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Synthesis, Characterization and Chemosensing Application of Schiff Base derived from 2-Pyridinecarboxaldehyde and 2-Nitroaniline and its Metal(II) Complexes

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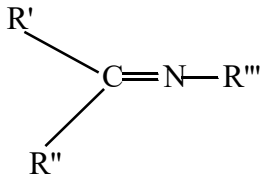
ABSTRACT

A Schiff base ligand was synthesized by refluxing 2-nitroaniline with 2-pyridinecarboxaldehyde in ethanol for three hours. The resulting ligand was further refluxed with metal(II) chlorides to yield Fe(II) and Cu(II) complexes in good yields, each exhibiting distinct colors. The ligand and its metal complexes were characterized using melting point, decomposition temperature, solubility tests, FTIR spectroscopy, magnetic susceptibility, conductivity measurements, and elemental analysis. Elemental data confirmed a 1:2 metal-to-ligand stoichiometry. The FTIR spectrum of the Schiff base displayed a strong absorption band at 1620 cm^{-1}, attributed to the azomethine (C=N) stretch, which shifted to lower frequencies in the complexes indicating coordination through the azomethine nitrogen. Conductivity studies revealed non-electrolytic behavior, while magnetic susceptibility measurements confirmed their paramagnetic nature. The Schiff base also exhibited potential as a fluorescence chemosensor, showing notable absorption changes at 295 and 395 nm and a visible color transition from light yellow to yellowish-brown, demonstrating high selectivity and sensitivity toward Cr^{3+} and Cd^{2+} ions compared to other cations. Antimicrobial screening showed that the metal(II) complexes possessed enhanced antibacterial and antifungal activities.

Keywords: Complexes, chemosensor, 2-pyridinecarboxaldehyde, Schiff base, azomethine, 2-nitroaniline,

1.0 NTRODUCTION

Schiff base (also known as imine or azomethine) is a chemical compound containing carbon and nitrogen double bond (-HC=N-) in which the nitrogen atom is connected to aryl or alkyl group. These compounds were named after Hugo Schiff, a German scientist, who first reported them in 1864 (Suleiman *et al.,* 2023). Schiff's bases have the following general structure (Figure 1.1)



Where R',R" and R"' are aryl or akyl group.

Fig. 1.1 :General structure of Schiff base

The sensing behavior of Schiff base chemosensors depends on the electronic configuration of the ligand and metal ion, as well as the rigidity and binding ability of the Schiff base structure, which determine its selectivity and sensitivity. Several mechanisms have been proposed for metal–ligand interactions, including chelation, intra/intermolecular charge transfer (ICT), hydrolysis, chelation-enhanced quenching (CHEQ), and ring-opening processes. Chemosensory responses mainly involve charge-transfer (CT) pathways such as ICT, metal–ligand charge transfer (MLCT), and twisted intramolecular charge transfer (TICT). In ICT systems, the signal changes with electron transfer efficiency, while MLCT occurs when electrons move from the ligand to the metal center. TICT involves excited-state electron transfer accompanied by molecular twisting and solvent relaxation.

1.1 Aim and Objectives

The aim of the research work is to synthesize and characterize some metal(II) Schiff base complexes derived from 2-pyridinecarboxaldehyde and 2-nitroaniline and to study their antimicrobial activities

The objectives of this research are to;

1. synthesize Schiff bases derived 2-pyridinecarboxaldehyde and 2-niroaniline
2. synthesize complexes of Fe(II), and Cu(II) with the synthesize Schiff base.
3. characterize the Schiff bases and their metal(II) complexes using the following technics:

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i. FT-IR,
ii. Magnetic susceptibility measurement
iii. Conductivity measurement
iv. Melting point/decomposition temperature
v. Solubility test
vi. Elemental analysis

4. study the application of the synthesize Schiff bases as fluorescent chemosensors for the detection of various metal cations, such as Cd^{2+}, Cr^{3+} in various materials
5. study the antibacterial and antifungal activities of the Schiff bases and their metal(II) complexes

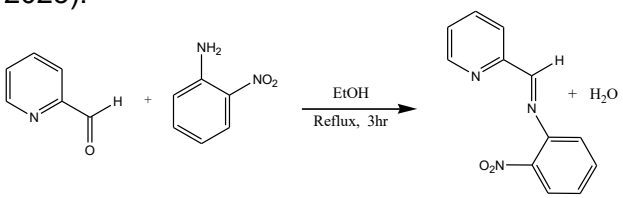
1.2 MATERIALS AND METHODS

All chemicals used were of analytical grade and used without further purification. Glassware was thoroughly cleaned, rinsed with distilled water, and oven-dried at 110 °C. Magnetic susceptibility of the complexes was measured using a Sherwood MK1 balance, while melting points and decomposition temperatures were determined with a Gallen kamp melting point apparatus (Gemini BV4947). FTIR spectra were recorded on an Agilent Cary 630 spectrophotometer, molar conductance was measured with a Jenway 4010 meter, and elemental analyses were conducted at OEA Labs, UK, using a CE Instruments EA1110 analyzer. The antimicrobial activity of the Schiff base and its complexes was evaluated in vitro against bacterial and fungal strains, using Ampiclox and Ketoconazole as reference drugs at Bayero University, Kano.

A 2-nitroaniline (0.01 mol, 1.37 g, 1.29 cm^3) solution in 20 cm^3 ethanol was added to the 20 cm^3ethanolic solution of 2-pyridinecarboxaldehyde (0.01 mol, 1.07g, 0.95cm^3) with stirring and the reaction was refluxed for 3 hours. The solid product was separated by filtration, purify by crystallization from ethanol, wash with diethyl ether, and dry in a desiccators over an hydrous calcium chloride (Salihu *et al*., 2022, Ogbonda *et al.,* 2023, Suleiman *et a*l., 2023)

1.2.0 Synthesis of the Schiff base

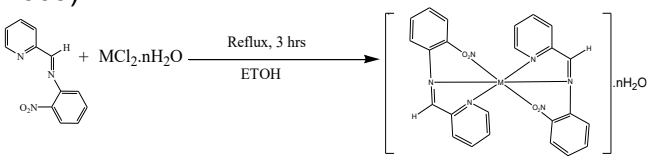
A 2-nitroaniline (0.01 mol, 1.37 g, 1.29 cm^3) solution in 20 cm^3 ethanol was added to the 20 cm^3 ethanolic solution of 2-pyridinecarboxaldehyde (0.01 mol, 1.07g, 0.95 cm^3) with stirring and the reaction was refluxed for 3 hours. The solid product was separated by filtration, purify by crystallization from ethanol, wash with diethyl ether, and dry in a desiccators over an hydrous calcium chloride (Salihu *et al*., 2022, Ogbonda *et al.,* 2023, Suleiman *et a*l., 2023).



Scheme 1: Synthesis of the Schiff base.

1.2.1 Synthesis of the Complexes

Schiff bases (0.01 mol, 1.37 g) solution in hot ethanol 20 cm^3 and ethanolic solution of the metal(II) chloride (0.01 mol), was mixed, stirred and reflux for 3 hours. The product obtained was then reduced to half its volume and left overnight. The product obtained was then filtered, recrystallise in ethanol, wash with cold diethyl ether and dried(Salihu *et al*., 2022, Suleiman *et a*l., 2023, Dasilva *et al.,* 2011, Munde *et al.,* 2009).



Where 1≤n≤2, andM = Co(II) and Ni(II),

Scheme: Synthesis of the complex

1.2.2 Preparing Cd^2 and Cr^{3+}solution for fluorescence detection using Schiff base chemosensors

A 0.89g $CdCl_2$ and $CrCl_3$ was weighed and dissolved in 100 cm^3 distilled water to obtain the stock solution, the stock solution was further dissolved in 100 cm^3 of buffer solution to prepare the working solution. Also, 1.78g of the Schiff base chemosensor was dissolved in DMSO, where equal volume of $CdCl_2$ and SB chemosensor were mixed together, and the mixture was incubated for 30 minutes to allow for complexes formation (Abu Taha *et al.,* 2025, Bharat *et al.,* 2023, Lianlian *et al.,* 2013).

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Compound	Colour	Decomposition Temperature(°C)	Melting Point(°C)	Percentage Yield (%)
Ligand, (L)	Yellow	–	93	
$[FeL_2].2H_2O$	Brown	170	–	88.42
$[CuL_2].2H_2O$	Brown	175	–	86.22

Compound	Water	Ethanol	Methanol	Acetonitrile	Ether	DMSO	DMF	nHexane
Ligand (L)	IS	SS	SS	IS	SS	S	S	IS
$[FeL_2].2H_2O$	IS	S	SS	IS	IS	S	S	IS
$[CuL_2].2H_2O$	IS	SS	SS	IS	IS	S	S	SS

1.2.3 Antibacterial and Antifungal Test

The method used by (Salihu*et al.,* 2022, Yusha'u and Salisu 2011) was used in atest, in which the schiff base and its metal(II)complexes were assayed by the ager disc diffusion method using cultures of *Staphylococcus aureus, Escherichia coli, Basillus subtilis*. The samples were separately dissolved in dimethylsulfoxide (DMSO) to have four different concentrations(500, 1000, 2000 and 4000) µg/disc. Each of these was separately placed on the surface of the culture and incubated at 37°C for 24.
The diameter of the zone of inhibition produced by the Schiff base ligand and its metal(II)complexes were taken and recorded. A similar procedure was applied in antifungal by using *Candida Albicans, Tinea Capitis, and Tinea Pedis* fungal isolates and incubated for 72 hours.

2.0 Results

Table 1:Physical properties of the Schiff base and its metal(II) complexes

L = Ligand($C_{12}H_9N_3O_2$)

Table 2: Solubility test of the SB and its metal(II) complexes

Key:
L = Ligand($C_{12}H_9N_3O_2$)
IS = Insoluble
S = Soluble
SS = Slightly Soluble
DMSO = Dimethyl sulfoxide
DMF = Dimethyl formamide

Table 3: Conductivity Measurement of the Metal(II) complexes in (10^{-3}) IN DMSO

Complex	Concentration ($moldm^{-3}$)	Specific Conductance ($Ohm^{-1}cm^{-1}$)	Molar Conductance ($Ohm^{-1}cm^2mol^{-1}$)
$[FeL_2].2H_2O$	1×10^{-3}	2.66×10^{-5}	26.66
$[CuL_2].2H_2O$	1×10^{-3}	2.00×10^{-5}	20.00

L = Ligand($C_{12}H_9N_3O_2$)

Table 4: Magnetic Parameters of Metal(II) Complexes

Complex	Magnetic Susceptibility	Molarmagnetic susceptibility	μ_{eff} (BM)
$[FeL_2].2H_2O$	7.4862×10^{-6}	3.1906×10^{-3}	2.76
$[CuL_2].2H_2O$	5.9550×10^{-7}	2.3682×10^{-4}	1.00

L = Ligand($C_{12}H_9N_3O_2$)

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Table 5: Infrared spectral data of the Schiff base and its metal(II) complexes

Complex	ν(OH) cm^{-1}	ν(C=N)cm^{-1}	ν(M–N) cm^{-1}	ν(M–O) cm^{-1}
Ligand (L)	3339	1620	–	–
$[FeL_2]$**.2**H_2O	3349	1568	745	428
$[CuL_2]$**.2**H_2O	3341	1594	741	443

L = Ligand($C_{12}H_9N_3O_2$)

Table 6: Microanalysis Data of the Schiff base and its metal(II) complexes

ound	M. wt. (g/mol)	% Calculated (Found)		
		C	H	N
	227.216	32.12(31.68)	2.48(1.98)	8.43(9.24)
$[FeL_2].2H_2O$	511.25	28.2(28.16)	2.51(1.76)	8.37(8.22)
$[CuL_2].2H_2O$	518.93	33.36(32.71)	2.48(2.23)	4.43(3.98)

L = Ligand($C_{12}H_9N_3O_2$)

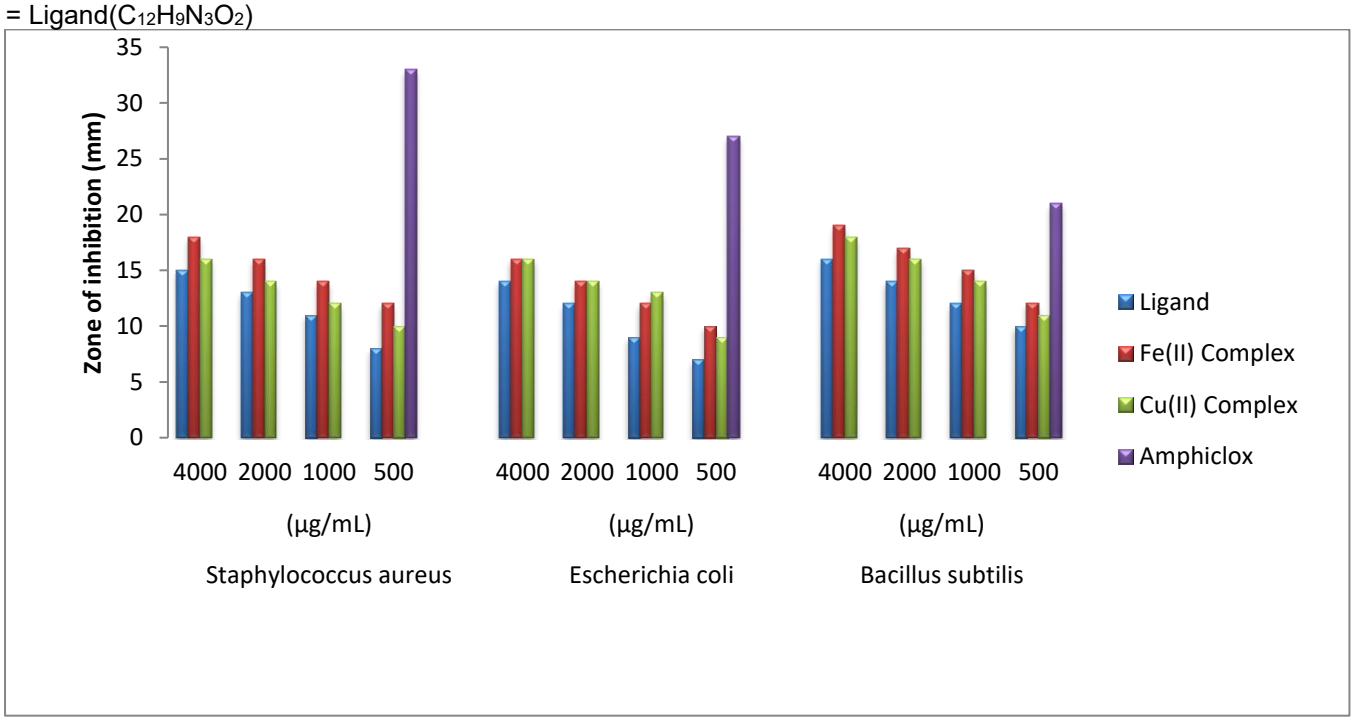


Figure 2: Antibacterial activities SB and its complex

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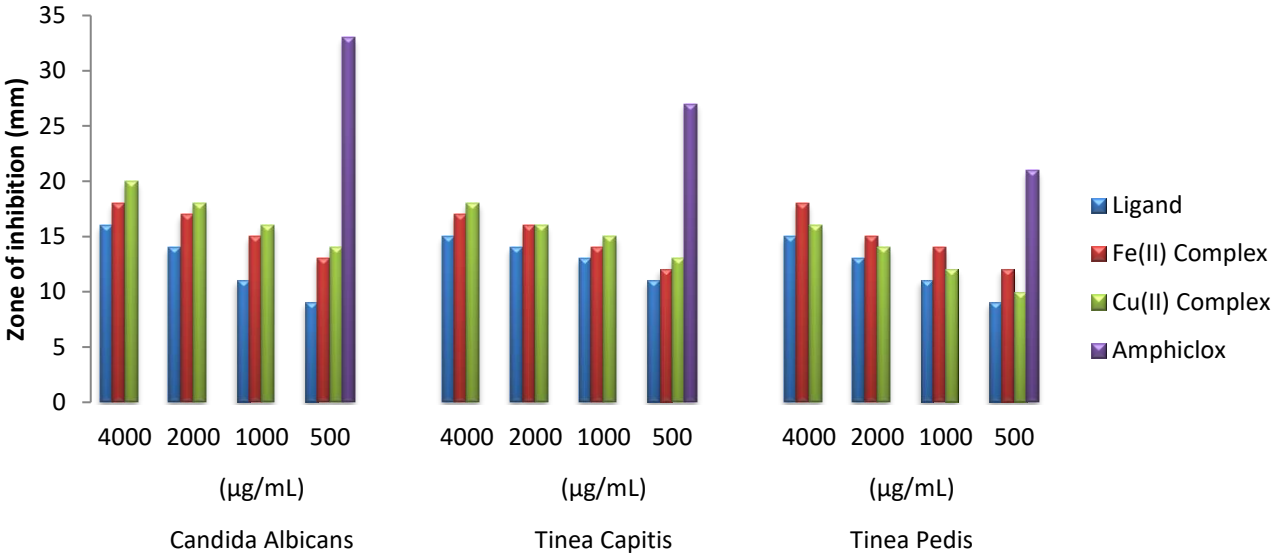


Figure 3: Antifungal activities SB and its complexes

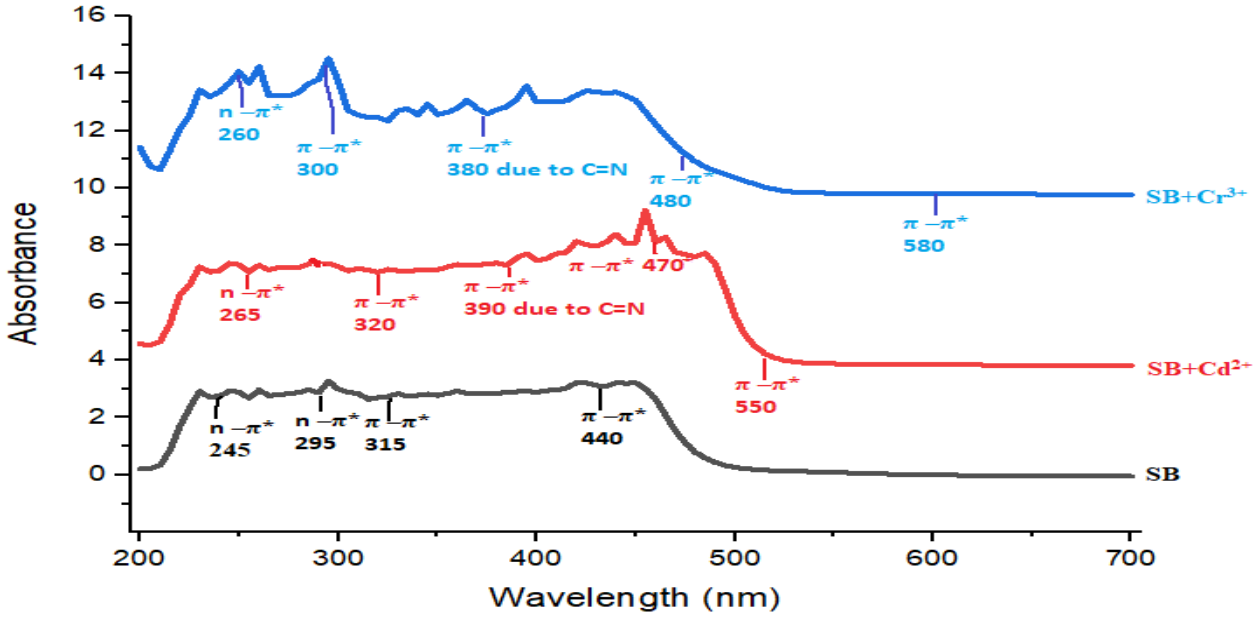


Figure 4: UV-Spectra of SB, $Cd^{2+}$and Cr^{3+}

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2.1 Discussion

The Schiff base and its metal(II) complexes were prepared in good yield, and their physical properties were analyzed. The percentage yield of the Schiff base was 90.81 % while that of the complexes were 88.42 and 86.22 %, which indicated that the methodology was very efficient for the synthesis. The Schiff base was yellow while the Mn(II) and Ni(II) complexes were brown and dark brown respectively, this showed that there is transformation and formation of the desired compounds.Also, it was found that the melting point of theSchiff base was 93^0C and the decomposition temperature of themetal(II) complexes were 170^0C and 175^OC, this is an indication that the complexes are thermally stable compared to the Schiff base which is due to the coordination of metal ion,as similar research was conducted and reported by (Salihu *et al*., 2021). This is presented in Table 1.The solubility test carried out for the Schiff base and their corresponding complexes in the following solvents, distilled water, ethanol, methanol, dimethylsulphoxide, diethyl ether, hexane and dimethylformamide. The Schiff base and the complexes are soluble in most of the highly polar solvent such as dimethylsulphoxide and dimethylformamide but poorly soluble in non-polar solvent; such as methanol and hexane, this is an indication that the compounds are polar in nature or there is a presence of polar group attached to the compounds, this is an indication that the compounds are polar in nature or there is a presence of polar group attached to the compounds as similar reported by (Salihu *et al.,* 2021*,* Gehad *et al.,* 2005). This result is contained in Table 2

The molar conductance of the metal(II)complexes wererelatively low and are in the range 26.66 and 20.00$\Omega^{-1}cm^2mol^{-1}$for Fe(II) and Cu(II)complexes. These low values suggest their non-electrolytic nature as similar reported by (Muzammil *et al.,* 2015). This contained in Table 3.The effective magnetic moments of the complexes were 2.76 and 1.00 B.M showing their paramagnetic nature. These magnetic susceptibility value of the complexes are consistent with octahedral geometry around the central metal ion as similar research was conducted and reported by (Salihu*et al.,* 2019, Gehad *et al.,* 2005). This contained in Table 4**.** The infrared spectra of the Schiff base showed a prominent band at 1620cm^{-1} due to ν(C=N) linkage appeared indicating that condensation between 2-nitroaniline and 2-pyridinecarboxaldehyde has taken place resulting into formation of the Schiff base. There is a shifting from 1620 cm^{-1} to range 1568-1594 cm^{-1} which indicate the formation of the complexes. Further evidence of the co-ordination of the Schiff base with metal ions was shown by the emergence of new peaks due to υ(M–N) in the range of 745-741 cm^{-1}, υ(M–O) in the range of 428- 443 cm^{-1}.The spectra showed a strong band at range of 3345-3347 cm^{-1}, suggesting the presence of free hydrogen in the metal complexes, a similar research was conducted and reported by Salihu *et al*., 2023. (Table 5)**.** The elemental analysis for C, H and N for all the complexes determined showed a good agreement between the calculated and observed values and the result suggested 1:2 metal to Schiff base as similar reported by (Salihu*et al.,* 2021, Abdullahi *et al.,* 2020). This result is contained in Table 6**.** The antibacterial activity of the Schiff base and their metal(II) complexes were determined using agar well diffusion method and the results presented in (Figure 2). The diameters of zone of inhibition were measured for each treatment. The Schiff base showed little activity at low concentration for most of the isolates (*Staphylococcus aureus, Escherichia coli, Basillus subtilis),* but relatively improved with an increase in the concentration of the compounds. Upon complexation, there has been increase in the activity, with most of the complexes showing significant inhibition which also increase with increase in concentration. Cu(II) complexe were found to be the most active, which indicate the tendency of this complex to be used as a potential drug (Salihu*et al.,* 2021, Siraj*et al.,* 2021).The antifungal activity of the Schiff base and their metal(II) complexes were also determined using agar well diffusion method and the results presented in figure 3. The diameters of zone of inhibition were measured for each treatment. The Schiff base showed little activity against all the fungal isolate (*Candida Albicans, Tinea Capitis,* and *Tinea Pedis*) at higher concentration but no activity at lower concentration. However, complexes recorded a much higher activity against all the tested fungal strains which increases with increases in concentration. However, they were found to be lower in activity than the Ketoconazole control used. (Siraj *et al.,* 2021).The UV–vis absorption

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studies of the Schiff base chemosensor derived from 2-nitroaniline and 2-pyridinecarboxaldehyde (SB1) were investigated and it showed absorptions peaks centered at 245 nm, with absorbance (2.919), 295 nm with an absorbance (3.275), 295 nm 315nm with absorbance (2.641), and 440nm (3.215), The absorption band at 245 nm, 295 nm, 315 nm, and 440 nm, corresponds to the n–π* and π –π* transition. These peaks of the SB chemosensor disappeared upon the addition of Cr^{3+}, and $Cd^{2+}$to SB solution, and new absorption bands 260, and 265. The band at 295 nm on the SB shifted bathochromically to 300 nm and 320 nm. Also, there is a red shift from 315 nm on the SB to 390 and 380 nm for Cd^{2+}, and Cr^{3+}. The bathochromic shift at 390 and 380 nm was due to the sensing ability of the chemosensors and the π –π* transition on C=N. The 480 and 470 nm appeared due to the π –π* for $Cr^{3+}$and Cd^{2+}, however, for the SB chemosensor there is hipsocshromic shift at 440 nm. These spectral changes occur due to the intramolecular charge transfer (ICT) process during the metal–ligand complex formation. However, there is Red shift and hypochromic shift for the absorbance on the all heavy metals. Hence, the increase in the wavelength and the decrease in the absorbance was due to the change in the chemical environment.

3.0 CONCLUSION

The Schiff base and its Fe(II) and Cu(II) complexes were prepared and characterized. Conductivity measurement showed that the complexes are non-electrolyte. The decomposition temperature of these Schiff base complexes revealed high values which is an indication of high stability. The solubility test carried out in various solvents showed they are all soluble in DMSO and DMF. IRspectroscopy shows the Schiff base is co-ordinated to the central metal ion through the N-atom of the azomethine. The positive values of the magnetic susceptibility of the complexes shows that the complexes are paramagnetic in nature. The elemental analysis of Schiff base and their metal complexes shows 2:1 ligand to metal ratio. The Schiff base chemosensor was able to sense and detect the selected heavy metal cations which showed that it can be used as an environmental remediation.The anti-microbial activity of the ligand and its metal complexesindicate that the metal(II) complexes were more active than the free Schiff base on one or more isolates. Even though the metalcomplexes were found to be more active than the free Schiff base,they were lower than the standard drugs (*ampicloxand**ketoconazole* for bacterial and fungal isolate respectively) used.

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