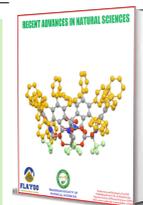


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One-pot synthesis and antimicrobial efficacy of polymeric schiff base metal complexes derived from o-phenylenediamine and terephthalaldehyde

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ABSTRACT

The antimicrobial efficacies of Schiff base Polymeric Ligand (SBPL) and Polymeric Schiff base Metal Complexes (PSBMCs) have been explored. Schiff base Polymeric Ligand (SBPL) and Polymeric Schiff base Metal Complexes (PSBMCs) derived from polycondensation of terephthalaldehyde, o-phenylenediamine and metal ions (M–L, Al³⁺, Cu²⁺, Zn²⁺, Fe³⁺) were synthesized in ethanol at room temperature through one-pot synthesis. The PSBL and PSBMCs synthesized were characterized using spectroscopic techniques: FT-IR (Fourier Transform Infrared) and Uv-vis (Ultraviolet visible). The spectroscopic data revealed that the azomethine functional group is involved in the complexation of metal ions with PSBL forming PSBMCs and geometry of PSBMCs is suggestive to be octahedral. The antibacterial and antifungi efficacies of SBPL and PSBMCs were examined on *Staphylococcus aureus*, *Escherichia coli*, *Aspergillus niger* and *Aspergillus flavus* employing the agar well diffusion method, Penicillin and omeprazole served as standard drug for antibacterial and antifungal activity respectively. The PSBMCs displayed promising activities in comparison with starting compound (SBPL). The Cu-L was found to be most potent antimicrobial agent against all tested bacterial and fungal.

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1. INTRODUCTION

Schiff bases are conceived to have imine (–CH=N–) functional group in which the nitrogen is attached to various groups such as aryl, alkyl, heterocyclic, or cycloalkyl, but not hydrogen [1–3]. A condensation reaction between a carbonyl compound (aldehyde or ketone) and a primary amine, typically in the presence of a catalyst like glacial acetic acid and favourable reaction con-

ditions, produces Schiff bases [4–7]. The imine group plays a vital role in their biological and chemical activities besides usefulness in polymer, corrosion and coordination chemistry [8–10]. Polymers with a system of conjugated –C=C– and –C=N– bonds in their main chain have been attracting the recognition of researchers due to their usefulness in diverse areas. Notable of interest are Schiff base polymers depicted by the presence of HC=N networking, synthesized by the polycondensation of diamines to series of dicarbonyl compounds [11]. Polymer-metal complexes are composed of metal ions coordinating to the polymer ligand by native bonds. Schiff base polymers and their

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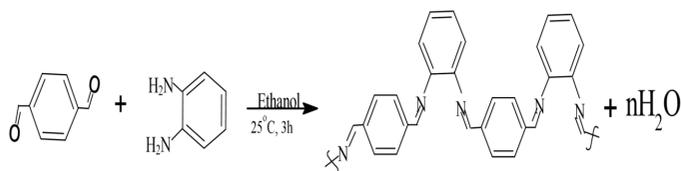


Figure 1. Proposed scheme for synthesis of polymeric Ligand (SBPL).

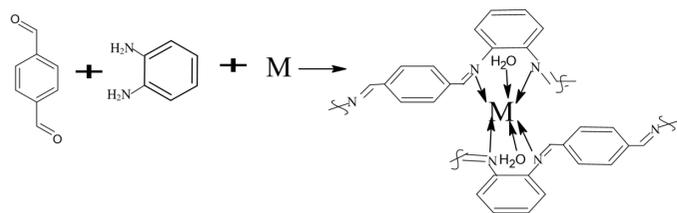


Figure 2. Proposed scheme for synthesis of polymeric metal complexes (PS-BMCs). Note: M= Al^{3+} , Cu^{2+} , Zn^{2+} , Fe^{3+} .

Table 1. Analytical data and physical properties of SBPL and PSBMCs

Compounds	Colour	Yield (g)	%Yield	Melting point ($^{\circ}\text{C}$)
L (SBPL)	Pale yellow	2.630	60	>300
Ni-L	Dark yellow	1.440	57	160–161
Al-L	Dark orange	1.585	63	244–246
Fe-L	Orange	1.786	66	281–282
Zn-L	Yellow	3.133	73	288–289
Cu-L	Grey	1.620	65	>300

metal complexes have demonstrated high mechanical strength, electrochemical and nonlinear optical properties [12] and have found wide applicability as pigment, dye, catalyst, intermediate in organic synthesis, a supercapacitor; exhibiting wide range of biological usefulness such as anti-fungal, anti-microbial, antimalaria, anti-proliferation, anti-inflammatory, anticancer, antiviral etc [2, 4, 7, 8, 13–15]. Many experimental studies have substantiated that dispensing the structurally remodeled medications, especially metal complexes is capable of demonstrating enhanced pharmacological and toxicological attributes besides equally displays better distinguishing characteristics [15]. Majority of transition metals have exhibited the capability to form stabilize complexes, hence demonstrating greater antimicrobial efficacy. This is as a result of their propensity to bind to nucleophilic (electron-rich) substances like DNA and proteins [16, 17]. Generally, results of array of researches have proved that Schiff base metal complexes are conceived to be more striking against microbes comparing to the parent compound alone [18–21]. The resistance to commercially accessible antimicrobial is alarmingly increasing; therefore, a need for urgent attention for newly antimicrobial agents to fight the resistance insurgence in pathogenic microbes [22].

Tetradentately coordinated Schiff base complexes of Cu (II), Co (II), and Ni (II) derived from 2-aminophenol/o-phenylenediamine and terephthalaldehyde had been reported and their antimicrobial potencies explored against chosen bacteria and fungi strains. The resultant outcomes indicated that the complexes of transition metal manifested improved antibacterial and antifungal activities [12]. Polymeric Schiff base Metal Complex using as Electrode for High-performance supercapacitor derived

from m-phenylenediamine and terephthalaldehyde had been synthesized and documented. The findings showed that the electrochemical efficiency of the metal complexes was substantially improved comparing to the ligand, specifically when the current density is 0.5 Ag^{-1} and Al-L displayed a better specific capacitance of 608.6 Fg^{-1} [23].

In this paper, Schiff base polymer Ligand (SBPL) and its corresponding metal complexes (Cu^{2+} , Ni^{2+} , Zn^{2+} , Fe^{3+} and Al^{3+}) have been prepared by one-pot synthesis polycondensation of o-phenylenediamine (OPDA) and Terephthalaldehyde (TPA) in ethanoic medium without any catalyst or tedious procedure. The physical properties of the polymers such as yields, solubility, and melting point and spectroscopic parameters are reported. The antimicrobial efficacy against selected bacterial and fungal (*Staphylococcus aureus*, *Escherichia coli*, *Aspergillus niger* and *Aspergillus flavus*) are studied as well.

2. EXPERIMENTAL

2.1. MATERIALS AND METHODS

All the reagents used were obtained from Sigma Aldrich and used without further purification. Melting point of the PSBL as well as that of PSBMCs were ascertained with Gallenkamp melting point apparatus. FTIR measurements were registered on Thermo Scientific Nicolet IS-5 with KBr in the range of $400\text{--}4000 \text{ cm}^{-1}$. The antimicrobial efficacy of polymeric Schiff base ligand and its metal complexes were sampled *in vitro* against bacteria and fungal such as *Staphylococcus aureus*, *Escherichia coli*, *Aspergillus flavus* and *Aspergillus niger* employing agar well diffusion method, Penicillin and Omeprazole as control for bacteria and fungi respectively, carried out at Microbiology department of Nigeria Stored Product Research Institute, Ilorin.

2.2. SYNTHESIS OF SCHIFF BASES POLYMERIC LIGAND (SBPL)

0.02 mol of OPDA (2.163 g) and TPA (2.683 g) each dissolved in 40 ml of ethanol were mixed together at ordinary condition employing magnetic stirring. The mixture of OPDA and TPA was further agitated continuously for 3 hours. The pale yellow precipitate of the ligand (SBPL) formed was harvested by filtration, washed thoroughly with ethanol and dried in a desiccator over silica gel. The Thin layer Chromatography (TLC) was used to monitor reaction's progress. The analytical data and proposed scheme of reaction are presented in Table 1 and Figure 1, respectively.

2.3. SYNTHESIS OF POLYMERIC SCHIFF BASE METAL COMPLEXES (PSBMCs)

One-pot synthesis is employed for the synthesis of PSBMCs. 0.02 mol of each hydrated nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 7.503 g), ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 5.950 g), ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 8.080 g), ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, 4.832 g) and ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 5.816 g) dissolved in ethanol, the solution was labeled as solution B. Thereafter, the B solution was slowly poured into the mixture solutions PSBL before precipitation appeared. The mixture further stirred for 3 hours at room temperature. Ultimately, Al-L, Zn-L, Cu-L, Ni-L, and Fe-L are obtained by filtration, washed and dried in a desiccator over silica gel. The purity of the product monitored

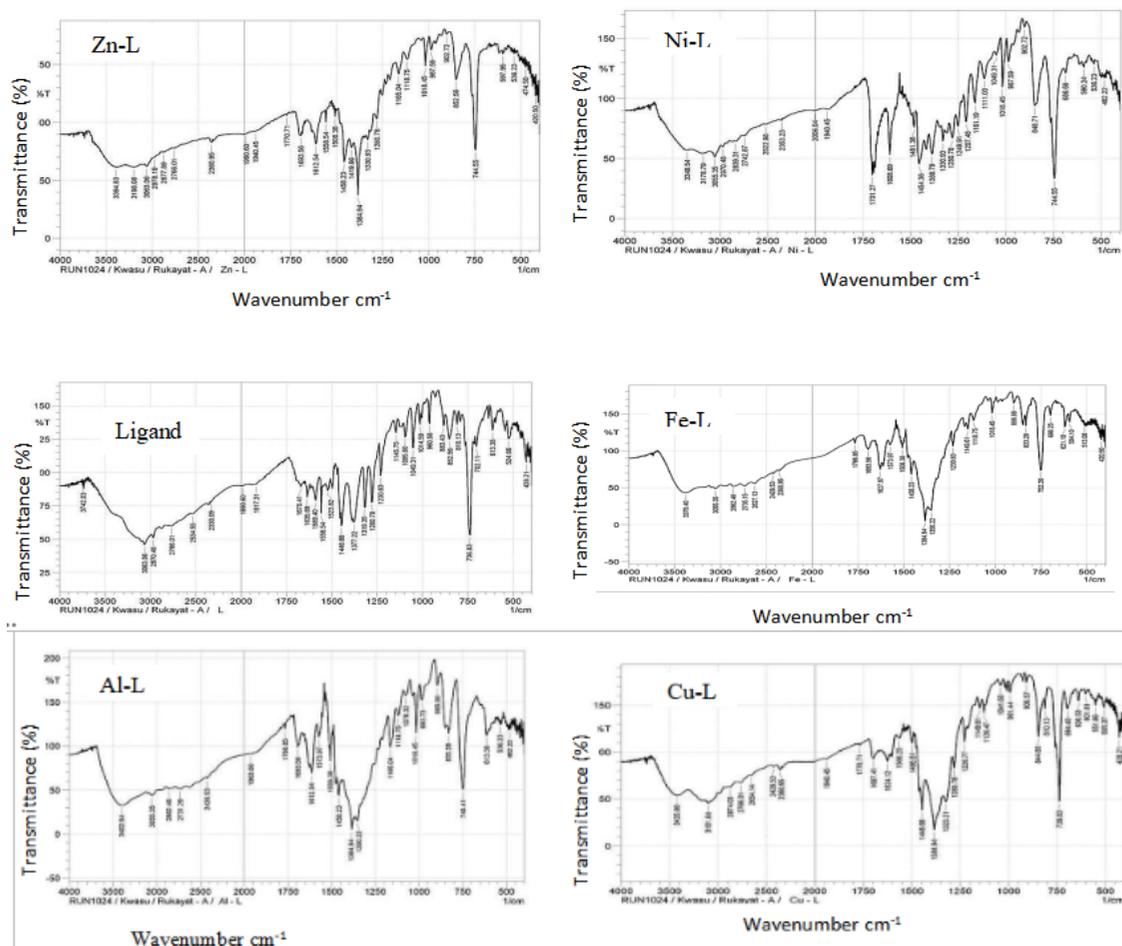


Figure 3. FTIR spectra of PSBL (L) and PSBMCs (Cu-L, Ni-L, Zn-L, Fe-L, Al-L).

Table 2. Solubility data of PSBL and PSBMCs.

Solvents/Compounds	Acetone		Methanol		DMSO		Ethanol		Distilled water		DMF		Acetonitrile	
	C	H	C	H	C	H	C	H	C	H	C	H	C	H
L (SBPL)	IS	PS	IS	PS	PS	S	IS	IS	IS	IS	S	S	IS	IS
Ni-L	IS	PS	IS	PS	S	S	IS	PS	IS	IS	S	S	IS	PS
Cu-L	IS	PS	IS	PS	S	S	IS	PS	IS	IS	S	S	IS	IS
Al-L	IS	IS	IS	PS	S	S	IS	IS	IS	IS	S	S	IS	IS
Fe-L	IS	IS	IS	IS	S	S	IS	IS	IS	IS	S	S	IS	IS
Zn-L	IS	IS	IS	IS	S	S	IS	IS	IS	IS	S	S	PS	PS

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

by TLC. The analytical data and proposed scheme of reaction are presented in Table 1 and Figure 2, respectively.

3. RESULTS AND DISCUSSION

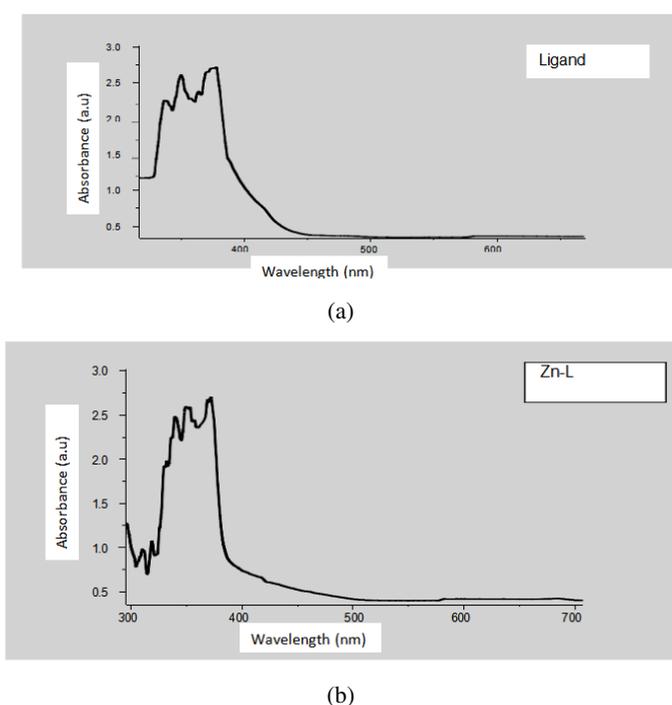
Analytical and solubility data of all synthesized polymers are given in Table 1 and 2, respectively, showing appreciable yield. The colour of the synthesized PSBMCs ranges from yellow, orange-grey, to dark coloration. The change in colour is a possible indication for complexation, as transition metals commonly form coloured complexes [24–26]. In the same vein, the melting ranges of PSBMCs attest to their purity as well as the formation of new compounds entirely different from the starting materials. The solubility test carried out using both polar and

non-polar solvents revealed that both the Schiff base polymer and its metal complexes are insoluble or partially soluble in distilled water, methanol, ethanol, acetone and acetonitrile but, soluble in DMSO and DMF due to the fact that they are polar aprotic solvents with high dielectric constant [12, 27].

The characteristics FTIR absorption peak as well as FTIR spectra of the PSBL and PSBMCs are summarized in Table 3 and Figure 3, respectively. The sharp absorption band discerned in the SBPL spectrum at 1635 cm^{-1} assignable to azomethine, $\nu(\text{C}=\text{N})$ group undergo bathochromic shift in the spectra of PSBMCs to $1627\text{--}1605\text{ cm}^{-1}$. This proposes the complexation of metal ions to the SBPL by nitrogen atom of azomethine group. This is further supported by the absorption wavenumber at 1319

Table 3. Characteristics FTIR absorption bands (cm^{-1}) of the SBPL and PSBMCs.

Compounds	$\nu(\text{C}=\text{N})$	$\nu(\text{C}-\text{C})$	$\nu(\text{H}_2\text{O})$	$\delta(\text{H}_2\text{O})$	$\nu(\text{C}-\text{H})$	$\nu(\text{C}-\text{N})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$
Ligand (PSBL)	1635	1446	-	-	2970 2766	1319	-	-
Ni-L	1605	1454	3348	849	2970 2766	1330	536	482
Cu-L	1624	1446	3425	845	2874 2766	1323	551	428
Al-L	1612	1458	3402	895	2863 2731	1350	536	482
Fe-L	1627	1458	3379		2862 2735	1350	513	474
Zn-L	1612	1458	3394	754	2978	1330	536	474

**Figure 4. UV spectra of ligand and Zn-L.**

cm^{-1} ascribable to $\nu(\text{C}-\text{N})$ which moved to higher frequency in the spectra of PSBMCs [28, 29]. The broaden band found in the spectrum of the polymeric Schiff ligand at 2970 and 2766 cm^{-1} due to symmetric and asymmetric stretching frequency of C-H bond which shifted in the spectra of the polymeric metal complexes owing to complexation [28, 30]. The broadly absorption bands ranging from 3425–3348 cm^{-1} noticeable in polymeric metal complexes' spectra are suggesting the appearance of water crystallization in the coordinating entity. This is further evidenced by out of plane bands observed between 745 and 895 cm^{-1} [12, 31, 32]. New bands observed in the fingerprint region which confirm Metal-Ligand coordination found in the scale of 536–551 and 474–482 cm^{-1} in the polymeric metal complexes' spectra ascribable to $\nu(\text{M}-\text{O})$ and $\nu(\text{M}-\text{N})$ respectively [33–38].

UV-vis absorption analysis was further explore in investigating the catenated framework of polymeric Schiff base ligand with its metal complexes, as in Table 4 and Figure 4. The identi-

Table 4. UV-visible spectroscopy results of SBPL and PSBMCs.

Compounds	$\lambda_{\text{max}}(\text{nm})$ (DMF)	$\lambda_{\text{max}}(\text{cm}^{-1})$ (DMF)	Band Assignment
Ligand (L)	250	40,000	$\pi-\pi^*$
	350	28,571	$n-\pi^*$
Ni-L	270	37,037	$\pi-\pi^*$
	388	25,773	$n-\pi^*$
	399	25,063	d-d transition
Cu-L	450	22,222	d-d transition
	280	35,714	$\pi-\pi^*$
	360	27,778	$n-\pi^*$
	401	24,938	d-d transition
Al-L	520	19,231	d-d transition
	270	37,037	$\pi-\pi^*$
	398	25,126	$n-\pi^*$
Fe-L	423	23,641	LMCT
	260	38,462	$\pi-\pi^*$
	359	27,855	$n-\pi^*$
Zn-L	450	22,222	d-d transition
	580	17,241	d-d transition
	270	37,037	$\pi-\pi^*$
	361	27,701	$n-\pi^*$
	420	23,810	LMCT

Key: LMCT (Ligand to Metal Charge Transfer).

fied absorption signals around 250 and 350 nm assigned to the $\pi-\pi^*$ transition due to the C=C of benzene ring and $n-\pi^*$ transition as result of non-bonding electron of azomethine-N atom respectively [12, 23]. These transitions decoded as inter-ligand charge transfers (ILCT) shifted to higher frequency in the PSBMCs' spectra as a result of complexation [25, 39, 40]. The absorption bands at 399 nm, 450 nm (Ni); 401 nm, 520 nm (Cu); 450 nm, 580 nm (Fe) are assigned to d-d transition while 423 nm (Al) and 420 nm (Zn) resulted from Ligand to Metal Charge Transfer (LMCT) [18, 22–24].

3.1. ANTIMICROBIAL ACTIVITIES OF POLYMERIC SCHIFF BASES AND ITS METAL COMPLEXES

The antimicrobial activities' results of the prepared compounds are specified in Table 5 and graphical representation provided in Figure 5 and Plate 6 as well. The antibacterial efficacy of the polymeric Schiff base and its corresponding complexes were studied against *Staphylococcus aureus*, *Escherichia coli* and antifungal against *Aspergillus niger*, *Aspergillus flavus* in DMF solvent. DMF displayed no zone of inhibition indicating its non-participation in the activity of synthesized polymers. The poly-

Table 5. Antimicrobial activity data of synthesized polymers.

Compounds		Concentrations ($\mu\text{g/ml}$)/Zone of Inhibition (mm)				
		10	20	30	40	50
Ligand	S.aureus	1.50 \pm 0.01	1.60 \pm 0.02	1.60 \pm 0.02	1.80 \pm 0.03	2.20 \pm 0.02
	E.coli	2.00 \pm 0.02	3.50 \pm 0.02	5.00 \pm 0.02	6.10 \pm 0.02	7.00 \pm 0.01
	Aspergillus Niger	1.80 \pm 0.02	2.20 \pm 0.03	2.50 \pm 0.02	2.80 \pm 0.01	2.90 \pm 0.02
	Aspergillus flavus	1.00 \pm 0.02	1.20 \pm 0.02	1.60 \pm 0.02	2.00 \pm 0.02	2.50 \pm 0.02
Al-L	S.aureus	5.00 \pm 0.01	6.40 \pm 0.02	10.00 \pm 0.02	12.10 \pm 0.02	14.00 \pm 0.02
	E.coli	3.10 \pm 0.01	5.50 \pm 0.02	7.30 \pm 0.02	7.00 \pm 0.02	9.00 \pm 0.02
	Aspergillus Niger	2.00 \pm 0.03	2.80 \pm 0.02	3.50 \pm 0.02	5.70 \pm 0.02	7.20 \pm 0.02
	Aspergillus flavus	1.70 \pm 0.01	2.30 \pm 0.02	2.50 \pm 0.02	2.90 \pm 0.02	3.10 \pm 0.01
Cu-L	S.aureus	5.00 \pm 0.01	6.40 \pm 0.02	10.00 \pm 0.02	12.10 \pm 0.02	14.00 \pm 0.02
	E.coli	6.50 \pm 0.02	7.00 \pm 0.03	9.50 \pm 0.02	9.90 \pm 0.01	11.50 \pm 0.02
	Aspergillus Niger	4.00 \pm 0.02	6.40 \pm 0.04	7.70 \pm 0.02	10.50 \pm 0.01	15.50 \pm 0.02
	Aspergillus flavus	1.80 \pm 0.02	2.50 \pm 0.02	3.50 \pm 0.03	8.50 \pm 0.02	10.00 \pm 0.01
Fe-L	S.aureus	2.50 \pm 0.02	3.10 \pm 0.02	4.80 \pm 0.02	7.00 \pm 0.03	7.60 \pm 0.02
	E.coli	3.40 \pm 0.02	4.00 \pm 0.04	6.10 \pm 0.02	8.00 \pm 0.02	10.00 \pm 0.02
	Aspergillus Niger	2.50 \pm 0.02	3.20 \pm 0.03	4.2 \pm 0.02	6.8 \pm 0.02	8.0 \pm 0.01
	Aspergillus flavus	2.00 \pm 0.02	2.60 \pm 0.03	4.50 \pm 0.02	5.80 \pm 0.02	7.00 \pm 0.02
Ni-L	S.aureus	0.00 \pm 0.00	0.00 \pm 0.00	0.00 \pm 0.00	3.90 \pm 0.02	5.00 \pm 0.02
	E.coli	0.00 \pm 0.00	0.00 \pm 0.00	5.30 \pm 0.02	6.00 \pm 0.02	8.10 \pm 0.02
	Aspergillus Niger	3.50 \pm 0.03	5.80 \pm 0.02	6.10 \pm 0.01	7.50 \pm 0.02	9.00 \pm 0.02
	Aspergillus flavus	1.40 \pm 0.01	3.50 \pm 0.02	4.80 \pm 0.02	7.00 \pm 0.02	8.50 \pm 0.02
Zn-L	S.aureus	0 \pm 0.00	0 \pm 0.00	0 \pm 0.00	0 \pm 0.00	3.00 \pm 0.01
	E.coli	0 \pm 0.00	0 \pm 0.00	0 \pm 0.00	2.10 \pm 0.02	4.00 \pm 0.02
	Aspergillus Niger	1.50 \pm 0.02	3.00 \pm 0.03	3.30 \pm 0.02	4.60 \pm 0.02	7.00 \pm 0.02
	Aspergillus flavus	2.50 \pm 0.04	3.40 \pm 0.02	4.00 \pm 0.02	6.00 \pm 0.02	7.80 \pm 0.02
Penicillin(P)		20.50 \pm 0.05				
Omeprazole(O)		16.50 \pm 0.03				

Key: $\mu\text{g/ml}$ (microgram per millimeter).

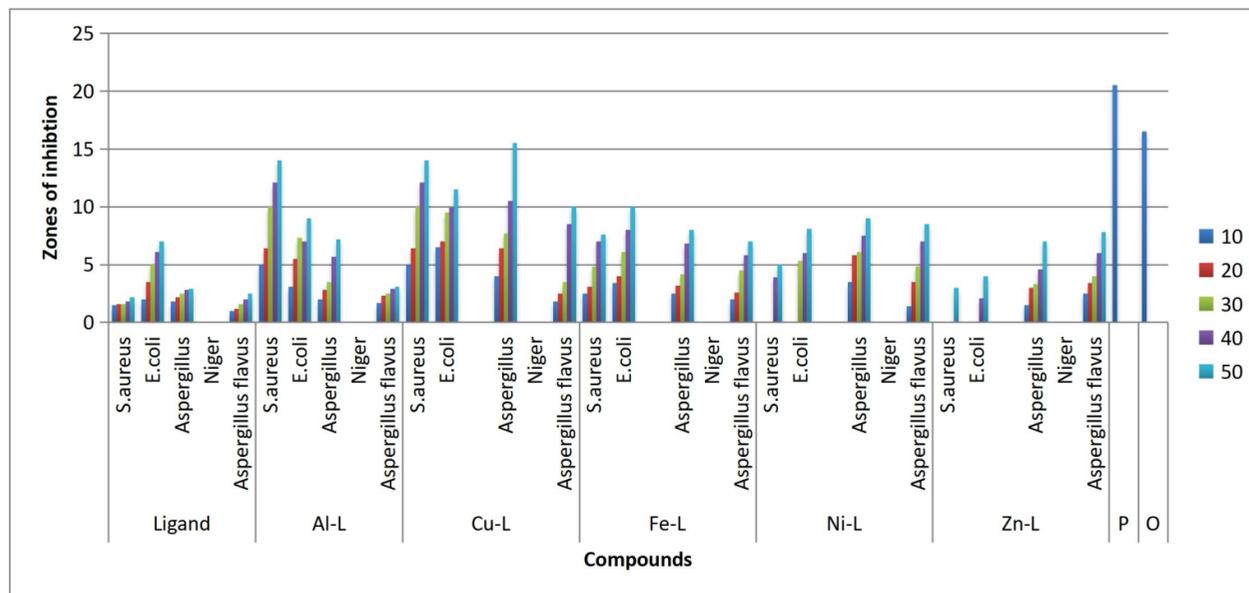


Figure 5. Graphical representation of biological activities data of synthesized polymers.

meric Schiff base and its polymeric coordination compounds demonstrated remarkable activity at high concentrations, the activity was found to be directly proportional to concentrations.

Generally, all the polymeric coordination compounds were found to be more efficacious antibacterial and antifungal agents compared to starting materials-polymer Schiff base [41, 42], but their



Figure 6. Antimicrobial plate showing the zone of inhibition.

effectiveness lower than control drugs employed. Cu-L was found to be most potent antimicrobial agent against all tested bacterial. This may have been due to the higher stability constant of Cu (II) than other transition metal used, according to the stability constant, the Cu (II) ion was made up of stronger interactions with N and O donor atoms, by which its lipophilic nature increased [20, 43]. This is also in accordance with the result reported by similar studies on Schiff base and their coordination compounds. This procedure of the antimicrobial activity of the polymeric coordination compounds can be rationalized with chelation theory which postulated that the polarity of the metal ion is demeaned to a considerable degree on account of overlap of the ligand orbital and partly sharing positive charge of the metal ion with non-bonding electron atoms. The delocalization of π -electrons over the entire chelates ring is augmented thereby reinforces the lipophilicity character of the metal complexes. Accordingly, permeation through lipid layers of microbes membrane is favoured thereby promote the obstructing of metal binding points of the enzymes of the pathogens [44–47].

4. CONCLUSION

Though the polymeric Schiff base metal complexes gotten from terephthalaldehyde and m-phenylenediamine have been synthesized but, antimicrobial studies not reported according to the available literature. In the present study, the Schiff base polymeric ligand (SBPL) and its metal complexes deduced from terephthalaldehyde and o-phenylenediamine are synthesized by one - pot reaction, characterized by physicochemical and spectroscopic techniques besides antimicrobial evaluation. The FTIR data revealed the absorption line at 1635 cm^{-1} assignable to azomethine bond of the SBPL. The band undergone change in the spectra of PSBMCs, possible indication of coordinating of metal ions to the ligand through azomethine functional group. The decomposition temperature of the PSBMCs demonstrated high values indicating high stability. The solubility test performed using various solvents established their absolute solubility in DMSO and DMF. Octahedral geometry is propounded for all PSBMCs based on spectroscopic data available. Antimicrobial activities' data of the synthesized polymers implied that the polymeric metal complexes efficacious than the Schiff base polymeric ligand at different concentration explored, however, the activities are lower than the standard drugs. These findings may lead to the development of metal - based antimicrobial agents.

DATA AVAILABILITY STATEMENT

The data are available with the corresponding author upon request.

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