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Characterization of Ni(II) and Cd(II) metal complexes using Schiff base ligand derived from 2-Thiophene carboxyldehyde and 2-Aminothiophenol

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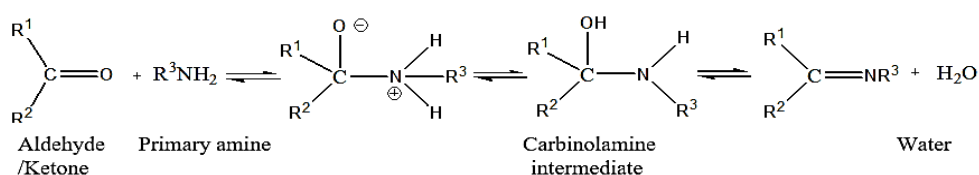
Abstract

Schiff base ligand derived from condensation of 2- aminothiophenol and 2- thiophene carboxyldehyde was synthesized and used for the preparation of Ni(II) and Cd(II) complexes. The synthesized ligand and complexes were analyzed by decomposition temperature, solubility, magnetic susceptibility, molar conductance and infrared spectra. The decomposition temperatures of the complexes are 212 and 221°C. Molar conductance values are 16.12 and 12.60 ohm⁻¹cm²mol⁻¹ respectively. New bands appeared in the IR spectra of the complexes in the range of 519 - 475 cm⁻¹ and 462 - 448 cm⁻¹ which indicate ν (M - N) and ν (M - S) vibrations respectively. Magnetic susceptibility measurement indicated that Ni (II) complex is paramagnetic while Cd(II) complex is diamagnetic. The solubility test revealed that all complexes and ligand are soluble in DMSO. The analytical data show the formation of 2:1 metal to ligand ratio for all complexes and suggested the formula [ML₂].nH₂O. The ligand and metal chelates have been studied for microbial activity using well diffusion method against selected bacteria and fungi. The results signify that Ni(II) and Cd(II) metal complexes inhibit more compared with Schiff base ligand against the same test organisms.

Keywords: Condensation, magnetic susceptibility, paramagnetic, 2-Aminothiophenol, 2-Thiophene carboxyldehyde, solubility, ligand

1. Introduction

Schiff bases are compound that contain azomethine group (-HC=N-) and were first reported by Hugo Schiff in 1864. Schiff bases form important class of nitrogen donor ligands and occupy eminent position amongst the recent achievements in the field of coordination Chemistry. They have the general structure of RN = CR' (Fig. 1). Where R and R' are aryl, alkyl, cycloalkyl or heterocyclic groups, which may be variously substituted. The bonding ability of ligands depends on the nature of atoms (which act as co-ordination sites), steric factor and electro negativity. If the ligand have functional group such as -OH or -COOH near the site of condensation, a very stable five or six membered chelate ring can be formed. Tridentate Schiff base ligands form comparatively stable complexes. Coordination behaviour of Schiff base ligand with transition metals play a significant role in biological and pharmacological activities, especially, transition metal complexes derived from amino acid Schiff bases. The formation of a Schiff base from an aldehydes or ketones is a reversible reaction and generally takes place under acid or base catalysis, or upon heating.



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The Schiff bases form metal complexes with d-block metals have been known to act as highly efficient catalysts in various syntheses and other useful reactions. Many Schiff base complexes

of ruthenium and palladium are used as catalyst in the syntheses of quality polymers. Imine complexes are used in the treatment of diabetes and AIDS. They aid in understanding the structure of biomolecules and biological processes taking place in the living organisms. They are used in the treatment of cancer and for immobilization of enzymes. Prepared metal (II) Schiff base complexes of Cu, Ni, Fe and Zn from salicylaldehyde and o-amino benzoic acid, the complexes were characterized by IR and NMR spectral analysis. The Schiff base and its complexes were tested for their antibacterial activity against *Pseudomonas aeruginosa*, *Proteus vulgaris*, *Proteus mirabilis*, *Klebsiella pneumonia* and *Staphylococcus aureus*, the results indicated that metal (II) Schiff base complexes are biologically more active than the Schiff base ligand against the same test organisms.

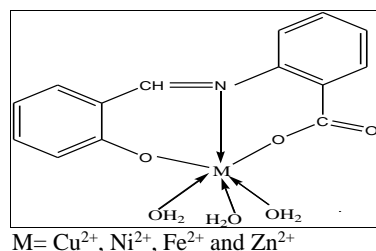


Fig 1: Triaqua- 2-[(2-oxo benzylidene) amino] benzoate Metal (II)

A series of Schiff base analogues of 4-aminoantipyrine analogues have been tested for bactericidal and cytotoxic activities against selected bacterial strains (*Klebsiella Pneumonia*, *Staphylococcus aureus*, *Cronobacter sakazakii*, *Citrobacter freundii*, *Salmonella enterica* and *Escherichia coli*) and brine shrimp (*Artemia salina*) nauplii, respectively. Of the compounds tested, two compounds showed a good inhibition of bacterial growth against *E. coli* and *C. sakazakii*, whereas three compounds demonstrated high cytotoxicity with LC₅₀ values of 225, 480, and 581 ppm, in a short term bioassay using *A. salina*,

The synthesis of Ni(II), Co(II) Cu(II) and Zn(II) complexes with Schiff base (3-ethoxy salicylidene amino benzoic acid) in alcoholic medium, prepared from 3-ethoxy salicylaldehyde and 2-amino benzoic acid. The complexes were non-electrolytes in dimethyl sulfoxide solvent (DMSO). The structure of the Schiff-base ligand and its metal complexes were confirmed by various spectroscopic studies like IR, UV-VIS, ¹H NMR, ¹³C NMR, ESI-mass spectra, elemental analysis, molar conductance, thermogravimetric studies and magnetic susceptibility measurements and six-coordinated geometry was assigned to these complexes.

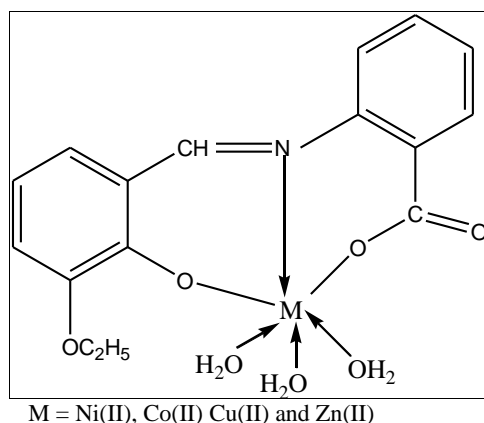


Fig 2: Triaqua- 2-[(3-ethoxy-2-oxo benzylidene) amino] benzoate Metal (II)

2. Materials and Methods

Reagents are of analytical grade purity and were obtained from Sigma Aldrich chemical limited. The glassware used were washed thoroughly with detergent, rinsed with distilled water and dried in an electrical oven. Melting point and decomposition temperature were determined using Gallenkamp melting point apparatus. Molar conductivity measurement was carried out using Jenway conductivity meter model 4010, while magnetic susceptibility measurement was done on MBS MKI magnetic susceptibility balance at 25 °C. IR spectral analysis was carried out using FTIR Cary 630 (Agilent Technology) model in the range of 4000 - 400cm⁻¹. Bacterial and fungal isolates were obtained and identified at the Department of Microbiology, Kano University of Science and Technology, Wudil.

2.1. Methods

2.1.1. Preparation of Schiff base

The Schiff base were prepared by mixing a solution of 2-aminothiophenol (2.50g, 0.1 mole) in 25ml of ethanol with 2-thiophene carboxylaldehyde (2.24g, 0.1 mole) in the same solvent. The reaction mixture was left under reflux for 2 hours. The product formed was separated by filtration, purified by crystallization from ethanol, and then dried in a desiccator over anhydrous calcium chloride.

2.1.2. Preparation of the metal complexes

The Ni (II) and Cd (II) complex of Schiff base was prepared by the addition of a solution of appropriate metal chloride (1mmol) in an ethanol water-mixture (1:1, 25ml) to the solution of schiff base (0.219g, 2mmol) in the same solvent (25ml). The resulting mixture was stirred under reflux for 1hour upon the complex precipitated and collected by filtration and washed with a 1:1 ethanol-water mixture.

2.2. Solubility test

The solubility test of the Schiff base, Ni (II) and Cd(II) metal complexes was carried out in some solvents, ethanol, methanol, dimethylsulphoxide (DMSO), dimethylformamide (DMF), n- hexane, diethyl ether, chloroform and distilled water. Small amount of Schiff base and metal(II) complexes were each added into the test tube followed by addition of the solvent. Their solubility was observed after shaking the test tube.

2.3. Melting point/decomposition temperature

The melting point of the Schiff base and the decomposition temperature of the Ni(II) and Cd(II) metal complexes were carried by taking small amount of each into a capillary tube. The tube was inserted into the Gallenkamp apparatus, the temperature at which the ligand melt and that which the complexes decompose were taken and Recorded

2.4. Conductivity measurement

0.003M solutions of the Ni(II) and Cd(II) complexes were prepared in DMSO and the molar conductance was determined by Janway, 4010 conductivity meter. All measurements were carried out at room temperature; the molar conductance value was obtained from the relation

$$\text{Molar conductance} = \frac{1000}{C} \times K$$

Where C = Molar concentrations
K = Specific conductance

2.5. Magnetic susceptibility measurement

The magnetic susceptibility of the Ni(II) and Cd(II) metal complexes was obtained from magnetic susceptibility balance. Each separate sample of the metal complex was placed into a capillary tube and then inserted into the magnetic susceptibility balance, the readings was then recorded. The gram magnetic moment is calculated using the relation.

$$X_g = \frac{CL(R - R_0)}{10^9 M}$$

Where

C = I, Constant of Proportionality

L = Sample length in the capillary tube (cm)

R = Reading obtained of sample placed in tube

R₀ = Reading obtained of the pre-weighed empty sample tube

M = W₂ - W₁, actual mass of sample in the tube (g)

The molar magnetic moment is calculated as

X_m = X_g × FW (g/mol)

The effective magnetic moment (B.M) is given by

μ_{eff} = 2.828 (X_m T)^{1/2}

T = Absolute temperature (25 °C)

2.6. Determination of percentage of water of crystallization in the complexes

About 0.2g of each of the Ni(II) and Cd(II) metal complexes was measured into a watch glass of known weight and placed in an oven at 110°C until a constant weight was obtained. The percentage composition of water in the complex was calculated using the below formula:

$$\frac{\text{Weight lost}}{\text{Weight of the complex taken}} \times 100$$

2.7. Determination of percentage of metal ions in the complexes

2.7.1. Digestion of metal complex

About 0.2g of each of the Ni(II) and Cd(II) metal(II) complex was placed in a 100cm³ beaker containing 25cm³ of distilled water to which 5cm³ of concentrated acid was added and then heated to about dryness. The contents in the beaker were allowed to cool to room temperature and 25cm³ of distilled water was added and the mixture was stirred before the filtrate was collected which contains the metal ions.

2.7.2: Estimation of nickel in Nickel(II) complex

Distilled water was added to the filtrate of digested nickel(II) complex to 97cm³. The filtrate was heated to 70-80 °C and alcoholic solution of 1% dimethylglyoxime (DMG) was added in slight excess, followed by immediate drop wise addition of dilute ammonia solution. The content was allowed to stand on steam bath for 30 minutes to ensure complete precipitation, before allow to cool and the precipitate separated, washed, dried at 110 °C and weight as [Ni(C₄H₇O₂N₂)₂].

2.7.2. Estimation of cadmium in Cadmium(II) complex

Distilled water was added to the filtrate obtained from the digested cadmium(II) complex to 75cm³, 1g of ammonium thiocyanate and 2cm³ of pyridine were added to the filtrate. The mixture was vigorously stirred until crystal separated out, which were allowed to stand for 15 minutes before washing with a solution made by dissolving 3.9g of potassium thiocyanate and 2.7g mercuric chloride in 100cm³ of distilled water. The precipitate was dried at 110°C and weight as [Cd(C₅H₅N)₂](SCN)₂.

2.8. Determination of the metal to ligand ratio in the complex compounds using Job's method of continuous variation

The number of coordinated Schiff base ligand in the metal ion were determined by Job's method in which 3 millimolar solution of the ligand and the metal(II) chloride were separately prepared. The following ligand to metal salt (mL); 1:15, 3:13, 5:11, 7:9, 9:7, 11:5, 13:3, 15:1 were taken from the ligand solution and each of the metal complexes. A total volume of 16ml was maintained (in the above order) throughout the process and mole fraction of the ligand was calculated in each mixture. The solutions of the metal chloride (blank) were scanned to obtained wave length of maximum absorption (λ_{max}) for each metal ion. The spectrophotometer was set at λ_{max} before taking the absorbance value. A plot of absorbance against mole fraction of the ligand, the number of coordinated ligand was determined using the following relation:

$$\bar{n} = \frac{X_i}{1 - X_i}$$

Where:

\bar{n} = number of coordinated ligand at maximum absorbance

X_i = mole fraction at maximum absorbance

2.9. Determination of empirical formular

The composition of each complex was determined from the known percentage of the metal ion and water content in the complex. The percentage composition of the ligand was obtained by adding percentage composition of the metal and water in the complex and subtracted from 100 to get that of ligand. The empirical formula of each of the complex was calculated using the percentage composition of the species involved.

2.10. Anti-bacterial studies

The antibacterial activity of the Schiff base ligand and its metal complexes was carried out by using bacterial isolates of *Staphylococcus aureus*, *streptococcus pneumoniae*, and *Escherichia coli*. The suspension of each microorganism was smeared on the surface of the solidified Muller-Hinton Agar (MHA) already poured into petri dishes. The Schiff base and the metal Complexes were separately dissolved in DMSO so as to have three distinct concentrations (60μg/disc, 30μg/disc and 15μg/disc) through serial dilution and placed on the surface of the culture media, incubated at 37 °C for 24 hours. Activities were determined by measuring (mm) the diameter of the zone of inhibition and compared with a standard drug (Ciprofloxacin) (Khan *et al.*, 2014) [13].

2.11. Anti-fungal studies

The antifungal activity of the Schiff base ligand and that of its metal complexes were tested against three pathogenic fungi; *Candida albicans*, *Aspergillus flavus*, and *Aspergillus fumigatus*, using disc diffusion method. Ketoconazole was used as standard fungicide and DMSO was used as a negative control (Khan *et al.*, 2014) [13]. The fungal suspension was smeared on the solidified Potato Dextrose Agar (PDA) already poured into petri dishes. The Schiff base and the metal Complexes were separately dissolved in DMSO to have three different concentrations (60μg/disc, 30μg/disc and 15μg/disc) per well. They were placed on the surface of the culture media and allowed to stand at room temperature for good 48 hours. Activities were determined by measuring (mm) the diameter of the zone of inhibition and compared with the standard.

3. Results and Discussions

3.1. Results

Results of the physical properties, characterization and microbial activities of the prepared Schiff base ligand, Ni(II) and Cd(II) metal (II) complexes are presented in the following tables.

Table 1: Physical properties of ligand, Ni(II) and Cd(II) complexes

Compound	Colour	% yield	M.P. (°C)	D. Temp (°C)
Ligand	Yellow	79	128	-
[NiL ₂].3H ₂ O	Light Brown	58	-	213
[CdL ₂].2H ₂ O	Yellow Brown	56	-	221

Where L = C₁₁H₉NS₂, M.P. = Melting point, D. Temp. = Decomposition temperature

Table 2: Solubility test of Schiff base ligand, Ni(II) and Cd(II) complexes

Solvents	Ligand	[NiL ₂]	[CdL ₂]
Water	IS	IS	IS
Methanol	S	SS	S
Ethanol	S	S	S
n-hexane	IS	IS	IS
Chloroform	S	IS	IS
Diethylether	S	S	SS
DMF	S	S	S
DMSO	S	S	S

L = C₁₁H₉NS₂, DMSO = Dimethylsulfoxide, DMF = Dimethylformamide, CCl₄ = Carbontetrachloride, S = Soluble, SS = Slightly soluble, IS = Insoluble

Table 3: IR spectra of the Schiff base and Ni(II) and Cd(II) complexes

Compounds	V(C=N) cm ⁻¹	V(M-S) cm ⁻¹	V(M-N) cm ⁻¹	V(C-S-C) cm ⁻¹	V(C-S) cm ⁻¹
Ligands	1689	-	-	852	-
[NiL ₂]	1659	462	519	829	765
[CdL ₂]	1633	448	475	832	765

L = C₁₁H₉NS₂

Table 4: Conductivity measurement data of 10⁻³ M Ni(II) and Cd(II) complexes in DMSO

Complexes	Electrical conductivity (ohm ⁻¹ cm ⁻¹) × 10 ⁻⁶	Molar conductance (ohm ⁻¹ cm ² mol ⁻¹)
[NiL ₂]	48.31 × 10 ⁻⁶	16.12
[CdL ₂]	37.81 × 10 ⁻⁶	12.60

L = C₁₁H₉NS₂

Table 5: Magnetic susceptibility data of Ni(II) and Cd(II) complexes

Complex	μ _{eff} (B.M.)	Magnetic property	Number of unpaired electrons
[NiL ₂]	2.86	Paramagnetic	2
[CdL ₂]	0.49	Diamagnetic	0

L = C₁₁H₉NS₂

Table 6: Determination of water of crystallization in the Ni(II) and Cd(II) complexes

Complex	Weight lost (g)	Percentage (%)	Number of water of crystallization
[NiL ₂]	0.017	8.50	2
[CdL ₂]	0.016	8.00	3

Table 7: Percentage (%) of metal ion in the Ni(II) and Cd(II) complexes

Complex	Percentage (%)
[NiL ₂]	12.74
[CdL ₂]	14.93

L = C₁₁H₉NS₂

Table 8: Empirical formula of the Ni(II) and Cd(II) complexes

Compound	% of Metal	% of Ligand	% of Water	Metal: Ligand ratio	Empirical formula
Ni(II) complex	12.74	78.76	8.50	1:2	[NiL ₂].2H ₂ O
Cd(II) complex	14.93	77.07	8.00	1:2	[CdL ₂].3H ₂ O

L = C₁₁H₉NS₂

Table 9: Mole fraction of the ligand and the absorbance values for Ni²⁺ ion at 540 nm

Ni ²⁺ : L ratio	Mole fraction	Absorbance
1:15	0.0625	0.0374
3:13	0.1875	0.0712
5:11	0.3125	0.0881
7:9	0.43715	0.1115
9:7	0.5625	0.1305
11:5	0.68754	0.1561
13:3	0.8125	0.0820
15:1	0.9375	0.0472

L = C₁₁H₉NS₂

Table 10: Mole fraction of the ligand and the absorbance values for Cd²⁺ ion at 635 nm

Cu ²⁺ : L ratio	Mole fraction	Absorbance
1:15	0.0625	0.1640
3:13	0.1875	0.1811
5:11	0.3125	0.3829
7:9	0.43715	0.5404
9:7	0.5625	0.5281
11:5	0.68754	0.7105
13:3	0.8125	0.5250
15:1	0.9375	0.2526

L = C₁₁H₉NS₂

Table 11: Antibacterial activity of the Schiff base, Ni(II) and Cd(II) complexes

Isolates	Compounds	Zone of inhibition (μg/ml)			Standard
		60	30	15	
<i>Staphylococcus aureus</i>	Ligand	9	8	7	29
	[NiL ₂]	13	9	7	
	[CdL ₂]	15	11	6	
<i>Streptococcus pneumoniae</i>	Ligand	8	6	6	19
	[NiL ₂]	14	11	6	
	[CdL ₂]	10	8	7	
<i>Escherichia coli</i>	Ligand	11	9	7	24
	[NiL ₂]	11	6	6	
	[CdL ₂]	13	8	6	

L = C₁₁H₉NS₂

Table 12: Antifungal activity of the Schiff base, Ni(ii) and Cd(ii) complexes

Isolates	Compounds	Zone of inhibition (μg/ml)			Standard
		60	30	15	
<i>Aspergillus fumigates</i>	Ligand	18	16	10	31
	[NiL ₂]	11	6	6	
	[CdL ₂]	17	10	7	
<i>Aspergillus flavus</i>	Ligand	13	10	7	26
	[NiL ₂]	21	17	13	
	[CdL ₂]	15	9	6	
<i>Candida albicans</i>	Ligand	11	8	6	29
	[NiL ₂]	14	11	8	
	[CdL ₂]	18	15	11	

L = C₁₁H₉NS₂

3.2. Discussion

The Schiff base ligand was prepared by condensation of 2-aminothiophenol and 2-thiophene carboxyldehyde to obtain

yellow crystal with high yield (75%) and melting point of 128 °C (table 4). The metal complexes, Ni(II) and Cd(II) complexes were synthesized and found to be of various colors with percentage composition of 57% and 56%. The decomposition temperature of the metal complexes range from 213 °C and 221 °C showing that the decomposition temperature of the metal (II) complexes is higher than that of melting point of the ligand indicating that complexation has taken place.

The solubility test carried out on the Schiff base showed that the Schiff base was soluble in methanol, ethanol, DMSO, DMF, diethylether, and chloroform but insoluble only in

water and n-hexane. However, the Ni(II) and Cd(II) metal complexes were soluble in ethanol, methanol, DMSO and DMF, but insoluble in water and n-hexane while slightly soluble in diethylether and chloroform (table 2).

The infrared spectral results of the ligand shows a band at 1689 cm^{-1} assigned to azomethine ν ($\text{C}=\text{N}$) vibration, this confirms condensation between amino group of 2-aminothiophenol and aldehyde group of 2-thiophene carboxyldehyde in the formation of Schiff base. The infrared spectral data of the Schiff base ligand, Ni(II) and Cd(II) metal complexes were listed in table 3.

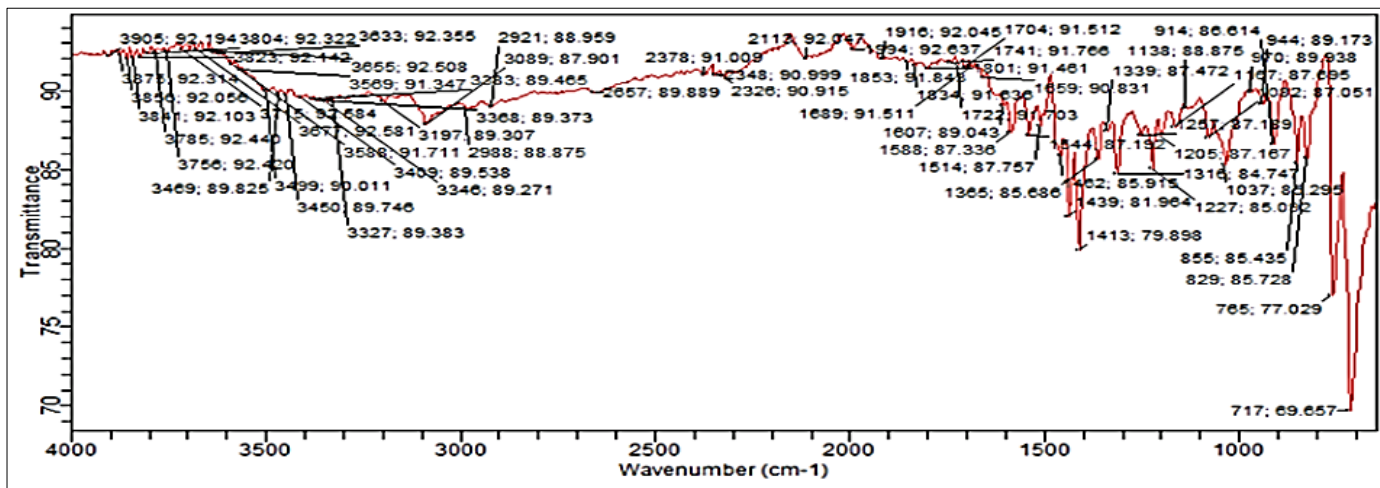


Fig 3: FT IR spectra of Ni(II) complex

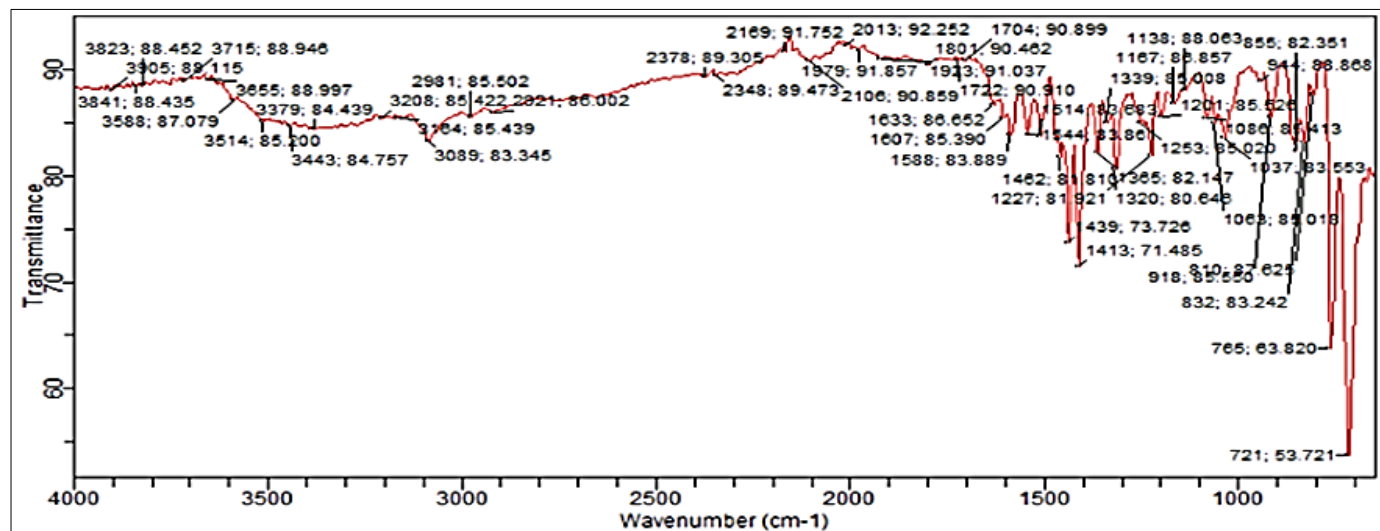


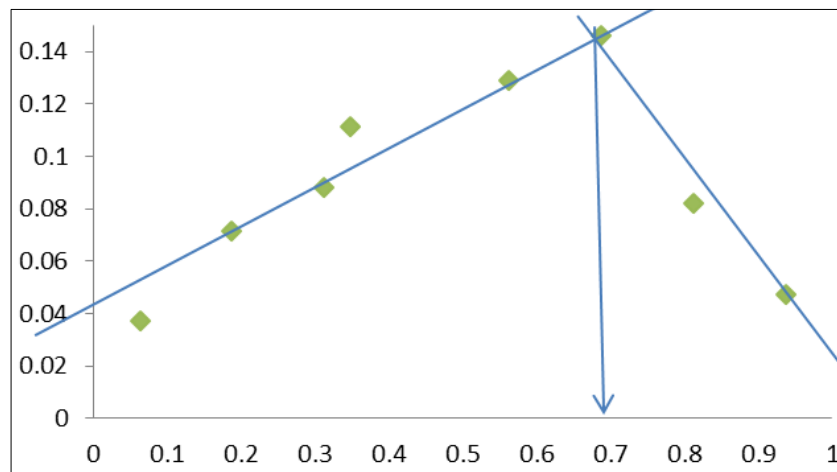
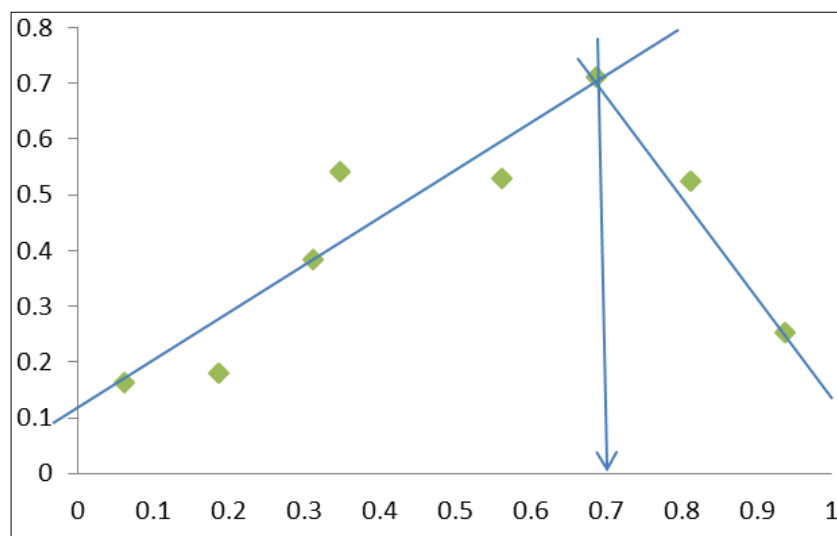
Fig 4: FT IR spectra of Cd(II) complex

The molar conductance of Ni(II) and Cd(II) metal complex was measured in dimethylsulfoxide (DMSO). The values obtained were 16.12 and 12.60 $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$ respectively in (table 4) which are relatively low, indicating the non-electrolytic nature of the metal complexes. The molar conductance ranges for non-electrolyte metal complexes in DMSO is 1-50 $\text{ohms}^{-1}\text{cm}^2\text{mol}^{-1}$

Magnetic susceptibility measurement values for Ni(II) and Cd(II) complexes at room temperature are arranged in table 5. The values Ni(II) complexes indicated it is paramagnetic because it shows the present of unpaired electrons, while Cd(II) complex is diamagnetic, showing the absence of unpaired electrons.

The metal-ligand ratio was determined by using Job's method

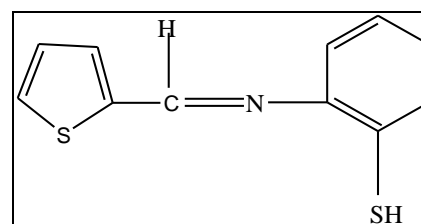
of continuous variation (UV-Visible). The results were presented in table 9 - 10. They revealed that the metal-ligand ratio was 1:2. Gravimetric analysis was used for the determination of the percentage of metal(II) ions in the complexes (table 7). The metal (II) Schiff base complexes show variation in the co-ordination number of water molecules. Cd(II) has three molecules of water, while Ni(II) has two water molecules. The empirical formulae of the complexes were determined from known values of percentage composition of metals, Schiff base and water of crystallization. The results revealed that the metal-ligand was 1:2 for all the complexes and suggested the formula $[\text{ML}_2] \cdot n\text{H}_2\text{O}$.

Fig 5: Ni²⁺ ion absorbance against mole fractionFig 6: Cd²⁺ ion absorbance against mole fraction

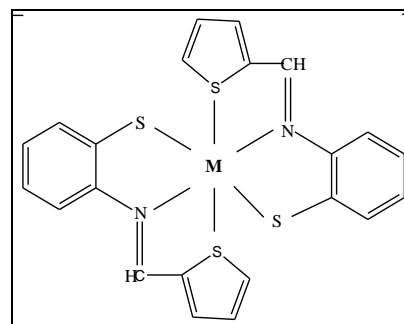
The antibacterial activity of the schiff base and metal complexes was carried out on three bacteria isolates, (*Staphylococcus aureus*, *Streptococcus pneumoniae* and *Escherichia coli*) using well diffusion method by taking DMSO as solvent (table 11). The result shows that both the ligand and metal complexes are found to be moderately effective against all tested bacteria, but the metal complexes exhibit higher antibacterial activity than the Schiff base and the activity increase with increase in concentration, this is probably due to chelation in the metal complexes. The Schiff base show activity against *Staphylococcus aureus* and *Escherichia coli* at all concentrations, but found to be active only at (60µg and 30µg) concentrations against *Staphylococcus aureu*, while the complexe of Ni (II) and Cd(II) is active at all concentrations. In *Escherichia coli*, Cd(II) complex is effective at (60µg and 30µg) only, while Ni(II) complex is active at (60µg) concentration only. However, in comparison to the standard drugs antibacterial activities of the Schiff base as well as the complexes is relatively low.

Antifungal studies were carried out by well diffusion technique on potato dextrose agar against three fungal isolate *Aspergillus fumigatus*, *Aspergillus flavus* and *Candida albicans* (table 11). The result of anti-fungal screening for the Schiff base and metal complexes revealed that both the Schiff base and Cd(II) complex show an activity against *Aspergillus fumigatus* isolate and the activity increase with increase in concentration, while the Ni(II) complex is active only at (60µg) concentration. Also, the Schiff base and the Ni (II)

complex show high activity against *Aspergillus flavus* at all concentrations, and the Cd(II) complex active at (60µg and 30µg) concentrations respectively. In *Candida albicans*, the Schiff base, Ni(II) and Cd(II) complexes shows an appreciable activity at all concentrations.



Scheme 1: Proposed structure of the ligand



Scheme 2: Proposed structure of the complex

4. Conclusion

The Schiff base and its metal complexes of Ni(II) and Cd(II) have been synthesized and studied by various analytical techniques. Job's method of Continuous variation shows that the metal-ligand ratio in all the complexes is 1:2. All the complexes are non-electrolytes in DMSO solvent. The decomposition temperature of Ni(II) and Cd(II) complexes indicated that complexation has taken places. Based on electrical conductivity data, Ni(II) Schiff base complexes is paramagnetic while Cd(II) complex is diamagnetic. The antimicrobial studies of the Schiff base and its metal(II) complexes reveals that the metal(II) complexes show better activity when compared to that of the ligand

5. Acknowledgement

I sincerely thank Almighty God for his grace upon me, may His peace, mercy and blessing be upon to His messenger Muhammad (S.A.W.) including his family and friends. My immense gratitude goes to my family especially my father Dalha Hamza, my mother Amina Sulaman for their endurance and prayers.

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