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Schiff Base Constructed from 2-Thiophene Carbaldehyde and O-Phenylenediamine and its Co (Ii), Ni (Ii), and Cu (Ii) Complexes: Synthesis, Characterisation, and Antimicrobial Studies

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ABSTRACT
Schiff base derived from 2-thiophene carbaldehyde and o-phenylenediamine was synthesized. The Schiff base underwent reflux with the corresponding metal (II) chlorides to produce Co(II), Ni(II), and Cu(II) complexes. These complexes were characterized through various analytical methods including FT-IR spectroscopy, determination of melting/decomposition temperatures, solubility assessments, magnetic susceptibility measurements, atomic absorption spectroscopy, conductivity analysis, and elemental analysis. In the FT-IR spectra of the Schiff base, a distinctive vibrational peak at 1622 cm^{-1} was observed, attributed to the azomethine υ(C=N) vibration frequencies. Interestingly, shifts in the vibrational bands (of azomethine) were noted in the complexes, indicating their involvement in complex formation. Absorption bands in the range of 713 – 769 cm^{-1} in the complexes were linked to M-N bond formation, confirming the presence of metal-nitrogen bonds. The complexes exhibited low molar conductance values (6–13 Ohm-1 cm^2 mol^{-1}), indicating their non-electrolytic nature. The Schiff base displayed a melting point of 120°C, whereas the complexes showed decomposition temperatures within the range of 155-196°C, signifying their thermal stability. The magnetic susceptibility values deduced suggested an octahedral geometry for all complexes, ranging between 2.24-3.12BM. Elemental analysis of the Schiff base and its metal(II) complexes for C, H, and N content revealed close agreement between observed and calculated percentages, suggesting a 1:2 metal-Schiff base ratio in all complexes. Qualitative tests confirmed the presence of chloride in the complexes, with estimated percentages falling between 4.62-7.90%. Furthermore, the synthesized Schiff base and its metal (II) complexes were evaluated for their antibacterial and antifungal activities against various microbial species. The results indicated that the metal complexes exhibited significant antimicrobial properties against *Escherichia coli, Staphylococcus Aureus, Klebsiella Pneumoniae, Pseudomonas aeruginosa, Candida albicans, and Aspergillus niger*.

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INTRODUCTION

Schiff bases, which are the result of condensation between primary amines and ketones or aldehydes, have a significant tendency to form stable complexes with a variety of transition metals (Lee, 1996). Their remarkable adaptability, simplicity of synthesis, and wide variety of uses highlight the chemistry's ongoing significance to modern researchers. Schiff base complexes are appealing in the field of bioinorganic chemistry because of their important contributions to the development of medicinal chemistry (Kumble et al., 2017). Numerous compounds with the fundamental azomethine (-C=N-) linkage have noteworthy antibacterial, antifungal, anticancer, antiviral, and antimalarial activities, indicating the critical role this functional group plays in biological activity (Arulmurugan et al., 2010; Annapoorani and Krishman, 2013). Furthermore, some Schiff base complexes with donor atoms of nitrogen and oxygen are very efficient stereospecific catalysts for a variety of organic and inorganic reactions, such as hydrolysis, reduction, and oxidation (Gupta and Sutar, 2008).

Numerous sulfa drug compounds were created, described, and tested for antibacterial activity (Fatima, 2015; Abdulbaset and Ahmad, 2014; Zahid et al., 2012). The extensive use of sulfa medications to treat a wide range of bacterial diseases is the reason for this increase in research activity (Brumfit and Hamilton, 1993). It has been demonstrated that adding more functional groups to the sulfa drug scaffold improves its functionality and strengthens its resistance to different bacterial strains. Additionally, there is growing evidence that the addition of metal ions by chelation significantly increases these medications' ability to combat infections (Jurca et al., 2017; Weaver et al., 2016).

MATERIALS AND METHODS

All of the analar-grade reagents and solvents used in the study were used without additional purification. After being carefully cleaned with soap and rinsed with distilled water, the glassware was desiccated at 110°C in a Gallankamp hot box oven. Using an electric meter balance, every measurement was done precisely. Agilent technician at the Department of Pure and Industrial Chemistry, Bayero University Kano (BUK), used the FUR CARY 630 model to record the infrared (IR) spectra of the ligand and its metal (II) complexes in the 400–1000 cm^{-1} and 650–4000 cm^{-1} ranges.

Using a gallankamp melting point device, the ligands and their metal (II) complexes' melting point and decomposition temperature were ascertained. The Janway4010 conductivity meter was used to measure the complexes' conductance. The Sherwood magnetic susceptibility balance was used to determine the magnetic susceptibility. The Central Laboratory of Umar Musa Yar'Adua University conducted elemental analyses for C, H, and N. For the production of the Schiff base, antimicrobial activity tests were conducted at Bayero University Kano's Department of Microbiology.

Synthesis of Schiff base

Two-thiophene carbaldehyde (0.05mole/4.67g) and O-phenylenediamine (0.025mol/2.7035g) in 50cm^3 of ethanol were refluxed for six hours. After that, the reaction mixture was let to cool for a full day. A precipitate was produced, which was then filtered, washed with ethanol, and dried for three days in a desiccator with $CaCl_2$ (Aly and Fathala, 2020).

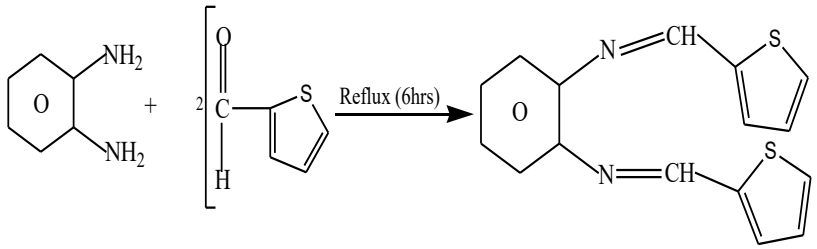


Figure 1: Orthophenylenediamine 2 – thiophene carbaldehyde Schiff base

Synthesis of Metal (II) Complexes

In a 50 cm³ ethanol solution, metal (II) salts (0.0025/mol) were separately mixed with ligands (0.005mol) and refluxed for six hours. The resultant mixture was then allowed to cool for a full day, which caused a solid precipitate to form. According to Aly and Fathala (2020), this precipitate was subsequently thoroughly cleaned using a solution of acetone and water, and then dried for two days in a desiccator filled with P_2O_5.

Antibacterial Test

Assessment of Antibacterial Efficacy The microbiological cultures collection at Bayero University Kano, Nigeria, provided clinical strains of Pseudomonas aeruginosa, Klebsiella pneumoniae, Staphylococcus aureus, and Escherichia coli. Cheesbrough's (2002) established microbiological techniques were used at the Microbiology Department to identify these strains. The Kirby-Bauer disc diffusion method was used to evaluate the antibacterial efficacy (CLSI, 2006). An overnight culture was suspended in a saline solution (0.85% NaCl) to create the bacterial inoculum, which was subsequently diluted to meet the McFarland turbidity standard of 0.5 (10^8 cells/mL). According to Yusha'u and Sadisu (2011), the produced inoculum was then swabbed onto the nutrient agar surface using a sterile cotton swab. In order to get four different concentrations (100 µg/mL, 200 µg/mL, 300 µg/mL, and 400 µg/mL) through serial dilution, the Schiff base and its complexes were individually dissolved in DMSO. After that, these solutions were poured onto sterile paper discs that had a diameter of 6.0 mm. The reference standard was Amoxicillin, a commercial antibiotic. The growth inhibition zones (in mm) surrounding the discs were measured and monitored after a 24-hour incubation period at 37°C. Yusha'u and Sadisu's (2011) standard was compared to the diameter of the inhibitory zone generated by the ligand and its complexes.

Antifungal Activity Test

Aspergillus niger and *Candida albicans* clinical isolates were obtained from the Microbiology Laboratory's culture collection at Bayero University Kano, Nigeria. Following the standard microbiological protocols described by Cheesbrough in 2002, the isolates were identified at the Department of Microbiology. Following the 2006 CLSI guidelines, the Kirby-Bauer disc diffusion experiment was used to evaluate the in vitro antifungal activity. The procedure outlined by Hassan et al. (2006) was followed for performing the inoculation technique. After pouring solidified Potato Dextrose Agar (PDA) into Petri plates, the prepared inoculum was applied to the surface of the PDA. Using a serial dilution technique, the ligand and its complexes were separately dissolved in dimethyl sulphoxide (DMSO) to produce four different concentrations (100µg/ml, 200µg/ml, 300µg/ml, and 400µg/ml) per disc. They were then placed on the potato dextrose agar surface of the culture media and let to incubate for 48 hours at room temperature. Nystatin, a common antifungal, was used as a reference to compare the diameter of the inhibitory zone produced by the ligand and the complexes (Hassan et al., 2006).

RESULTS AND DISCUSSION

Results of the analysis are presented in the tables below:

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

Compound	Colour	Decomposition temp (^{0}C)	Melting Point (^{0}C)	Percentage Yield %
Ligand	Yellow	-	120	90.20%
$[CoL_2Cl_2]$	Sea Blue	189	-	68.42%
$[NiL_2Cl_2]$	Burnt umber	155	-	66.92%
$[CuL_2Cl_2]$	Light Green	196	-	72.53%

L = Ligand

Table 2: Solubility Test of the Ligand and its Metal (II) Complexes

Compound	Distilled Water	MeOH	EtoH	Chloroform	Acetone	Nitro Benzene	CCl_4	DMF	DMSO	Ether	Pet Ether	Acetonitrile
Ligand	IS	SS	SS	SS	S	SS	IS	S	S	IS	IS	S
$[CoL_2Cl_2]$	IS	SS	SS	S	S	SS	S	S	S	IS	IS	SS
$[NiL_2Cl_2]$	IS	SS	SS	SS	S	SS	SS	S	S	IS	IS	SS
$[CuL_2Cl_2]$	IS	SS	SS	S	S	S	S	S	S	SS	IS	SS

Key: L = Ligand $C_{16}N_2S_2H_{12}$
S = Soluble
SS = Slightly Soluble
IS = Insoluble

Table 3: Conductivity Measurement of Complexes in 1x10m^{-3} DMSO

Complex	Concentration (dm^{-3})	Specific conductance (Ohm^{-1} Cm^{-1})	Molar conductance Ohm^{-1} cm^2 mol^{-1}
$[CoL_2Cl_2]$	1 x 10^{-3}	13 x 10^{-6}	13
$[NiL_2Cl_2]$	1 x 10^{-3}	6 x 10^{-6}	6
$[CuL_2Cl_2]$	1 x 10^{-3}	7 x 10^{-6}	7

L = Ligand $C_{16}N_2S_2H_{12}$

Table 4: Infrared Spectral Data of the Ligand and its Metal (II) Complexes

Compound	υ (C=N) cm^{-1}	υ (M-N) cm^{-1}	υ (M-Cl) cm^{-1}
Ligand	1622	-	-
$[CoL_2Cl_2]$	1611	481	538
$[NiL_2Cl_2]$.	1640	432	566
$[CuL_2Cl_2]$	1659	418	577

L = Ligand $C_{16}N_2S_2H_{12}$

Table 5: Elemental Analysis (CHN) of the Ligand and its Metal (II) Complexes

Compound	C	H	N
Ligand	64.77 (64.87)	4.05 (4.35)	2.88 (2.67)
$[CoL_2Cl_2]$	54.21(53.09)	3.62 (3.31)	6.83 (7.71)
$[NiL_2Cl_2]$	54.83 (53.11)	3.54 (3.31)	8.21 (7.74)
$[CuL_2Cl_2]$	53.25 (52.75)	3.93 (3.29)	1.33 (7.69)

L = Ligand $C_{16}N_2S_2H_{12}$
The values in the parenthesis are the calculated ones.

Table 6: Magnetic Parameters of Metal (II) Complexes

Compound	Magnetic susceptibility Xg($erg.G^{-2}$ g^{-1})	Molar Magnetic Susceptibility Xg($erg.G^{-2}$ mol^{-1})	B.M (μ_{eff})
$[CoL_2Cl_2]$	3.40 x10^{-6}	2.5 x10^{-3}	3.12
$[NiL_2Cl_2]$	3.67 x10^{-6}	2.65 x10^{-3}	2.52
$[CuL_2Cl_2]$	2.88 x10^{-6}	2.1 x10^{-3}	2.24

Table 7: Antifungal Activities of Metal (II) Complexes against some Fungal Species

	Candida albican			*Aspergillusniger*		
Compound/conc./(ug/disc)	300	200	100	300	200	100
$[CoL_2Cl_2]$	15	10	8	12	12	9
$[NiL_2Cl_2]$	9	6	6	10	9	9
$[CuL_2Cl_2]$	13	9	6	12	10	6
Nystatin (50mg/ml)	22	23	20	20	20	18

Table 8: Antibacterial Activities of Metal Complexes against some Fungal Species

Micro Organism	*Escherichia Coli*				*Staphylococcus Aureus*				*Klebsiella Pneumoniae*				*Pseudomonas aeruginosa*			
Compound/Conc./(ug/disc)	400	300	200	100	400	300	200	100	400	300	200	100	400	300	200	100
$[CoL_2Cl_2].6H_2O$	12	11	6	6	11	10	10	6	12	11	9	6	13	10	9	9
$[NiL_2Cl_2].3H_2O$	13	6	6	6	12	12	9	7	10	6	6	6	14	10	6	6
$[CuL_2Cl_2].H_2O$	13	10	6	6	12	6	6	6	14	12	10	9	12	12	9	7
Amoxicillin	29				33				30				31			

Discussion

O-phenylenediamine and 2-thiophene carbaldehyde were condensed to create the Schiff base. This produced a yellowish compound with a melting point of 120°C and a high yield of 92.20%. As shown in Table 1, compounds of various colours were produced by the subsequent synthesis of complexes from the reaction of the Schiff base with different metal (II) chlorides. These complexes' total yields, which varied from 66.92% to 72.53%, showed effective formation.

The stability range, as determined by the breakdown temperature analysis, was 155°C to 196°C.

Tests of the metal (II) complexes' and the ligand's solubility in various solvents (Table 2) revealed that the complexes were soluble in DMSO and DMF, but insoluble in ether and CCl_4.

The complexes' non-electrolytic character was suggested by the low values of molar conductance measurements (Table 3) in DMSO, which ranged from 6 to 13 Ohm^{-1} cm^2 mol^{-1}.

The Schiff base's infrared spectral data (Table 4) showed a vibrational peak at 1622 cm^{-1} that was linked to the azomethine V(C=N) vibration frequencies. The complexes' involvement in complex formation was indicated by the band's shift to a range of 1611–1659 cm^{-1}. The presence of metal-nitrogen bonds was confirmed by the attribution of absorption bands between 418 and 481 cm^{-1} to M-N bond formation. Additionally, the creation of M-Cl bonds was associated with absorption bands in the 538–577 cm^{-1} region.

A constant 1:2 metal-Schiff base ratio was found in all complexes, according to elemental analysis (Table 5) of the Schiff base and its metal (II) complexes for H, C, and N content. The calculated and observed percentages agreed quite well. For the metal (II) complexes,

Magnetic susceptibility measurements at room temperature (Table 6) showed magnetic moments between 2.24 and 3.12BM, indicating paramagnetic characteristics and an octahedral geometry in line with values documented in the literature for octahedral complexes (Angelici, 1977).

Significant antifungal action against fungal species was shown by the complexes, especially at higher concentrations (Table 7). Likewise, at high concentrations, the complexes demonstrated strong antibacterial activity against bacterial species (Table 8).

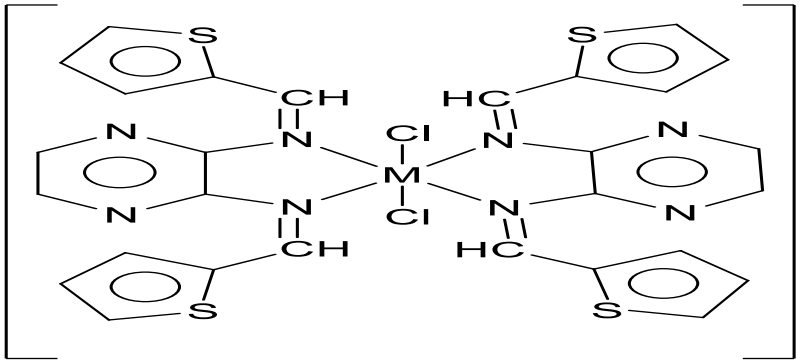


Figure 2: The proposed general structure of the complexes where M is Co, Ni or Cu

Conclusion

A Schiff base derived from 2-thiophene carbaldehyde and o-phenylenediamine, and its Co(II), Ni(II) and Cu(II) complexes, were synthesised and characterised by FT-IR, CHN analysis, molar conductivity, magnetic susceptibility, and solubility and thermal measurements. The ν(C=N) band shifted from 1622 cm^{-1} in the free ligand to 1611–1659 cm^{-1} in the complexes. New bands assigned to ν(M–N) (418–481 cm^{-1}) and ν(M–Cl) (538–577 cm^{-1}) appeared, indicating coordination through the azomethine nitrogen. Low molar conductance (6–13 Ω^{-1} cm^2 mol^{-1} in DMSO) indicated non-electrolytic behaviour, consistent with coordinated chloride. The analytical data were consistent with $[ML_2Cl_2]$ stoichiometry. Magnetic moments (2.24–3.12 BM) suggested an octahedral geometry, which still needs confirmation by electronic spectra. The complexes decomposed at 155–196 °C, indicating moderate thermal stability. In vitro screening showed moderate antibacterial (inhibition zones up to 14 mm) and antifungal (up to 15 mm) activity at the highest concentrations tested, lower than amoxicillin and nystatin.

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