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SYNTHESIS, SPECTRAL STUDY, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF COBALT (II) COMPLEX OF CHALCONE OF PYRIDINE-2-CARBALDEHYDE

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ABSTRACT: The synthesis of Cobalt(II) metal complex has been synthesized by using novel Chalcone of Pyridine-2-Carbaldehyde. The ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and Pyridine-2-Carbaldehyd. The structure of the complex has been characterized by the analytical data, conductivity measurement, magnetic moment, UV-Vis spectra, thermal studies and XRD. Analytical data shows 1:2 stoichiometry and the magnetic moment, TG-DTA and XRD suggests that Co(II) complex has octahedral geometry. The presence of coordinated water molecules in Co(II) complex is confirmed by thermal studies. The conductivity data reveals that the complex is non electrolyte. Antimicrobial study of complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards. The Co(II) complex shows moderate to good Antibacterial and Antifungal activity.

KEYWORDS: Antimicrobial activities, TG-DTA study, XRD, Physico-chemical property, Magnetic Susceptibility and Conductivity.

I. INTRODUCTION

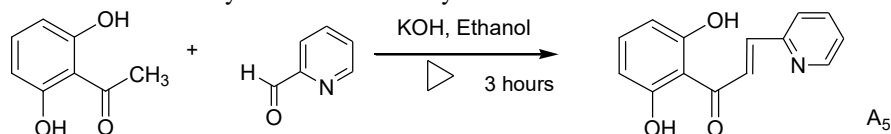
Chalcones constitute an important group of natural products, which has two aromatic rings joined by α, β unsaturated carbonyl system. The name chalcone is given by Kostanecki and Tambar [1]. The $>CO-C=C<$ moiety imparts biological characteristics to the Chalcones. Such α, β -unsaturated carbonyl group in chalcone is found to be responsible for their antimicrobial activity [2]. Chalcone is a common natural pigment and one of the important intermediate in the biosynthesis of flavonoids[3]. The metal complexes possess interesting biochemical properties, such as antitumor, antioxidant, and antimalarial, anti-fungal and antimicrobial activities [4]. The magnetic moment, TG-DTA supports the octahedral geometry of the metal complex of chalcone.

II. MATERIALS AND METHODS

2.1 Synthesis of Chalcone of Pyridine-2-Carbaldehyde :

The reagents used for preparation of Chalcone of Pyridine-2-Carbaldehyde are of A.R. grade. 2,6-dihydroxy acetophenone (0.01 mol) and pyridine-2-carbaldehyde (0.01 mol) are dissolved in ethyl alcohol (25 mL) and then potassium hydroxide 10 mL (40%) were added to it. The reaction mixture was heated for 3 hours till yellow-brown color was obtained. The progress of reaction was monitored by TLC, after completion of reaction the content was poured into ice cold water and then acidified by dil. HCl the solid obtained was filtered and the crude product was recrystallized from ethyl alcohol to give pyridine chalcone [5].

Figure-1: Synthesis of Chalcone of Pyridine-2-Carbaldehyde



2.2 Synthesis of Metal Complex:

The solution of 0.02 mole of Chalcone of Pyridine-2-Carbaldehyde was taken in round bottom flask containing 30 ml of anhydrous methanolic solution and boiled for 10 minutes. A hot solution of 0.01 mole, of

Cobalt Acetate in 20 ml of methanol was added drop wise to the solution of the chalcone of Pyridine-2-Carbaldehyde to this reaction mixture, 10% alcoholic ammonia was added up to slightly alkaline pH. The complex was precipitated at 8 pH range. The pH 8-10 range was definite for these complexes [6]. The content was stirred on magnetic stirrer for one hour. The solid metal complex separated out and washed with methanol three to four times. The melting point of the complex was determined by Thiele's melting apparatus. The reactions of formation of Co (II) complex is shown in **Figure-2**.

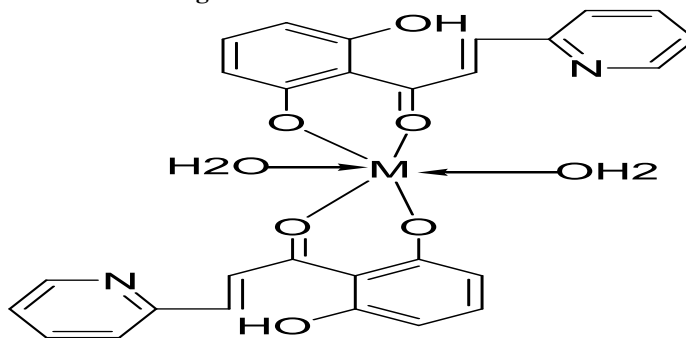


Figure-2: Metal complex of Cobalt (II) with Chalcone of Pyridine-2-Carbaldehyde
R= -H, M= Co(II)

III. RESULTS AND DISCUSSION

3.1 Physical parameters:

Metal complex of Cobalt (II) with Chalcone of Pyridine-2-Carbaldehyde was Coffee brown in color. The complex was precipitated at 8 pH range, having Melting point 330⁰C. The complex is insoluble in water and soluble in DMSO, DMF [7].

3.2 CHO analysis:

The carbon, hydrogen, oxygen, Cobalt metal percentage in Co (II) complex of chalcone measured at SAIF Cochin, Kerala. The calculated and measured values of CHO analysis are matching and are given in the **Table-1**.

Table-1: Study CHO analysis synthesized Co (II) complex

Metal complex	Chemical formula	Mol. Wt.	Elemental analysis : % found (calculated)						
			C	H	N	O	S	X(Br)	M
Co (II) Complex	[C ₂₈ H ₂₄ O ₈ N ₂ Co]	575	58.49 (58.44)	4.23 (4.20)	4.84 (4.86)	22.28 (22.24)	-	-	10.26 (10.24)

3.3 Magnetic susceptibility, solution conductivity and electronic absorption spectral data

Magnetic susceptibility:

The high spin Co(II) ion have d⁷ configuration. The high spin octahedral complexes show the magnetic moment up to 5.2 B.M. at room temperature. The magnetic moment of Co(II) complexes in the present investigation all the Co(II) complexes contain a paramagnetic d⁷ species. All complexes showed magnetic susceptibility values. This observation is in agreement with previously reported magnetic properties [8].

Table-2 Magnetic susceptibility, solution conductivity and electronic absorption spectral data Magnetic susceptibility:

Co (II) Complexes	Molar Conductance $\text{Ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$	μ_{eff} (B.M.)	Absorption Maxima cm^{-1} (nm)	
			${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$	Charge Transfer
Chalcone	5.69	4.64	20833 (480)	26315 (380)

Solution conductivity and electronic absorption spectral data:

The solution conductivities of 10^{-3} M solution of metal complex in DMSO were measured on EQUIPTRONICS digital conductivity meter EQ - 660 with $20 \mu\Omega$ to $200 \mu\Omega$ at 298K temperature. They are insoluble in water and soluble in DMSO, DMF. The low solution conductivity of 10^{-3} M solutions of Co(II) complexes in DMSO indicates their non-electrolytic nature.

Figure-3.

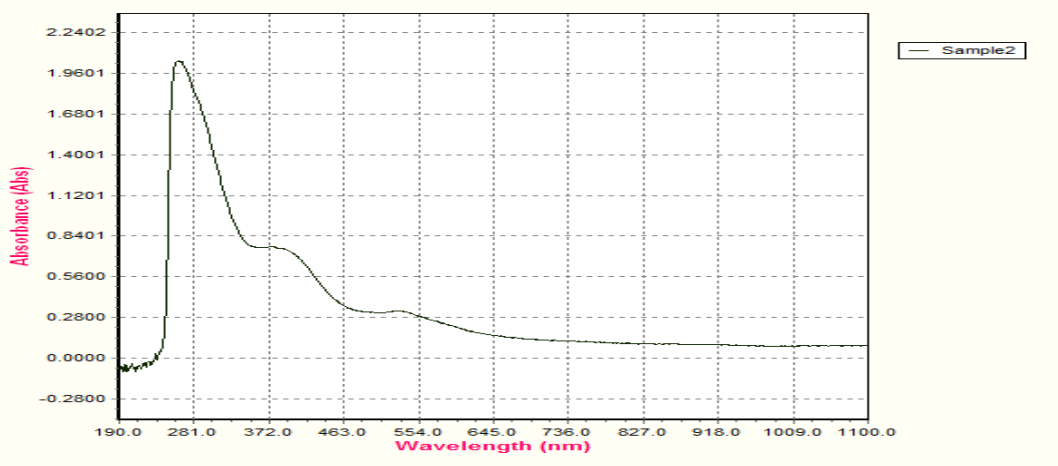


Figure-3: Electronic absorption spectrum

The electronic spectra of Co(II) complexes studied in the present investigation exhibit absorption bands at 22271 to 25641 and 26315 to 32258 cm^{-1} assigned to ${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$ and charge transfer band respectively[9].

3.4 Thermal analysis Co (II) complex of Pyridine-2-Carbaldehyde :

The simultaneous thermo gravimetric, differential thermal analysis of Co (II) complex Pyridine-2-Carbaldehyde was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000 at SAIF, Cochin, Kerala. The heating rate was $10^\circ/\text{min}$ and flow rate of nitrogen 50 ml/min. The reference substance used was $\alpha \text{Al}_2\text{O}_3$ in platinum crucible and sample weighed in the range of 4-12 mg. The thermogram of Co (II) complex Chalcone of Pyridine-2-Carbaldehyde is presented in figure-4. This curve reveals that there is presence of lattice as well as coordinated water in the complex.

The TG curve of Co(II) complex of Chalcone of Pyridine-2-Carbaldehyde In the first weight loss at 79.13°C indicating presence of lattice water with weight loss 5.40% (calc. wt. loss 5.50%). The second loss due to coordinated water molecule liberates from the complex, takes place at 190°C . In the third step loss of 23.40% (calc. wt. loss 24.00%) up to temperature in the range $300-400^\circ\text{C}$. This is supported by endothermic peak in DTA at 258.19°C . This may be due to decomposition of non-coordinated part of two pyridine ring fragments. In the fourth step, the loses of two fragments of $\text{C}_6\text{H}_5\text{O}_2$ take place with the major mass reduction, loss of 37.55% (calc. wt. loss 36.80%) which is authenticated by a broad endotherm in DTA curve at 329.89°C due to loss of coordinated part of a metal complex. The next step decomposition occurs with mass loss of 16.33% (calc. wt. loss 17.13%) in the range $370-430^\circ\text{C}$, due to loss of two enone part of chalcones fragment. In the last step after that formation of metal oxide is indicated by constant weight loss 7.6% beyond 550°C .

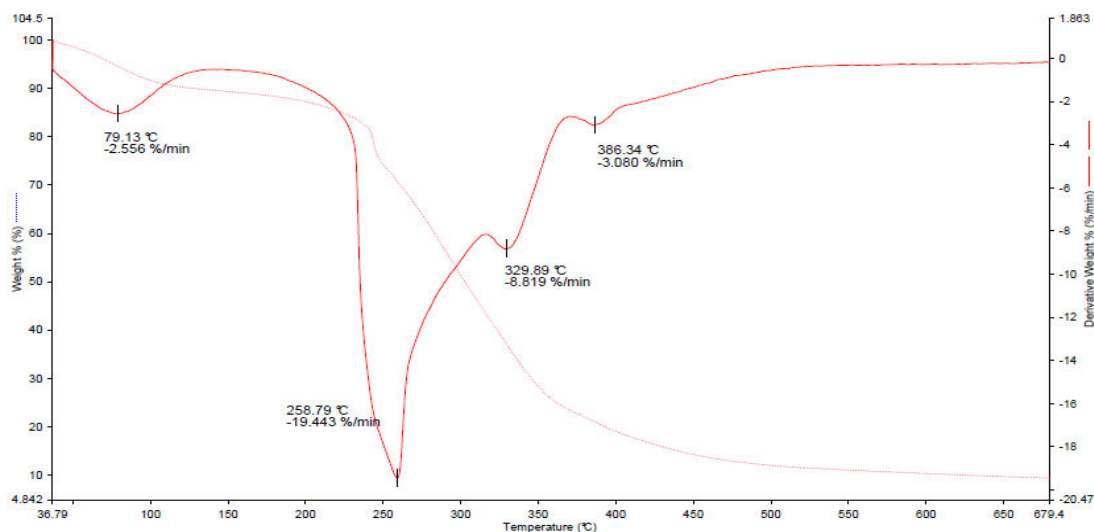


Figure-4: TG-DTA curve of Co (II) complex of Chalcone of Pyridine-2-Carbaldehyde

Thermodynamic and Kinetic Parameters

Akahira [10], first introduced that decomposition and kinetic studies of thermal reactions are useful in determining thermodynamic and kinetic parameters like free energy, entropy change, activation energy, pre-exponential factor. Thermal decomposition studies of materials are useful in predicting thermal stability (Table-3).

The negative values of the entropy of activation (ΔS) indicate that the metal complex is thermally stable. ΔG is positive for the complexes revealing that the free energy of the final residue is higher than that of the initial complex, and all decomposition steps are non-spontaneous processes. Also, the value of free energy of activation, ΔG increases significantly for the subsequent decomposition stages of a given complex [11]

Table-3: Thermodynamic and Kinetic Parameters of Co (II) complex of Chalcone of Pyridine-2-Carbaldehyde

Metal complex	Method	Step	Decomp. Temp.	Order of Reaction	Ea(KJ mol ⁻¹)	ΔS (KJ mol ⁻¹)	ΔG (KJ mol ⁻¹)	Z (S ⁻¹)	Correlation Coefficient (r)
A ₅ Co	H-M C-R	I	340	0.9	13.49 46.11	-160.96 -131.64	24.94 55.49	48465.57 1647974	0.998 0.997
		II	591	0.9	33.67 38.01	-162.38 -119.27	45.24 46.51	40867.05 7295489.70	0.930 0.998

3.5 X-ray diffraction spectral studies of metal complex of Co (II) complex of Chalcone of Pyridine-2-Carbaldehyde:

The XRD spectral study has been done at SAIF, Cochin Kerala. The standard deviation observed for Co (II) is 0.030 which is within the permissible limit of 2%. The observed and calculated densities are 1.2465gcm⁻³ and 1.3873 gcm⁻³ respectively. The volume is found to be 218.55 Å³ and complex crystallizes in the Tetragonal system with 1 atom per unit cell. The lattice parameters are a = 4.9165Å, b = 4.9165Å, c = 5.4031Å, $\alpha=90^\circ$, $\beta=90^\circ$, $\gamma=90^\circ$

3.6 Indexed X-ray Diffraction Data of Co(II) Complex of Chalcone of Pyridine-2-Carbaldehyde

Peak No	2θ observed	2θ calculated	d observed	d calculated	Miller Indices of Planes			Relative Intensities (%)
					h	k	l	
1	16.487	16.492	5.37237	5.45732	0	0	1	4.25
2	37.406	37.502	2.40221	2.47543	0	-1	2	2.68
3	51.809	51.713	1.76320	1.86549	0	2	2	4.83
4	52.213	52.199	1.75052	1.75962	-1	2	2	4.51
5	57.115	57.149	1.61136	1.69847	3	-1	0	4.98
6	63.438	63.543	1.46514	1.42849	-1	-3	0	5.96
7	64.594	64.603	1.44168	1.47536	-3	-1	1	4.52
8	66.162	66.179	1.41127	1.54376	0	2	3	3.69
9	66.970	66.754	1.39618	1.54793	1	-3	2	6.74
10	67.585	67.904	1.38496	1.46211	2	-1	3	4.39
11	70.606	70.674	1.32293	1.76530	-3	-1	2	5.64
12	71.206	71.008	1.32316	1.35672	-1	0	4	4.74
13	74.227	74.675	1.27660	1.54333	-1	-1	4	5.18

Unit cell data and crystal lattice parameters

a (Å) = 4.9165

b (Å) = 4.9165

c (Å) = 5.4031

α = 90°

β = 90°

γ = 90°

Standard deviation (%) = 0.030

Volume (V) = 218.55 Å³

Density (obs.) = 1.2465 gcm⁻³

Density (cal.) = 1.3873 gcm⁻³

Z = 4

Crystal system = Tetragonal

Space group = P2/m

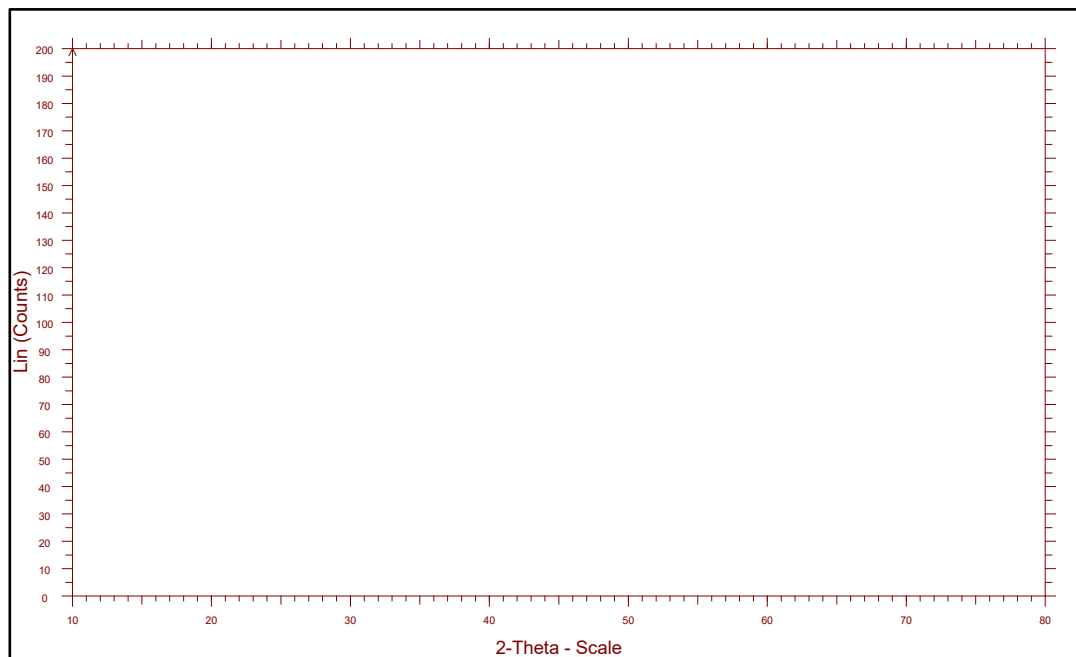


Figure-5: X-ray diffractogram of Co (II) complex of Chalcone of Pyridine-2-Carbaldehyde

3.7 Antimicrobial activity:

Antimicrobial activity was assayed by cup plate agar diffusion method by measuring inhibition zones in mm. In vitro antimicrobial activity of all synthesized compounds and standard have been evaluated against strains of The fungal toxicity of Co (II) complex was studied *in vitro* against *Aspergillus niger* ATCC 16404, *Saccharomyces cerevisiae* ATCC 9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration.

The antibacterial activity of Co (II) complex was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus* ATCC 6538, *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739, *Salmonella typhi* ATCC9207, *Shigella boydii* ATCC 12034, *Enterobacter aerogenes* ATCC13048, *Pseudomonas aerogenosa* ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this study.

From the results of antimicrobial activity of ligands and complex it is clear that the complex shows enhanced activity than ligand. The increase in antimicrobial activity is due to faster diffusion of metal complexes as a whole through the cell membrane or due to the combined activity of the metal and ligands [12].

IV. CONCLUSION

The Co (II) complex was colored, soluble in most of the organic solvent. The stoichiometry ratios of the metal complexes are obtained has been found to be 1:2. Solution conductivity of this metal complex reveals nonelectrolytic nature. The electronic spectral data, magnetic moment, TG-DTA suggests that Co (II) has Octahedral geometry. The CHO analysis gives C, H, and O percentage in the metal complex. From the antimicrobial activity of ligand and complex it is clear that the complex shows enhanced antimicrobial activity than ligand.

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