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# Synthesis, Characterization and Antimicrobial Study of Manganese (II) Complex of 2-(Furan-2-Yl)-5-Hydroxy-4 H-Chromen-4-Ones

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## ABSTRACT

The synthesis of Manganese (II) metal complex has been synthesized by using novel 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand. The ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and 5-methylfurfural. The structure of the complex has been characterized by the analytical data, conductivity measurement, magnetic moment, UV-Vis spectra, IR and XRD analysis. Analytical data shows 1:2 stoichiometry and the magnetic moment, suggests that Mn (II) complex has octahedral geometry. The conductivity data revels 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 Mn (II) complex shows moderate to good Antibacterial and Antifungal activity.

**Keywords**: IR, XRD study, Physico-chemical property, Magnetic Susceptibility and Conductivity, Antimicrobial activities.

## I. INTRODUCTION

Chalcone is a generic term given to compounds bearing the 1, 3-diphenyl-2-propen-1-one framework and belong to the flavonoid family[1-3]. 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[4]. The $\alpha$, β-unsaturated carbonyl group in chalcone is found to be responsible for their antimicrobial activity [5]. The metal complexes possess interesting biochemical properties, such as antitumor, antioxidant, and antimalerial, anti-fungal and antimicrobial activities [6]. All crystals of a substance possess the same elements of symmetry. The computer program, used for indexing data was powder-X [7]. The X-ray powder diffractogram of the metal complex was used for the structural characterization and determination of lattice dimensions.

## II. MATERIALS AND METHODS

### 2.1 Synthesis of Chalcone:

The reagents used for preparation Chalcone are of A.R. grade. A mixture of 2,6-dihydroxy acetophenone (0.01 mol) and 2-furaldehyde (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 it gives chalcone [8].

### 2.2 Synthesis of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand:

Take Chalcone (0.01 mol) and then it was dissolved in 20 ml DMSO, to this catalytic quantity of iodine was added. Contents were refluxed for one hour, the progress of the reaction was monitored by TLC and the reaction mixture was left overnight. It was then poured on ice cold water, the separated solid was filtered washed with cold water followed by a dilute sodium-thiosulphate solution. The product was crystallized from ethanol it gives a flavones [9].

### 2.3 Synthesis of Metal Complex:

The solution of 0.02 mole of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand 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 Mn Acetate, 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 [10]. 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 Mn (II) complex is shown in **Figure-1**.

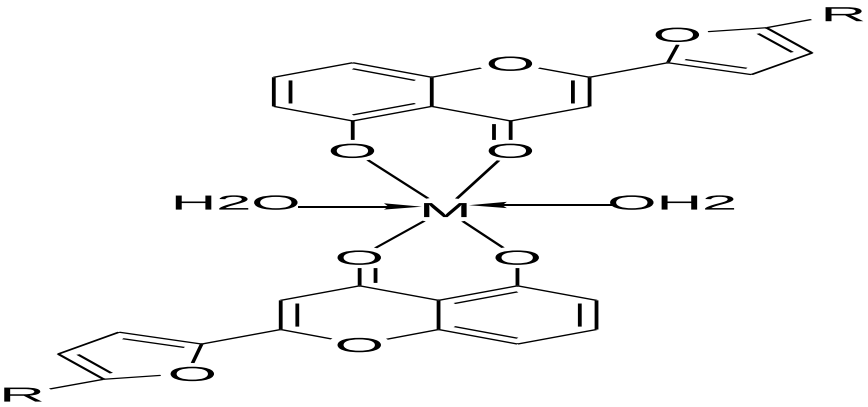


**Figure-1:** Metal complex of Manganese (II) with 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand
**R= -H, M= Mn (II)**

## III. RESULTS AND DISCUSSION

### 3.1 Physical parameters:

Metal complex of Manganese (II) with 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand was Blackish brown in color. The complex was precipitated at 8 pH range, having Melting point 320$^0$C. The complex is insoluble in water and soluble in DMSO, DMF [11].

### 3.2 CHO analysis:

The carbon, hydrogen, oxygen, Manganese metal percentage in Mn (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 Mn (II) complex

| Metal complex | Chemical formula | Mol. Wt. | Elemental analysis : % found (calculated) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | C | H | N | O | S | X(Br) | M |
| Mn (II) Complex | $[C_{26}H_{18}O_{10}Mn]$ | 545 | 57.30 (57.26) | 3.32 (3.33) | - | 29.30 (29.34) | - | - | 10.09 (10.07) |

**3.3 Magnetic susceptibility, solution conductivity and electronic absorption spectral data Magnetic susceptibility:**

The magnetic moment of Mn(II) complexes in the present investigation are in the range which is almost close to the spin only value of 5.92 B.M. These values are in good agreement with the moment reported for mononuclear high spin octahedral Mn(II) complexes by earlier workers.

| Mn(II) Complexes | Molar Conductance $Ohm^{-1} cm^2 mol^{-1}$ | $\mu_{eff}$ (B.M.) | Absorption Maxima $cm^{-1}$ (nm) | | |
|---|---|---|---|---|---|
| | | | $^6A_{1g} \rightarrow {}^4T_{2g}$ (G) | $^6A_{1g} \rightarrow {}^4A_{1g}$ (G), $^4E_g$ | Charge Transfer |
| Flavone | 6.97 | 5.97 | 24154(414) | 27624(362) | 29673(337) |

**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 μΩ to 200 μΩ at 298K temperature. They are insoluble in water and soluble in DMSO, DMF. The low solution conductivity of $10^{-3}$ M solutions of Mn(II) complexes in DMSO indicates their non-electrolytic nature.

The electronic absorption spectra of Mn(II) complexes were showed three bands at 19,120 to 25000 $cm^{-1}$, 25125 to 27700 $cm^{-1}$, and 28993 to 30581 $cm^{-1}$ assignable to $^6A_{1g} \rightarrow {}^4T_{2g}$(G), $^6A_{1g} \rightarrow {}^4E_{1g}$ or $^6A_{1g} \rightarrow {}^4T_{1g}$ (G) and charge transfer indicating octahedral geometry around the metal ion[12-13].

**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. 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 Manganese (II) with 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand was recorded on a Perkin- Elmer Spectrum RX-IFTIR Spectrophotometer in the range 4000-400 $cm^{-1}$ (**Table-2**) using potassium bromide pellet at CIL, Chandigarh, Punjab. The stretching frequency of metal complex of Manganese (II) with 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand is represented in table number (2) and the IR spectrum in **Figure-2.**

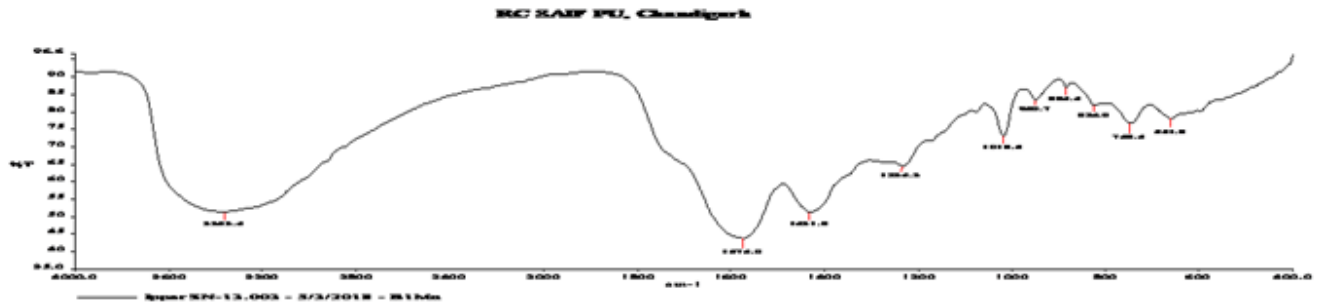

**Figure-2:** IR spectrum of metal complex of Manganese (II) with 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand

| **Peak No** | **2θ observed** | **2θ calculated** | **d observed** | d calculated | Miller Indices of Planes | | | **Relative Intensities (%)** |
|---|---|---|---|---|---|---|---|---|
| | | | | | h | K | L | |
| 1 | 16.413 | 16.345 | 5.39657 | 5.67843 | 0 | 0 | 1 | 100 |
| 2 | 18.223 | 18.324 | 4.86422 | 4.85438 | 0 | -1 | 0 | 62.63 |
| 3 | 18.238 | 18.248 | 4.86032 | 4.54789 | -1 | 0 | 0 | 14.80 |
| 4 | 23.901 | 23.894 | 3.72000 | 3.45873 | -2 | -1 | 0 | 19.18 |
| 5 | 23.932 | 23.432 | 3.71526 | 3.56739 | -1 | 0 | 1 | 13.77 |
| 6 | 23.965 | 23.119 | 3.71021 | 3.54893 | 1 | -1 | 0 | 5.9 |
| 7 | 24.083 | 24.345 | 3.69229 | 3.76593 | 1 | -1 | 1 | 8.37 |
| 8 | 25.145 | 25.438 | 3.53828 | 3.86739 | 0 | 1 | 1 | 4.5 |
| 9 | 33.323 | 33.659 | 2.68664 | 2.56789 | 1 | 1 | 1 | 7.75 |
| 10 | 36.929 | 36.547 | 2.43231 | 2.47632 | 0 | -2 | 0 | 23.71 |
| 11 | 46.135 | 46.432 | 1.96599 | 1.94678 | -1 | -2 | 1 | 13.19 |
| 12 | 48.158 | 48.321 | 1.88016 | 1.45680 | 1 | 2 | 1 | 4.9 |
| 13 | 49.057 | 49.489 | 1.85550 | 1.85409 | -2 | 2 | 0 | 2.31 |

**Table-2: IR spectral data Manganese (II) complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand:**

### 3.6 X-ray diffraction spectral studies of metal complex of Mn (II) complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand:

The XRD spectral study has been done at SAIF, Cochin Kerala. The standard deviation observed for Mn(II) is 0.042 which is within the permissible limit of 2%. The observed and calculated densities are 0.8615 $gcm^{-3}$ and 0.8609 $gcm^{-3}$ respectively. The volume is found to be 1546.5 $Å^3$ and complex crystallizes in the monoclinic system with 1 atom per unit cell. The lattice parameters are a = 7.9163 Å, b = 4.9165Å, c = 8.4089Å, α=90˚, β= 102˚, γ=90˚.

### 3.7 Indexed X-ray Diffraction Data of Mn(II) Complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand. Table-3:

| **Ligand/Metal complexes** | **υ (OH) $cm^{-1}$** | **υ (H2O) $cm^{-1}$** | **υ (-CO-CH=CH-) $cm^{-1}$** | **υ (-C=O in pyron ring) $cm^{-1}$** | **υ (C-O-C) $cm^{-1}$** | **υ (C=C) $cm^{-1}$** | **Aromatic Ring (C=C) $cm^{-1}$** | **υ (M-O) $cm^{-1}$** |
|---|---|---|---|---|---|---|---|---|
| [Mn(B1)2] | - | 3363 | - | 1574 | 1019 | 1431 | 1234 | 661 |

**Unit cell data and crystal lattice parameters for** Mn (II)**:**

Unit cell data and crystal lattice parameters

a (Å) = 7.9163 Volume (V) = 1546.5 $Å^3$

b (Å) = 4.9165 Density (obs.) = 0.8615 $gcm^{-3}$

c (Å) = 8.4089 Density (cal.) = 0.8609 $gcm^{-3}$

α = 90˚ Z = 1

β = 102˚ Crystal system= Monoclinic

γ= 90˚ Space group = P2/m

Standard deviation (%) = 0.042

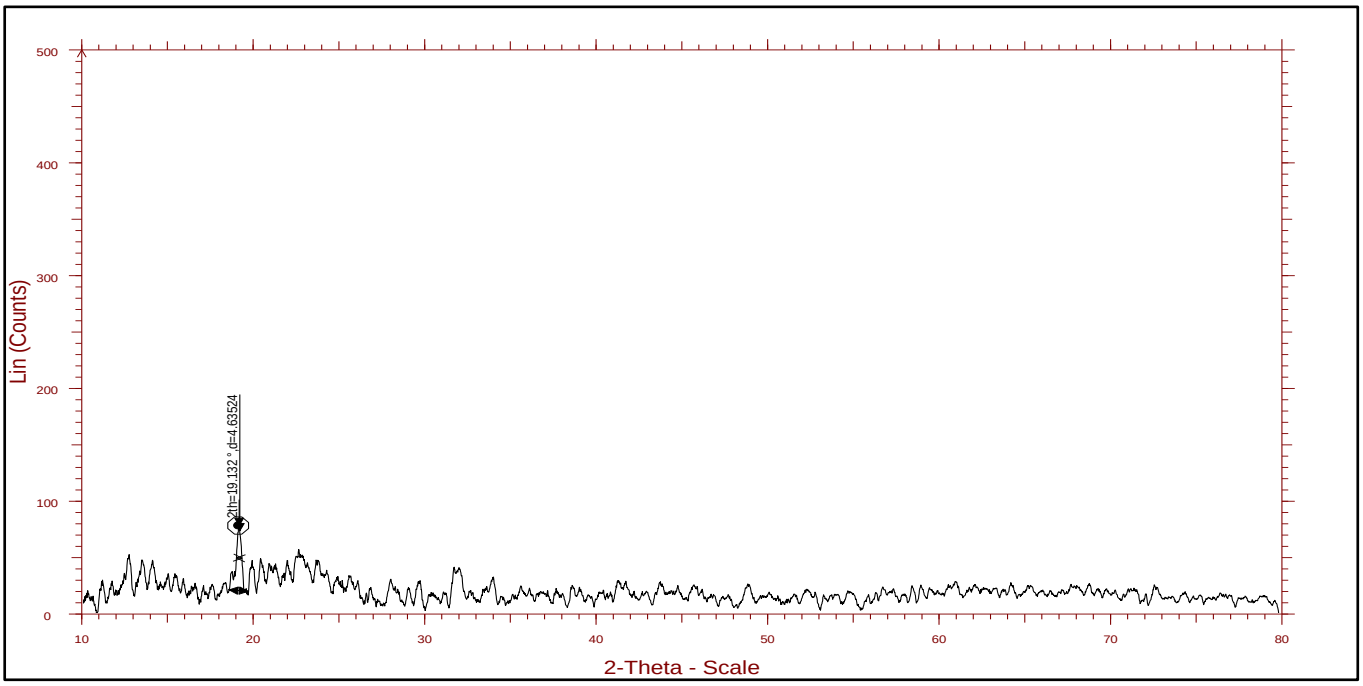


**Figure-3:** X-ray diffractogram of Mn (II) complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand

### 3.8 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 Mn (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 Mn (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 [14].

## IV. CONCLUSION

The Mn (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 Mn (II) has Octahedral geometry. The CHO analysis gives C, H, and O percentage in the metal complex. The XRD parameters shows that the structure of Mn (II) is Monoclinic and has space group = P2/m. From the antimicrobial activity of ligand and complex it is clear that the complex shows enhanced antimicrobial activity than ligand.

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