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Synthesis, Characterization And Antimicrobial Study Of Nickel(II) Complex Of (E)-1-(2,6-Dihydroxyphenyl)-3-(5-Methylfuran-2-Yl)Prop-2-En-1-One

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Abstract:

The synthesis of Nickel (II) metal complex has been synthesized by using novel (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-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, thermal studies and XRD analysis. Analytical data shows 1:2 stoichiometry and the magnetic moment, TG-DTA suggests that Ni(II) complex has octahedral geometry. The presence of coordinated water molecules in Ni (II) complex 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 Ni (II) complex shows moderate to good Antibacterial and Antifungal activity.

Keywords: Antimicrobial activities, TG-DTA, XRD study, 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 antimalerial, anti-fungal and antimicrobial activities [3]. The magnetic moment, TG-DTA supports the octahedral

geometry of the metal complex of chalcone. All crystals of a substance possess the same elements of symmetry. The computer program, used for indexing data was powder-X [4].The X-ray powder diffractogram of the metal complex was used for the structural characterization and determination of lattice dimensions.

2. Materials and Methods:

2.1 Synthesis of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one ligand:

The reagents used for preparation of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one are of A.R. grade. The mixture of 2,6-dihydroxy acetophenone (0.01 mol) and 5-methyl-Furaldehyde (0.01 mol) is dissolved in ethanol (20 mL) and then sodium hydroxide 10 mL (40%) was added to it. The mixture was stirred for overnight till brown color ppt was observed. The progress of the reaction was monitored by TLC, from thin layer chromatography the completion of the reaction is observed. After completion of the reaction, the contents were poured into ice cold water and then acidified by dil. HCl. The solid obtained was filtered and crude product was recrystallized from ethanol to give the chalcone [5].

2.2 Synthesis of Metal Complex:

The solution of 0.02 mole of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)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 Nickel Nitrate 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 [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 Ni(II) complex is shown in **Figure-1**.

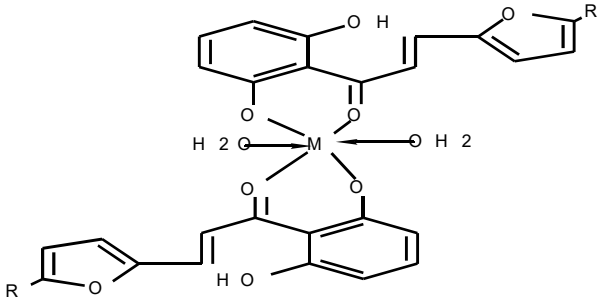

Figure-1: Metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methyl furan-2-yl)prop-2-en-1-one

3. Results and Discussion:

3.1 Physical parameters:

Metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one was reddish brown in color. The complex was precipitated at 8 pH range, having Melting point 280^0C. The complex is insoluble in water and soluble in DMSO, DMF [7].

3.2 CHO analysis:

The carbon, hydrogen, oxygen, Nickel metal percentage in Ni (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 Ni (II) complex

Metal complex	Chemical formula	Mol. Wt.	Elemental analysis : % found (calculated)						
			C	H	N	O	S	X(Br)	M
Nickel (II)	$[C_{28}H_{26}O_{10}Ni]$	581	57.86 (65.02)	4.50 (5.06)	-	27.52 (18.56)	-	-	10.09 (11.34)

3.3 Magnetic susceptibility, solution conductivity and electronic absorption spectral data

Magnetic susceptibility:

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 conductivity values in DMSO solution (10^{-3} M) are indicates non-electrolytic nature. The Solution conductivity was (

Figure-2: Electronic absorption spectrum

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 Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)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 metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one is represented in table number (2) and the IR spectrum in **Figure-3.**

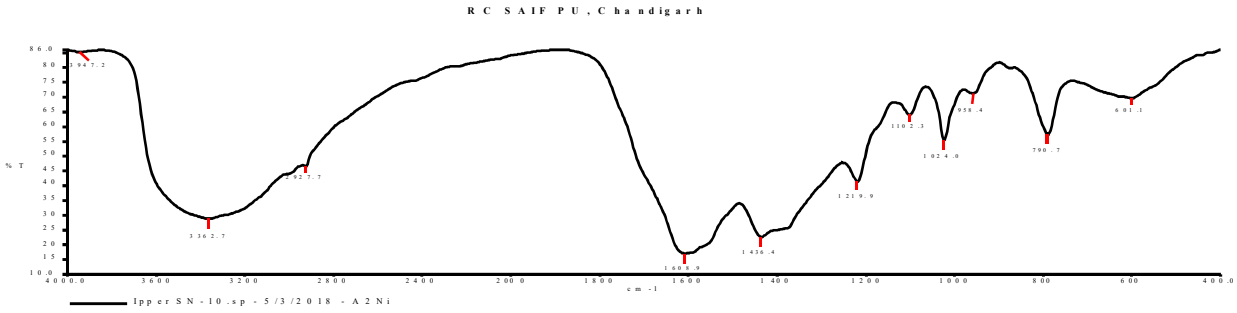

Figure-3: IR spectrum of metal complex of Nickel (II) with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one

Table-2: IR spectral data Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one:

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}
$[Ni(A_2)_2]$	2927	3362	1608	-	1024	1436	1219	601

3.5 Thermal analysis Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one:

The simultaneous thermo gravimetric, differential thermal analysis of Ni(II) complex (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)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 weighed in the range of 4-12 mg. The thermogram of Ni(II) complex (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)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 Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one (**Figure-4**) shows high thermal stability and two-step decomposition. The first step within temperature range 240-330˚C with mass loss of 40.36% (calc. wt. loss 41.03%) due to two fragments of $C_6H_5O_2$ take place. This is confirmed by an endothermic peak at 275.86˚C in DTA curve. The second step decomposition observed within 380-420˚C with a weight loss of 46.32%. This may be due to the loss of two fragment of 5-methyl-furan and enone part of chalcone of metal complex. Beyond that TG curve attains a constant weight loss 14.44% level corresponding to their metal oxide that is NiO.

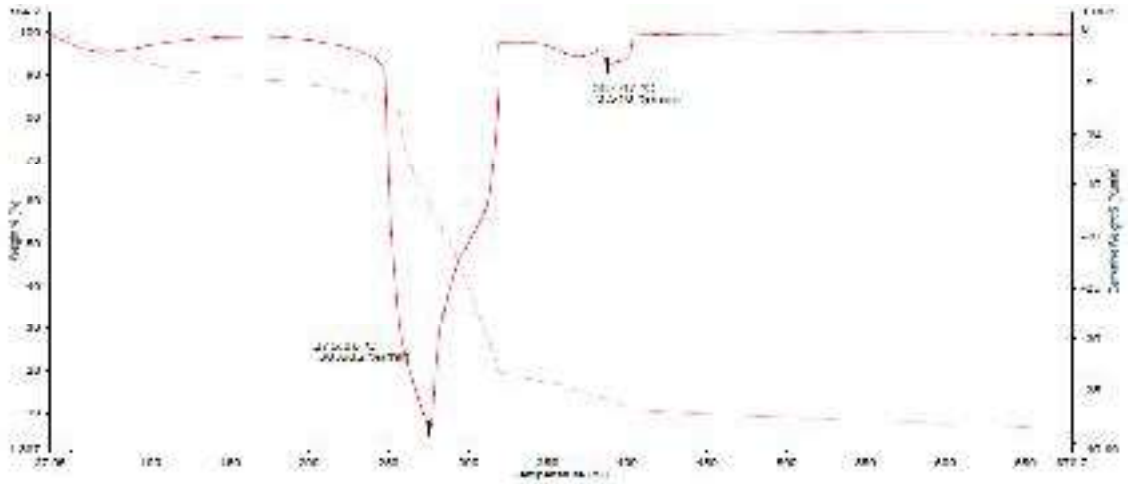

Figure-4: TG-DTA curve of Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one

Thermodynamic and Kinetic Parameters

Akahira [11], 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[12].

Table-3: Thermodynamic and Kinetic Parameters of Ni(II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)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)
A_2Ni	H-M C-R	I	290	0.9	13.42 70.96	-161.70 -138.30	24.89 80.81	47529.95 738243	0.999 0.998
	H-M C-R	II	580	0.9	6.73 8.69	-171.09 -86.94	18.93 14.89	14335.18 355917865.6	0.987 0.996

3.6 X-ray diffraction spectral studies of metal complex of Ni (II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one:

The XRD spectral study has been done at SAIF, Cochin Kerala. The standard deviation observed for A_2Ni is 0.023% which is within the permissible limit of 2%. The observed and calculated densities are 1.9583 gcm^{-3} and 1.7392 gcm^{-3} respectively. The volume is found to be 339.43 $Å^3$ and complex crystallizes in the monoclinic system with 1 atom per unit cell. The lattice parameters are a=13.345 Å, b=8.252 Å, c= 9.756 Å, α=90°, β= 109.78°, γ=90°.

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

Unit cell data and crystal lattice parameters

a (Å) = 13.345, b (Å) = 8.252, c (Å) = 9.765, α = 90°, β = 109.78°, γ= 90°, Standard deviation (%) = 0.23, Volume (V) = 339.43 $Å^3$, Density (obs.) = 1.9583 gcm^{-3}, Density (cal.) = 1.7392 gcm^{-3}, Z = 4 Crystal system= Monoclinic, Space group = P2/m. (**Figure-5**)

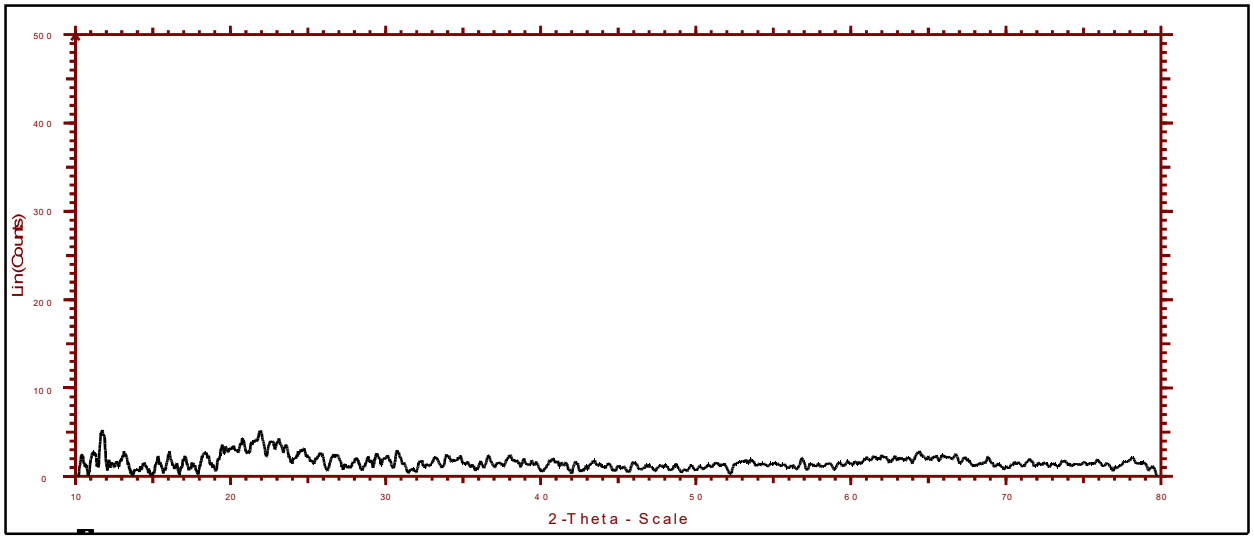

Figure-5: X-ray diffractogram of Ni (II) complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one

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 Ni(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 Ni(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 [13].

Conclusion:

The Ni (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 Ni(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 Ni(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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