



SYNTHESIS AND CHARACTERIZATION OF IRON (II) AND NICKEL (II) SCHIFF BASE COMPLEXES

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ABSTRACT

The complexes of iron (II) and nickel (II) with schiff base derived from benzoin and 2-amino benzoic acid have been prepared. Solubility, melting point, decomposition temperature, conductance measurement, infrared (IR) and UV – Visible spectrophotometric studies were used in characterizing the compounds. The melting point of the schiff base determined is 120°C. The decomposition temperatures of iron (II) and nickel (II) complexes are 152°C and 155°C, while the molar conductance values are 11.2 and 10.7 ohm⁻¹cm²mol⁻¹, respectively. The UV-Visible spectrophotometric analysis revealed 1:1 (metal-ligand) stoichiometry for the two complexes.

Keywords: Schiff base, conductivity, azomethine, benzoin, spectrophotometry.

INTRODUCTION

When an aldehyde or a ketone is condensed with a primary amine, a Schiff base is produced, which is a compound containing azomethine group, $R-C=N-$ (Holm *et al.*, 1966; Hobday and Smith, 1972). It has been known that a variety of metal ions on interaction with schiff bases yield chelates, for example; Holm *et al.* (1966) reported the synthesis and magnetic studies on schiff base complexes of copper (II). Recently, Xishi *et al.* (2003) reported the synthesis and characterization of Mn (II), Co (II) and Cu (II) complexes with a novel schiff base ligand derived from 2, 2' - bis(p-methoxyphenylamine) and salicylic aldehyde. Transition metal schiff base complexes have been found to play a vital role in medicine, biological systems and industries. The field of medicine has witnessed an increase in the number of complexes with therapeutic value, for example, cobalt (III) schiff base complexes are potential antiviral agents, cis-dichlorodiamineplatinum (II) is an anti-cancer agent and copper (II) schiff base complex is an anti-tubercular agent (Lippard, 1994; Bleomink and Reedi, 1996). The use of atom transfer radical cyclisation mediated by copper (II) schiff base complexes to furnish nitrogen heterocycles most of which are biologically active molecules and also the use of copper schiff base catalyst in carbon based radical cyclisation reactions were recently investigated (Clerk and Jones, 1989; Clerk *et al.*, 1998; Clerk *et al.*, 1999). The complex compound formed by schiff base derived from benzoin and 2-amino benzoic acid with cobalt (II) salt has been reported (Mahaptra *et al.*, 1977). This paper reports the preparation and characterization of two new complex compounds formed by iron (II) and nickel (II) metal ions with the schiff base.

MATERIALS AND METHODS

The solvents and chemicals used in this work were of Analar grade. All the glass wares used were washed thoroughly with distilled water and dried in an oven. Weighing was carried out on electric metler balance, model AB 54. Melting point, decomposition temperature and coordinated water were determined on Gallenkamp melting point apparatus. Infrared spectral analyses were recorded using Fourier Transformed IR, Genesis series model in Nujol within 400-4000 cm⁻¹. Electrical conductivity measurements were carried out using conductivity meter model Jenway 4010, while UV- visible spectral measurements were done on a Pye Unicam UV-visible spectrophotometer.

Preparation of Benzoin

Into a 500cm³ round bottom flask was added 65cm³ of rectified spirit, 47.5cm³ of benzaldehyde and a solution of 5g of sodium cyanide in 50cm³ of water. A condenser was then attached and the mixture refluxed on a steam bath for half an hour. The flask and its contents were cooled in an ice-bath to precipitate pale yellow crystals, which were suction filtered, washed with cold water, dried and then recrystallized from 40cm³ of hot rectified spirit (Mahaptra *et al.*, 1977).

Preparation of the Schiff Base Ligand

Into an ethanolic solution of the prepared benzoin (0.01mol) and 2-amino benzoic acid (0.01mol) in a 500cm³ round bottom flask, 3.0g of anhydrous sodium acetate was added. The mixture was refluxed for an hour on a steam bath. To precipitate the grayish white product, the content was poured into an ice-cold water which was separated and recrystallized from rectified spirit, suction filtered, washed with water, and dried in a vacuum dessicator (Mahaptra *et al.*, 1977).

Preparation of the Complexes

Ethanol solutions of metal chloride (0.025mol) and schiff base (0.05mol) were mixed and the resulting mixture followed by dropwise addition of ammonia until the metal chelate separated, which were then suction filtered, washed with ethanol and ether before dried in vacuum dessicator. The crystals were recrystallized from rectified sprit and dried (Mahaptra *et al.*, 1977).

RESULTS AND DISCUSSION

The benzoin and Schiff base have been prepared as reported (Mahaptra, *et al.*, 1977; Vogel, 1978), they are pale yellow and grayish white, respectively. The iron (II) and nickel (II) Schiff base complexes have been prepared using the adopted synthetic procedure reported by Mahaptra, *et al.* (1977), the compounds are brown and blue, respectively. The melting point temperatures of the benzoin and the schiff base are 137°C and 120°C, respectively, which are in agreement with the literature values (Vogel, 1978; Mahaptra, *et al.*, 1977). The decomposition temperatures of iron (II) and nickel (II) Schiff base complexes are 152°C and 155°C, respectively, which present fairly stable complex compounds. The percentage by weight (Table 1) of iron and nickel in the complexes are 12.60 and 12.90, respectively, and these revealed 1:1 metal to ligand ratio. The coordinated water analysis (Table 2) of the complexes indicated three water molecules per molecular formula, which is in complete agreement with the report on cobalt (II) schiff base complex (Mahaptra *et al.*, 1977). The complexes are insoluble in water and common organic solvents, but are readily soluble in

dimethylsulphoxide (DMSO). The molar conductance values (Table 3) of iron (II) and nickel (II) schiff base complexes in $1 \times 10^{-3}M$ DMSO solution were 11.2 and 10.7 $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$, respectively, suggesting their non-ionic nature (Geary, 1971). The IR spectrum (Table 4) of the free schiff base shows bands of medium intensity at 1210, 1590, and 1670 cm^{-1} which are for $\nu(\text{C-O})$, $\nu(\text{C=N})$ and $\nu(\text{C=O})$, respectively (Silverstein and Bassler 1967; Freedman, 1961; Nakamoto, 1963). The same three bands were also noticed in the two complexes, which confirmed coordination of the Schiff base to the respective metal ions. The bands in the regions 511-558 cm^{-1} and 470-478 cm^{-1} are assigned to $\nu(\text{M-N})$ and $\nu(\text{M-O})$ stretching vibrations, respectively (Mahaptra *et al.*, 1977), confirming coordination of the schiff base to the metal ions. The broad bands in the region 3350-3355 cm^{-1} observed in the two complexes have been established to be hydrated due to the broad bands in the region 3350—3355 cm^{-1} (Patel and Agwara, 1990). The coordinated water analysis of the complexes indicated three water molecules per molecular formula, which is in complete agreement with the report on cobalt (II) schiff base complex (Mahaptra *et al.*, 1977).

The spectrophotometric analysis (Table 4) revealed 1:1 (metal-ligand) stoichiometry in the two complex compounds, which is common feature in the first row transition metals (Holm and O’Connon, 1971; Mahaptra *et al.*, 1977). Therefore, from the analyses carried out in this work, the proposed general molecular structure of iron (II) and nickel (II) schiff base complexes is as presented below;

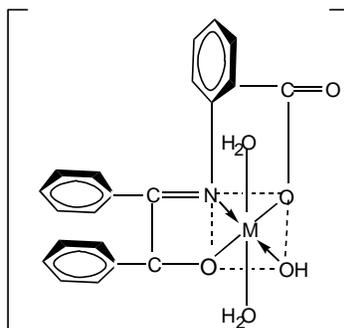


Figure 1: The proposed general Structure of iron (II) and nickel (II) Schiff base complexes.

Table 1: Percentage Composition by Weight of Metal in the Complex

S/No.	Compound	%Metal Calculated	%Metal Observed
1	[FeL(H ₂ O) ₃]	12.49	12.60
2	[NiL(H ₂ O) ₃]	13.23	12.90

Table 2: Percentage Composition by Weight of Water in the Complexes

S/No.	Compound	%Water Calculated	% Water Observed
1	[FeL(H ₂ O) ₃]	12.25	11.19
2	[NiL(H ₂ O) ₃]	12.17	12.12

Table 3: Conductivity Measurement of the Complexes in DMSO (1 x 10⁻³M)

S/No.	Compound	Specific Conductance (k)	Molar Conductance (Ohm ⁻¹ cm ² mol ⁻¹)
1	[FeL(H ₂ O) ₃]	9.8x10 ⁻⁶	11.20
2	[NiL(H ₂ O) ₃]	9.3x10 ⁻⁶	10.70

Table 4: Infrared Spectral Data of the Schiff Base (L) and the Complexes

S/No.	Compound	$\nu(\text{C}=\text{N})$ (cm ⁻¹)	$\nu(\text{C}-\text{O})$ (cm ⁻¹)	$\nu(\text{O}-\text{H})$ (cm ⁻¹)	$\nu(\text{M}-\text{N})$ (cm ⁻¹)	$\nu(\text{M}-\text{O})$ (cm ⁻¹)
1	Schiff Base	1590	1210	-	-	-
2	[FeL(H ₂ O) ₃]	1595	1208	3350	511	470
3	[NiL(H ₂ O) ₃]	1596	1279	3355	558	467

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