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Synthesis, Characterization of Mn(II) Complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one Ligand as Antibacterial and Antifungal Agents

ABSTRACT

Mn(II) 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 using analytical data such as, conductivity measurement, magnetic moment, XRD analysis, UV-Vis and Infra Red spectral studies. Analytical data shows 1:2 stoichiometric ratio 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 synthesized Mn(II) complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards. The Mn(II) complex shows promising Antibacterial and Antifungal activities.

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

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1. 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 are 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 α, 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, antibacterial, antimalerial, antifungal and antimicrobial activities [6-7]. All crystals of a substance possess the same elements of symmetry. The computer program, used for indexing data was powder-X [8]. The X-ray powder diffractogram of the metal complex was used for the structural characterization and determination of lattice dimensions.

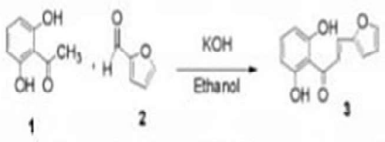
2. Materials and Methods:

The reagent used for preparation of Chalcone is A.R. grade.

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

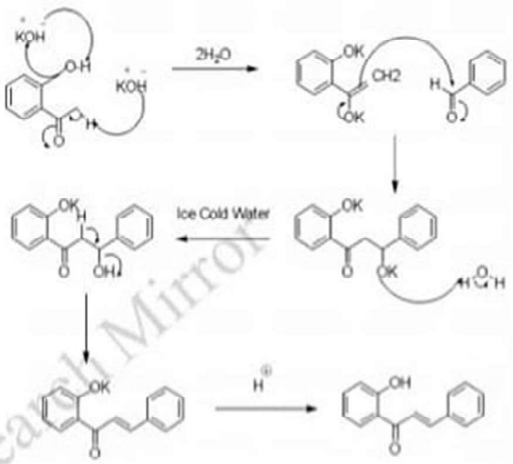
A mixture of 2,6-dihydroxy acetophenone (**1**) (0.01 mol) and 2-furaldehyde (**2**) (0.01 mol) are dissolved in ethanol (20 mL) and then solution of potassium hydroxide 10 mL (15%) were added to it. The mixture was stirred for overnight.

The progress of the reaction was monitored by TLC using eluent Petroleum ether: Ethyl acetate (7:3). It was 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 (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one (**3**) chalcone [9] (**Scheme-1**).



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

General Mechanism of Chalcone formation:

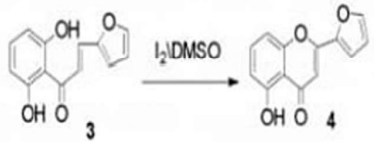


2.2 Synthesis of 2-(furan-2-yl)-5-hydroxy-4*H*-chromen-4-one ligand:

Chalcone (**3**) (0.01 mol) taken in round bottom flask and then it was dissolved in 20 ml DMSO solvent and catalytic amount of iodine was added. Contents were refluxed for one hour; the progress of the reaction was monitored by Thin Layer Chromatography using eluent Petroleum ether: Ethyl acetate (7:3).

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 2-(furan-2-yl)-5-hydroxy-4*H*-chromen-4-one (Flavone) (**4**) was crystallized from ethanol [10].

(Scheme-2)

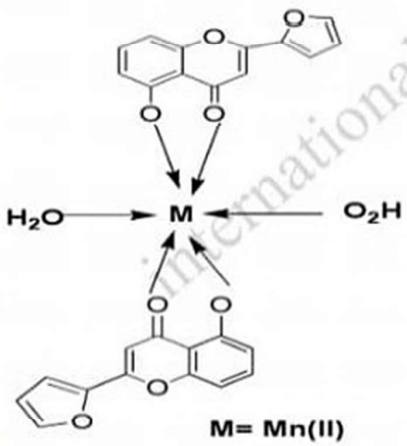


Scheme-2: Synthesis of 2-(furan-2-yl)-5-hydroxy-4*H*-chromen-4-one

2.3 Synthesis of Mn(II) Metal Complex:

The solution of 0.02 mole of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one (**4**) ligand was taken in round bottom flask containing 30 ml of anhydrous methanolic solution and boiled for 10 minutes. A hot solution 0.01 mole of Manganese Acetate in 20 ml of methanol was added drop wise. 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 this complex [11].

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 **Structure-1**.



Structure-1:

Metal complex of Manganese (II) with 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand

3. 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°C. The complex is insoluble in water and soluble in DMSO, DMF [12].

3.2 Elemental 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 C, H, [O] analysis are matching and are given in the **Table-1**.

Table-1: Study of C, H, [O] analysis of 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	$[C_{30}H_{18}O_{10}Mn]$	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:

3.3.1 Magnetic Susceptibility:

The magnetic moment of Mn(II) complex 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 (**Table-2**).

Table-2: Magnetic susceptibility, conductivity and electronic absorption spectral study of synthesized Mn(II) complex:

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

3.3.2 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 µ&! to 200 µ&! 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 $^6A_{1g}$'!$^4T_{2g}$(G), $^6A_{1g}$'!$^4E_{1g}$ or $^6A_{1g}$'!$^4T_{1g}$ (G) and charge transfer indicating octahedral geometry around the metal ion[13-14].

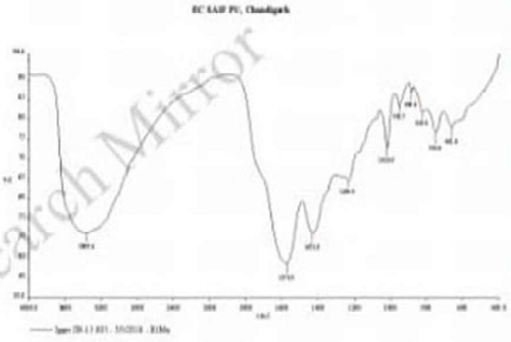
3.4 Infra Red Spectral Study of Mn(II) Complex:

The IR spectrum of α, β-unsaturated carbonyl group has characteristic bands of chalcone at prominent bands between 1625 to 1650 cm^{-1}. 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-3**) using potassium bromide pellet at CIL, Chandigarh, Punjab.

Table-3: IR spectral data Manganese (II) complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand:

Ligand/ Metal complexes	υ (OH) cm^{-1}	υ (H_2O) 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(II) Complex	-	3363	-	1574	1019	1431	1234	661

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 (3) and the IR spectrum in **Graph-1.**



Graph-1: IR spectrum of metal complex of Manganese (II) with 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand

3.6 X-ray diffraction spectral studies 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 synthesized Mn(II) complex 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=90Ï, β= 102Ï, γ=90Ï (Table-4) & (Graph-2).

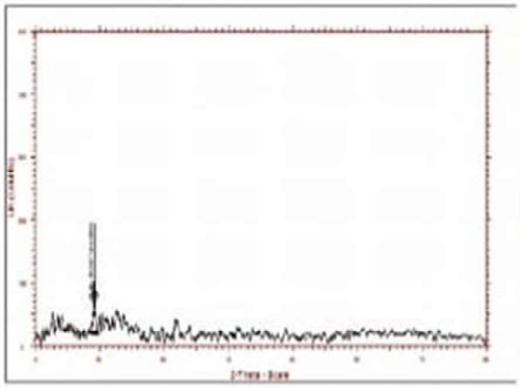
Table-4:
X-ray Diffraction Study of Mn(II) Complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand:

3.7 Indexed X-Ray Diffraction Data of Mn(II) Complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand:

Sr. 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

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

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 = 90Ú
Z = 1
β = 102Ú
Crystal system= Monoclinic
γ= 90Ú
Space group = P2/m
Standard deviation (%) = 0.042



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

3.7 Antimicrobial Activity of Mn(II) Complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand:

Antimicrobial activity of synthesized Mn(II) complex was assayed by cup plate agar diffusion method by measuring inhibition zones in mm.

3.7.1 Antibacterial Study of Synthesized Mn(II) Complex:

The synthesized Mn(II) Complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand was studied for evaluating antibacterial activity were used Gram positive and Gram negative bacterial pathogens. *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633, *Staphylococcus aureus* ATCC 6538 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.

3.7.2 Antifungal Study of Synthesized Mn(II) Complex:

In vitro antifungal activity of all synthesized Metal complex and standard have been evaluated against strains of the fungal toxicity of Mn(II) complex was studied *in vitro* against *Aspergillus niger* ATCC 16404, *Candida albicans* ATCC10231, *Saccharomyces cerevisiae* ATCC 9763 are fungal pathogens at fixed 1% concentration.

From the results of antimicrobial activity of Mn(II) Complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand. It is clear that the complex shows promising antimicrobial 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 [15].

4. Conclusion:

Synthesized Mn(II) Complex of 2-(furan-2-yl)-5-hydroxy-4H-chromen-4-one ligand was colored, soluble in organic solvent. The stoichiometry 1:2 a ratio of the metal complex is obtained has been found. Solution conductivity of Mn(II) Complex reveals nonelectrolytic nature. On the basis of magnetic moment, TG-DTA, electronic spectral data studies shows that Mn(II) complex has Octahedral geometry. The elemental analysis CHO gives C, H, and [O] percentage in the metal complex.

The X-Ray Diffraction parameters shows that the structure of Mn(II) is Monoclinic and has space group = P2/m. From the Antibacterial and Antifungal activities of ligand and complex it is clear that the complex shows enhanced antimicrobial activity than ligand.

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