



SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF COBALT (II) COMPLEXES OF SOME 2-HYDROXY-4, 5-DIMETHYL SUBSTITUTED CHALCONES

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ABSTRACT

The present paper deals with synthesis and characterization of metal complex of some novel 2-hydroxy-4,5-dimethyl substituted chalcones. The substituted chalcones were prepared by reacting 2-hydroxy-4,5-dimethyl acetophenone with corresponding aromatic aldehydes like 2-chlorobenzaldehyde, 3-bromobenzaldehyde and 4-cyanobenzaldehyde. The Cobalt (II) metal complexes of general formula ML_2 with newly prepared chalcones synthesized and characterized by several physicochemical techniques like Melting point, Elemental analysis, molar conductance, magnetic susceptibility, Electronic absorption and Infrared spectral studies. The analytical data confirmed 1:2 stoichiometry of M:L and the electronic spectral data suggest that all Co (II) complexes have octahedral geometry. The conductivity data show that all these complexes are non-electrolytes. Furthermore, biological activities of complexes with selected bacterial strain carried and the results have been compared with commercial standards.

KEYWORDS : Transition metal complexes, Chalcones, Spectral characterization and biological activities

INTRODUCTION:

The chemistry of chalcones generated intensive scientific studies throughout the world, specially interesting for their biological applications. Chalcones are coloured compounds¹ because of the presence of the chromophore and auxochromes. Chalcones are of a great interest because they have a unique structural feature² of having a $>C=O$ functional group in conjugation with $>C=C<$ and the whole molecule is in conjugation. The chalcones are α,β -unsaturated ketones containing the reactive keto ethylene group $-CO-CH=CH-$ which makes it biologically active. Some substituted chalcones and their derivatives have been reported to possess some interesting³ biological properties such as antibacterial, antifungal, insecticidal, ulcerogenic, anticancer, anti-inflammatory, anaesthetic, analgesic, antimalarial, antioxidant etc.

Chalcones are also key precursor^{4,5} in the synthesis of many biologically important heterocycles. Transition metal ions are found in several biological species and are reported to play an important role in different enzymatic and physiological reactions, the interaction of chalcones with metal ions may also change the antioxidant properties and also biological effects of the chalcones⁶.

MATERIALS:

All the chemicals and reagents were of AR grade and used without further purification. Ethanol, DMF, $CoSO_4 \cdot 7H_2O$, 2-hydroxy-4, 5-dimethyl acetophenone, 2-chlorobenzaldehyde, 3-bromobenzaldehyde, 4-fluorobenzaldehyde and 4-cyanobenzaldehyde etc were used in this work.

METHODS:

1) Synthesis of transition metal complexes:

The substituted chalcones were prepared⁷ by reacting 2-hydroxy-4,5-dimethyl acetophenone with corresponding aromatic aldehydes like 2-chlorobenzaldehyde, 3-bromobenzaldehyde, 4-fluorobenzaldehyde and 4-cyanobenzaldehyde in presence of alkaline medium at room temperature.

The transition metal complexes were prepared by mixing corresponding chalcone ligands with Cobalt sulphate in the ratio of 1:2. A warm ethanolic solution of $CoSO_4 \cdot 7H_2O$ (0.01 mole) was added to ethanolic solution (0.02 mole) of corresponding chalcone ligands. The pH of solution adjusted to 7-8 using aqueous ammonia Solution. The mixture was heated under reflux for 2-3 hrs. The

complex thus obtained was filtered and washed with excess of ethanol and dried at room temperature.

2) Biological Studies:

The complexes were screened for antibacterial activity against gram positive *Staphylococcus aureus*, gram negative *Escherichia coli* and for antifungal activity against *Aspergillus niger* by following methods.

a) Antibacterial activity:

The minimum inhibitory concentration (MIC) of the chalcone ligands and their complexes was carried using Broth dilution method⁸. The stock solution of all the complexes was prepared by dissolving 10 mg of complex in 10 ml DMF. The complex solution was serially diluted to give a concentration of 512, 256, 128, 64, 32 and 16 $\mu g/ml$ in test tubes containing 1 ml sterile nutrient broth. Then, the tubes were inoculated with 100 μl of bacterial suspension in the saline and incubated at 37°C for 24 hrs. A tube containing nutrient broth only was seeded with the test organism to serve as control. All the tubes were then incubated at 37°C for 24 h and then examined for growth by observing O.D. at 600nm.

b) Antifungal activity by Disc Transfer Technique:

The measurement of antifungal activity by disk transfer technique was carried using 6 mm diameter disks. Single disc was aseptically inoculated with sabouraud's dextrose agar medium containing desired concentration of the test complex. Test and control (without complex) were inoculated for 3-4 days at 28°C and zone of inhibition was calculated using standard technique.

RESULT AND DISCUSSION:

1) Characterisation of complexes:

All the complexes are stable at room temperature insoluble in water and most of the common organic solvents but soluble in DMF and DMSO. The elemental analysis data of its complexes are in good agreement with proposed molecular formulas. The thermogram of Co (II) complexes shows the presence of two hydrated water molecule. The analytical data of the complexes (Table-1) indicates that their stoichiometry may be represented as 1:2 metal to ligand ratio. The molar conductance values of the complexes in DMF solvents suggesting their non-electrolytic nature.

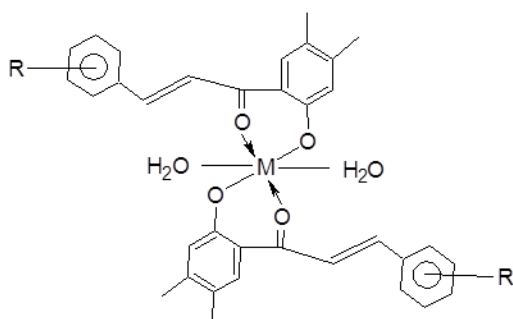
The μ_{eff} values at room temperature for Co (II) complexes have magnetic moment values in the range of 4.39 to 5.14 B.M. These values are expected for octahedral geometry of Co (II) complexes at room temperature and further supported by electronic spectral data.

Table-1 Elemental Analysis, Magnetic data of Co(II)-Chalcone Complexes

Complex	Molecular Formula	Mol. Wt. (gm/mol)	Color	M.P. °C	Yield (%)	Elemental analysis found/Calculated %				μ_{eff} [BM]	Molar Conductance (mho.cm ² .mol ⁻¹)
						Carbon	Hydrogen	Halogen	Metal		
Co(II)-2-Chloro Complex	[Co(II)(C ₁₇ H ₁₄ O ₂ Cl) ₂ .2H ₂ O]	630.42	Gray	>300	58	64.78 (64.72)	4.48 (4.51)	11.25 (11.21)	9.35 (9.38)	5.11	0.005
Co(II)-3-Bromo Complex	[Co(II)(C ₁₇ H ₁₄ O ₂ Br) ₂ .2H ₂ O]	719.32	Yellow	>300	66	56.77 (56.82)	3.92 (3.90)	22.22 (22.18)	8.19 (8.26)	5.03	0.003
Co(II)-4-Fluoro Complex	[Co(II)(C ₁₇ H ₁₄ O ₂ F) ₂ .2H ₂ O]	597.57	Gray	>300	61	68.34 (68.39)	4.72 (4.76)	6.36 (6.32)	9.96 (9.96)	4.78	0.007
Co(II)-4-Cyano Complex	[Co(II)(C ₁₈ H ₁₄ O ₂ N) ₂ .2H ₂ O]	611.55	Orange	>300	59	70.70 (70.76)	4.61 (4.61)	-	9.64 (9.58)	4.43	0.008

Table-2 :: Some Important IR Bands in cm⁻¹ of the Co (II) chalcones-Metal Complexes

Complex		$\nu_{\text{(O-H)}}$ (H ₂ O)	$\nu_{\text{C=O}}$ (Phenol)	$\nu_{\text{Ph-C=C}}$	$\nu_{\text{C=O}}$	$\nu_{\text{M-O}}$
Co(II)-2-Chloro Chalcone Complex	[Co(II)(C ₁₇ H ₁₄ O ₂ Cl) ₂ .2H ₂ O]	3263	1188	1572	1641	647
Co(II)-3-Bromo Chalcone Complex	[Co(II)(C ₁₇ H ₁₄ O ₂ Br) ₂ .2H ₂ O]	3329	1188	1577	1647	678
Co(II)-4-Fluoro Chalcone Complex	[Co(II)(C ₁₇ H ₁₄ O ₂ F) ₂ .2H ₂ O]	3357	1190	1575	1653	622
Co(II)-4-Cyano Chalcone Complex	[Co(II)(C ₁₈ H ₁₄ O ₂ N) ₂ .2H ₂ O]	3298	1188	1573	1641	654



Where M – Cobalt, R – 2-Chloro, 3-Bromo, 4-Fluoro, 4-Cyano Chalcone.

Figure 1: Proposed structure of the complexes**Table-3 : Antimicrobial activity of synthesized Ligands and Co(II)Complexes**

Samples	Antibacterial		Antifungal
	E. coli	S. aureus	A. Niger
	(MIC- $\mu\text{g/ml}$)		(ZOI- mm)
DMF	-	-	-
Ampicillin	4	8	-
Fluconazole	-	-	30 \pm 02
2-hydroxy-4,5-dimethyl-2-chloro chalcone	>512	>512	-
2-hydroxy-4,5-dimethyl-3-bromo chalcone	16	128	-
2-hydroxy-4,5-dimethyl-4-fluoro chalcone	16	256	08 \pm 0.2
2-hydroxy-4,5-dimethyl-4-cyano chalcone	128	218	09 \pm 0.2
Co(II)-2-chloro chalcone Complex	16	16	-
Co(II)-3-bromo chalcone Complex	256	128	07 \pm 0.2
Co(II)-4-fluoro chalcone Complex	64	256	07 \pm 0.2
Co(II)-4-cyano chalcone Complex	>512	128	07 \pm 0.2

In addition to this keeping in view of the increasing problems of antimicrobial resistance complexes were screened for their antibacterial and antifungal activities.

The antimicrobial activity of all the complexes is less than that of standard Ampicillin and Fluconazole, Shown in (Table-3).

CONCLUSION:

The elemental analysis, magnetic susceptibility, IR, ¹H NMR and

Physical Properties observations suggest the octahedral geometry for the Co (II) complexes and also exhibit the antimicrobial activity.

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