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PHENOTHIAZINE CHALCONE COMPLEXES: SYNTHETIC AND STRUCTURAL STUDIES

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ABSTRACT

Twelve transition metal complexes of substituted phenothiazine chalcones were synthesized by condensing phenothiazine chalcones with transition metal halides. The chalcone can be prepared by reacting 2-acetyl phenothiazine with P-chloro benzaldehyde and P-methoxy benzaldehyde. Further the synthesized ligands and the metal complexes were characterized and analysed by using different elemental and spectral analysis viz IR, ^{1}H-NMR, TGA-DTA, Magenetic Moment, Molar Coductivity etc. The analytical data confirm the stoichiometry of metal to ligand as 1:2. The molar conductivity data confirms the non electrolytic nature of all the metal complexes. The 1H-NMR spectra of complexes shows broad peaks due to complex pattern of splitting. The thermal study shows the presence of coordinated water molecule in some complexes. The Co (II), Ni (II), Fe (II), and Zn (II) shows octahedral geometry whereas Mn(II) and Cu (II) shows tetrahedral geometry.

KEYWORDS: 2-acetyl phenothiazine chalcones, Transition Metal Complexes, Spectral analysis.

INTRODUCTION

There is a great interest in synthesis and characterization of ligands which contain O, N sequence and their metal complexes. Chalcones were synthesized by the condensation of aromatic ketones with aromatic aldehyde in presence of acidic and basic media. Chalcone derivatives are associated with some important biological activities such as antibacterial[1], antifungal[2], anticancer[3], antileishmanial, antimalerial[4], scaffold[5] and potent Tyrosinase activity.[6] Chalcones are more potent bioactive compounds.[7] The bidentate chalcones containing O, N donar sequence have been tried for complexation with transition metals.

The search for a new sequence has resulted by the condensation of chalcones. The neutral bidentates are shown to form octahedral and tetrahedral complexes with transition metals, where two ligands encompass the metal ion in a octahedral or tetrahedral array. In this paper we wish to report the synthesis and characterization of Co(II), Cu(II), Ni(II), Mn(II), Zn(II), and Fe(II) complexes obtained from the condensation of the metal halides with the substituted phenothiazine chalcones.

MATERIAL AND METHOD

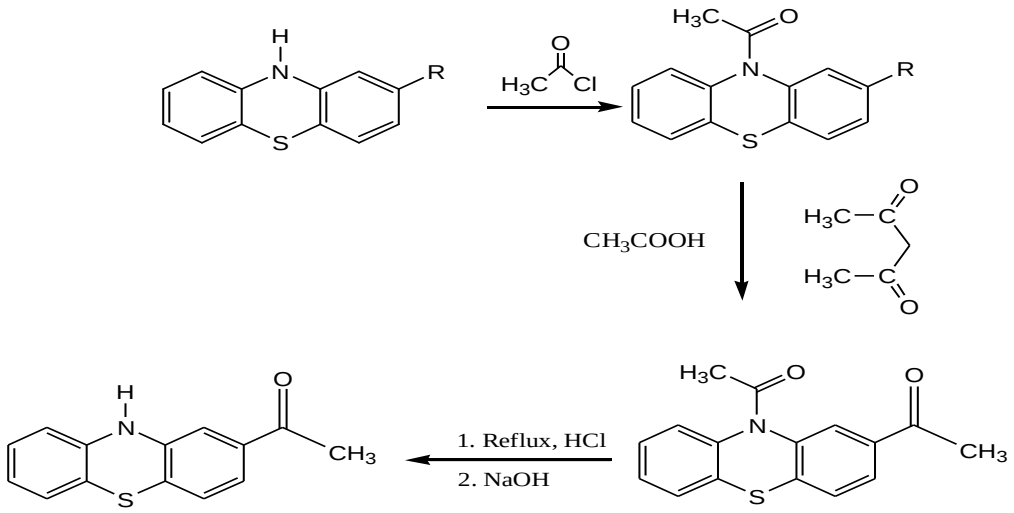
Chemicals used for the synthesis of ligands and the complexes were of AR grade. The completion of the reaction was checked by TLC. The melting points were taken in an open capillary tube and were uncorrected.

Synthesis of Chalcones

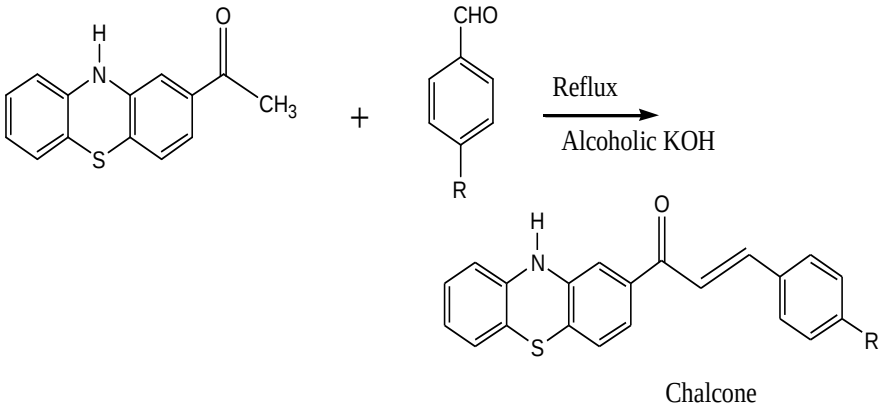
To the solution of 2-acetyl phenothiazine in methanol (50 ml) P-chloro benzaldehyde and P-Methoxy benzaldehyed were added in presence of 40% methanolic KOH (10 ml) and refluxed the reaction mixture for 3 hours on water bath. After completion of the reaction, the reaction mixture was kept at room temperature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

Filter the reaction mixture then acidified the filtrate with dil. HCl. The solid obtained was filtered at sunction pump washed with cold water and recrystallised by using methanol.

Scheme
I.



II.



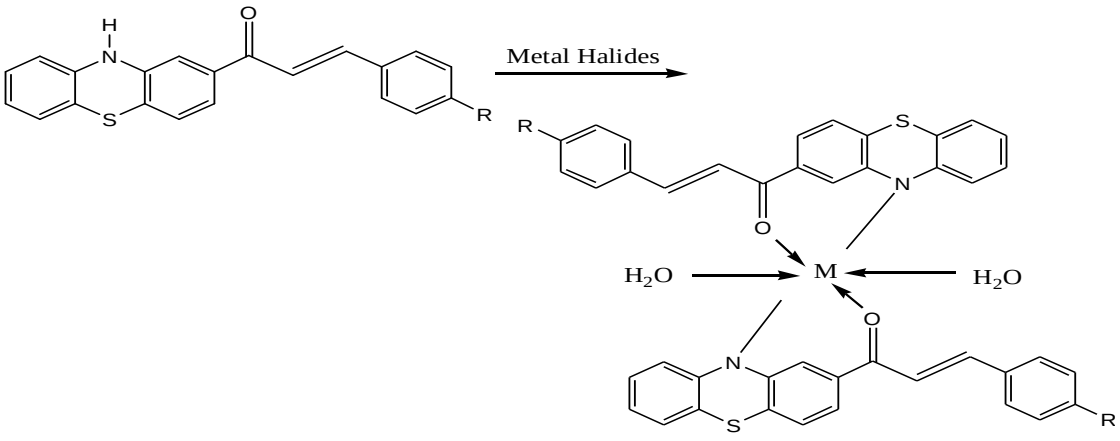
Chalcone

Where
R=- Cl, -OCH_3

Synthesis of metal complexes

The ligands and the metal halides were taken in 2:1 ratio i.e. 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



Where
M= Zn, Co, Ni, Cu, Fe, Mn

RESULT AND DISCUSSION

The complexes are coloured, stable at room temperature, insoluble in water and in 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 3432 and 3328 Cm^{-1} due to -NH stretch. In the IR spectra of complexes this band appears as an intense broad band near 3650-3830 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 1642 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 422-446 Cm^{-1}. The literature survey support such interpretation.

^{1}H-NMR Spectra

^{1}H-NMR Spectra of complexes and ligands shows well resolved signals. Due to complex formation there observed broad peaks in ^{1}H-NMR spectra of complexes. The peak in the ^{1}H-NMR spectra of ligands near at d 8.6 (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 2.72 B.M. for Zn (II) and 4.05 B.M. for Co (II) complexes, 5.28 B.M. for Ni (II), 2.32 B.M. for Cu (II) and 2.12 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 16.14-28.04 SCm^{2} Mol^{-1} for Zn(II) complexes and 12.25- 24.22 SCm^{2} Mol^{-1} for Co(II), Ni(II) complexes and in the range of 20.09-21.11 SCm^{2} Mol^{-1} for Cu(II) and Fe(II) complexes suggesting their non electrolytic nature.

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

Sr. No.	Molecular formula	Molecular weight	colour	M.P.	Molar conductivity	μ eff
1	$C_{21}H_{14}ONSCl$	205	Yellow	89	---	---
2	$C_{22}H_{16}O_2NS$	187	Dark yellow	76	---	---
3	$[C_{21}H_{14}ONSCl]_2$ Zn	440	brown	232	16.14	2.72
4	$[C_{21}H_{14}ONSCl]_2$ Co	437	Pinkish brown	254	12.25	4.05
5	$[C_{21}H_{14}ONSCl]_2$ Ni	438	Brown	286	20.09	5.28
6	$[C_{21}H_{14}ONSCl]_2$ Cu	439	Greenish brown	204	13.25	2.32
7	$[C_{21}H_{14}ONSCl]_2$ Fe	436	Brown	187	14.36	2.12
8	$[C_{21}H_{14}ONSCl]_2$ Mn	435	Dark brown	215	12.89	2.35
9	$[C_{22}H_{16}O_2NS]_2$ Zn	404	Dark yellow	189	18.21	2.81
10	$[C_{22}H_{16}O_2NS]_2$ Co	401	Orange	234	13.46	4.12
11	$[C_{22}H_{16}O_2NS]_2$ Ni	402	Brown	179	21.11	5.02
12	$[C_{22}H_{16}O_2NS]_2$ Cu	403	Light green	267	13.81	2.51
13	$[C_{22}H_{16}O_2NS]_2$ Fe	400	Green	230	14.02	2.01
14	$[C_{22}H_{16}O_2NS]_2$ Mn	399	Brown	192	12.03	2.60

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 mononegative bidentate 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 hence shows the octahedral geometry.

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