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Synthesis and Antimicrobial Efficacies of Bidentate Schiff Metal Complexes Derived From Terephthalaldehyde and 2-Aminophenol

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ABSTRACT: The objective of this paper was to investigate the synthesis and antimicrobial efficacies of Bidentate Schiff metal complexes of Mn (II), Co (II), Ni (II), Cu (II) and Zn (II) complexes derived from equimolar ratio of terephthalaldehyde (TPA) and 2-aminophenol (AMP) The ligand and complexes were characterized using physicochemical techniques - melting point, solubility, conductivity and spectroscopic parameters - FTIR and UV-Visible. FTIR spectrum of the Schiff base ligand showed absorption bands at 1671 cm^{-1} which shifted to 1689 - 1698 cm^{-1} in the spectra of metal complexes indicating of coordination of metal ion to the ligand through azomethine N-atom. In the same vein, the sharp absorption bands at 3323 - 3366 cm^{-1} in the spectra of metal complexes showed coordinated water of crystallization. Square planar geometry was proposed for all metal complexes based on the available spectroscopic data obtained. The antimicrobial results showed that both the ligands and the metal complexes inhibited bacterial and fungi growth, more effective than some of the standard antimicrobial drugs used. The Cu (II) complex exhibited encouraging antibacterial activities against all strains of microbes, the ligand, Mn (II), and Co (II) metal complexes were only active against *staphylococcus aureus*, *Escherichia Coli*, and *Candida albicans* while Ni (II) complex showed no activity against both bacteria and fungi strains. MIC (Mininum Inhibitory Concentration) of Manganese complex is 20 mg/ml while MIC that prevents the visible growth of the tested organisms for Schiff base ligand, Copper and Cobalt complexes is 40 mg/ml.

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Schiff bases are organic compounds characterized by the general formula $R_2C=NR'$, where nitrogen atom linked to an aryl or alkyl group, but not hydrogen (Kostova, 2013), usually synthesized through the condensation reaction of primary amines and carbonyl compounds. These compounds have gained popularity in recent years due to their ability to bind to various metal ions forming coordination complexes exhibiting both medical and non-medical properties (El-Barasi *et al.*, 2023, Liu, 2018). As a result of their stability under various oxidative/reductive conditions and through structural modifications they demonstrated diverse chemical, optical and magnetic properties (Al Zoubi, 2016, Cozzi, 2004). The interactions between these donor ligands and metal ions create complexes with differing geometries, which recent literatures have shown to be potentially more active biologically

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(Siva, 2011). Hence, transition metal chelation of these Lewis bases can significantly influence the bioactivities of these ligands. This has led to the synthesis of various complexes with various properties, such as antibacterial, antifungal, anticancer, antioxidant, anti-inflammatory, antimalarial and antiviral activities (Afradi, 2016, Bader, 2010). Besides, they also serve as catalysts in reactions such as polymerization reactions, reduction of thionyl chloride and oxidation of organic compounds (Lashanizadegan and Jamshidbeigi, 2011), possess propensity to be used in areas such as electrochemistry, bioinorganic, catalysis, separation processes, metallic deactivators and environmental chemistry (Ziyad *et al*., 2011). The incessant resistance of micro-organisms against certain antimicrobial drugs and alarming increase in multidrug-resistant bacteria and fungi prompted the initiation of development of transition metal-based agents as promising pharmacological application that can offer unique therapeutic alternatives. (Ibrahim *et al.*, 2021; Sadi *et al.,* 2017). In recent times, a lot of research works have been reported on complexes of Schiff base containing 2-aminophenol and/or terephthalaldehyde (Suleiman, 2023; Savitha and Vedanayaki, 2021; Jabbi *et al.,* 2020; Shaygan *et al.*, 2018, Hassan, 2013). Shaygan *et al.* (2018) reported a Schiff base derived from ortho-substituted anilines and terephthalaldehyde with Cobalt (II) metal ion. Saranyan *et al.* (2020) reported the synthesis of tetradentate Schiff base complexes of Cu (II), Co (II), and Ni (II) through the condensation of 2-aminophenol/o-phenylenediamine and terephthalaldehyde. The antimicrobial studies of the Schiff base ligands and their complexes against selected bacteria and fungi strains revealed that the transition metal complexes possess good antibacterial and antifungal activities than the Schiff base ligands.

Here we reported the synthesis and antimicrobial efficacies of bidentate Schiff metal complexes of Mn (II), Co (II), Ni (II), Cu (II) and Zn (II) obtained by condensing terephthalaldehyde and 2-aminophenol in ethanolic medium. The Schiff base ligand was synthesized by ordinary cold stirring method as against the refluxing method obtainable in the available literatures.

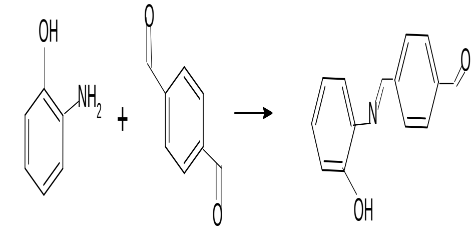
MATERIALS AND METHODS

Physical measurements: All reagents are of analytical grade and were used as supplied by Sigma-Aldrich, USA without further purification. Melting points were measured using a Stuart SMP110 melting point apparatus. The FTIR spectra was obtained using the Thermo scientific Nicolet IS-5 FTIR spectrophotometer using KBr pellet in the range of 400 – 4000 cm^{-1}. Conductance was recorded using LIDA instrument model DDS-307 conductivity meter at 32 ^oC. The UV-Visible spectra of the ligand and their complexes were recorded on Beckman Coulter Du730 UV/visible spectrophotometer available at Chemistry Laboratory, University of Ilorin, Ilorin, Kwara State, Nigeria. Pure clinical cultures of *Staphylococcus aureus* (SA1 and SA2), *Escherichia coli* (EC1 and EC2), *Klebsiella oxytoca* (KO1 and KO2), *Citrobacter freundii* (CF1 and CF2), *Proteus mirabilis* (PM1 and PM2), *Salmonella typhi* (ST1) and *Candida albicans* (CA1 and CA2) as well as typed isolates of *S. aureus* (ATCC 29213), *E. coli* (ATCC 25922), *S. typhi* (ATCC 6539) and *C. albicans* (ATCC 10231) were collected from the Laboratory Unit of the Department of Pharmaceutical Microbiology and Biotechnology, Faculty of Pharmaceutical Sciences, University of Ilorin, Kwara State.

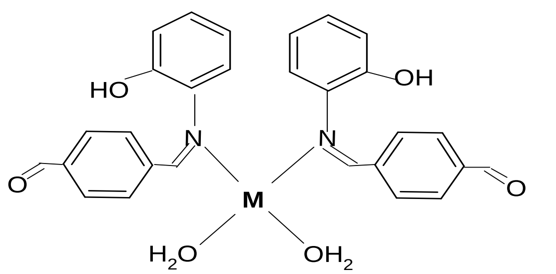
Synthesis of Schiff Base 4-((2-hydroxyphenylimino) methyl) benzaldehyde: Condensation of terephthalaldehyde with 2-aminophenol was done in 1:1 molar ratio. (2.18g, 2 mmol) 2-aminophenol (AMP) and (2.68 g, 2 mmol) terephthalaldehyde (TPA) each dissolved in 15 ml of ethanol was added together while stirring. The mixture was stirred continuously for 2 hours on the magnetic stirrer and light brownish solution obtained was allowed to evaporate slowly at room temperature. A light brown precipitate formed was then separated out by filtration, washed twice with ethanol followed by recrystallization in methanol and dried in a desiccator over silica gel. The analytical data of Schiff base (SB) ligand is contained in Table 1 and the proposed reaction scheme for the synthesis of Schiff base is presented in scheme 1.

Synthesis of metal (II) Complexes: The preparation of Schiff metal complexes followed 2:1 molar ratio of the synthesized Schiff base and the respective metal chloride salts.

1 mmol of the respective metal chloride salts, $MCl_2.XH_2O$ ($MnCl_2.4H_2O$, $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$ and $ZnCl_2.2H_2O$) dissolved in 15 ml of ethanol was added to the ethanolic solution of the preformed Schiff base ligand, SB (0.4508 g, 2 mmol). The mixture was refluxed for 3 hours. The colored precipitate formed in each case was filtered, washed with ethanol and dried in desiccator over silica gel. The analytical data is presented in Table 1 and the proposed reaction is schematically depicted in scheme 2.



Scheme 1: Proposed reaction scheme for the synthesis of Schiff base Ligand, 4-((2-hydroxyphenyliminomethyl) benzaldehyde (SB)



Scheme 2: Proposed reaction scheme for the synthesis of metal complexes
Where M = Mn (II), Co (II), Ni (II), Cu (II), or Zn (II).

RESULTS AND DISCUSSION

The physical and analytical data of the compounds are presented in Table 1. The terephthalaldehyde and 2-aminophenol yielded the ligand with percentage yield of 61 % and a sharp melting point of 213-214 ℃ indicating its purity and relative thermal stability. The formation and purity of Schiff base metal complexes can be inferred from differences in the colour formation and sharp melting point respectively. The yields have range from 54-76 %. The conductivity of the ligand and its metal (II) complexes in DMF, suggested the non-electrolytic nature of the products expect Mn complex. (Mathur 2014). The solubility test data contained in Table 2 showed that both the Schiff base and its metal complexes are practically insoluble in distilled water, soluble in DMSO, ethyl acetate and partially soluble in other solvents. The major FTIR absorption peaks of the ligand and its metal (II) complexes are presented Table 3. The solid-state FTIR spectrum of the SB showed absorption bands at 1671 cm^{-1} assignable to (C=N) stretching vibration band. The band shifted to 1689 - 1698 cm^{-1} in the spectra of metal complexes signifying a possible indication of coordination of metal ion to the ligand through azomethine group (Suleiman *et al.*, 2023; Saranyan *et al.,* 2020; Turan *et al.,* 2019).The coordination of azomethine N-atom to the metal ions can be buttressed by absorption band at 1367 cm^{-1} on the spectrum of the ligand attributed to v(C – N) which undergone bathochromic shift (1380 -1383 cm^{-1}) in the spectra of metal complexes as a result of complexation (Ali *et al.,* 2015; Raman *et al.,* 2004). The sharp absorption bands at 3323 - 3366 cm^{-1} and 924 – 967 cm-[1] in the spectra of metal complexes can be ascribed to $v(H_2O)$ and $\delta(H_2O)$ respectively indicating the presence of coordinated water molecules in the coordinating sphere (Ali *et al.,* 2015). The sharp band at 3044 cm^{-1} which is prominent in the spectra of the ligand and the metal complexes showed that the phenolic hydroxy group did not involve in complexation.

The non-involvement of phenolic-OH in complexation is supported by absorption bands at 1242 and 1287 cm^{-1} due to v(C–O) which remain constant in the spectra of the ligand besides its metal complexes. In the same vein, the absorption peaks at 1776 cm^{-1} (Otuokere *et al.,*2022) assignable to carbonyl aldehyde v(CHO) which is observable in the spectra of the ligand as well as that of the metal complexes confirmed that only one aldehyde of terephthalaldehyde condensed with the 2-aminophenol in the formation of the Schiff base ligand. This further attest to the 1:1 (terephthalaldehyde:2-aminophenol) formulation of the Schiff base ligand. The bands which confirm M – L coordination observed in the regions 516 - 518 cm^{-1} and 448 - 449 cm^{-1} correspond to the v(M— N) and v(M—O) bands respectively. (Suleiman *et al.*, 2023; Osunniran *et al.*, 2022; Saritha and Metilda, 2019). The UV-visible absorption bands and their assignment for the ligands and metal complexes are contained in Table 4. Schiff base ligand showed a strong band in the regions 250 and 300 nm which correspond to π→π* (C = C) of the aromatic benzene and n→π* non-bonding electron on the Nitrogen atom of azomethine groups respectively.

Table 1: Analytical Data and Physical Properties of Schiff Base and its Metal (II) Complexes.

SB and metal complexes	Molecular formular	Mol. Mass (g/mol)	Colour	Yield (g)	Yield (%)	Melting pt. temp. (°C)	Conductivity (μs/cm)
Ligand (SB)	$C_{14}H_{11}NO_2$	225.24	Yellowish Brown	1.371	61	213 -214	614
$Mn(SB)_2.2H_2O$	$MnC_{28}H_{28}N_2O_6$	543.72	Light Brown	0.3826	76	167 -169	1097
$Co(SB)_2.2H_2O$	$CoC_{28}H_{28}N_2O_6$	547.71	Brown	0.3602	71	180 – 182	609
$Ni(SB)_2.2H_2O$	$NiC_{28}H_{28}N_2O_6$	547.47	Deep Brown	0.2795	54	179 -181	615
$Cu(SB)_2.2H_2O$	$CuC_{28}H_{28}N_2O_6$	552.32	Dark	0.3687	72	>300	610
$Zn(SB)_2.2H_2O$	$ZnC_{28}H_{28}N_2O_6$	554.16	Dark	0.2789	55	190 - 191	663

Table 2: Solubility Data of Schiff Base and its Metal (II) Complexes.

Solvent/ Compound	Methanol		Ethanol		Ethyl Acetate		DMSO		Distilled water		Chloroform		Acetonitrile		N-Hexane		Toluene	
	C	**H**	**C**	**H**	**C**	**H**	**C**	**H**	**C**	**H**	**C**	**H**	**C**	**H**	**C**	**H**	**C**	**H**
Ligand (SB)	PS	S	PS	S	S	S	S	S	IS	IS	S	S	S	S	PS	PS	PS	S
$Mn(SB)_2.2H_2O$	PS	S	PS	S	S	S	S	S	IS	IS	S	S	PS	S	PS	PS	PS	PS
$Co(SB)_2.2H_2O$	IS	PS	IS	PS	S	S	PS	S	IS	IS	S	S	PS	S	PS	PS	PS	S
$Ni(SB)_2.2H_2O$	PS	S	PS	S	S	S	S	S	IS	IS	PS	S	S	S	PS	PS	S	S
$Cu(SB)_2.2H_2O$	PS	S	PS	S	PS	S	S	S	IS	IS	S	S	PS	S	IS	PS	S	S
$Zn(SB)_2.2H_2O$	IS	PS	IS	S	S	S	S	S	IS	IS	PS	S	IS	S	IS	PS	PS	S

H = hot, **C** = cold, **PS** = partially soluble, **IS** = insoluble and **S** = soluble.

Table 3: Characteristics FTIR Absorption Bands (cm^{-1}) of the Schiff Base (SB) and its Metal Complexes

Samples	***v*(C=N)**	***v*(C-N)**	***v*(O-H)**	***v*(H_2O)**	**δ(H_2O)**	***v*(C=O)**	***v*(C-O)**	***v*(M-N)**	***v*(M-O)**
Ligand (SB)	1671	1367	3044	-	-	1776	1287 1242	-	-
$Mn(SB)_2.2H_2O$	1689	1380	3044	3366	924 967	1776	1287	516	448
$Co(SB)_2.2H_2O$	1695	1380	3044	3366	924 966	1776	1287 1242	517	448
$Ni(SB)_2.2H_2O$	1695	1380	3044	3366	924 966	1775	1287 1242	517	448
$Cu(SB)_2.2H_2O$	1698	1380	3044	3367	924 966	1776	1288 1242	518	448
$Zn(SB)_2.2H_2O$	1689	1383	3041	3329	927 963	1776	1288 1244	518	449

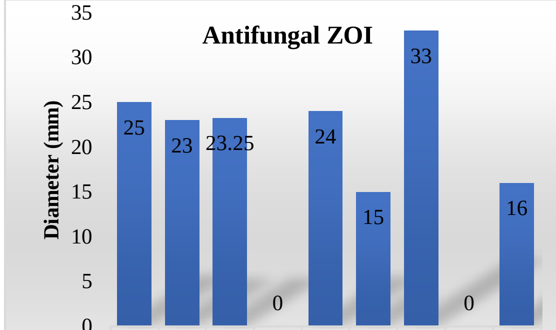
Table 4: UV-Visible Spectroscopy Results

COMPOUNDS	**Λmax (nm) (DMSO)**	**Λmax (cm^{-1}) (DMSO)**	**Assignment**
Ligand (SB)	250	40,000	$\pi-\pi^*$
	300	33,333	$n-\pi^*$
$Mn(SB)_2.2H_2O$	302	33,113	$\pi-\pi^*$
	380	26,316	$n-\pi^*$
	440	22,727	d-d transition
	510	19,608	d-d transition
$Co(SB)_2.2H_2O$	280	35,714	$\pi-\pi^*$
	350	28,571	$n-\pi^*$
	440	22,727	d-d transition
	510	19,608	d-d transition
$Ni(SB)_2.2H_2O$	320	31,250	$\pi-\pi^*$
	380	26,316	$n-\pi^*$
	440	22,727	d-d transition
	540	18,519	d-d transition
$Cu(SB)_2.2H_2O$	280	35,714	$\pi-\pi^*$
	360	27,778	$n-\pi^*$
	410	24,390	d-d transition
	560	17,857	d-d transition
$Zn(SB)_2.2H_2O$	220	45,455	$\pi-\pi^*$
	300	33,333	$n-\pi^*$
	380	26,316	$n-\pi^*$
	440	22,727	Charge-transfer
	480	20,833	Charge-transfer

The bathochromic shifting of the bands in the electronic spectra of the metal complex, indicated the coordination of the azomethine nitrogen of C= N to the metal center. The metal complexes of Mn (II), Co (II), Ni (II), Cu (II) and Zn (II) showed a non-ligand wide band in the visible region at 440-540 nm which could be attributed to d – d transition. (Turan *et al.,* 2019). The bands at 440 and 480 nm in Zn complexes are assigned to ligand-to-metal charge transfer as d-d transition is not possible since there is no empty or partially filled d-orbital in Zn ion.

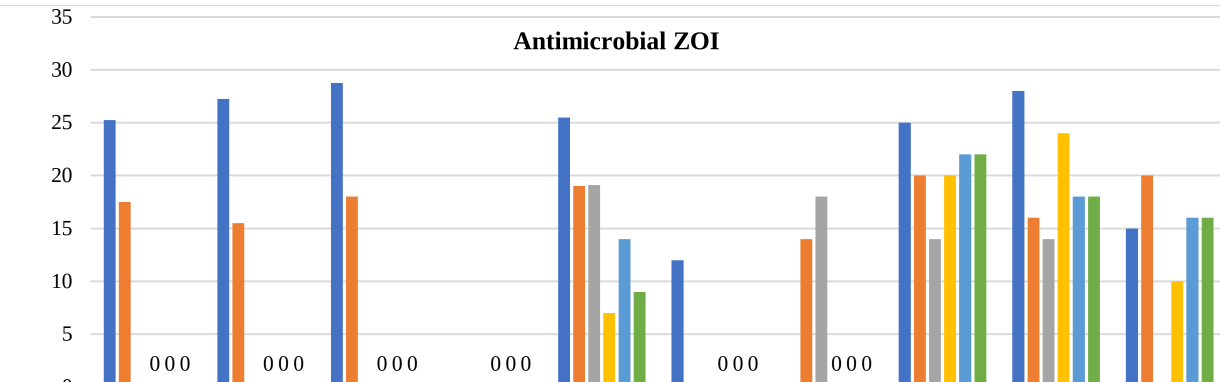
Antimicrobial Studies: ZOI/MIC (Zone of Inhibition/Minimum Inhibitory Concentration): Paper disk diffusion method was used for the assay according to the methods described by (Ogba *et al.*, 2017) with slight modifications from the Kirby-Bauer diffusion method described by (Balouiri *et al.,* 2016). The SB and its metal complexes showed higher zones of inhibition than the standard drugs, Fluconazole (25µg) and Flucytocin(10µg) except for Nystatin (100 units) which have higher zone of inhibition, when screened against the fungus *Candida albican*. (Figure 1). Likewise, the screening of the SB and its metal complexes showed very promising zone of inhibition against some selected clinical cultures of *Staphylococcus aureus (SA)*, *Escherichia coli* (EC), *Klebsiella oxytoca* (KO), *Citrobacter freundii* (CF), *Proteus mirabilis* (PM). The antibacterial ZOI for the strains mentioned were compared with standard drugs; Gentamicin (10µg), Imipenem (10 µg), Ofloxacin (5 µg) and Amoxicillin clavulanic (30µg). (Figure 2). MIC (Minimum Inhibitory Concentration) screenings were done at serial concentrations of 20, 40 and 80 mg/mL of the SB and its metal complexes against *Staphylococcus aureus, Escherichia coli, and Candida albican.*

The MIC results revealed that the lowest concentration of $Mn(SB)_2.2H_2O$, $Zn(SB)_2.2H_2O$ and $Cu(SB)_2.2H_2O$, $Co(SB)_2.2H_2O$, SB, that can prevent the visible growth of the tested organisms is 20 mg/ml and 40 mg/ml respectively as contained in Figure 3. This shows that Mn and Zn complexes are more efficacious than others. In the same vein, the MCB (Minimum Bactericidal Concentration) values of Mn (II), Zn (II) and Co (II), Cu (II) complexes against *Candida albican, Escherichia coli* are 40 mg/ml and 80 mg/ml respectively as depicted in Figure 3.



	SB	Mn L2	Co L2	NiL 2	Cu L2	ZnL 2	Nystatin	Fluconazole	Flucytocin
CA	25	23	23.3	0	24	15	33	0	16

Fig. 1: Antifungal Zone of inhibition (mm) of the SB, its metal complexes and some selected drugs used as control.
Key: - *Candida albican* (CA)



	SB	MnL2	CoL2	NiL2	CuL2	ZnL2	CN	IMP	OXF	AMC
SA	25.25	27.25	28.75	0	25.5	12	0	25	28	15
EC	17.5	15.5	18	0	19	0	14	20	16	20
ST	0	0	0	0	19.1	0	18	14	14	0
KO	0	0	0	0	7	0	0	20	24	10
PM	0	0	0	0	14	0	0	22	18	16
CF	0	0	0	0	9	0	0	22	18	16

Fig. 2: Antibacterial Zone of inhibition (mm) of the SB, its metal complexes and some selected drugs used as control.
Keys: - *Staphylococcus aureus* (SA); - *Klebsiella oxytoca* (KO); -*Escherichia coli* (EC) -*Proteus mirabilis* (PM 2); -*Salmonella typhi* (ST); -*Citrobacter freundii* (CF); *OXF – Ofloxacin (5 µg) *IMP – Imipenem (10 µg) *CN- Gentamicin (10 µg) *AMC – Amoxicillin clavulanic (30 µg)

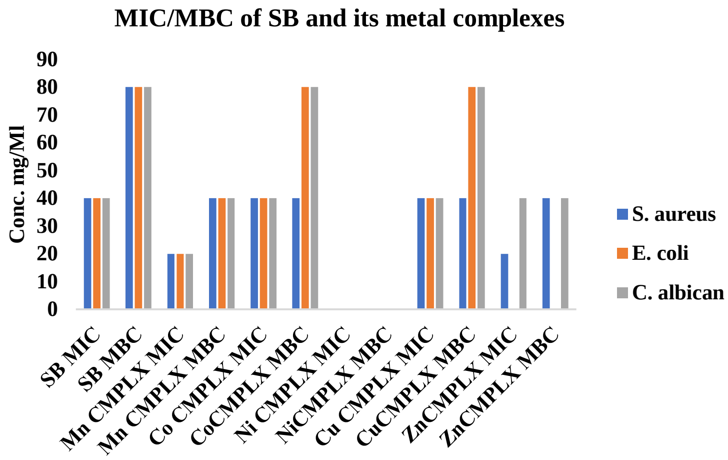


Fig. 3: MIC/MBC of the SB and its metal complexes

Key: *SB (Schiff Base); MIC (Minimum Inhibitory Concentration); MBC (Minimum Bactericidal Concentration) CMPLX (Complex)*

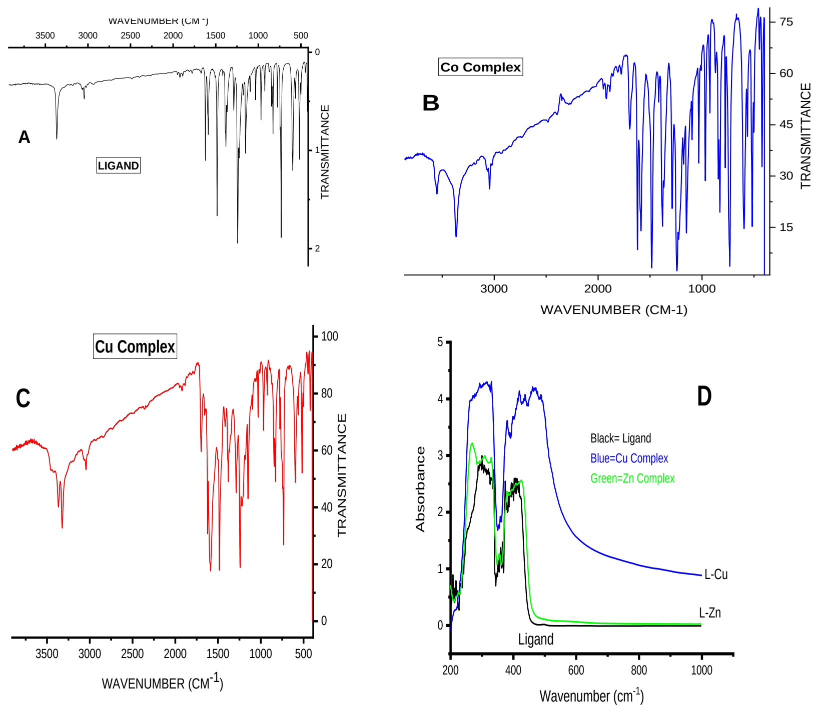


Fig. 4: FTIR spectra of the ligand, Co and Cu complexes (A, B and C) and the UV-vis spectra of the ligand, Cu and Zn complexes (D).

Conclusion: Bidentate Schiff base and Mn (II), Co (II), Ni (II), Cu (II), or Zn (II) complexes have been successfully synthesized from the condensation reaction of terephthalaldehyde and 2-aminophenol by cold stirring, characterized and antimicrobial efficacy determined. The spectroscopic data available revealed that metal ions bonded to the ligand through azomethine group and square planar geometry formulated as $[M(SB)_2(H_2O)_2]$ is proposed for the metal complexes. The Cu (II) complex exhibited very high antibacterial activities against all strains of bacteria. The ligand and Mn (II), Co (II) and Zn (II) metal complexes were only active against *staphylococcus aureus*, *Escherichia Coli* and *Candida Albicans* but not against *Klebsiella oxytoca*, *Citrobacter Freundii*, *Salmonella typhi and Proteus mirabilis*. Therefore, the ligand (SB) and its Cu (II), Mn (II), Co (II), and Zn (II) complexes are recommended for further studies that may lead to the discovery of potent antimicrobial agents.

Declaration of Conflict of Interest: The authors declare no conflict of interest.

Data Availability: Data are available upon request from the corresponding author.

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