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Synthesis and Spectroscopic Characterization of Mn(II) and Fe(II) Complexes With A Schiff Base Derived from4-(N, N-Dimethylamino) Benzaldehyde and 2-Aminophenol

Abubakar Abdullahi Ahmed[1]* and Yusuf Yunusa[2]

[1]Government Unity Secondary School, Basirka, Jigawa State, Nigeria
[2]Federal College of Animal Health and Production Technology, National Veterinary Research Institute, Vom, Plateau State, Nigeria

Abstract : The Schiff base ligand (DBAP) was synthesized by condensation reaction of 2-aminophenol and 4-(N,N-dimethylamino)benzaldehydein 1:1 molar ratio in an ethanolic medium. The metal(II) complexes were formed by refluxing the chloride salts of the metals with the Schiff base ligand. TheSchiff base and its complexes were characterized by melting point/decomposition temperature, solubility test, elemental analysis, molar conductance measurement, infrared spectral analysis and magnetic susceptibility measurement. The ligand and the complexes were coloured, non-hygroscopic and air stable. The elemental analysis data of the complexes showed the formation of 1:2 metal - ligand ratio. The conductivity measurement data revealed that the complexes are non-electrolytes. The infrared data indicated the bidentate nature of the Schiff base ligand coordinated with the metal ions via the nitrogen atom of the azomethine(C=N) and oxygen atom of the hydroxyl group after deprotonation. The magnetic moment values of the complexes suggested a paramagnetic phenomenon around a four- coordinate tetrahedral geometry.
Keywords : Schiff base ligand, infrared, magnetic moment.

Introduction

Schiff bases have been known since 1864 when Hugo Schiff reported the condensation of amines with carbonyl compounds. The common structural feature of these compounds is the azomethine group with a general formula RHC=N-R^1, where R and R^1 are alkyl, aryl, cyclo alkyls or heterocyclic groups which may be variously substituted[1].

Ni(II) chelete of Schiff base derived from4-dimethylaminobenzaldehyde and cysteine has been synthesized and characterized.has been synthesized and characterized[2].hasbeen synthesized and characterized. Cu(II), Zn(II) and Cd(II) metal complexes of Schiff base derived from 2-aminobenzoic acid and4-(N,N-dimethylamino)benzaldehyde were synthesized. The complexes were investigated by several physicochemical techniques such as elemental analysis, IR and electronic spectra, molar conductance and magnetic moment measurements[3].

Due to paucity of information, this work aims at synthesizing and characterizing Mn(II) and Fe(II) Schiff base complexes derived from 2-aminophenol and 4-(N,N-dimethylamino)benzaldehyde .

Experimental

Materials and Methods

All glass wares used were properly washed with detergent, rinsed with distilled water and dried in an oven. All chemicals used in this work were of Analar grade and used as supplied without further purification. All weighing were carried out on college B154 Metler Toledo electric balance. Melting point and decomposition temperatures were determined on Stuart SMP 10 melting point apparatus. IR spectra measurements were recorded using FTIR Nicolel IS10 Thermoscientific, in the region 4000-400$cm^{-1.}$ Electrical conductivity measurements were carried out using Siemens WPA CM35 Conductivity meter. Magnetic susceptibility measurements were carried out using Sherwood MK1Magnetic susceptibility balance, and Pascal's diamagnetic correctionconstants were applied. The metal contents were determined using Atomic Absorption spectrophotometer 210 VGP. The elemental analysis of CHN was carried out at OEA labs.,Callington, United Kingdom using a CE instruments (thermo) EA1110 Elemental Analyser using Xperience software.

Preparation of the Schiff base ligand (DBAP)

The Schiff base ligand was prepared by adding 75cm^3ethanolic solution of 2-aminophenol (5.46g, 0.05mol) to the same volume of ethanolic solution of 4-(N,N-dimethylamino)benzaldehyde (6.85g, 0.05mol). The mixture was refluxed with stirring for 3 hours. The resulting solution was evaporated to half its volume and the precipitated product was separated, washed twice with 15cm^3ethanol and dried over anhydrous $CaCl_2$ in a desiccator[3].

Synthesis of the metal(II) complexes

The complexes were synthesized by adding 0.015mol (3.6g) of the Schiff base ligand (DBAP) dissolved in 75cm^3 hot ethanol to 75cm^3ethanolic solution of 0.0075 mol of the metal(II) chlorides and were separately refluxed with stirring for 1 hour. On cooling to room temperature, the coloured complexes precipitated out, were separated, washed with 15cm^3 ethanol and dried over anhydrous $CaCl_2$ in a desiccator[3].

Results

Table 1: Physical Properties of the Schiff base and its Metal(II) Complexes

Compound	M. wt (g/mol)	Colour	% yield	M.P. (^oC)	D. Temp. (^oC)	Molar Conductivity ($ohm^{-1}cm^2mol^{-1}$)	μ_{eff}(B. M)
DBAP	240.15	Cadmium Orange	64.72	119	-	-	-
$[Mn(DBAP)_2].11H_2O$	731.24	Brown	58.17	-	136	12.64	7.06
$[Fe(DBAP)_2].14H_2O$	786.15	Bistre	58.60	-	148	18.53	5.97

Where DBAP is $C_{15}H_{15}N_2O$, M.P= Melting point, D. Temp.= Decomposition temperature, M. wt= molecular weight

Table 2: Solubility of the Schiff base and its Metal(II) Complexes

Compounds	Solvents								
	Acetone	CCl_4	Chloroform	DMF	DMSO	Ethanol	Methanol	Nitro benzene	water
DBAP	S	SS	S	S	S	S	S	S	IS
$[Mn(DBAP)_2].11H_2O$	SS	SS	SS	S	S	SS	S	SS	IS
$[Fe(DBAP)_2].14H_2O$	SS	IS	SS	S	S	SS	S	SS	IS

KEY: IS=Insoluble, S=Soluble, SS= Slightly soluble

Table 3: Microanalysis Data of the Schiff base and its Metal(II) Complexes.

Compound	M. wt. (g/mol)	% Found (Calculated)			
		C	H	N	M
DBAP	240.15	74.68 (74.97)	6.81 (6.71)	11.52(11.66)	-
$[Mn(DBAP)_2].11H_2O$	731.24	49.14 (49.27)	5.32 (7.11)	7.67 (7.66)	7.46(7.51)
$[Fe(DBAP)_2].14H_2O$	786.15	45.74 (45.83)	4.19 (7.38)	6.83 (7.12)	6.94 (7.10)

Where DBAP is $C_{15}H_{15}N_2O$, M. Wt. = Molecular Weight

Table 4: Relevant Infra red Frequencies (cm^{-1}) of the Schiff base and its Metal(II) Complexes.

Compounds	*v*(OH) cm^{-1} Phenolic	*v*(OH) cm^{-1} water	*v*(C=N) cm^{-1}	*v*(C-O) cm^{-1}	*v*(M-N) cm^{-1}	*v*(M-O) cm^{-1}
DBAP	3335.14	-	1615.10	1374.16	-	-
$[Mn(DBAP)_2].11H_2O$	-	3282.63	1585.18	1365.53	458.83	442.00
$[Fe(DBAP)_2].14H_2O$	-	3313.98	1588.48	1362.39	548.25	471.95

Where DBAP is $C_{15}H_{15}N_2O$

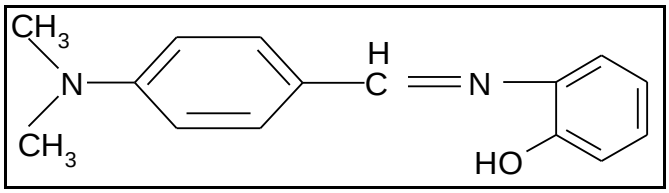

Fig. 1: proposed structure of the Schiff base

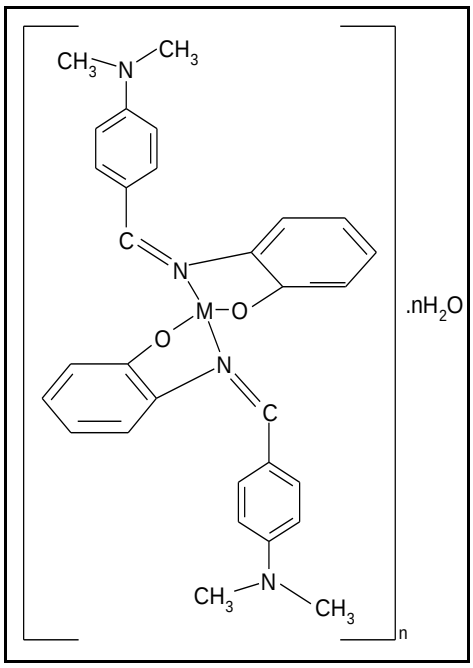

Fig. 2: proposed structure of Mn(II) and Fe(II) complexes

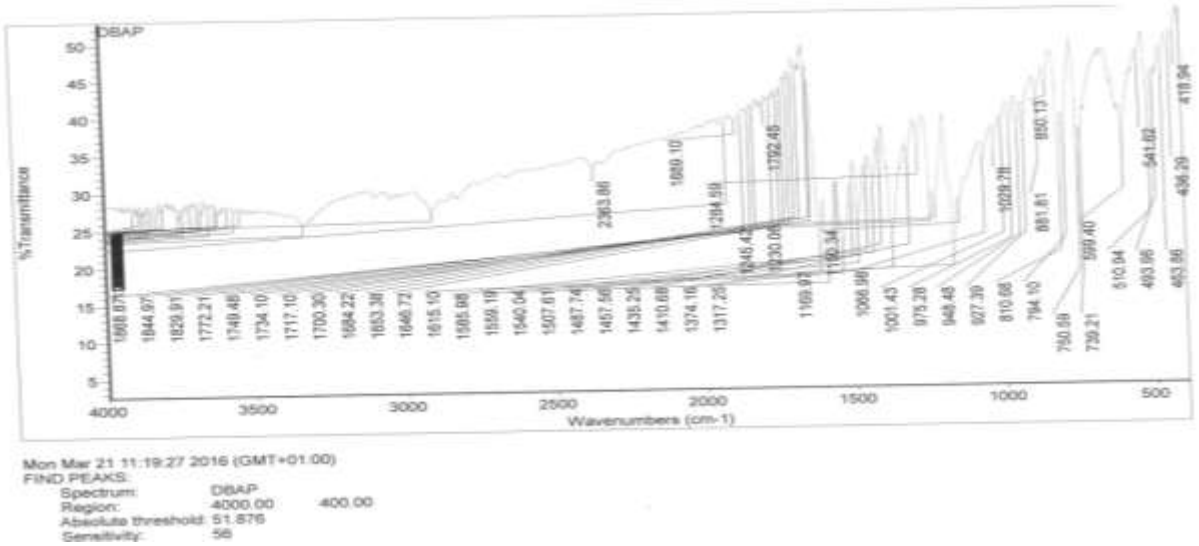

Fig. 3: IR spectrum of Schiff Base

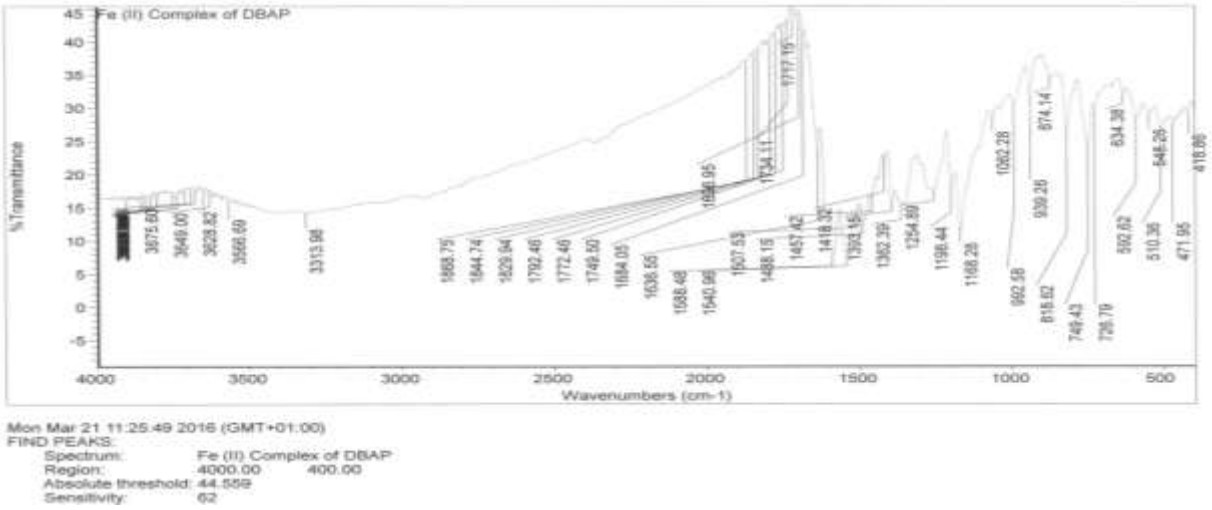

Fig 4: IR Spectrum of Fe(II) Complex

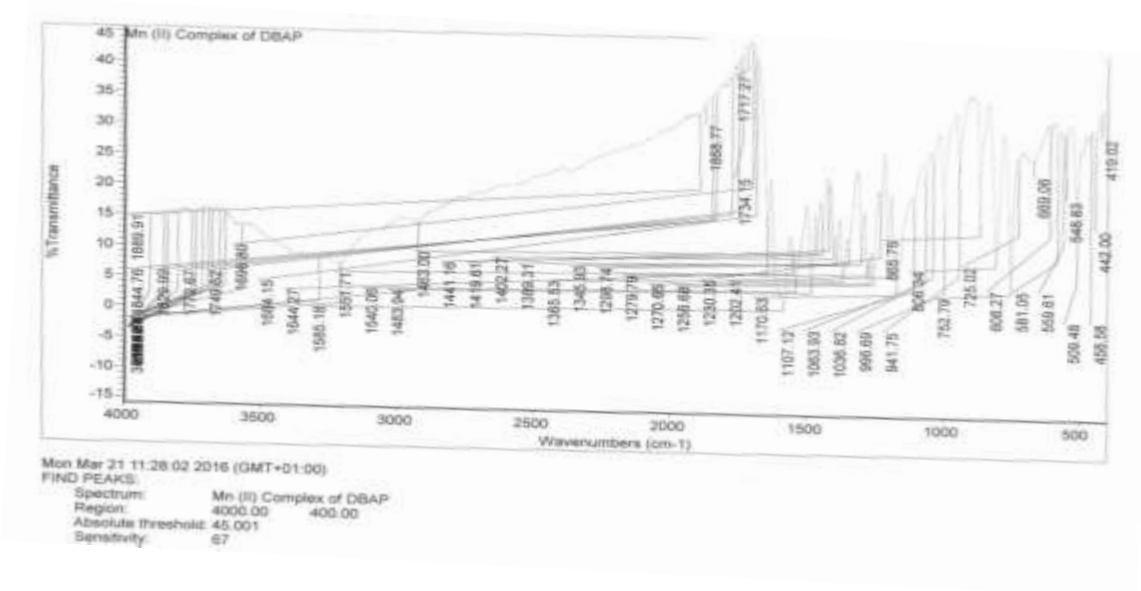

Fig. 5: IR Spectrum of Mn(II) Complex

Discussion

The prepared Schiff base and its metal(II) complexes gave good yield, ranging from 58.17-64.72%. The Schiff base was cadmium orange in colour while the Mn(II) and Fe(II) complexes are brown and bistre respectively. The melting point of the Schiff base was found to be 119^{o}Cwhile the decomposition temperatures of the complexes are 136^{o}C and 148^{o}C.

Molar conductivity

The molar conductance of the complexes was determined. It was found to be 12.64 and 18.53 ohm^{-1}cm^{2}mol^{-1}for the Mn(II) and Fe(II) complexes respectively. These low values indicated that the complexes are non-electrolytes[4].

Magnetic Moment

The effective magnetic moments of the complexes were calculated. The magnetic moment of 7.06 and 5.97 B.M observed for Mn(II) and Fe(II) complexes are indicative of five and four unpaired electrons respectively in a tetrahedral environment[5]. The physical properties are presented in Table 1.

Solubility Test

The solubility of the Schiff base and its metal(II) complexes were determined in water and some common organic solvents. The Schiff base was found to be soluble in all the solvents used except carbontetrachloride and water. The complexes were soluble in dimethylsulphoxide,dimethylformamide and methanol but insoluble in water and slightly soluble in the other solvents used. The results are presented in Table 2.

Microanalysis

The elemental analysis of the Schiff base and its metal(II) complexes were determined. The found and calculated values were fairly in good agreement. The elemental analysis data of the Schiff base suggested the formation of $C_{15}H_{15}N_2O$ while that of the complexes revealed the formation of$[Mn(DBAP)_2].11H_2O$ and $[Fe(DBAP)_2].14H_2O$. The complexes are formed in 1:2 M:L ratio. The results are presented in Table 3.

Infrared Spectra

The infrared spectrum of the Schiff base showed a band due to the phenolic *ν*(OH) stretching vibration at ≃3335 cm^{-1}. This band disappeared in the spectra of the complexes suggesting deprotonation and involvement of the oxygen atom in coordination[6]. The band at ≃1615 cm^{-1} in the free ligand is attributed to the *ν*(C=N) stretching vibration. The shifting of this band to lower frequencies of ≃1585 and 1589 cm^{-1} in the complexes suggested the coordination of metal atoms via the nitrogen atom of the azomethine[7,8]. The *ν*(C-O) phenolic stretching of the Schiff base is observed at≃ 1374cm^{-1} which got shifted to lower frequencies of ≃1366 and 1362 cm^{-1} in the complexes. This is indicative of coordination through the phenolic oxygen[9].Further conclusive evidence of the coordination of the Schiff base with the metals was shown by the appearance of weak low frequency new bands at≃459 and 548 cm^{-1} assigned to *ν*(M-N) stretching vibration, and at≃ 442 and 472 cm^{-1} assigned to *ν*(M-O) stretching vibration[10,11]. The broad bands at≃3283 and 3314 cm^{-1} in the spectra of the complexes are attributable to water of hydration[12]. The results are presented in Table 4.

Conclusion

The Schiff base and its Mn(II) and Fe(II) complexes were synthesized and characterized. The elemental analysis data confirmed 1:2metal - ligand ratio. The conductivity measurement data revealed that the complexes are non-electrolytes. The infrared data indicated the bidentate nature of the Schiff base ligand coordinated with the metal ions via the nitrogen atom of the azomethine and oxygen atom of the hydroxyl group after deprotonation. The magnetic moment of the complexes suggested a paramagnetic phenomenon around a four-coordinate tetrahedral geometry. From the analytical data and available information in the literature, the following structures are proposed.

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