Research Article

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Synthesis and characterization of Ni(II) complex with Schiff base derived from benzophenone and 2-aminophenol

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ABSTRACT

A Schiff base ligand was obtained when benzophenone and 2-aminophenol reacted with each other under normal circumstances. Interaction of the Schiff base ligand with $NiCl_2.6H_2O$ gave rise to hydrated, ash coloured complex of high thermostability. Characterization using some physicochemical techniques such as solubility, melting point, molar conductivity and FT-IR were carried out. The results revealed the solubility of the synthesized compounds in different solvents. The water of crystallization was calculated to constitute 2.8 %, consistent with 1 water molecule of crystallization. On subjection to conductivity measurement, an observed molar conductance of 2.55 $ohm^{-1}cm^2mol^{-1}$ proved that the complex is a non-electrolyte. The FT-IR spectrum of the ligand exhibits a band at 1684.88 cm^{-1} attributed to *ν*C=N which shifted to a higher frequency in the spectrum of the complex. This shifting indicates that the ligand coordinates to the Ni(II) ion *via* the azomethine nitrogen. On the basis of spectral analysis, the complex could be formulated as $[NiL_2].H_2O$. It could be inferred that the ligand behaved as monoanionic bidentate ligand with the azomethine nitrogen (N) and phenol oxygen (O) as the coordination sites assuming a four-coordinate square planar geometry.

1. Introduction

Schiff bases are organic compounds, considered to be a subclass of imines, which may be secondary aldimines or ketimines depending on the nature of the parent carbonyl compounds, which are synthesized by nucleophilic addition of aliphatic or aromatic amines with carbonyl compounds forming intermediate hemiaminals followed by elimination of water, the reaction often being catalyzed in acid medium. They have the general formula R-CH=NR' where $R' \neq H$. The presence of the azomethine function in the Schiff base compounds renders them as potential candidates for forming a wide range of complex compounds with both transition and non-transition metal ions [1,2]. In recent times, plenty works have been reported complexes of Schiff base containing 2-aminophenol with mostly aldehyde [3-12]. Subbaraj et al. [13] reported Schiff bases derived from substituted benzophenone and aniline with metal(II) ions (Mn, Co, Ni Cu and Zn).

Paucity of information in the literature on the synthesis of Schiff base derivable from benzophenone and 2-aminophenol with its Ni(II) complex as well as the chelation behaviour of the Schiff base towards metal(II) ion has prompted the research work.

2. Results and Discussion

The physical and analytical data of the compounds are presented in Table 1. The benzophenone and 2-aminophenol yielded the ligand with percentage yield of 24.38 % and a sharp melting point of 220 ℃ indicating its purity and relative thermal stability. The interaction of the ligand with $NiCl_2.6H_2O$ produced an ash coloured complex with percentage yield of 39.74 %. The formation of this coloured complex might be due to d-d transition or nature of the ligand [14]. The molar conductivity of the Ni(II) complex in DMF was calculated as 2.55 $Ohm^{-1}cm^2mol^{-1}$. This low value indicates non-electrolyte hence no anions are present [15].

The solubility of the Schiff base and its Ni(II) complex was studied in different polar and non-polar solvents. The result shows that the ligand was soluble in hot and cold acetone, insoluble in hot and cold chloroform and slightly soluble in hot diethyl ether. It also showed that the

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complex was insoluble in hot and cold distilled water, acetone and chloroform. It was slightly soluble in hot and cold diethyl ether but soluble in hot ethanol. This could be attributed to the interaction between the hydrogen ion in these compounds under investigation and the oxygen atom in the solvents which results in the formation of hydrogen bond [16].

Table 1. Physiochemical Properties of Schiff base and Ni(II) Complex

Compound	F. weight ($gmol^{-1}$)	Colour	% Yield	M.P/D.T (^0C)	Molar Conductivity ($\Omega^{-1}cm^2mol^{-1}$)
Schiff Base	273.35	Light Brown	24.38	220	-
$[NiL_2].H_2O$	623.38	Ash	39.74	340	2.55

The percentages nickel and water of crystallization is presented in Table 2. It portrayed that the percentage of water of crystallization found is 3 % while the percentage of water of crystallization calculated is 2.8 %. It also showed that the number of water molecules is 1 hence the complex is hydrated. The experimental and theoretical value of the nickel are in close agreement. The data further showed 1:2 Metal-Ligand ratio.

Table 2. Metal and water content in the complex

Complex	% Found (Calculated)	
	Ni	H_2O
$[NiL_2].H_2O$	9.41(8.96)	3.00(2.80)

The FT-IR spectra are shown in figures 1 and 2 and the selected bands are presented as in Table 3. The FT-IR spectra of starting materials- benzophenone and 2-aminophenol showed bands at 1681.38 cm^{-1} and 3375.73 cm^{-1}, respectively, which are characteristics of $_{\nu}C=O$ and $-NH_2$ respectively [17]. The disappearance of these peaks and manifestation of new strong band at 1624.88 cm^{-1} attributable to $_{\nu}C=N$. Stretching vibration is an attestation of conversion of the carbonyl (C=O) and amine (NH_2) groups of the starting materials into Schiff base [18]. In order to identify the ligation behavior of the Schiff base ligand towards the metal ion, the FT-IR spectrum of the Schiff base is compared with that of the complex. On complexation, the $_{\nu}C=N$ peak which appears at 1624.88 cm^{-1} in the free ligand has shifted to higher frequency of 1626.39 cm^{-1} and the band due to $_{\nu}OH$ in the ligand (3803.03 cm^{-1}) disappeared in the spectrum of the complex with the shifting of phenolic $_{\nu}C-O$ band from 1295.34 cm^{-1} to 1311.85 cm^{-1}. These shifting indicates that the Schiff base ligand coordinates to the Ni(II) ion via the azomethine nitrogen and phenolic oxygen atoms [13]. It is further confirmed by the formation of two new peaks at 664.88 cm^{-1} and 767.83 cm^{-1} corresponding to $_{\nu}Ni-O$ and $_{\nu}Ni-N$ vibrations respectively [19]. The broad band at 3275.70 cm^{-1} is assigned to the existence of water molecules [20]. It could be inferred that the ligand behaved as monoanionic bidentate ligand with the azomethine nitrogen (N) and phenol oxygen (O) as the coordination sites, and that the Ni(II) ion complex assumed a four coordinates square planar geometry [21].

Table 3. Relevant IR Bands (cm^{-1}) of the Schiff Base and Ni(II) Complex

Compound	IR Bands (cm^{-1})							
	νNH_2	νOH	νH_2O	$\nu C=O$	$\nu C=N$	$\nu M-O$	$\nu M-N$	$\nu C-O$
Benzophenone	-	-	-	1681.3	-	-	-	-
2-aminophenol	3375.7	3302.7	-	-	-	-	-	-
Schiff base	-	3803.0	-	-	1624.8	-	-	1295.3
$[NiL_2].H_2O$	-	-	3275.7	-	1626.3	664.8	767.8	1311.8

L=Schiff base = $C_{19}H_{15}NO$

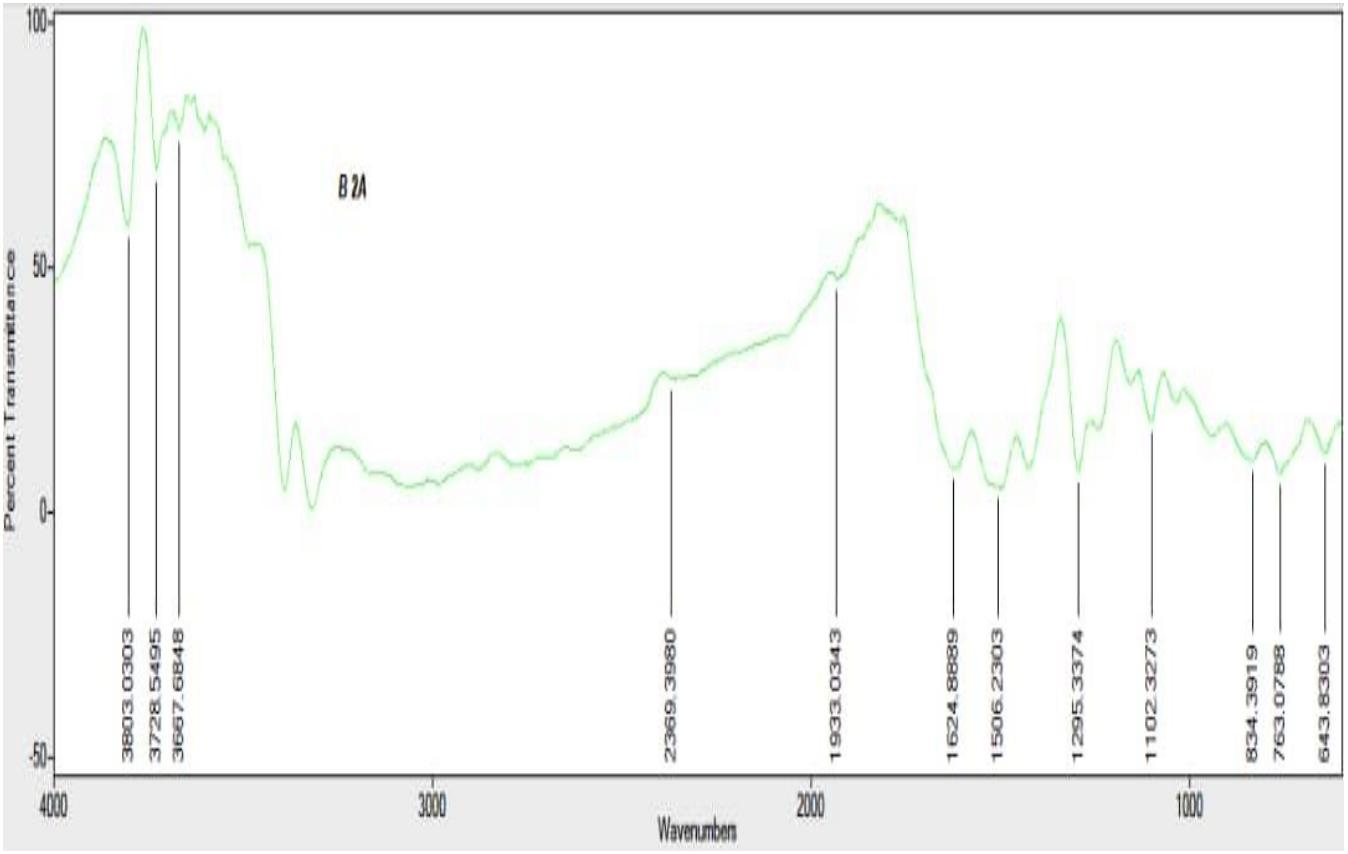


Fig. 1. FT-IR Spectrum of the Schiff base Ligand

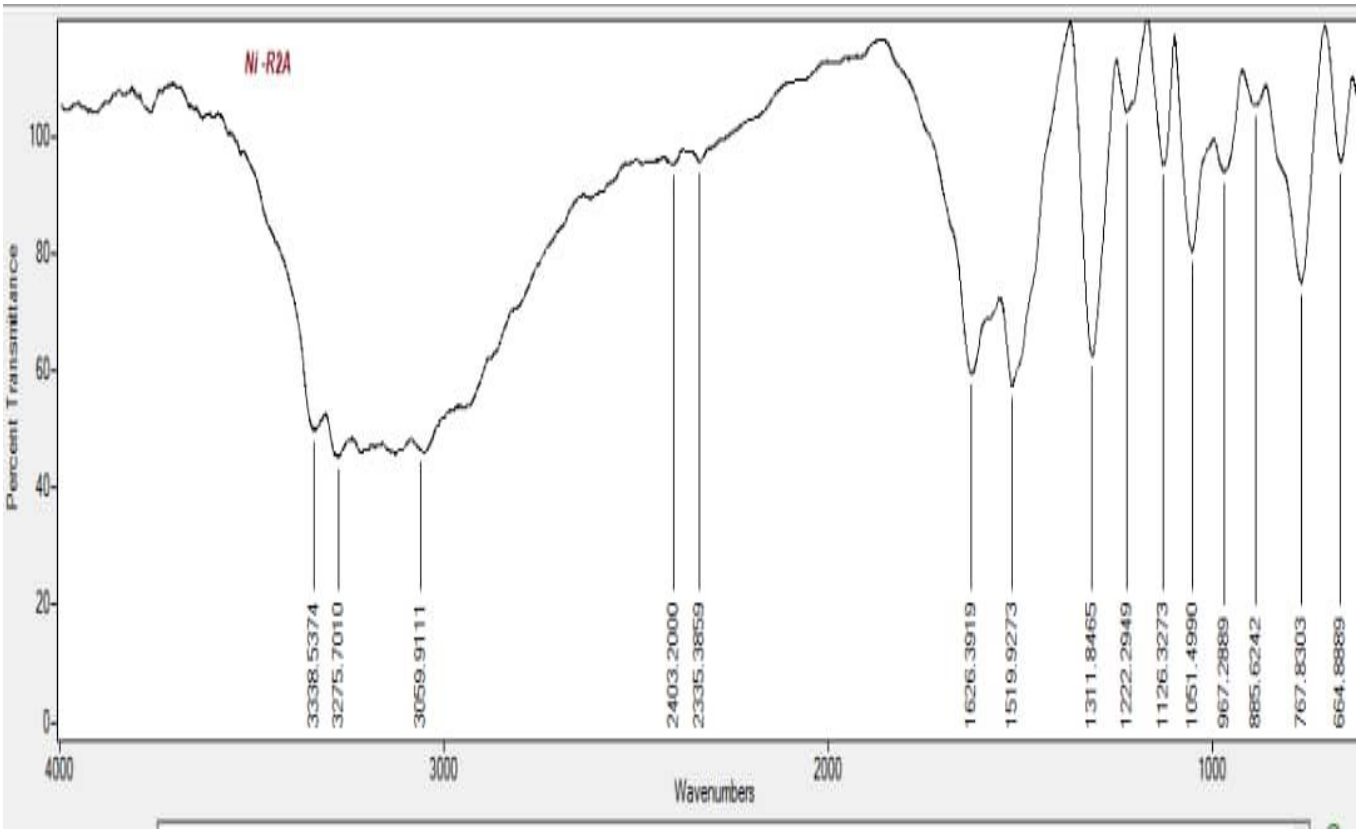


Fig. 2. FT-IR Spectra of Ni(II) Complex

Table 4. Determination of ratio of Ni(II), Ligand and Water of Crystallization in the complex

Species	**Ni**	**Ligand (L)**	**H_2O**
Percentage composition by mass	9.41 %	87.59 %	3 %
Divide by smallest quotient	$\frac{9.41}{58.69}$	$\frac{87.59}{273.25}$	$\frac{3}{18}$
Mole Ratio	0.1603	0.3206	0.1666
	$\frac{0.1603}{0.1603}$	$\frac{0.3206}{0.1603}$	$\frac{0.1666}{0.1603}$
Ratio	1	2	1

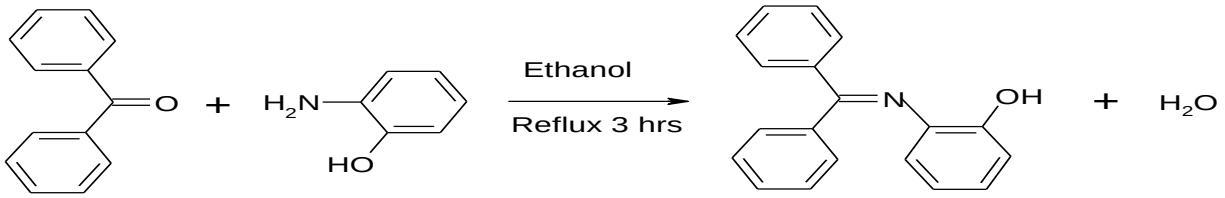
3. Experimental

3.1. Physical measurements

Chemicals of analytical grade were used as supplied without further purification, viz benzophenone, 2-aminophenol (BDH), ethanol, DMF**,** distilled water, dichloromethane, acetone, tetrachloromethane, diethyl ether, and benzene. Melting points were measured on an Electrothermal 9100 apparatus. The IR spectrum for each compound was obtained using the BUCK Scientific Fourier transform IR model M 530 spectrophotometer. Conductance was recorded using LIDA instrument model DDS-307 conductivity meter at 32 ^oC [25]. All solvents used were dried and distilled according to standard procedures.

3.2. Synthesis of Benzophenone-2-Aminophenol Schiff Base

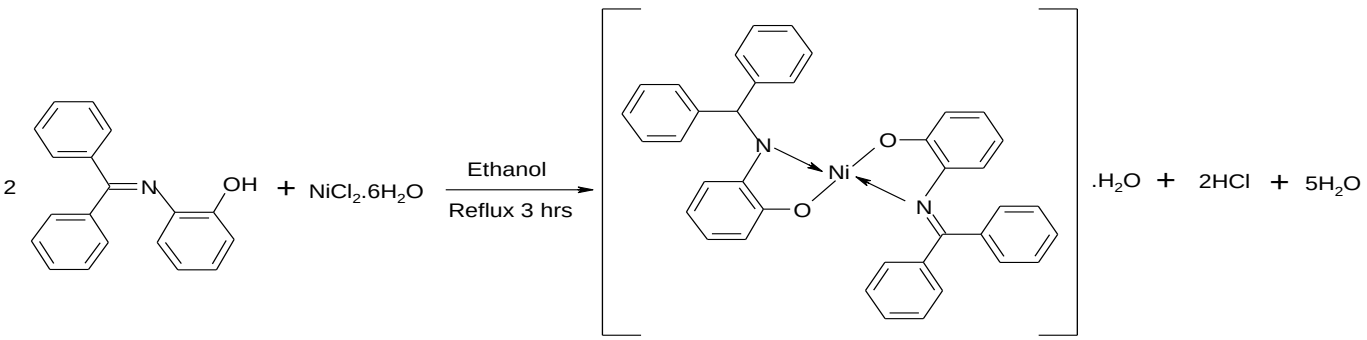
Benzophenone (10.933 g, 60 mmol), diluted with 40 ml ethanol was added in portions to 2-aminophenol (6.548 g, 60 mmol) dissolved in 40 ml ethanol. The mixture was refluxed for 3 hours and a light brown colour solution was obtained [3]. The reaction is depicted in scheme 1.



Scheme 1. synthesis pathway of the Schiff base

3.3. Synthesis of Ni(II) Complex

Nickel(II) chloride hexahydrate (5.594 g, 2.5 mmol) dissolved in 40 ml ethanol was added to the prepared solution of the Schiff base ligand (1.367 g, 5 mmol) also dissolved in 40 ml ethanol. The reaction mixture was refluxed for 3 hours. It was filtered and the filtrate on cooling to room temperature yielded the complex as an ash coloured powdery residue [3]. The reaction is schematically depicted (scheme 2).



Scheme 2. Synthesis of Ni(II) Complex

3.4. Determination of water of crystallization

Exactly 0.2 g of the complex was weighed into a watch glass of known weight and placed in a thermostat oven at a temperature of 110 ^{o}C, the complex was left in the oven for 2 hours. It was weighed and placed again in the oven until constant weight was obtained [22]. The % was calculated using equation 1.

$$\% \text{ water of crystallization} = \frac{W3 - W2}{W1} \times 100 \ \% \qquad (1)$$

Where W_1 = Original weight of complex (g), W_2 = Watch glass + Original weight of complex before heating in oven (g), W_3 = Watch glass + Complex after heating to constant weight (g)

3.5 Determination of empirical formula of the Ni(II) complex

The composition of the complex was determined from the known percentages of the nickel ion and water content in the complex. the percentage of the ligand was obtained by subtracting the sum of the percentages of metal and water contents from 100 %. The empirical formula was calculated using the percentage composition of the species involved by obtaining the moles and corresponding mole ratios [23]. The result is presented in Table 4. The Ni(II) ion was precipitated as a complex of dimethylglyoxime after acid digestion of the Ni(II) Schiff base complex [24].

4. Conclusion

Ni(II) complex have been synthesized from Schiff base derived from the condensation reaction of benzophenone and 2-aminophenol. The Schiff base and the corresponding Ni(II) complex were characterized. The ligand and its metal complex were soluble in some organic solvent used. The molar conductivity value revealed that the complex is non-electrolytic in nature. It was established from combined results of the chemical and physical analysis as well as FT-IR spectra that the ligand employed in this work coordinated with Ni(II) through the azomethine nitrogen and phenolic oxygen after deprotonation and proposed to have a square planar geometry formulated as $[Ni(L)_2].H_2O$.

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