast aluminum, cast iron, steel, brass and copper. This compound is also useful for automobile engines. [2] Phthalic acid and its derivatives also used as anticorrosive, anti-deposit and biocide agent for protection of aqueous heating and cooling systems. [3]

Carter and co-workers have done extensive works on thermal absorption of phthalic digesters in rats. [4] It is found that metal complexes of phthalic acid have antiseptic properties on films, fibers and fabrics. The uranium complexes of phthalic acid have an enhanced sensitivity applied in radiation because of the atomic number dependence of the photoelectric effect. [5] The complexes of zinc with phthalic acid act as a anticorrosive coating compound and marine growth inhibitors. [6]

An exhaustive studies have been carried out on the metal complexes of phthalic acid in solution as well as in solid from. [7-13] Brzyska and co-workers recorded IR and X-ray spectra of thorium (IV) complexes with phthalic acid. [14] The cobalt salts of phthalic acid and other organic acid are used as a decoluring materials. [15] The lead salts of phthalic acid have been used as sealing materials having resistance to gasoline and alcohol, good adhesive to plated steel. [16]

Previously, we studied electronic properties of N_2O_4 Schiff base ligand containing metal complexes of Cd(II), Pd(II), Hg(II) and Zr(IV) [17-19], also synthesis and characterization of mixed ligand coordinating Co(II), Cu(II), Ni(II), Cr(III) and Fe(III) complexes with amino acid and hetero cyclic amines. [20-23] In this study, several new mixed ligand complexes of Cobalt(II) ions with phthalic acid and nitrogen containing heterocyclic bases have been prepared and characterized on the basis of elemental analysis, conductivity and magnetic measurements and infrared spectral and electronic spectroscopic studies. The antimicrobial activity of the synthesized complexes also reported.

2. Experimental

2.1 *Measurements and materials*

Electronic spectra were recorded on a Thermo electron Nicolet evolution 300 UV-Vis spectrophotometer. All chemicals were commercial products and were used as supplied.

2.2. *General method for the preparation of Co(II) complexes:*

General method for the preparation of the Co(II) complexes of [Co(DPH)L$_4$]. Where L = Pyridine, Quinoline, 2-Picoline and Iso-quinoline; and DPH = Deprotonated phthalic acid

An ethanolic solution of Co(II) chloride (1 m mole) and deprotonated phthalic acid (1 m mole) were mixed in the calculated ratio with constant stirring but no precipitate was observed. Then 25 ml of an ethanolic solution of L (4 m mole) was added to the resulting mixture and heat on a magnetic regulator hot plate with constant stirring. The volume of the solution was reduced to one half and allowed to cool. The precipitate formed and were filtered, washed several times with ethanol and then dried in a desiccator over anhydrous $CaCl_2$.

3. Results and Discussion

All the complexes are non-hygroscopic and stable at room temperature. The Co(II) complexes are insoluble in common organic solvents but are soluble in DMSO, DMF and $CHCl_3$.

3.1 *Elemental analysis and conductivity measurement*

The analytical data and their physical properties of the complexes are given in Table 1. The molar conductance of 10^{-3}M solution of the complexes in DMSO were measured at 30 °C. The molar conductance values are in the range 6.8 to 9.5 Ω^{-1} cm^2 mole^{-1} (Table 1). The molar conductance values indicates that the compound are non-electrolytic in nature.

Table 1: Selected physical properties of Co(II) complexes

Complexes	**Colour**	**Meting point or decomposition temperature (± 5°C)**	**Molar conductance (ohm^{-1} cm^2 mol^{-1})**	**(μ_{eff}) B.M.**
[Co(DPH)(Py)$_4$]	Green	254	9.5	3.96
[Co(DPH)(Q)$_4$]	Deep blue	185	7.6	3.96
[Co(DPH)(IQ)$_4$]	Light blue	260	7.9	3.90
[Co(DPH)(2-Pic)$_4$]	Deep blue	235	6.8	3.98

DPH = Deprotonated phthalic acid, Py = C_5H_5N, IQ = C_9H_7N, Q = C_9H_7N, 2-Pic=C_6H_7N

3.2 *IR spectral studies*

The IR spectrum of the complexes is shown in table 2. In the IR spectrum of Co(II) complexes shown a strong absorption band at (1576 – 1676) cm^{-1} arises due to ν(C = O) modes. The medium absorption bands in the region (1374 - 1320) cm^{-1} due to ν(C - O) modes have been observed. Further the presence of (M - O) bonding is evident from the appearance of ν(M - O) modes at (493 to 590) cm^{-1} in the spectra of the complexes. The complexes also display bands at (405 – 467) cm^{-1} due to ν(M - N) modes. The in-plane and out-of-plane ring deformation modes of heterocyclic amines observed at 680 and 620 cm^{-1} respectively undergo a positive shift in mixed ligand complexes confirming their coordination through nitrogen. The presence of metal nitrogen bonding in the complexes is evident from the appearance of ν(M-N) modes at 493-410 cm^{-1} in the spectra of the complexes and ν(M-O) appearance at 673-760).

Table 2: Selected IR spectral data of Co(II) complexes

Complexes	**ν(C = O) cm^{-1}**	**ν(C - O) cm^{-1}**	**ν(C=N) cm^{-1}**	**ν(M-O) cm^{-1}**	**ν(M-N) cm^{-1}**
[Co(DPH)(Py)$_4$]	1576	1374	1604	494	467
[Co(DPH)(Q)$_4$]	1561	1366	1586	511	455
[Co(DPH)(IQ)$_4$]	1548	1359	1589	523	405
[Co(DPH)(2-Pic)$_4$]	1676	1365	1578	590	405

DPH = Deprotonated phthalic acid, Py = C_5H_5N, IQ = C_9H_7N, Q = C_9H_7N, 2-Pic=C_6H_7N

3.3 *Magnetic Moment and Electronic Spectra*

The Co(II) complexes magnetic moment were found in the range 3.90 to 3.98 B.M. (Table 1) at room temperature, in agreement three unpaired electron (paramagnetic) with octahedral geometry. The magnetic moment of Co(II) complexes varies from 3.90 to 4.15 B.M. which indicate that these complexes are paramagnetic having three unpaired electrons with a octahedral geometry.

All the compounds under investigation are found to be paramagnetic and their selected electronic spectral components are given in Table 3. The electronic spectra of the complexes gave three bands at around 380 nm, 420 nm, and 560 nm corresponding to the transition $^4T_{2g}\rightarrow{}^4T_{1g}$ and $^4T_{2g}\rightarrow{}^4T_{1g}(P)$ and charge transfer band respectively. These spectra indicated the octahedral stereochemistry of Co(II) complexes.

Table 3: Electronic spectral data of Co(II) complexes

Complexes	**Band I**	**Band II**	**Band III**
[Co(DPH)(Py)$_4$]	380	420	560
[Co(DPH)(Q)$_4$]	390	446	555
[Co(DPH)(IQ)$_4$]	385	425	556
[Co(DPH)(2-Pic)$_4$]	395	430	550

DPH = Deprotonated phthalic acid, Py = C_5H_5N, IQ = C_9H_7N, Q = C_9H_7N, 2-Pic=C_6H_7N

Table 4: Data of the Elemental analysis of the complexes

Complexes	Metal %	Carbon %	Nitrogen %	Hydrogen %
$[Co(DPH)(Py)_4]$	10.92 (11.04)	62.33 (62.47)	10.39 (10.51)	4.48 (4.62)
$[Co(DPH)(Q)_4]$	7.97 (8.07)	71.45 (71.55)	7.58 (7.68)	5.45 (5.47)
$[Co(DPH)(IQ)_4]$	7.97 (8.11)	71.45 (71.56)	7.58 (7.59)	5.45 (5.51)
$[Co(DPH)(2\text{-}Pic)_4]$	9.90 (9.97)	64.54 (64.63)	9.41 (9.52)	5.42 (5.57)

DPH = Deprotonated phthalic acid, Py = C_5H_5N, IQ = C_9H_7N, Q = C_9H_7N, 2-Pic=C_6H_7N

4. Antibacterial and antifungal Screening

Metal complexes play an important role in regulating biological activities. The disk diffusion method was employed for the in vitro study of antibacterial effects against one Gram positive and two Gram negative bacteria. On the other hand antifungal activities of the metal complexes against two pathogenic fungi *Candida albicans (*Human Pathogens) and *Aspergillus species* (Plant Pathogens) were measured. The results revealed that the complexes are more microbial toxic than the free metal ions or ligands. All the complexes shown moderate to strong antimicrobial activities and the zone of inhibition observed shown in Table 5 and 6.

Table 5: Antibacterial activity

Compounds	Zone of inhibition, diameter in mm		
	For *Escherichia coli*	For *Shigella sonnei*	For *Bacillus subtilis*
$[Co(DPH)(Py)_4]$	9	10	8
$[Co(DPH)(2\text{-}Pic)_4]$	12	14	12
$[Co(DPH)(Q)_4]$	10	15	14
$[Co(DPH)(IQ)_4]$	20	19	17
Kanamycin disc	20	20	22

DPH = Deprotonated phthalic acid, Py = C_5H_5N, IQ = C_9H_7N, Q = C_9H_7N, 2-Pic=C_6H_7N

Table 6: Antifungal activity

Compounds	Zone of inhibition, diameter in mm	
	For *Candida albicans*	For *Aspergillus species*
$[Co(DPH)(Py)_4]$	10	12
$[Co(DPH)(2\text{-}Pic)_4]$	11	13
$[Co(DPH)(Q)_4]$	11	16
$[Co(DPH)(IQ)_4]$	18	14
Nystatin	19	20

DPH = Deprotonated phthalic acid, Py = C_5H_5N, IQ = C_9H_7N, Q = C_9H_7N, 2-Pic=C_6H_7N

5. Conclusion

Magnetic susceptibility measurement indicated the paramagnetic nature of the complexes. The IR spectral data showed the ligand coordinate with the metal through O and N atoms. The electronic spectral data were in conformity with the octahedral structure Co(II) complexes. Based on these facts a structure of complex has been proposed as shown in Figure 1.

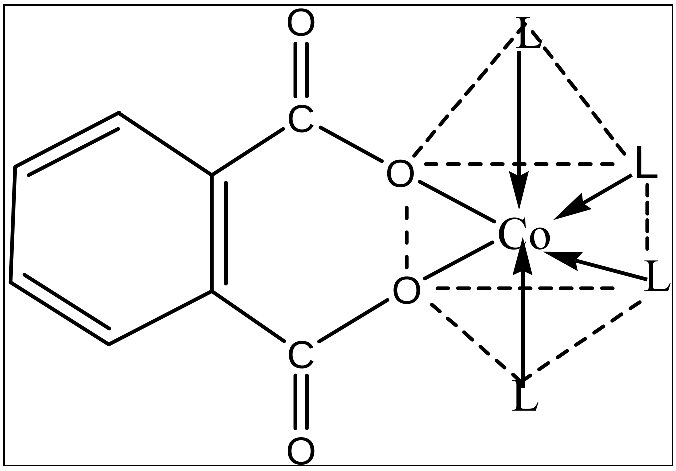

Fig 1: Proposed Structure of $[Co(DPH)L_4]$ complexes
Where, L = Pyridine, Quinoline, 2-Picoline and iso-quinoline,

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