



SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL STUDY OF COPPER (II) COMPLEX OF (E)-3-(FURAN-2-YL)-1-(2,6-DIHYDROXYPHENYL)PROP-2-EN-1-ONE

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ABSTRACT The synthesis of Copper (II) metal complex has been synthesized by using novel (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one ligand. The ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and 2-furfural. The structure of the complex has been characterized by the analytical data, conductivity measurement, magnetic moment, UV-Vis spectrum, IR spectrum. Analytical data shows 1:2 stoichiometry and the magnetic moment, suggests that Cu (II) complex has octahedral geometry. 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 Cu (II) complex shows moderate to good Antibacterial and Antifungal activity.

KEYWORDS : Antimicrobial activities, UV-Vis spectrum, IR spectrum, Mass Spectrum, Physico-chemical property, Magnetic Susceptibility and Conductivity.

1. 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]. The metal complexes possess interesting biochemical properties, such as antitumor, antioxidant, and antimicrobial, anti-fungal and antimicrobial activities[3]. The magnetic moment supports the octahedral geometry of the metal complex of chalcone.

2. MATERIALS AND METHODS:

2.1 Synthesis of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one ligand:

The reagents used for preparation of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one are of A.R. grade. A mixture of 2,6-dihydroxy acetophenone (0.01 mol) and 2-furfuraldehyde (0.01 mol) are dissolved in ethanol (20 mL) and then solution of potassium hydroxide 10 mL (15%) were added to it. The mixture was stirred for overnight. The progress of the reaction was monitored by TLC. It was then poured on ice cold water and acidified with dilute HCl. The coffee brown solid was precipitates, filtered and washed with water and recrystallized from ethanol [4].

2.2 Synthesis of Metal Complex:

The solution of 0.02 mole of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl) prop-2-en-1-one 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 Copper Sulphate in 20 ml of methanol was added drop wise to the solution of the chalcone of 2-furfural 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 [5]. 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 Cu (II) complex is shown in Figure-1.

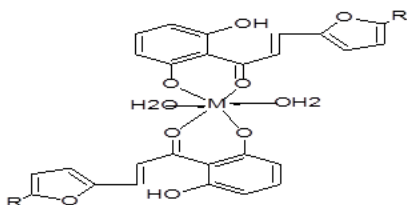


Figure-1: Metal complex of Copper (II) with (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one R = -H, M= Cu (II)

3. RESULTS AND DISCUSSION:

3.1 Physical parameters:

Metal complex of Copper (II) with (E)-3-(furan-2-yl)-1-(2,6-

dihydroxyphenyl)prop-2-en-1-one was Coffee brown in color. The complex was precipitated at 8 pH range, having Melting point 280°C. The complex is insoluble in water and soluble in DMSO, DMF [6].

3.2 CHO analysis:

The carbon, hydrogen, oxygen, Copper metal percentage in Cu (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 Cu (II) complex

| Metal complex | Chemical formula | Mol. Wt. | Elemental analysis : % found (calculated) | | | | | | |
|---------------|---|----------|---|-------------|---|---------------|---|-------|---------------|
| | | | C | H | N | O | S | X(Br) | M |
| Copper (II) | [C ₂₆ H ₂₂ O ₂] ₂ Cu | 558 | 55.96 (63.21) | 3.97 (4.48) | - | 28.67 (19.43) | - | - | 11.38 (12.86) |

3.3 Magnetic susceptibility, solution conductivity and electronic absorption spectral data

Magnetic susceptibility: Table-2.

| Mn(II) Complex | Molar Conductance Ohm ⁻¹ cm ² mol ⁻¹ | μ_{eff} (B.M.) | Absorption Maxima cm ⁻¹ (nm) |
|----------------|---|--------------------|--|
| Cu(II) Complex | 2.97 | 2.13 | ² B _{1g} → ² E _g Charge Transfer 23474(426) 33557.05 (298) |

The observed magnetic moment values of Cu(II) complexes in the present investigation are found to be in the range 1.81 to 2.2 B.M. at room temperature corresponding to one unpaired electron. Magnetic moments are slightly higher than the spin only value (1.73 B.M.).

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 Cu (II) complexes in DMSO indicates their non-electrolytic nature.

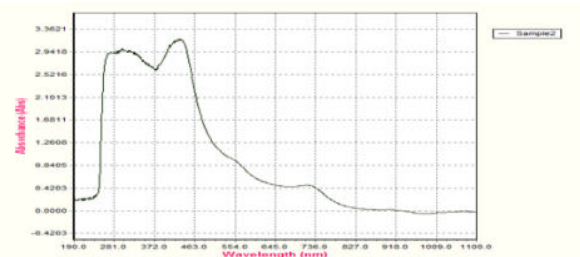


Figure-2: Electronic absorption spectrum

In the present investigation, Cu(II) show UV transition band in the range 24154 to 26560 cm⁻¹ which is attributed to ²B_{1g} → ²E_g and charge transfer band observed in the range 35026-39370 cm⁻¹ indicating distorted octahedral geometry around the Cu(II) ion. [7-8].

3.4 Infra red spectrum:

The IR spectrum of α , β -unsaturated carbonyl group has characteristic bands of chalcone at prominent bands between 1625 to 1650 per cm [9]. The characteristic peaks in infra red spectrum give the presence of particular functional group. The region at which other absorption bands appear depends on the type of aromatic / hetero-aromatic rings as well as the substituent present on these rings. The infrared spectrum of metal complex of Copper (II) with (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one was recorded on a Perkin- Elmer Spectrum RX-IFTIR Spectrophotometer in the range 4000-400 cm^{-1} (Table-3) using potassium bromide pellet at CIL, Chandigarh, Punjab. The stretching frequency of metal complex of Copper (II) with (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one is represented in table number (2) and the IR spectrum in Figure-3.

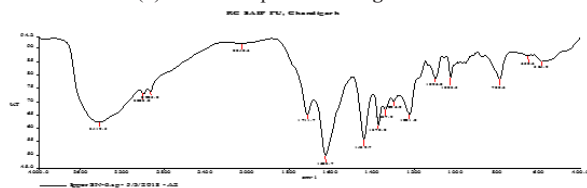


Figure-3: IR spectrum of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

Table-3: IR spectral data of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one:

| Molecule | $\nu(\text{O-H})$ Enolic | $(-\text{CO}-\text{CH}=\text{H}-\alpha, \beta\text{-unsaturated carbonyl group})$ | Carbonyl group in pyron ring ($-\text{C}=\text{O}$) | $(\text{C}-\text{O}-\text{C})$ Stretching Frequency | $(\text{C}=\text{C})$ Stretching Frequency | Aromatic Ring $(\text{C}=\text{C})$ Stretching Frequency | Ar-H Stretching Frequency | $-\text{NO}_2$ stretching frequency |
|----------|--------------------------|---|---|---|--|--|---------------------------|-------------------------------------|
| Ligand | 3420 | 1652 | - | 1096 | 1575 | 1457 | 2920 | - |

3.5 Mass spectrum data of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one:

Mass spectroscopy is the most accurate technique for the determination of molecular weight of compound. In this technique matter is bombarded with highly energetic electrons. Then matter absorbs or ejects electrons, from it. When it ejects electrons charged species are formed. The (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one have 231 as molecular weight which is confirmed due to following mass spectrum of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one.

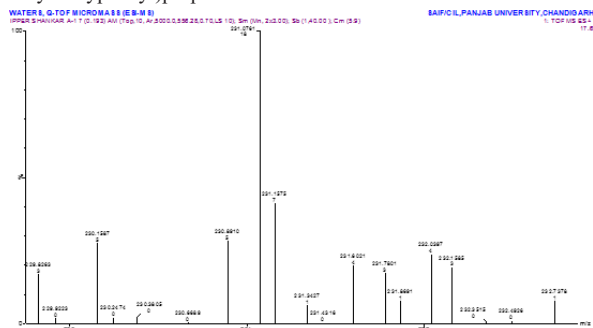


Figure-4: Mass spectrum of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

3.6 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 Cu (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 Cu (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 [10].

CONCLUSION:

The Cu (II) complex was Coffee brown 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, suggests that Cu (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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