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CONTENTS

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Synthesis, Characterization and Antimicrobial study of Manganese (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 Manganese (II) metal complex **1** 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-furaldehyde. The structure of the complex has been characterized by the analytical data, conductivity measurement, magnetic moment, UV-Vis spectra, and thermal studies. Analytical data shows 1:2 stoichiometry and the magnetic moment, TG-DTA suggests that Mn(II) complex has octahedral geometry. The presence of coordinated water molecules in Mn (II) complex **1** is confirmed by thermal studies. 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 **1** shows moderate to good Antibacterial and Antifungal activity.

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

I. INTRODUCTION

Chalcones and their metal complexes play an important role in modern coordination chemistry. These compounds possessing novel structural features, interesting spectral and magnetic properties, have been observed of intensive research due to their importance in medical, agriculture, analytical, biological and industrial fields. In recent years a number of β-dicarbonyl compounds in which the carbonyl function bonded to olefinic linkage have gained considerable importance mainly because of the fact that such compounds are structurally related to the active chemical constituents of several traditional medicinal plants[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 metal complexes

possess interesting biochemical properties, such as antitumor, antioxidant, and antimalerial, anti-fungal and antimicrobial activities [5]. The magnetic moment, TG-DTA supports the octahedral geometry of the metal complex of chalcone.

II. 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-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 to give the chalcone [6].

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 Manganese Acetate in 20 ml of methanol was added drop wise to the solution of the chalcone of 5-methylfurfural 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 [7]. 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 **1** is shown in **Figure-1**.

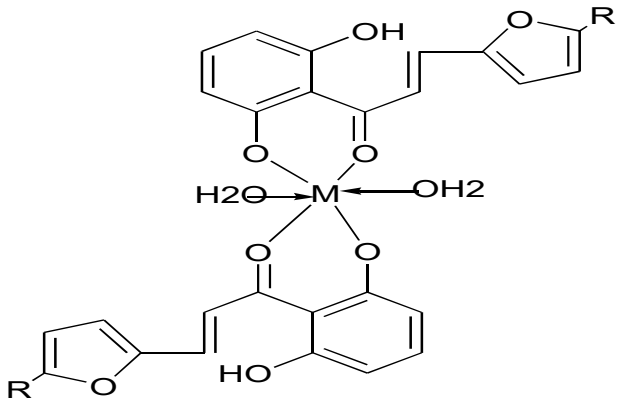

Figure-1: Metal complex **1** of Manganese (II) with (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one
R= -H, M= Mn(II)

III. RESULTS AND DISCUSSION

3.1. Physical parameters:

Metal complex **1** of Manganese (II) with (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one was 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 [8].

3.2. CHO analysis:

The carbon, hydrogen, oxygen, Manganese metal percentage in Mn (II) complex **1** 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 of synthesized Mn (II) complex **1**

Metal complex	Chemical formula	Mol. Wt.	Elemental analysis : % found (calculated)						
			C	H	N	O	S	X(Br)	M
Mn (II) Complex	$[C_{26}H_{22}O_{10}Mn]$	549	56.84 (64.33)	4.03 (4.57)	-	29.12 (19.78)	-	-	9.99 (11.32)

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

The magnetic moment of Mn (II) complex **1** 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) complex **1** by earlier workers [9].

Table-2: Study magnetic susceptibility, solution conductivity and electronic absorption of synthesized Mn (II) complex 1 of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

Mn(II) 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
1	2.12	5.86	24937(401)	28571(350)	32154(311)

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) complex **1** in DMSO indicates their non-electrolytic nature.

Figure-2.

Figure-2: Electronic absorption spectrum of Mn (II) complex **1** of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

The electronic absorption spectra of Mn(II) complex **1** **Table-1:** Study CHO analysis synthesized Mn (II) complex **1** 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 [10-11].

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 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 Mn (II) complex **1** of (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-2**) using potassium bromide pellet at CIL, Chandigarh, Punjab. The stretching frequency of (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.**

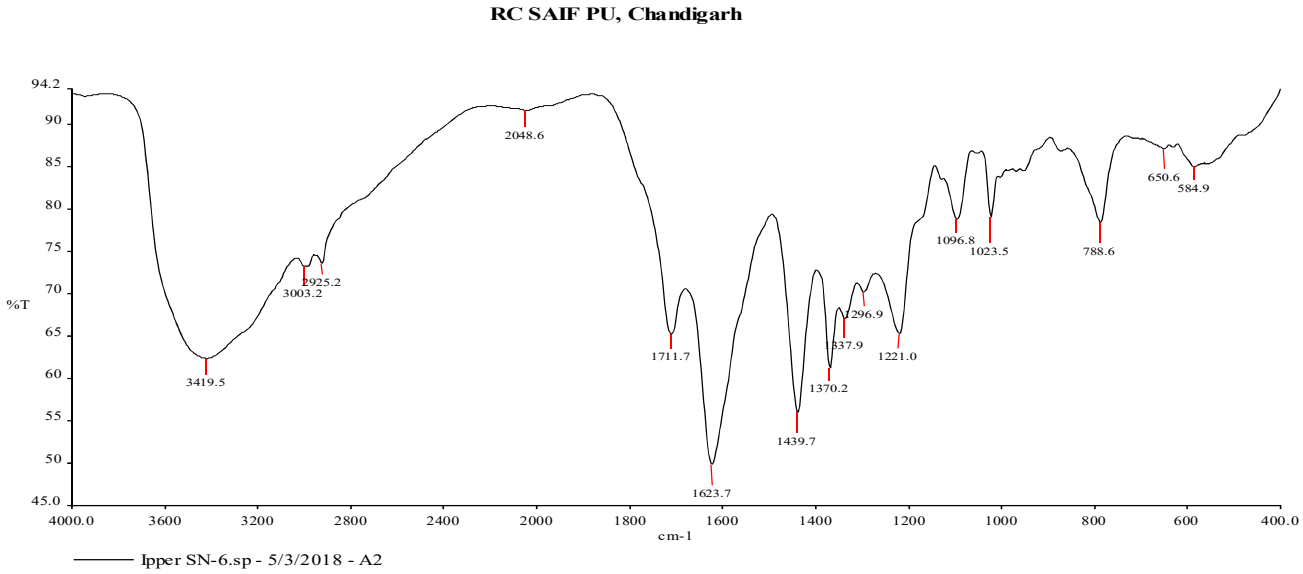

Figure-3: IR spectrum of Mn (II) complex **1** of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

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

Molecule	υ(OH) Enolic	(-CO-CH=CH-) α,β-unsaturated carbonyl group	Carbonyl group (-C=O in pyron ring)	(C-O-C) Stretching Frequency	(C=C) Stretching Frequency	Aromatic Ring (C=C) Stretching Frequency	Ar-H Stretching Frequency	$-NO_2$ stretching frequency
Ligand	3420	1652	-	1096	1575	1457	2920	-

3.5. Thermal analysis Mn (II) complex 1 of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

The simultaneous thermo gravimetric, differential thermal analysis of Mn (II) complex **1** of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one was performed in an inert nitrogen atmosphere on Perkin Elmer STA

6000 at SAIF, Cochin, Kerala. The heating rate was 10˚/min and flow rate of nitrogen 50 ml/min. The reference substance used was α Al_2O_3 in platinum crucible and sample weighted in the range of 4-12 mg. The thermogram of Mn (II) complex (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one is presented in figure-2. This curve reveals that there is presence of lattice as well as coordinated water in the complex.

The thermogram of Mn (II) complex **1** of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one shows first weight loss at 60˚C indicating presence of lattice water. The second loss due to the coordinated water molecule liberated, from the complex. The anhydrous compound undergoes four step decomposition. In the first two steps, decomposition occurs due to loss of non-coordinated part of ligand. The first step shows decomposition within a temperature of range from 240-330˚C with mass loss of 39.29%, which is supported by a sharp an endothermic peak at 259˚C in DTA curve. It may be due to half decomposition of non-coordinated part of ligand. In the second step, decomposition observed at about 350-400˚C with the weight loss of 33.78% in TG curve. This is supported by an endothermic peak at 380˚C. This may be due to decomposition of remaining coordinated part of ligand. Beyond 600˚C there is a formation of MnO as indicated by constant weight loss of in TG-DTA curve.

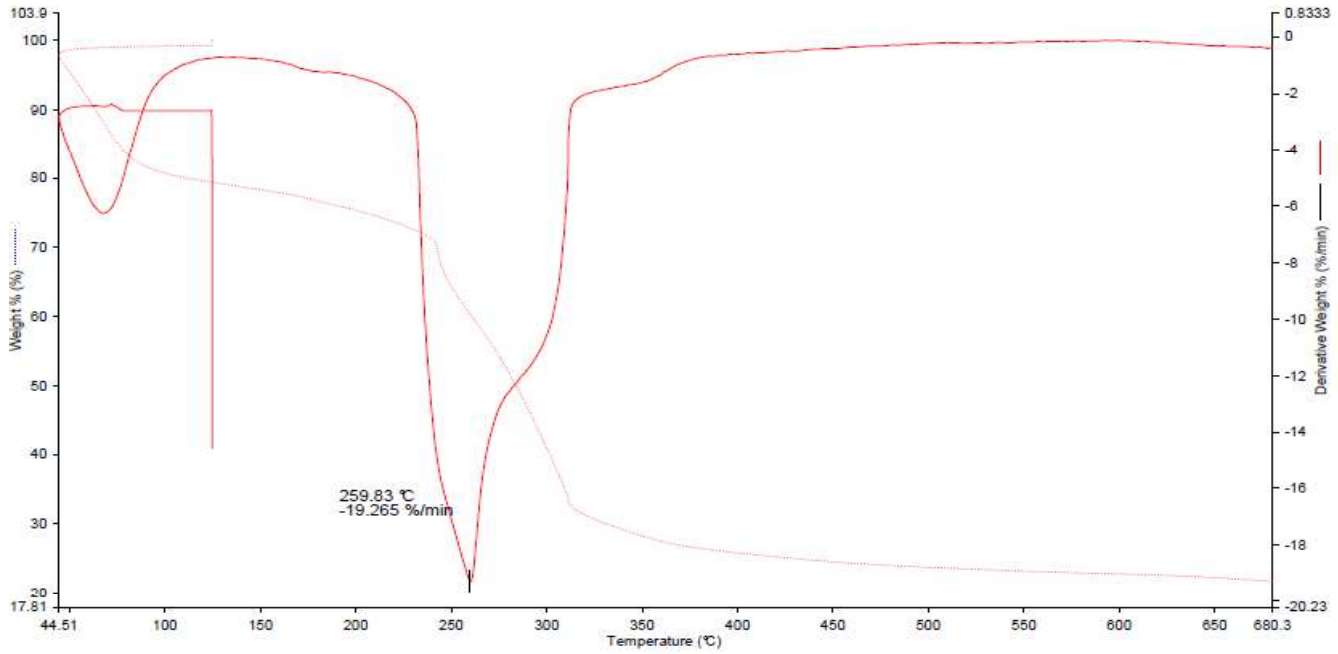

Figure-4: TG-DTA curve of Mn (II) complex **1** of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

3.6. Thermodynamic and Kinetic Parameters

Akahira [12], 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 [13]

Table-3: Thermodynamic and Kinetic Parameters of Mn (II) complex 1 of (E)-3-(furan-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

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)
Mn (II) complex	H-M C-R	I	300	0.5	26.84 21.75	-153.91 -97.64	37.80 28.70	112973.9 98100737	0.907 0.989
	H-M C-R	II	450	0.5	6.73 4.09	-172.89 -83.66	19.06 9.96	11545.4 527754555.2	0.999 0.997

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 Mn (II) complex **1** 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 **1** 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 **1** 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 non-electrolytic 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. From the antimicrobial activity of ligand and complex it is clear that the complex shows enhanced antimicrobial activity than ligand.

V. ACKNOWLEDGEMENT

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Chalcone Biological Significance and Synthesis A Review

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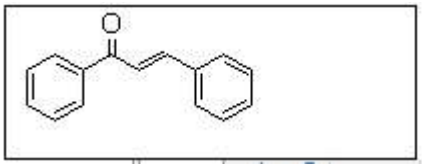
ABSTRACT

Chalcone is an aromatic ketone that forms the central core of many important biological compounds. The biogenetic building blocks of favonoids and isofavonoids, which are abundant in plants, are called chalcones. Chalcones are active lead molecules in the search for new drugs in medicinal chemistry. Here, we review the biological significance and synthesis of natural and synthetic chalcones.

Keywords: Chalcones, Antibacterial activity, Antidiabetic activity, Claisen–Schmidt condensation, Witting reaction

I. INTRODUCTION

Chalcones are the building blocks of several natural compounds.[1-2] The word "chalcone" is derived from the Greek word "chalcos", meaning "bronze", which results from the colors of most natural chalcones.[3] Chalcones are 1, 3-diaryl-2-propen-1-ones with various substitution patterns that exist in cis and trans isomeric forms, with the trans form being thermodynamically advantageous.They are represented as



On their aryl rings, they have different substituents. Some of these substitutes affect the biological properties that chalcones have. However, the key pharmacophore is believed to be the, α,β -unsaturated ketone moiety.[4] Naturally occurring chalcones and their synthetic analogues have been documented to comprise a wide range the biological activity. They are therefore high in demand as the starting components in the synthesis of a number of different heterocyclic compounds. Chalcones have been utilized as medicine for thousands of years to treat a variety of pharmacological conditions through the use of plants and herbs. There are many chalcone-containing drugs that have received clinical use approval. Metochalcone was previously employed as a choleretic medication, whereas sofalcone was used as an antiulcer and mucoprotective medicine (Figure 1)[5, 6]