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‘रॉयल’ या सहामयि प्रसिध्द झालेली मते मुख्य संपादक संपादक मंडळ व सल्लागार मंडळास मान्य असतीलच असे नाही. या नियतकालिकात प्रसिध्द करण्यात आलेली लेखकांची मते ही त्याची वैयक्तिक मते आहेत. तसेच शोधनिबंधाची जबाबदारी स्वतः लेखकावर राहिल.

हे नियतकालिक मालक मुद्रक प्रकाशक विनय शंकरराव हातोलें यांनी अजिंठा कॉम्प्युटर अँड प्रिंटर्स जयसिंगपूरा विद्यापीठ गेट औरंगाबाद येथे मुद्रित व प्रकाशित केले.

5. Physico-Chemical Studies of Complexes of MN (II) with Cinnamylidene Acetophenone (Olefinic Ligand)

Ms. Suvarna S. Nalage

Department of Chemistry, Shree Shivaji Arts, Comm. and Science College, Rajura,
Dist - Chandrapur (M.S.).

Abstract

Some novel transition metal [Mn(II), Fe(II), Co(II), Ni(II), Cu(II) and Zn (II)] complexes of Cinnamylidene acetophenone have been prepared. To study the structural aspects of these metal chelates various Physico – Chemical methods have been used i.e. elemental analysis, infrared spectra, electronic spectra, magnetic studies, conductivity measurement, Thermal studies etc. The present paper includes synthesis of ligand, preparation of Mn(II) complex and its systematic structural study.

Keywords: Cinnamylidene acetophenone, Mn(II) complex, Magnetic properties, IR study of complex, Elemental analysis

Introduction

Transition metal compounds present a vast array of structures, along with a full diversity of properties and chemistry. This diversity offers rich opportunities for their applications in organic synthesis and in selective catalytic processes. However, the huge variety of coordination chemistry results in an increasing complexity of its study.

There are increasing number of reactions in which metal ion participate, presumably as catalyst. And in which it is supposed that the catalytic action takes place through metal complexes^{1,2}, involving one or all of the reactants.

There are various kinds of ligand which can bind with various metal ions to form complexes.

Commonly α , β - unsaturated aromatic ketone is known as chalcone. Chalcones are very reactive compounds and owe their reactivity to keto – enolic ($O=C-C=C$) type of conjugated double bond system present in the molecule. Some chalcones are found to exist free in nature, i.e. butein, hyssopin etc. Harborne reported 3 naturally occurring chalcones aglycones. Chalcones are also useful in deciding the structure of many naturally occurring pigments, e.g. fistein, hemlock and tannin. They are also used as intermediates for the preparation of substances having therapeutic value⁴. In the present work, transition metal complex is prepared by using

cinnamylidene acetophenone with Mn(II). To study the structural aspects of this metal chelate various physico-chemical methods have been used i.e. infrared spectra, electronic spectra, magnetic studies, conductivity measurement etc.

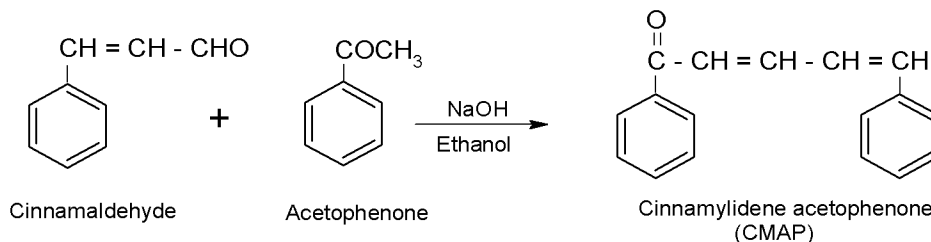
Experimental

Chemicals

The chemicals used were analytical grade. Water and ethyl alcohol were doubly distilled.

Synthesis of Ligand

The ligand used in the present work is not commercially available. However, it was prepared by the method reported earlier⁵ as below. 0.16 mol of acetophenone was taken in a 500 ml beaker. To this 0.16 mol of cinnamaldehyde was added. The reaction mixture was cooled in an ice bath. To this cold solution 60 ml of ethyl alcohol and 100 ml of 0.27 mol of NaOH were added with continues stirring. Temperature of the solution was kept between 15- 30 C. Stirring was continued for about half- one hour. A lot of yellow sediment was separated out. The mixture was then allowed to stand for overnight. It was then filtered, washed several times with water and dried. The crude product was recrystallized from ethyl alcohol. It was then weighed and melting point was noted. The melting point of cinnamylidene acetophenone was found to be 1010C.



Synthesis of Metal Complex: Manganese acetate was dissolved in minimum quantity of ethyl alcohol and 2 to 3 drops of acetic acid was added to get its clear solutions. A hot ethanolic clear solution of ligand was gradually added to the hot ethanolic clear solution of metal salt in the molar proportion of 1:1 with constant stirring. The reaction mixture was then digested on water bath for about 2 hours. A coloured complex was filtered, washed several times with hot water and ethyl alcohol and dried in a desiccator over calcium chloride.

Analysis of the Complex

A) Elemental analysis: Estimation of metal content has been carried out by classical method⁶. Estimation of elements (C,H) has been carried out at RSIC, Punjab University, Chandigarh. Elemental analysis provides information about the percentage of basic elements like carbon, hydrogen, nitrogen and sulphur, which form the skeletal backbone of the ligand and the metal complexes. For this about 0.1 to 0.2 g of complex was weighed accurately in a clean dry

platinum crucible and heated strongly in air for about 5-6 hours to attain the constant reduced weight and metal was weighed in the form of the stable oxide.

B) Infrared Spectra: Infrared spectra of both ligand and complex are recorded in solid, liquid or as Nujol mull emulsions of a sample. In the solid state, the most common material used for making the pellets is KBr for studies in the range $4000 - 400 \text{ cm}^{-1}$. In the present study, infrared spectra of ligand and its complex as KBr pellets were scanned on Shimadzu 8101 A FTIR Spectrophotometer from department of pharmacy, RTMNU, Nagpur for the range of $4000 - 400 \text{ cm}^{-1}$.

C) Electronic Spectra: In the present study, absorption spectra of ligand was recorded in DMSO on a Shimadzu UV 3101 PC spectrophotometer at department of chemistry, RTMNU, Nagpur and diffused reflectance spectra of complex was recorded on Cintra spectrophotometer at department of chemistry, RTMNU, Nagpur

D) Molar conductance measurement: The electrical conductance measurement has been carried out in DMSO using a Toshniwal conductivity bridge and a dip type cell calibrated with KCl solution

E) Magnetic susceptibility measurement: Magnetic susceptibility measurement at room temperature were carried out by a Gouy's method.

Results and Discussion

Elemental analysis: In the present investigation it was observed that Mn (II) forms the coordination complex with CMAP. Molecular weight of the complex could not be determined by conventional methods like osmometry, viscometry etc., due to solubility problems. Elemental analysis (Table 1.1) suggests a 2:1 metal ligand stoichiometry for the coordination complex.

Table 1.1: Analytical and Physical data for the ligand and Mn(II) complex

Analytical and Physical data		C ₁₇ H ₁₄ O (CMAP)	[Mn(II) ₂ (C ₁₇ H ₁₄ O)(CH ₃ COO) ₂ 4H ₂ O] H ₂ O
FW		234	552
Colour		Pale Yellow	Brown
% Analysis found (Cal)	Mn	-	19.18 (19.93)
	C	87.12 (87.17)	44.1 (45.65)
	H	5.78 (5.98)	5.33 (5.97)
Geometry of complex		-	Octahedral
Molar Conductance $\times 10^{-4} \text{ mhos cm}^2 \text{ mole}^{-1}$			4.96

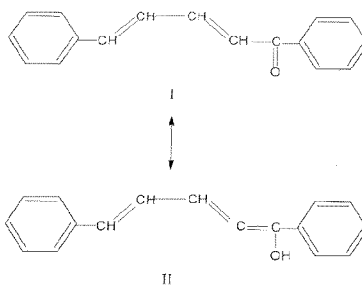
Infrared Spectra of CMAP and its Mn (II) complex: Various significant bands of CMAP and its Mn(II) complex have been assigned with the aid of literature data and these assignments are given in table 1.2

Table 1.2: Infrared Spectral data of CMAP and Mn (II) – CMAP Complex

Bands	CMAP	Mn - CMAP
C=C	1599 st	1437s
(COO ⁻) asym		1560s
(COO ⁻) sym		1404s
$\Delta(\text{COO})$		156
C=O	1655 st	-
(-CH=CH-)trans	997 st	-
$\nu(\text{H}_2\text{O})$		3420b
$\delta(\text{H}_2\text{O})$		860w
Mn – O		617w
Mn - C		441m

m – medium, **w** – Weak, **b**- broad, **s** – Sharp, **st** - Strong

It can be concluded from the table that the C-C bonds are involved in the metal- ligand bonding in Mn(II) complex. IR spectrum of ligand (CMAP) displays broad medium intensity band at 3450 cm assignable to ν (O-H) vibration along with a strong, sharp band at 1655 cm assignable to C=O vibration. Therefore, it can be concluded that the ligand exists in the following tautomeric forms.



Complete disappearance of C=O stretching band and retention of a shoulder around 2850 cm⁻¹, a characteristic frequency of enolic -OH stretch indicates the existence of enolic form II of ligand in metal chelate. The spectrum displays a sharp and strong intensity band at 1599 cm⁻¹ assignable to C = C stretching vibration. The shifting of this band to lower frequency side (150 - 160 cm⁻¹) in the complex suggests strong coordination through C = C bond. In the complex, $\nu(\text{C}=\text{C})$ appears as medium intensity band at 1437cm⁻¹. The coordination through C=C bond is further supported by the disappearance of a strong band at 997 cm corresponding to out of plane C- H bending in conjugated trans- trans system.

Nakamoto⁷ reported that the carboxylate give a strong asymmetric stretching band near 1650- 1550 cm^{-1} and a weaker stretching band near 1400 cm^{-1} . The difference in these frequencies (Δ) was correlated to the nature of metal - ligand bonding of carboxylate group. It was concluded that as the covalent character of the Metal-O bond increases; the carbonyl group becomes more asymmetrical which results in the greater difference in the stretching frequencies. As the strength of O- Metal bond increases, the symmetry of the COO- group increases and the difference (Δ) decreases.

In the present investigation a band observed at 1560 cm^{-1} can be assigned to asymmetric stretching of COO- group and a band at 1404 cm^{-1} can be assigned to symmetric stretching vibration of COO- group. Further comparatively small separation value, Δ (COO), of 156 cm^{-1} suggests the bidentate nature of the acetate group⁸.

The IR spectrum of this metal complex displays a weak band in 441 cm^{-1} region which can be assignable to Mn-C bond⁹. Similarly, weak to medium intensity bands in 617 cm^{-1} can be assigned to Mn-O bond.

In general water of hydration absorbs at 3300-3450 cm^{-1} ¹⁰ A broad band observed in the metal complex at 3420 cm^{-1} can be assigned as coordinated water in this complex. A weak band at 860 cm^{-1} can be assigned to rocking mode of coordinated water. No separate band for ν (O- H) observed as stretching modes of water of hydration overlaps it. IR spectra (wavenumbers are in cm^{-1}) of the ligand and Mn(II) complex are shown in Fig 1.1 and 1.2 resp.

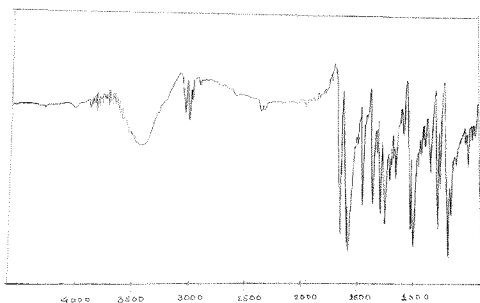


Fig 1.1: IR Spectra of CMAP

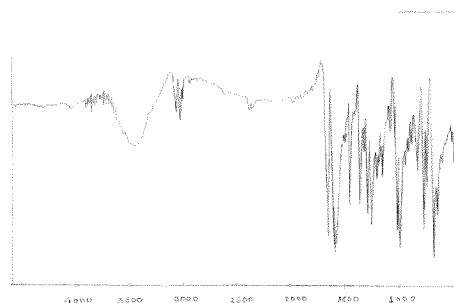


Fig 1.2: IR Spectra of Mn - CMAP

Magnetic Moment and Electronic Spectra: The electronic spectra of the complex was recorded in DMSO solution. The magnetic and electronic spectral data are given in table 1.3

Magnetic Property: A magnetic susceptibility of Mn (II) complex was measured at room temperature using Guoy method. From the knowledge of magnetic susceptibility, magnetic moment was calculated. The magnetic moment value of Mn (II) chelate was found to be closed to 4.89 B.M. The sub abnormal value of magnetic moment than that of expected spin only value might be attributed to direct Mn – Mn interaction indicating high spin octahedral geometry¹¹

Electronic Properties: Diffused reflectance spectra of Mn (II) complex, under present investigation was studied in the range of 250- 750 nm. This spectrum is incorporated in Fig. 1.4. Observed transitions and their probable assignments are shown in table 1.3. Electronic spectra of Mn (II) complex shows three weak bands at 16.94, 18.18 and 21.73 kK which may be due to ${}^6A_1(S) \rightarrow {}^4T_1(G)$, ${}^6A_1(S) \rightarrow {}^4T_2(G)$ and ${}^6A_1(S) \rightarrow {}^4A_1(G)$ transitions respectively. The extremely low intensities of these bands are indicative of octahedral geometry as a consequence of their doubly forbidden nature¹². This is in agreement with the findings of Mehta et al¹³.

Table 1.3: Magnetic moment and Assignment of Solid State Reflectance Spectra of Mn CMAP complex

μ_{eff} (B.M.)	Absorption Bands		Assignments
	nm	kK	
4.89	460	21.73	${}^6A_1(S) \rightarrow {}^4A_1(G)$
	550	18.18	${}^6A_1(S) \rightarrow {}^4T_2(G)$
	590	16.94	${}^6A_1(S) \rightarrow {}^4T_1(G)$

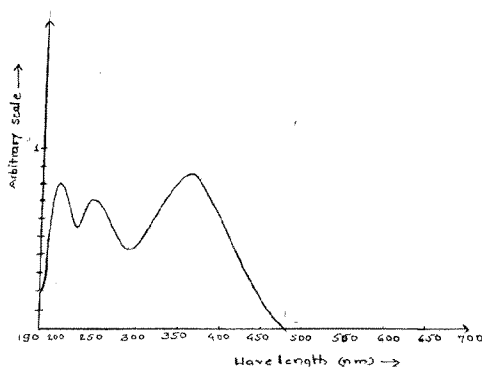


Fig 1.3: Absorption spectra of CMAP

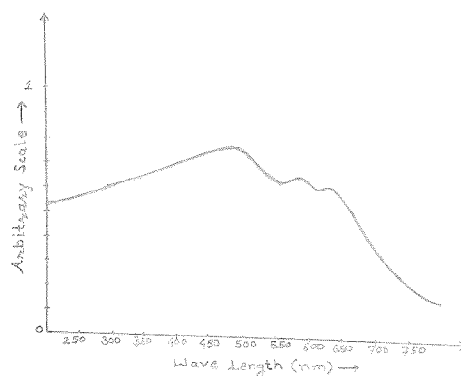
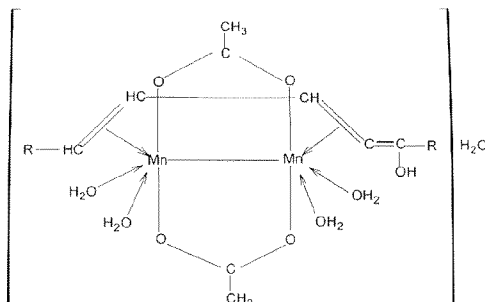


Fig 1.4: Solid State Reflectance Spectra of Mn – CMAP complex

Conclusion

Elemental analysis of Mn (II) shows the composition 2:1 (M: L). Its non electrolytic nature has been indicated by the low values of molar conductance in DMSO. The magnetic moment value of this complex suggested to have octahedral geometry. Octahedral geometry is also supported by electronic spectra of the complex. The changes in stretching frequencies in the IR spectra of the ligand and its complex indicates that C=C bonds of ligand are involved in coordination. As well as acetate groups are also found to involved in coordination with the metal ion. Mn - oxygen and Mn- Carbon bond formation is further ascertained by the appearance of new

bands in the low frequency region. Thus, on the basis of elemental analysis, conductance measurements, magnetic, spectral and IR data, the complex is suggested to have octahedral stereochemistry. Higher percentage of metal in the complex indicates its dimeric nature.



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