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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF NICKEL (II) COMPLEXES OF SOME 2-HYDROXY-4, 5-DIMETHYL SUBSTITUTED CHALCONES

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ABSTRACT The present paper deals with synthesis and characterization of metal complex of some novel 2-hydroxy-4, 5-dimethyl substituted chalcones. The substituted chalcones were prepared by reacting 2-hydroxy-4, 5-dimethyl acetophenone with corresponding aromatic aldehydes like 2-chorobenzaldehyde, 3-bromobenzaldehyde, 4-fluorobenzaldehyde and 4-cyanobenzaldehyde. The Nickel (II) metal complexes of general formula ML2 with newly prepared chalcones synthesized and characterized by several physiochemical techniques like Melting point, Elemental analysis, molar conductance, magnetic susceptibility, Electronic absorption and Infrared spectral studies. The analytical data confirmed 1:2 stoichiometry of M: L and the electronic spectral data suggest that all Ni (II) complexes have octahedral geometry. The conductivity data show that all these complexes are non-electrolytes. Furthermore, biological activities of complexes with selected bacterial strain carried and the results have been compared with commercial standards.

KEYWORDS : Transition metal complexes, Chalcones, Spectral characterization and biological activities

INTRODUCTION:

The chemistry of chalcones generated intensive scientific studies throughout the world, specially interesting for their biological applications. Chalcones are coloured compounds[1] because of the presence of the chromophore and auxochromes. Chalcones are of a great interest because they have a unique structural feature[2] of having a >C=O functional group in conjugation with >C=C < and the whole molecule is in conjugation. The chalcones are α,β-unsaturated ketones containing the reactive keto ethylene group –CO–CH=CH which makes it biologically active. Some substituted chalcones and their derivatives have been reported to possess some interesting[3] biological properties such as antibacterial, antifungal, insecticidal, ulcerogenic, anticancer, anti-inflammatory, anaesthetic, analgesic, antimalarial, antioxidant etc.

Chalcones are also key precursor[4,5] in the synthesis of many biologically important heterocycles. Transition metal ions are found in several biological species and are reported to play an important role in different enzymatic and physiological reactions, the interaction of chalcones with metal ions may also change the antioxidant properties and also biological effects of the chalcones[6].

MATERIALS:

All the chemicals and reagents were of AR grade and used without further purification. Ethanol, DMF, $NiSO_4.6H_2O$, 2-hydroxy-4, 5-dimethyl acetophenone, 2-chorobenzaldehyde, 3-bromobenzaldehyde, 4-fluorobenzaldehyde and 4-cyanobenzaldehyde etc were used in this work.

METHODS:

1) Synthesis of transition metal complexes:

The substituted chalcones were prepared[7] by reacting 2-hydroxy-4,5-dimethyl acetophenone with corresponding aromatic aldehydes like 2-chorobenzaldehyde, 3-bromobenzaldehyde, 4-fluorobenzaldehyde and 4-cyanobenzaldehyde in presence of alkaline medium at room temperature.

The transition metal complexes were prepared by mixing corresponding chalcone ligands with Nickel sulphate in the ratio of 1:2. A warm ethanolic solution of $NiSO_4.6H_2O$ (0.01mole) was added to ethanolic solution (0.02 mole) of corresponding chalcone ligands. The pH of solution adjusted to 7-8 using aqueous ammonia Solution. The mixture was heated under reflux for 2-3 hrs. The complex thus obtained was filtered and washed with excess of ethanol and dried at room temperature.

2) Biological Studies:

The complexes were screened for antibacterial activity against gram positive *Staphylococcus aureus*, gram negative *Escherichia coli and for antifungal activity against Aspergillus niger by following methods.*

a) Antibacterial activity:

The minimum inhibitory concentration(MIC) of the chalcone ligands and their complexes was carried using Broth dilution method[8]. The stock solution of all the complexes was prepared by dissolving 10 mg of complex in 10 ml DMF. The complex solution was serially diluted to give a concentration of 512, 256, 128, 64, 32and 16 μg/ml in test tubes containing 1 ml sterile nutrient broth. Then, the tubes were inoculated with 100 μl of bacterial suspension in the saline and incubated at 37°C for 24 hrs. A tube containing nutrient broth only was seeded with the test organism to serve as control. All the tubes were then incubated at 37°C for 24 h and then examined for growth by observing O.D. at 600nm.

b) Antifungal activity by Disc Transfer Technique:

The measurement of antifungal activity by disk transfer technique was carried using 6 mm diameter disks. Single disc was aseptically inoculated with sabouraud's dextrose agar medium containing desired concentration of the test complex.Test and control (without complex) were inoculated for 3-4 days at 28°C and zone of inhibition was calculated using standard technique.

RESULT AND DISSCUSSION:

1) Characterisation of complexes:

All the complexes are stable at room temperature insoluble in water and most of the common organic solvents but soluble in DMF and DMSO. The elemental analysis data of its complexes are in good agreement with proposed molecular formulas. The thermogram of Ni (II) complexes shows the presence of two hydrated water molecule. The analytical data of the complexes (Table-1) indicates that their stoichiometry may be represented as 1:2 metal to ligand ratio. The molar conductance values of the complexes in DMF solvents suggesting their non-electrolytic nature.

The μ_{eff} values at room temperature for Ni (II) complexes have magnetic moment values in the range of 2.80 to 3.20 B.M. These values are expected for octahedral geometry of Ni (II) complexes at room temperature and further supported by electronic spectral data.

Table-1 Elemental Analysis, Magnetic data of Ni(II)-Chalcone Complexes

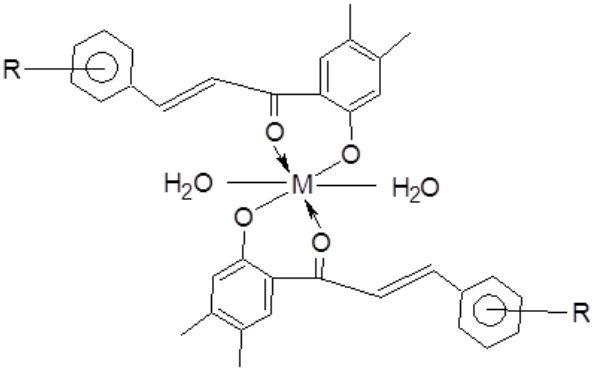
Complex	Molecular Formula	Mol. Wt. (gm/mol)	Color	M.P. °C	Yield (%)	Elemental analysis found/Calculated %				μ_{eff} [BM]	Molar Conductance (mho.cm^2. mol^{-1})
						Carbon	Hydrogen	Halogen	Metal		
Ni(II)-2-Chloro Complex	$[Ni(II)(C_{17}H_{14}O_2Cl)_2.2H_2O]$	630.18	Brown	>300	60	64.80 (64.85)	4.48 (4.51)	11.25 (11.16)	9.31 (9.37)	2.83	0.002
Ni(II)-3-Bromo Complex	$[Ni(II)(C_{17}H_{14}O_2Br)_2.2H_2O]$	719.09	Yellow	>300	63	56.79 (56.83)	3.92 (3.97)	22.22 (22.26)	8.16 (8.11)	3.20	0.004
Ni(II)-4-Fluoro Complex	$[Ni(II)(C_{17}H_{14}O_2F)_2.2H_2O]$	597.27	Green	>300	62	68.37 (68.42)	4.73 (4.69)	6.36 (6.341)	9.83 (9.74)	2.91	0.002
Ni(II)-4-Cyano Complex	$[Ni(II)(C_{18}H_{14}O_2N)_2.2H_2O]$	611.31	Green	>300	73	70.73 (70.79)	4.62 (4.57)	-	9.60 (9.44)	2.88	0.009

Infra-Red:

The IR Spectra of all the Ni (II) –Chalcones Complexes have been recorded in KBr pellets in the region of 4000-400 cm$^{-1.}$ On FTIR Spectrophotometer Shimadzu (Model).The IR spectral data along with the possible assignments of chalcone complexes are presented in Table 2.The evidence drowns for the formation of complexes has been done by using their analytical data, spectral techniques and magnetic studies. Thus on the basis of above results the possible geometry around the metal ion was evidently confirmed in such a way that the Ni (II) complexes form octahedral geometries

Table-2 : Some Important IR Bands in cm^{-1}of the Ni (II) chalcones-Metal Complexes

Complex		$\nu_{(O-H)}$ (H_2O)	ν_{C-O} (Phenol)	$\nu_{Ph-C=C}$	$\nu_{C=O}$	ν_{M-O}
Ni(II)-2-Chloro Chalcone Complex	$[Ni(II)(C_{17}H_{14}O_2Cl)_2.2H_2O]$	3265	1188	1572	1641	638
Ni(II)-3-Bromo Chalcone Complex	$[Ni(II)(C_{17}H_{14}O_2Br)_2.2H_2O]$	3351	1188	1577	1647	651
Ni(II)-4-Fluoro Chalcone Complex	$[Ni(II)(C_{17}H_{14}O_2F)_2.2H_2O]$	3301	1190	1575	1653	698
Ni(II)-4-Cyano Chalcone Complex	$[Ni(II)(C_{18}H_{14}O_2N)_2.2H_2O]$	3278	1188	1573	1641	687



Where M – Nickel, R – 2- Chloro, 3-Bromo, 4-Fluoro, 4-Cyano Chalcone.

Figure 1: Proposed structure of the complexes

Table-3 : Antimicrobial activity of synthesized Ligands and Ni (II) Complexes

Samples	Antibacterial		Antifungal
	E. coli	S. aureus	A. Niger
	(MIC- µg/ml)		(ZOI- mm)
DMF	-	-	-
Ampicillin	4	8	-
Fluconazole	-	-	30 ± 02
2-hydroxy-4,5-dimethly-2-chloro chalcone	>512	>512	-
2-hydroxy-4,5-dimethly-3-bromo chalcone	16	128	-
2-hydroxy-4,5-dimethly-4-fluoro chalcone	16	256	08 ± 0.2
2-hydroxy-4,5-dimethly-4-cyano chalcone	128	218	09 ± 0.2
Ni(II)-2-chloro chalcone Complex	>512	>512	-
Ni(II)-3-bromo chalcone Complex	-	-	-
Ni(II)-4-fluoro chalcone Complex	>512	>512	-
Ni(II)-4-cyano chalcone Complex	16	>512	14 ± 0.2

In addition to this keeping in view of the increasing problems of antimicrobial resistance complexes were screened for their antibacterial and antifungal activities.

The antimicrobial activity of all the complexes is less than that of standard Ampicillin and Fluconazole, Shown in (Table-3).

CONCLUSION:

The elemental analysis, magnetic susceptibility, IR, 1H NMR and Physical Properties observations suggest the octahedral geometry for the Ni (II) complexes and also exhibit the antimicrobial activity.

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