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Synthesis and Antimicrobial Activity of Some Phenothiazine Chalcones and Its Metal Complexes

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Abstract : A series of six transition metal complexes of phenothiazine chalcones were synthesized. All the synthesized ligands and its metal complexes were analysed by elemental and spectral analysis Viz IR, ¹H-NMR, TGA-DTA, magenetic moment and molar conductivity. The analytical data confirm the metal to ligand stoichiometry as 1:2. The ¹H-NMR spectra of the complexes shows broad peaks due to complex pattern of splitting. The molar conductivity data confirms the non electrolytic nature of all the complexes. The Co (II), Ni(II), Fe(II), shows octahedral geometry whereas Zn (II) and Cu(II) shows tetrahedral geometry.

Keywords : Phenothiazine Chalcones, Metal Complexes, Spectral analysis.

Introduction

There is a great interest in synthesis and characterization of ligands which contain O, N, S sequence and their metal complexes. Chalcones were synthesized by the condensation of acetophenones with aromatic aldehyde in the presence of acidic and basic media. The chalcones shows remarkable antimicrobial activities^{1,2}, they belong to flavonoid family. Chalcones have displayed an impressive array of biological activities among which, antitumour³, anticancer⁴, antimalarial⁵, Biocedal⁶, antityrosinase⁷, antiinflammotory⁸, Scaffold⁹, antihyperglycemic¹⁰ activities have been reported. Chalcones are also key precursors in the synthesis of many important biologically important heterocyclic compounds. The presence of α - β unsaturated keto function in chalcone is found to be responsible for their biological activities. Thus the synthesis of chalcones has generated vast interest to organic as well as for medicinal chemist.

So keeping all these points in mind we synthesized new phenothiazine chalcones

Experimental

All the chemicals used for the synthetic work were of A.R. grade. The solvents used were purified by standard methods. All the melting points were determined in an open capillary tube and are uncorrected. Completion of the reaction was monitored by thin layer chromatography on pre-coated sheets of silica gel-G.

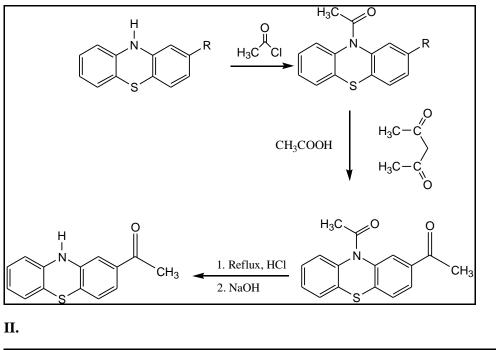
a. Synthesis of chalcones:

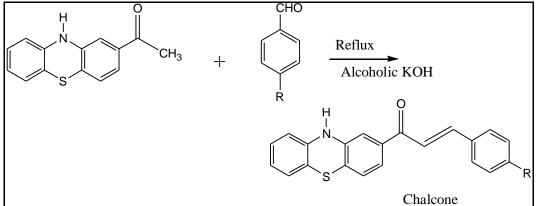
To the solution of 2-acetyl phenothiazine in absolute methanol (50 ml) benzaldehyde was added in presence of 40% methanolic KOH (10 ml) and refluxed for 3 hours on water bath. After refluxing the reaction mixture was kept at room temperature for 1 hour.

Filter the reaction mixture to remove insoluble byproducts, then acidified the filtrate with Dil. HCl. The solid obtained was filtered at sunction pump, washed with cold water and recrystallise the solid using methanol to give the chalcone i.e. ligand

Scheme:

I.

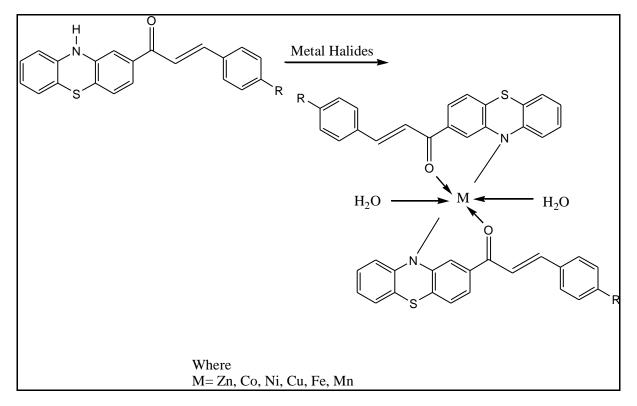




b. Synthesis of metal complexes

The ligand (0.02 mole) and the metal salt (0.01 mole) in 50 ml methanol was refluxed for 2 hours in a reaction flask. The solid mass separated was filtered through a sintered glass crucible (G4) and the residue was washed several times with hot methanol until the washings were free of the excess of ligand. Analytical and physical data is given in table-I

Scheme



Result and discussion

All the complexes are stable at room temperature, insoluble in water and common organic solvents but soluble in DMF and DMSO. The analytical data of the complexes indicates their stoichiometry as 1:2 metal to ligand ratio

IR Spectra:

The ligand showed a sharp peak at 3352 and 3378 Cm^{-1} due to -NH stretch. In the IR spectra of complexes this band appears as an intense broad band near 3680-3800 Cm^{-1} due to coordination of N-H bond along with the metal.

In the IR spectra of all the ligands an intense band appearing around 1653 Cm^{-1} is attributed to V(C=O) this band is shifted to lower wave number in the spectra of the complexes indicating coordination through oxygen of (-C=O-) group. The V(M-O) band was observed in the complexes around 440-455 Cm^{-1} . The literature survey support such interpretation

¹H-NMR Spectra

¹H-NMR Spectra of complexes and ligands shows well resolved signals. Due to complex formation there observed broad peaks in 1H-NMR spectra of complexes.In the ¹H-NMR spectrum of complexes the signal due to –NH proton is absent, suggesting the deprotonation of the –NH group attached to aromatic ring in the chalcone.The peak in the ¹H-NMR spectra of ligands near at d 8.8 (S, 1H, -NH) is disappear in the complex spectra confirming the coordination of the ligand to the metal ion through this Nitrogen atom.

Magnetic Moment

The Magnetic Moment values of the complexes are in the range of 2.90 B.M. for Zn(II) and 4.32 B.M. for Co(II) complexes, 5.08 B.M. for Ni (II), 2.15 B.M. for Cu (II) and 2.02 B.M. for Fe (II) which actually observed for the octahedral geometry of Zn(II) and Co(II), Ni(II) complexes and square planar geometry for the Cu(II) complexes

Molar Conductivity

The Molar conductance values of the complexes are in the range of $15.02-27.05 \text{ SCm}^2 \text{ Mol}^{-1}$ for Zn(II) complexes and $12.08-24.02 \text{ SCm}^2 \text{ Mol}^{-1}$ for Co(II), Ni(II) complexes and in the range of $20.11-20.15 \text{ SCm}^2 \text{ Mol}^{-1}$ for Cu(II) and Fe(II) complexes suggesting their non electrolytic nature.

Sr.	Molecular formula	Molecular	colour	M.P.	Molar	μ eff
No.		weight			conductivity	
1	$C_{21}H_{14}ONS$	171	Light	76		
			brown			
2	$(C_{21}H_{14}ONS)_2Zn$	372	Yellow	206	15.03	2.90
3	$(C_{21}H_{14}ONS)_2 Co$	369	Brown	242	13.20	4.31
4	$(C_{21}H_{14}ONS)_2$ Ni	370	Orange	249	12.08	5.08
5	$(C_{21}H_{14}ONS)_2Cu$	371	Light	262	20.15	2.15
			Green			
6	$(C_{21}H_{14}ONS)_2$ Fe	368	Dark	210	20.11	2.20
			Brown			
7	$(C_{21}H_{14}ONS)_2Mn$	367	Brown	194	12.60	

Table-I: Physical and Analytical data of the ligand and complexes

Conclusion

All the transition metal complexes are coloured insoluble in most of the organic solvents but soluble in DMSO and DMF. The stoichiometric ratio of metal to ligand is 1:2. The IR spectral data indicate that all ligands act as mononegativebidentate species towards all the complexes. Molar conductivity data shows the non electrolytic nature of the complexes. Thermal analysis of Zn(II), Fe(II), Ni(II), Co(II) complexes confirms that there are two moles of coordinated water.

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