International Journal of Pharmacy and Biological Sciences
ISSN: 2321-3272 (Print), ISSN: 2230-7605 (Online)
IJPBS | Volume 8 | Special Issue 4- Chem Fest | 2018 | 36-48
2nd National Conference on Emerging Trends and Future Challenges
In Chemical Sciences-Chem Fest-18
Held at Shanmuga Industries Arts and Science College, Tiruvannamalai, 05th Oct 2018
| Seminar Proceedings | Research Article | Biological Sciences |Open Access |MCI Approved|
|UGC Approved Journal|

SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL ACTIVITY OF INDOLE-3-CARBOXALDEHYDE SCHIFF BASE Hg (II) AND Zr (IV) COMPLEXES

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ABSTRACT

Schiff base have been prepared from Indole-3-Carboxaldehyde and 1,4 Diaminobutane. Ligand are synthesized in a 1:2 mol ratio, and their Hg (II) and Zr (IV) complexes in a 1:1 mol ratio (Ligand:Metal). Ligand and metal complexes were characterized by FTIR, UV,^{1}H NMR, SEM and Antimicrobial activity. The compounds were confirmed by the presence of an imine band stretching in the 1630-1640cm^{-1} and νMetal –N and metal ion to chlorine at <600 cm^{-1}. The ^{1}H NMR of Ligand a singlet signal at δ 7.72 ppm observed isomethine nitrogen (s, C=N) on complexation Hg (II) and Zr (IV) are observed δ 7.21 ppm, δ 7.65 ppm. The UV Vis spectrum of ligand show band at 241 nm on complexation Hg (II) and Zr (IV) shows longer wavelengths at 395 nm and 407 nm. Surface morphologies were analyzed with SEM. Generally, the Schiff base ligand had a smooth surface morphology, while the Hg (II) and Zr (IV) complexes presented heterogeneous features with smooth and rough surface regions and Antimicrobial activity the free ligand and their metal complexes have been screened for their in vitro biological activities against bacteria and fungi. The metal complexes show more potent activities compared with Schiff base ligand.

KEY WORDS

Antimicrobial activities, Indole-3-carboxaldehyde, Schiff base, Mercury (II), Zirconium (IV), 1,4 Diaminobutane.

INTRODUCTION:

The Schiff bases are considered a very important class of ligands, which form complexes with many metals[1]. These complexes have wide applications in some biological respects, analytical fields, organic catalysis, oxygen carriers and as corrosion inhibitors in especially acidic environments for various alloys and metals[2]. Schiff bases have a large number of synthetic uses in organic chemistry. Acylation of Schiff bases by acid anhydrides, acid chlorides and acyl cyanides is initiated by attack at the Nitrogen atoms leads to net addition of the acylating agent to the carbon – nitrogen double bond. Reactions of this type have been put to good use in natural product synthesis[3].

Schiff bases appear to be an important intermediate in a number of enzymatic reactions involving interaction of an enzyme with an amino (or) a carbonyl group of the substrate one of the most important structural feature of these Compounds is the azomethine group with the general formula RCH=N-R1, where R and R1 are alkyl, aryl, cyclo alkyl (or) heterocyclic groups whih may be substituted. These compounds are also known as anils, imines (or) azomethines[4]. several studies showed that the presence of a ione pair of electrons in an sp2 hybridized orbital of nitrogen atom of the azomethie group is of considerable chemical and biological importance. Because of the relative easiness of preparation, synthetic flexibility, and the special property of C=N group, Schiff bases are generally excellent chelating agents, especially when a functional group like –OH (or) SH group is present close to the azomethine group so as to form a five (or) six-member ring with the metal ion. Nowadays the research field dealing with Schiff base coordination chemistry has expanded enormously[5].

However, few studies have been performed regarding Hg^{2+} and Zr^{2+} complexes with Schiff base derived from Indole – 3- carboxaldehyde. Mercury and zirconium play an important role in biological and chemical process[6,7]. Mercury also forms useful amalgams with many metals, which find various applications in diverse fields. Due to the numerous applications and the toxic nature of amalgams and mercury compounds[8]. The toxicity of merury depends on its occurring forms; organomerurials such as methyl mercury are more toxic than elemental mercury and other inorganic mercury compounds. Mercury contains ligands e.g: Mercuric and mercuros are known form stable complexes with class b metal ions, such as gold(I), and Se (II) because mercury is considered to be a soft Lewis base[9].

Zirconium compared to mercury it has low toxicity; zirconium can be taken into the body by eating food, drinking water. Zirconium is not well absorbed into the bodly with only about 0.27 of the amount ingested being absorbed into the blood stream through the intestines. The biomedical applications of zirconium bearing compounds are used dental implants and crowns, knee and hip replacements, middle ear ossicwar chain reconstruction and other restorative and prosthetic devices[10].

In this paper we report on a series of new Hg (II) and Zr (IV) complexes with Schiff bases ligand derived from indole -3- carboxaldehyde which display moderate until good anti-bacterial and antifungal activities[11,12].

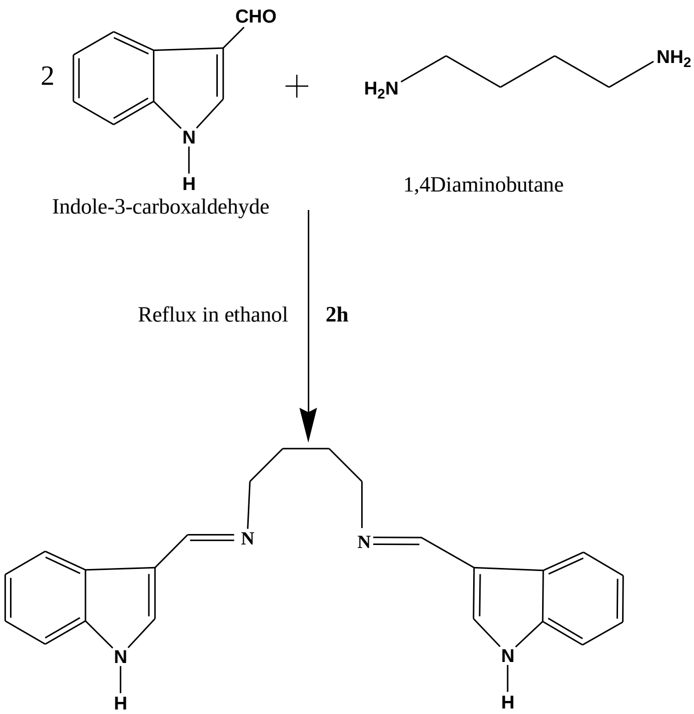
2. MATERIALS AND METHODS:

2.1 Materials

Indole-3-carboxaldehyde, 1,4 diaminobutane and ethanol were purchased from sigma Aldrich and used without solvents were purchased from Merck.

2:2 Synthesis of Schiff base Ligand

1,4 –diaminobutane (putrescine) (5.806g,40mmol) was added to a solution of idole-3-carboxaldehyde (3.52g, 40mmol) in absolute ethanol 50ml in 1:2 ratio. The reaction mixture was refluxing the solution for 2h. it was filtered. The resulting dark green colour solid product was extracted with absolute ethanol, purified by recrystallization from ethanol, washed with ethanol and then dried[13] scheme 1.



Scheme 1

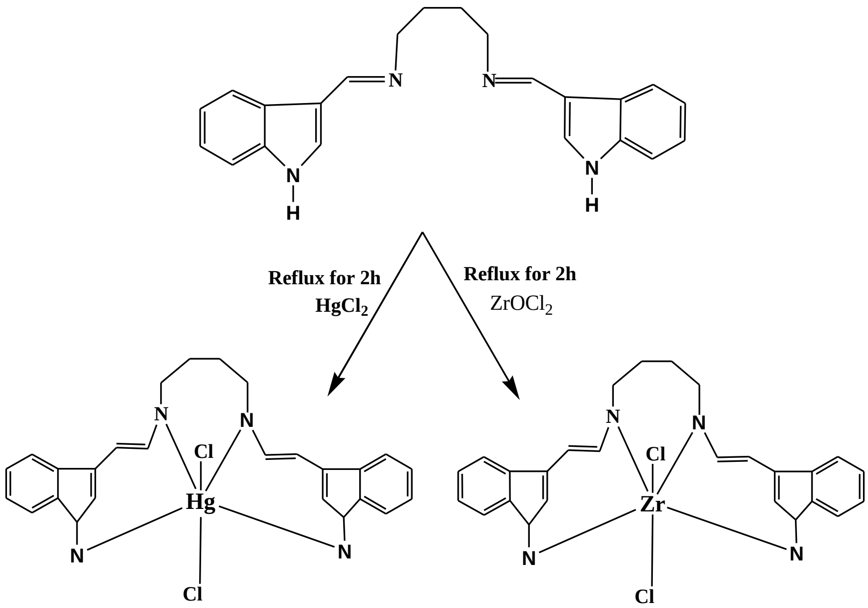
2:3 Syntheses of the metal complexes

2:3:1 Synthesis of Hg (II) complex

The Hg (II) complex was prepared by adding a ethanolic solution of $HgCl_2$ (0.549g,2mmol) in small quantities with stirring to hot ethanolic solution of the Schiff base ligand, (0.684g,2mmol) in ethanol (50ml) was added drop wise. After the addition was completed. The mixture was stirred and heated to reflux for 2h. the volume of the reaction mixture was reduced to 20-30 ml. the light reddish brown colour solid complex which separated out was suction filtered washed first with aqueous ethanol and finally with ether and dried. Scheme 2.

2:3:2 Synthesis of Zr (IV) Complex

Similarly, ZrCl2 (0.644g, 2mmol) the mixture was stirred and heated to reflux 2h. The light greenish colour solid crystals are separated out was suction filtered washed first with aqueous ethanol and finally with ether and dried[14]. Scheme 3.



Scheme – 2 **Scheme - 3**

2:4 Characterization of Schiff base ligand and metal complexes

The FTIR spectra were recorded in Perkin-Elmer spectrophotometer in the range of 400cm-1 to 4000cm-1 in KBr phase. To observe optical property separated for Schiff base ligand and metal complexes were periodically analyzed with UV-Vis spectrometer (JSCOV-670) using the range 200 to 800 nm. The 1H NMR spectra were recorded in DMSO d6 400 MHZ (Bruker) spectrometer. And chemical shifts calculated in ppm with respect to TMS (δ=0). The shape of the Schiff base ligand and metal complexes were observed by SEM using model EVO18 (ZEISS).

2.5 Biological Activity

The synthesized schiff base and metal complexes were screened for antibacterial and antifungal activity.

2.5.1. Antibacterial Activity Study

Characteristics of the medium:

The main characteristics of the medium were to support the growth of the organisms normally tested and not contain antagonist of antimicrobial activity. The medium must allow free diffusion of plant extract from the well.

The sterilized medium was poured into a Petri dish in a uniform thickness and kept aside for solidification. Using sterilized swabs, even distribution of lawn culture was prepared using bacteria such as *Escherichia coil, Staphylococcus aureus* and *Bacillus cereus* in Muller Hinton agar (MHA) Plates[15].

Muller Hinton Agar (MHA) and Sabouraud's dextrose Agar (SDA) were prepared with lawn culture using desired test organism and the ingredients are Dextrose 4.0g, Mycological peptone 1.0g, Agar 2.0g, Distilled water 100ml and P^H 5.0 medium. The inoculated plates were kept aside for few minutes. Using well cutter 4 wells were made in those plates at required distance. In each step of well cutting, the well cutter was thoroughly wiped with alcohol. Using sterilized micropipette, 20 µl of compounds are ligand and metal complexes extract were added in the three different wells and in another well the same volume of *ciprofloxacin* was taken as reference.

After diffusion, the plates were incubated at 37°C for 24 hours. After incubation, the inhibition of growth was analyzed. The antibacterial activity was evaluated by measuring the diameter of the zone of inhibition in mm against the test fungal strain. The tests were carried out in triplicates.

Muller Hinton Agar (MHA) and Sabouraud's dextrose Agar (SDA) were prepared with lawn culture using desired test organisms and the ingredients are used dextrose(4.0g), Mycological peptone(1.0g), Agar(2.0g), Distilled water (100 ml) and medium of P^H (5.0). The inoculated plates were kept aside for few minutes[16]. Using well cutter 4 wells were made in those plates at required distance. In each step of well cutting, the well cutter was thoroughly wiped with alcohol. Using sterilized micropipette, 20 µl of compounds are ligand and metal complexes extract were added in the three different wells and in another well the same volume of *ciprofloxacin* was taken as reference.

After diffusion, the plates were incubated at 37°C for 24 hours. After incubation, the inhibition of growth was analyzed. The antibacterial activity was evaluated by measuring the diameter of the zone of inhibition in mm against the test fungal strain. The tests were carried out in triplicates.

<u>The agar well diffusion method:</u>

Simple susceptibility screening test using with agar-well diffusion method as adapted earlier was used. Each microorganism was suspended in Brain Heart Infusion (BHI) broth and diluted to approximately 10^6 colony forming unit(cfu)/ml. They were "flood-inoculated" onto the surface of BHI agar and *Sabouraud Dextrose* Agar (SDA) and then dried. For *C. albicans* and *C. tropicalis*, SDA was used Five-millimeter diameter wells were cut from the agar using a sterile cork-borer, and 100 µl of the sample's solutions were delivered into the wells. The plates were incubated for 18 hours at 35°C. Antimicrobial activity was evaluated by measuring the zone of inhibition against the test microorganisms. *Ceftaazidime* (Fortum) (10 µg) and *Trifulcan (*5 µg) were the standard drugs for antibacterial and antifungal activities, respectively. Acetone was used as solvent control. The tests were carried out in duplicates. Results were interpreted terms of diameter of inhibition zone in mm.

The estimation of the Minimal Inhibitory Concentration (MIC) and Minimal Bactericidal Concentration (MBC) was carried out by the broth dilution method. Dilutions of essential oil from 2.0 to 0.075 mg/ml were used. Test bacteria culture was used at the concentration of 105 CFU/ml. MIC values were taken as the lowest essential oil concentration that prevents visible bacterial growth after 24 hrs of incubation at 37°C, and MBC as the lowest concentration that completely inhibited bacterial growth. Chloramphenicol was used as reference and appropriate controls with no essential oil were used. Each experiment was made three times.

The bactericidal kinetic assay was performed by using appropriate concentrations of essential oil (corresponding to ½ MIC and MBC).

2.5.2. Anti-Fungal Activity:

The sterilized medium was poured to a Petri in a uniform thickness and kept aside for solidification and the ingredients are Beef extract 0.2g, Peptoe 1.75g, Starch 0.15g, Agar 1.70g, Distilled water 100ml and P^H 7.5 medium. Using sterilized swabs, even distribution of lawn culture was prepared using desired fungi such as *Aspergillus niger*, *Candida albicans* and *Aspergillus fumigatus* in SDA plates[17].

The inoculated plates were kept aside for few minutes. Using with well cutter, four wells were made in those plates at required distance. In each step of well cutting, the well cutter was thoroughly wiped with alcohol. Using sterilized micropipette, 20 ml of compounds A, B, C extract were added in the three different wells and in another well the same volume of Amphoterecin - B or *ketoconazale* was taken as reference.

After diffusion, the plates were incubated at temperature for 24 hours for yeast like fungus and 48-72 hours for molds. After incubation, the inhibition of growth was analyzed. The antifungal activity was evaluated by measuring the diameter of the zone of inhibition in mm against the test fungal strain. The tests were carried out in triplicates.

Determination of Minimum Inhibitory Concentration (MIC):

One ml of extract (1mg/ml) was incorporated into one ml of nutrient broth and *sabouraud's* dextrose broth and was serially diluted to obtain concentration of 1000 µg/ml, 125 µg/ml, 62.5 µg/ml, 31.25 µg/ml respectively. 20 µl of the inoculum was added to each of the test tubes. The tube without the extract served as control. The tubes were incubated at room temperature and readings were recorded after a period of 24 hrs for bacteria and 3 days for fungi.MIC was recorded as the lowest concentration of the extract at which no visible growth of the bacterial and fungal occurred after a period of seven days incubation.

3. RESULTS

3:1 UV:Vis Spectra analysis:

The UV-Vis spectrum of Ligands shows band at 241 nm Fig. 1(a), The adsorption Hg (II) spectrum metal complex shows band at 395 nm Fig. 1 (b) and Zr(IV) spectrum of adsorption band shows at 407 nm Fig. 1(c) with a shoulder band around longer wavelength. The complex Hg (II) and Zr (IV) two adsorption bands can be safely assigned to the chare transfer transitions and compared to the ligand adsorption bands with two metal complexes are adsorbed band shows at longer wavelengths[18,19].

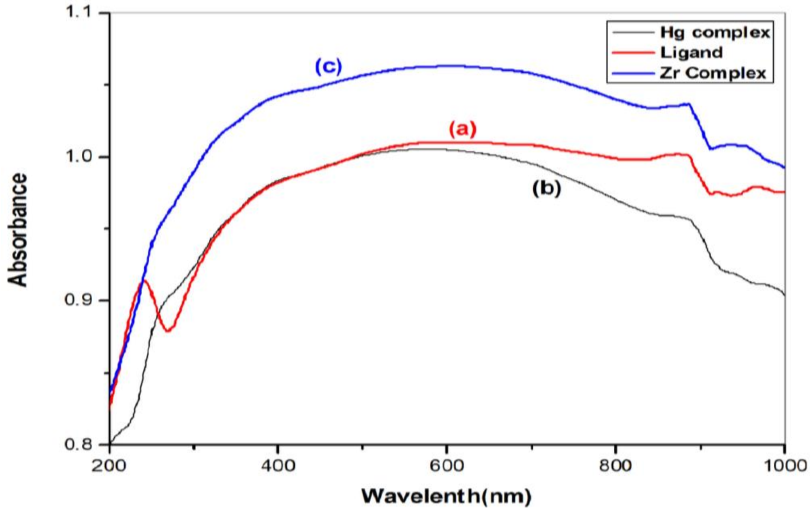


Fig. 1. UV: Vis Spectra (a) Schiff base ligand; (b) Hg (II) complex; (c) Zr (IV) complex

3:2 FTIR-spectral Analysis

FT-IR analysis was performed to the ligand and metal complexes before and after complex preparations, the spectrum obtained for ligand shows bands at 3402 cm^{-1},2922 cm^{-1},1628 cm^{-1},1575 cm^{-1},1488 cm^{-1},1324 cm^{-1},1234 cm^{-1} and 740 cm^{-1}.The Hg(II) complex shows bands at 3428 cm^{-1},1613 cm^{-1},1328 cm^{-1},1212 cm^{-1},746 cm^{-1},566 cm^{-1} and 464 cm^{-1} and Zr(IV) complex shows bands at 3438 cm^{-1},1616 cm^{-1},1329 cm^{-1},1226 cm^{-1},747 cm^{-1},549 cm^{-1} and 493 cm^{-1}.

Table 1:IR data of the Schiff and their metal complexes (cm^{-1})

Compound	ν (N-H)	ν (C=N)	ν (C-N)	ν (M-Cl)	ν (M-N)	Phenyl ring Vibrations
Ligand	3402	1625	1324	-	-	740
Hg (II) Complex	3428	1613	1328	566	464	746
Zr (IV) Complex	3438	1616	1329	549	493	747

The study of IR stretching frequencies of the ligand and metal complexes will give an idea about mode of bonding in complexes and they also indicate the arrangement of atom in space and bond angles most of the assignments of IR absorptions frequencies have been based on the concept of group frequency. In large molecule factors both external as well as internal influence the force constant and determine the precise position of the infrared adsorption bands. On complexation with metal ions, the characteristic IR frequencies of the coordinating groups are influenced by the force constant of the metal ligand bond resulting in the shifting of the group frequencies. These shifts are useful in identifying the coordination sites. The important vibrating group in a free Schiff base ligand in naturally the imino group. The strong absorption due to this found to lie in the region C=N 1628 cm^{-1}. However, on complexation the group frequency of the free azomethine is altered. The strong adsorption band shows that 3402 cm^{-1} in N-H stretching vibration. The band shows 2922cm^{-1} in the presence of alkane C-H stretching frequency. The bond 1575 cm^{-1} show the presence of aromatic C=C stretching vibration and other bands shows in shift base ligand in adsorption bands at stretching vibrations are C-N band in 1324 cm^{-1} and the band 740 cm^{-1} shows that phenyl ring vibration[20]. Fig2 (a)

The brown colour indole-3-carbaldehyde, 1,4 diaminobutane to Hg (II) if obtained from Hg (II) chloride and shiff base. The complex is soluble in alcohol. The infrared spectra of this complex show broad band centered around 3428cm^{-1}. This is assigned to the coordination water molecule N-H stretching vibration. The infrared spectrum of the Schiff base ligand shows a strong band at 1628 cm^{-1}, which assigned to the C=N stretching vibration. On complexation this band shifted to 1613 cm^{-1}. This indicates that the azomethine nitrogen is coordinated to the metal ion. The band in 1324 cm^{-1} in C-N aromatic stretching frequency shifted to 1328 cm^{-1} in metal complexation. The band shows 740 cm^{-1} in phenyl ring vibration shows higher frequency in metal complex 746 cm^{-1} and the new bond show that 566 and 464 cm^{-1} range indicating the formation of a linkage between the nitrogen and metal ion to chlorine[21]. Fig 2(b).

Similarly, the light reddish brown colour of Zirconium (II) complex if obtained from Zirconium (II) chloride and schiff base and the complex is also soluble in alcohol. The IR spectrum of this complex show a broad band centered around 3438 cm^{-1} which is assigned to the coordinated N-H stretching frequency. The IR spectrum of the Schiff base ligand shows a strong band at 1628 cm^{-1}, which is assigned to the C=N stretching vibration on complexation this band shifted lower frequecy1616 cm^{-1}. This indicates that the azomethine nitrogen is coordinated to the metal ion. The band 1324 cm^{-1} in C-N aromatic stretching vibration shifted to 1329 cm^{-1}in metal complexation. The band shows 747 cm^{-1} in Zirconium metal complex in phenyl ring vibration to higher than the sshiff base ligand and the new band show that 493-549 cm^{-1} range indicating the formation of a linkage between nitrogen and metal ion to chlorine[22]. Fig 2(c).

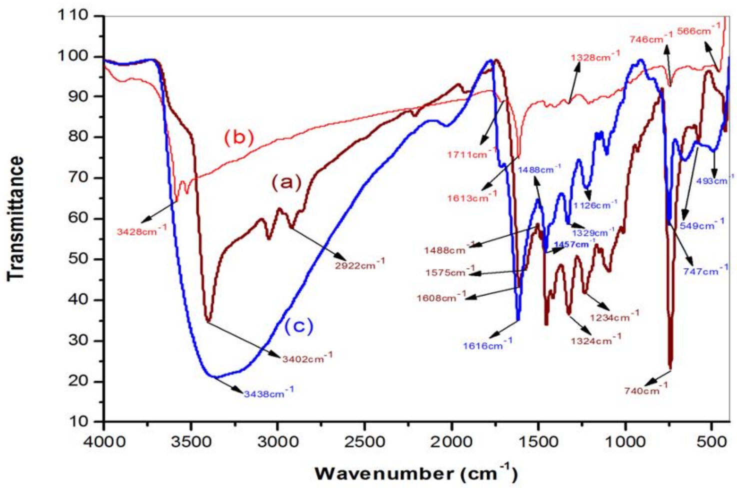


Fig. 2. FT-IR Spectra (a) Schiff base ligand; (b) Hg (II) complex; (c) Zr (IV) complex

3:3 ^{1}H NMR Spectral Analysis

The nuclear magnetic resonance spectra the signals of the respective protons of the complexes are verified on the basis of its chemical shifts, multiplicities and coupling constants. The ^{1}H NMR spectra of ligand Hg(II) and Zr (IV) complexes were recorded in DMSO –D6 ^{1}H NMR spectrum ligand shows signals due to N-H at δ 11.61 in the free ligand (S 1H,N-H) and a singlet signal at δ7.72 ppm observed in the spectrum for the free ligand shows azomethine nitrogen (S, C=N). The down field δ7.55 and δ7.24 exhibits doublet in one aromatic hydrogen (d, 1H, Ar H) and the deshielding aromatic hydrogen show triplet in δ 7.15 (t,1H). The low field δ 6.84 present in the free ligand singlet one hydrogen (S, 1H). The upfield low frequency shielding protons are present δ1.23 and δ 3.06 multiplet shows that free ligand[23]. (Fig 3 (a).

The 1H NMR spectrum of the Hg (II) complex in DMSO d6 solvent confirms it's proposed structure and the complex shows a broad signal at δ 8.13 ppm due to imine protons a broad peak at δ 1.15 due to the hydrogen resonance whilst and another Hydrogen protons in metal complex appear at δ 2.67 ppm. The multiplet resonances in the range δ 2.18 -2.67 ppm are related to butane protons and the signals of aromatic protons of the ligand and metal complex appear in the region δ 7.08 – 7.21 ppm. Fig 3 (b).

Similarly, the H^1 NMR spectrum of the Zr (IV) complex shows that broad signal at δ7.65 ppm due to the imine protons, a broad peak at δ 1.55 ppm due to the protons in free ligand and another proton in metal complex appear at δ 2.81 ppm. The multiplet resonances in the range δ 2.50 -2.51ppm are related to butane protons in the ligands and aromatic protons of metal complex appear in the region δ 7.08 – 7.21 ppm[24]. Fig. 3(c).

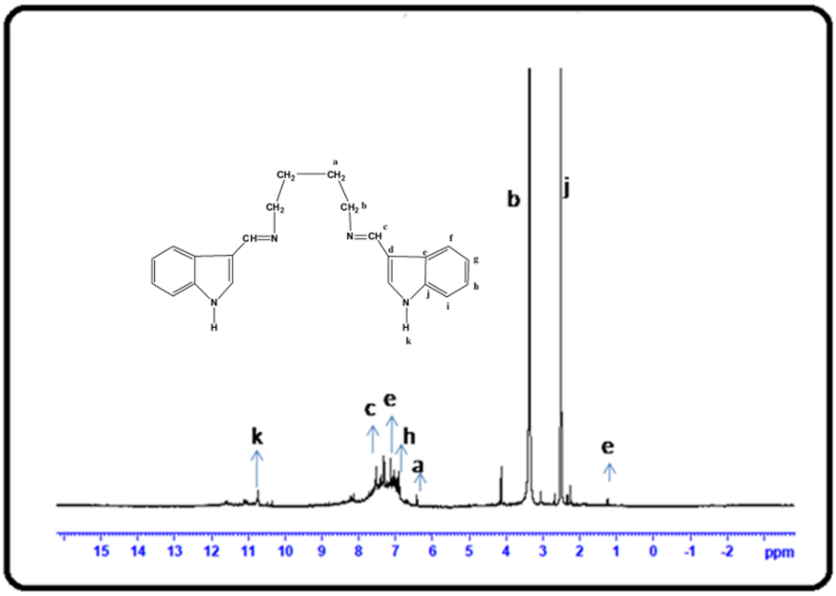


Fig. 3. (a) ^{1}H NMR spectrum of ligand

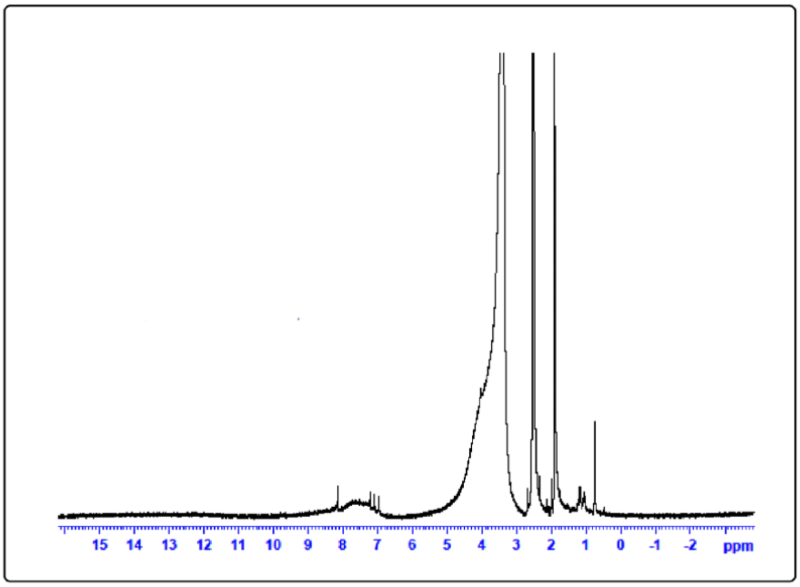


Fig. 3. (b) ^{1}H NMR spectrum of Hg (II) complex

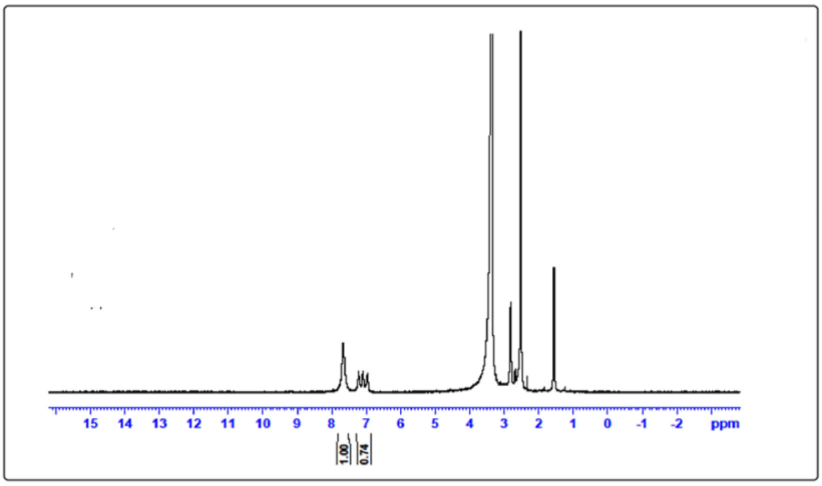


Fig. 3. (c) ^{1}**H NMR spectrum of Zr (IV) complex**

3.4 Scanning Electron Microscopy (SEM) Analysis

The SEM images of ligand, Hg (II) and Zr (II) complexes are presented in respectively as a representative example, Generally the Schiff base ligand host a smooth surface morphology, Fig 4 (a). While the complexes presented heterogeneous features, with smooth and rough surface regions. The complexes were also with SEM in which was possible to verify brighter regions evidencing the presence of the metallic species are very rough surfaces[25]. Fig.4. (b, c)

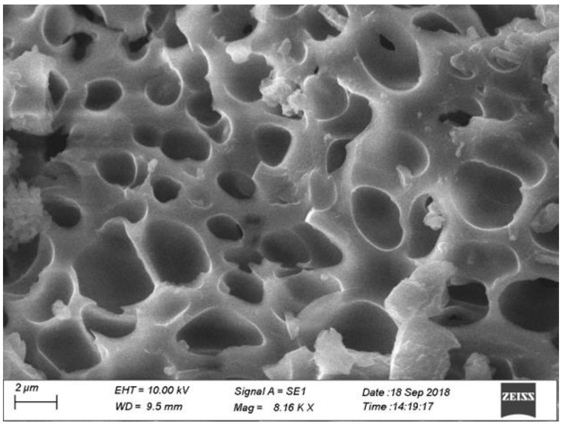


Fig. 4. (a) SEM image of ligand

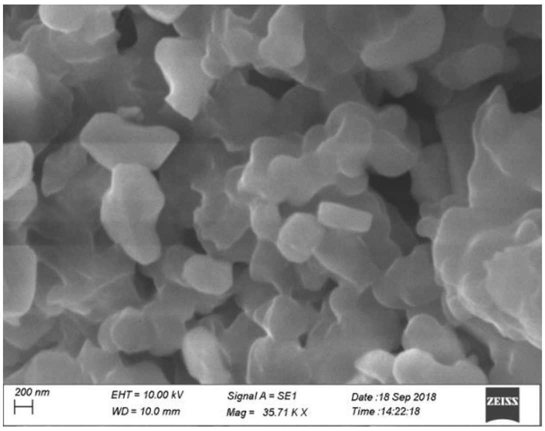


Fig. 4. (b) SEM image of Hg (II) Complex

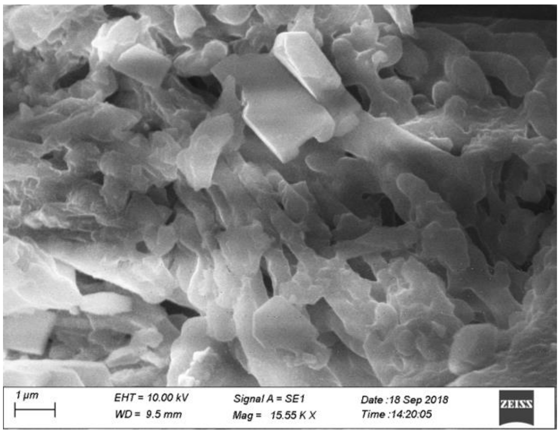


Fig. 4. (c) SEM image of Zr (IV) Complex

3.5 Antibacterial Activity

The results of the antibacterial screening of the Schiff base ligand, Hg (II) and Zr (IV) complexes at a concentration of 20mg/ml against all bacteria have been found. The inhibition zones were measured in mm and results are shown in Table 2.

Table 2. The Antibacterial activity of ligand, Hg (II) and Zr (IV) complexs against tested microorganisms.

S.No	Microorganisms	Zone of inhibition (mm)				
		Control	Ligand	Hg (II) complex	Zr (IV) complex	Ciprofloxazion
1.	*B. cereus*	-	-	26	8	25
2.	*S.aureus*	-	-	33	14	26
3.	*E.coli*	-	-	32	7	24

The results of antimicrobial screening, indicate that Schiff base ligand, Hg (II) and Zr (IV) complexes significant activity against *Bacillus Cereus(B.cereus)*, *Staphylococcus aureus (S.aureus)* and *Escherichia coli(E.coli)*. The high zone of inhibition shows Hg (II) complex in *Staphylococcus aures(S.aures)* 33 mm and Zr (IV) complex show that 14 mm and reference material *Ciprofloxazin* show that inhibition 26 mm. the *E. coli* bacteria shows that zone of inhibition in Hg (II) complex 32 mm, Zr (IV) complex inhibition of 24 mm and the *B. cereus* bacteria shows that the zone of inhibition Hg(II) complex 26 mm, Zr (IV) complex 8 mm and reference material *Ciprofloxazin* zone of inhibition 25mm. Fig 5 (b). The zone of inhibition only shows selected metal complexes, but no zone of inhibition shows that Schiff base ligand[26]. Fig. 5 (a,b,c)

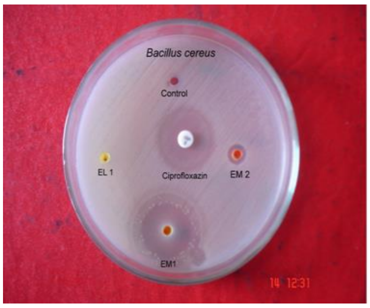


Fig.5. (a)Antibacterial activity of Ligand (EL_1), Hg (II) complex (EM1) and Zr (IV) complex (EM2) against *B. cereus*

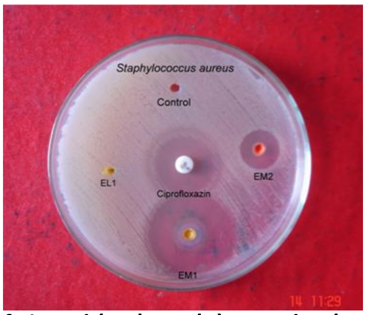


Fig. 5. (b)Antibacterial activity of Ligand (EL_1), Hg (II) complex (EM1) and Zr (IV) complex (EM2) against *S.aureus*

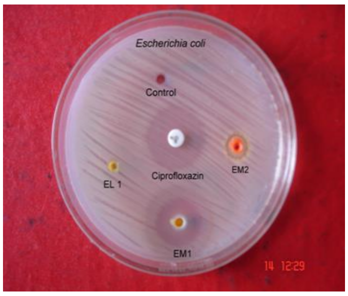
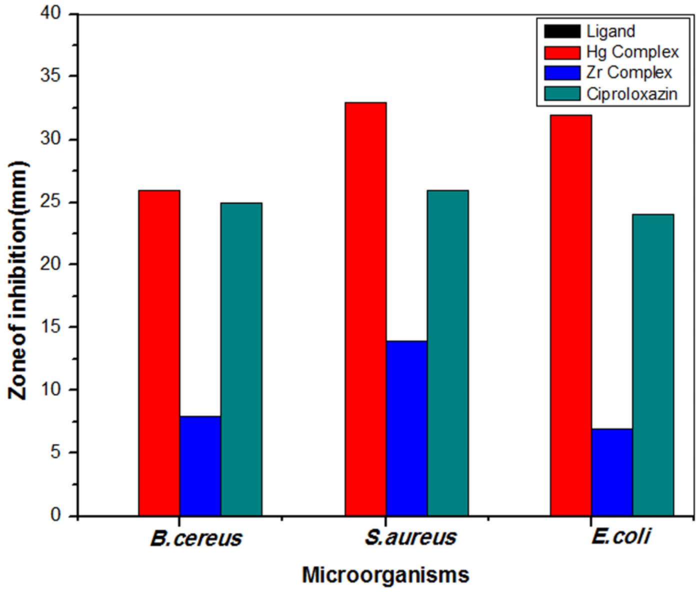


Fig. 5. (c)Antibacterial activity of Ligand (EL_1), Hg (II) complex (EM1) and Zr (IV) complex (EM2) against *E. coli*

Graph 1: Zone of inhibition (mm) of Antibacterial Activity



3.6 Anti-Fungal Activity:

The antifungal screening effect of the synthesized Schiff base ligand, Hg (II) complex and Zr (IV) complex were evaluated against *Aspergillus niger (A.niger), Candida albicans (C.albicans)* and *Aspergillus Fumigatus (A.fumigatus)* using *Amphoterecing – B* standard anti-fungal reference by well diffusion method. The inhibition zones were measured in mm and results are shown in Table. 3.

Table 3: The Antigungal activity of ligand, Hg (II) and Zr (IV) complexs against tested microorganisms.

S.No	Microorganisms	Zone of inhibition (mm)				
		Control	Ligand	Hg (II) complex	Zr (IV) complex	Ciprofloxazion
1.	*A.niger*	-	-	40	8	14
2.	*A.fumigatus*	-	-	35	14	12
3.	*C.albicans*	-	-	37	12	-

The results showed that the Hg (II) complex 40 mm in *A. niger* and antifungal reference *Amphoterecin –B* Show at 14mm. The zone of inhibition in *C. albicans* show 37mm and reference *Amphoterecin –B* show at 14mm. The zone of inhibition in *C. albicans* show 37 mm and reference *Amphoterecin – B* indicate at no zone of inhibition and the *A. fumigatus* shows that zone of inhibition 35 mm and *Amphoterecin – B* at 12 mm. Similarly Zr (IV) complex show that zone of inhibition in 12 mm and *A.niger* 8 mm. The standard antifungal reference *Amphoterecin – B* shows that zone of inhibition *A.niger* 14 mm, *A.fumigatus* 12 mm and *C.albicans* show that no zone of inhibition. Therefore the results showed that the Hg (II) complex than the Zr (IV) complex exhibited potent inhibitory activity against all the three Fungal strains[27,28]. Fig. 6 (a,b,c)

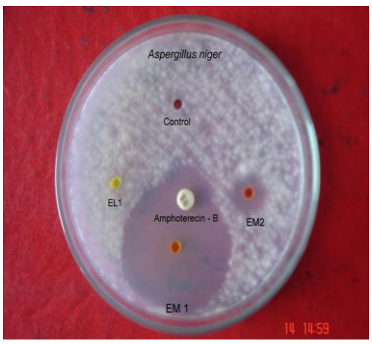


Fig. 6 (a) Antifungal activity of Ligand (EL_1), Hg (II) complex (EM1) and Zr (IV) complex (EM2) against *A.niger*

Fig. 6 (b) Antifungal activity of Ligand (EL_1), Hg (II) complex (EM1) and Zr (IV) complex (EM2) against *A.fumigatus*

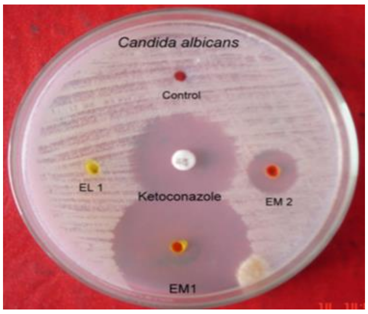
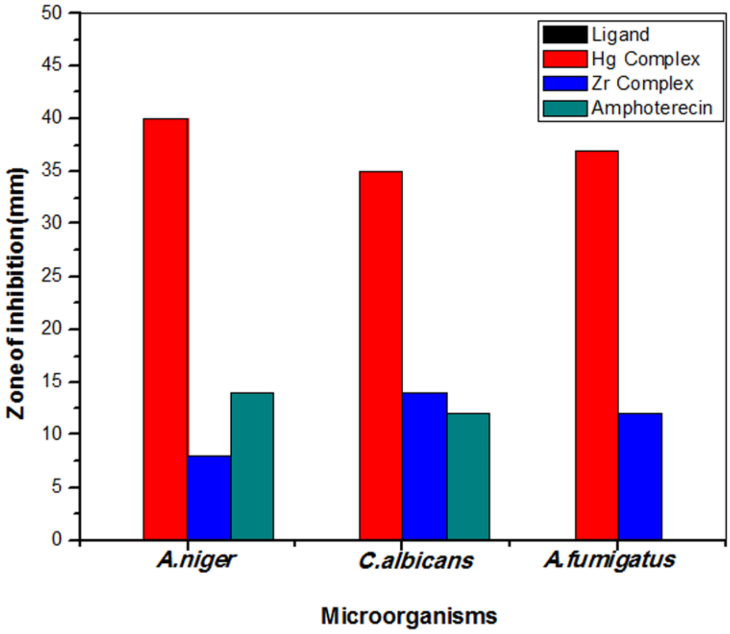


Fig. 6. (c) Antifungal activity of Ligand (EL_1), Hg (II) complex (EM1) and Zr (IV) complex (EM2) against *C. albicans*

Graph 2: Zone of inhibition (mm) of Antibacterial Activity



4. DISCUSSION

The new Schiff base ligand was prepared by the 1:2 ratio reaction between Indole-3-Carboxaldehyde and 1,4 diaminobutane in ethanol. The complexes Hg(II) and Zr (IV) were synthesized by the reaction of the ligand with the appropriate chlorine salts in a 1:1 molar ratio. The complexes were characterized by optical and elemental analysis. The UV- Vis spectrum of ligand show band at 241 nm and the adsorption spectrum of zirconium complex indicate that at higher wavelength 407 nm than Hg (II) complex show at 395 nm. The two metal complexes are adsorbed band shows at longer wavelengths compare to Schiff base ligand due to charge transfer transitions are involved.

The infrared spectrum of ligand shows a strong intensity band at 1628cm^{-1}, which may reasonably be assigned to the imine function (C=N) in the Schiff base ligand. The strong adsorption band shows that 3402cm^{-1} show the presence of aromatic C=C stretching vibration and other bands shows in Schiff base lignad in adsorption band at stretching vibrations are C-N band in 1324 cm^{-1}, C-C band at 1234 cm-1 and the band 740cm-1 show that phenyl ring vibration. The IR spectra of the complexes show a sharp band in the 1633-1638 cm^{-1} region, which is attributed to the ν(C=N) stretch, indicating coordination of the azomethine nitrogen to the meatls . Bands at 1328-1329 cm^{-1} associated with ν(C-N) vibrations from the aromatic rings are also present in the spectra and the new bands shows the two complexes at 464-566cm^{-1} and 493-549cm^{-1} the formation of a linkage between nitrogen and metal ion to chlorine.

The 1H NMR spectrum ligand shows signals due to N-H at δ 11.61 ppm in the free ligand (S ,1H N-H) and a singlet signal at δ7.72 ppm observed in the spectrum for the free lignad shows azomethine nitrogen (S, C=N) and the down field δ 7.55-7.24 ppm exhibits doublet in one aromatic hydrogen (d 1H, ArH) a broad peak 1.23 ppm due to the Ha resonance whilst Hb protons appear peak at δ3.06 ppm.

The SEM images of ligand, Hg (II) and Zr (II) complexes are presented in respectively as a representative example, Generally the Schiff base ligand host a smooth surface morphology, while the complexes presented heterogeneous features, with smooth and rough surface regions. The complexes were also with SEM in which was possible to verify brighter regions evidencing the presence of the metallic species are very rough surfaces.

The antimicrobial activity of both ligand and tow metal complexes are screening of against bacteria and fungi the good zone of inhibiton of bacteria *S.aures* in Hg(II) complex show at 33 mm and reference material *ciprofloxazin* show that inhibition 26nm. Then the Zr(IV) complex show that S.aures 14 mm and reference material *ciprofloxazin* show that inhibition zone at 26 mm. The antifungal activity the high zone of inhibition of Fungi *A.niger* in Hg (II) complex show at 40 mm and antifungal referecnce material *amphoterecin – B* show at 14 mm that the Zr (IV) complex show that *C.albicans* in 37 mm and *Amphotercin- B* no zone of inhibition. These compounds exhibited significant activity against all tested microorganism.

CONCLUSION

The Schiff base ligands from readily and in high yield upon reacting Indole-3-carboxaldehyde. These Schiff base readily coordinate to Hg (II) , Zr (IV) complexes most of the Hg(II) complex as well as $HgCl_2$, exhibit moderate antibacterial activity and good antifungal activity.

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