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Exploring Bismuth (III) Sulpha Drugs Schiff Base Coordination Complexes: Design, Synthesis, Structural, Spectroscopic Characterization, and Biological Evaluations

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Abstract

The present study aimed to prepare and then evaluate the tetra and coordinated Bismuth (III) complexes [$BiCl_2$ (L)] (LH = N, O donor Schiff base employed by the condensation of 1-acetyl-2-naphthol and 2-acetyl-1-naphthol using sulpha drugs as ligand) were derived by the reaction of bismuth (III) trichloride and sodium salt of an N, O donor Schiff base ligand and characterized. The authenticity of all the synthesized ligands and their Bi (III) complexes had been identified by microanalysis, various spectroscopic techniques like FT-IR, ^{1}H NMR, and elemental analysis. The spectral evidence of the complexes has revealed the bidentate complexing nature of the Schiff base through phenolic oxygen and azomethine nitrogen atoms. The Schiff base ligand and Bismuth (III) complexes were assessed for antimicrobial activity. Assessment of antimicrobial activity against Gram-negative & Gram-positive bacteria and fungi has demonstrated that the complex (III) was more active than the Schiff base.

Keywords

Antimicrobial, Bismuth (III) complexes, Schiff base, Spectral evidence

1. Introduction

Schiff bases are considered a very important class of organometallic chemistry, which has wide applications in many biological aspects (antibacterial, antifungal, antitumor, anticancer, anticorrosion, and anti-inflammatory [1-4]. A perusal of the literature reveals that condensation products of sulpha drugs and carbonyl compounds are not only good bacteriostatic but are also good complexing agents. The efficacy of the sulpha drug Schiff bases can be enhanced upon coordination with a suitable metal ion, due to the presence of the SO_2NH moiety as an important toxophoric function [5-6]. Recently, several reports have appeared detailing structural investigations of bismuth (III) halide complexes with donor ligands and the relation of observed structures to the stereochemical activity of the lone pair of electrons on the bismuth atoms [7-9]. Despite the widespread use of bismuth compounds in medicine and the efforts devoted to developing new bismuth compounds for the treatment of a variety of diseases due to their high effectiveness and low toxicity in the treatment of a variety of antitumor activity [10], pharmacology [11], catalytically activity and microbial infections, including syphilis, gastritis and colitis [12]. The cytotoxic and antiproliferative activities of bismuth (III) complexes have been investigated [13-17].

In the present work keeping all these things in consideration I am very enthusiastic to synthesize bismuth (III) Schiff bases complexes [$BiCl_2$ (L)] with N, O donor Schiff bases (L^1H-L^6H) derived from sulpha drugs (sulphadimidine, sulphaguinidine and sulphadiazene) and 1-acetyl-2-naphthol and 2-acetyl-1-naphthol were obtained. The antimicrobial activity of the bismuth (III) complex was investigated against the growth of Gram-positive *Staphylococcus aureus*, Gram-negative *Escherichia coli* bacteria and *Aspergillus niger*, *Rhizopus phaseoli* fungi. The structures of the ligand and bismuth (III) complexes were characterized and also discussed by elemental analysis, molar conductance, IR, and NMR spectra.

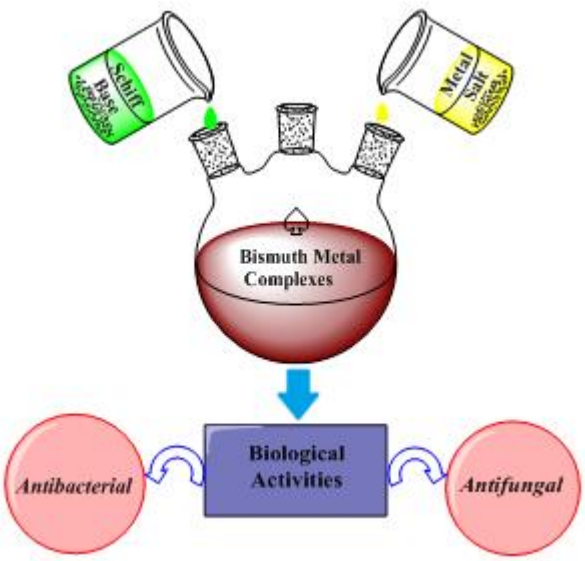

Figure 1. Schematic presentation of the application ofSchiff bases complexes.

2. Experimental

2.1. Materials

All chemicals and solvents used for the synthesis were of reagent grade. 1-acetyl-2-naphthol, 2-acetyl-1-naphthol, and sulpha drugs (Aldrich), bismuth trichloride were obtained commercially and used as received.

2.2. Physical Techniques

All the reactions were carried out under strictly anhydrous conditions. IR spectra were acquired using a model Shimadzu Spectrophotometer by preparing in KBr medium on an FTIR spectrometer. Nitrogen was estimated by Kjeldal's method. Molecular weights were determined by Rast's camphor method. Molar conductance measurements were made in anhydrous DMF at 34±1° using a Systronics Model 305 conductivity bridge. Melting points were measured by the melting points apparatus.

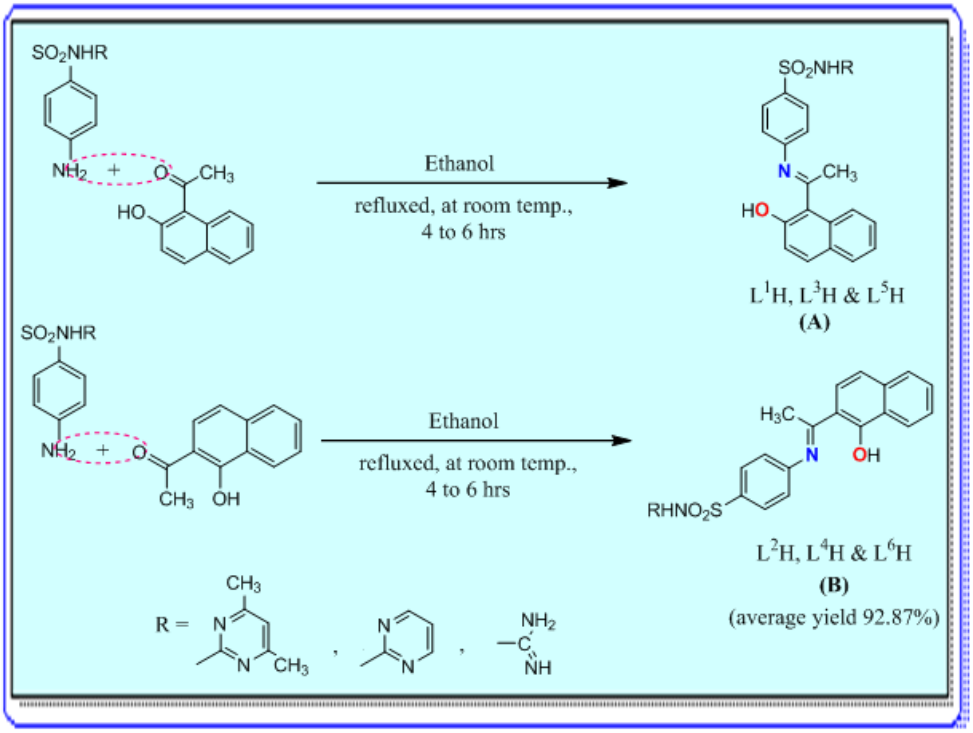

Figure 2. Scheme for the preparation of ligands

2.3. Synthesis of Ligands

1-acetyl-2-naphtholsulphadimidine (L^1H) was synthesized by the condensation of 1-acetyl-2-naphthol with sulpha drug. Schiff bases were prepared by the condensation of various sulpha drugs with 1-acetyl-2-naphthol and 2-acetyl-1-naphthol. The ligands used were L^2H (2-acetyl-1-naphtholsulphadimidine), L^3H (1-acetyl-2-naphtholsulphadiazene), L^4H (2-acetyl-1-naphtholsulphadiazene), L^5H (1-acetyl-2-naphtholsulphaguinidine), L^6H (2-acetyl-1-naphthol sulphaguinidine). Reactants were taken in a 1:1 molar ratio using ethanolic as a reaction medium in the presence of sodium acetate. The solution was refluxed in a water bath from 4 to 6 hours and then allowed to cool at room temperature. The dried crystalline solids were purified by washing with ethanol and recrystallization with acetone. The physical properties and analytical data are

recorded and the structure of ligands (type A and B) proposed in the present work is given in Fig.2.
1-acetyle-2-napthol sulphadimidine ($C_{24}H_{22}N_4O_3S$)[L^1H], Light Yellow Solid, m.p. 180 ºC; 2-acetyle-1-napthol sulphadimidine ($C_{24}H_{22}N_4O_3S$)[L^2H], Pale YellowSolid, m.p. 184 ºC; 1-acetyle-2-naptholsulphadiazine($C_{22}H_{18}N_4O_3S$)[L^3H],Light BrownSolid, m.p. 178 ºC; 2-acetyle-1-naptholsulphadiazine ($C_{22}H_{18}N_4O_3S$)[L^4H], YellowishPowder, m.p. 186 ºC;1-acetyle-2-naptholsulphaguanidine($C_{19}H_{18}N_4O_3S$)[L^5H], Light Brown Solid, m.p. 210 ºC; 2-acetyle-1-naptholsulphaguanidine ($C_{19}H_{18}N_4O_3S$)[L^6H], Light Brown Solid, m.p. 205 ºC.

2.4. Synthesis of Complexes

The sodium salt of the monofunctional bidentate ligand ($L^{1\text{-}6}H$) was dissolved in methanol in a round bottom flask. At the same time, bismuth (III) chloride was dissolved separately in dry methanol. Then, bismuth (III) chloride solution was added dropwise into the flask containing the ligand solution in 1:1 molar ratios. The contents were refluxed for about 4-5 hours. The solid compound obtained was filtered, washed repeatedly with ethanol and dried over anhydrous $CaCl_2$ in a desiccator. The white sodium chloride formed during the reaction was removed by filtration. The purity of the compounds was checked by TLC using silica gel-G as an adsorbent. The physical properties and analysis of these complexes are listed (Figure 3 & 4).
Bi [$C_{24}H_{21}Cl_2N_4O_3S$]**(a(i))**:Yellowsolid; yield 77%; m.p. 220 ºC; Calcd. : N, 7.72; S, 4.42; Cl, 9.77; Bi, 28.81(%), Found: N, 7.36; S, 4.26; Cl, 9.58; Bi, 28.81(%); Mol. wt. Calcd. : 725.40, Found: 723.98.
Bi [$C_{24}H_{21}Cl_2N_4O_3S$]**(b(ii))**:Creamysolid; yield 79%; m.p. 178 ºC; Calcd. : N, 7.72; S, 4.42; Cl, 9.77; Bi, 28.81 (%), Found: N, 7.48; S, 4.20; Cl, 9.72; Bi, 28.36 (%); Mol. wt. Calcd. : 725.40, Found: 724.12.
Bi[$C_{22}H_{17}Cl_2N_4O_3S$]**(a(iii))**: Brownsolid; yield 82%; m.p. 168 ºC; Calcd. : N, 8.03; S, 4.60; Cl, 10.17; Bi, 29.97 (%), Found: N, 7.88; S, 4.56; Cl, 10.06; Bi, 29.56 (%); Mol. wt. Calcd. : 697.35, Found:696.34.
Bi [$C_{22}H_{17}Cl_2N_4O_3S$]**(b(iv))**: Light Yellowsolid; yield 80%; m.p. 208 ºC; Calcd. : N, 8.03; S, 4.60; Cl, 10.17; Bi, 29.97 (%), Found: N, 7.98; S, 4.28; Cl, 10.12; Bi, 29.26 (%); Mol. wt. Calcd. : 697.35, Found:695.99.
Bi [$C_{19}H_{17}Cl_2N_4O_3S$]**(a(v))**: Orangesolid; yield 74%; m.p. 235 ºC; Calcd. : N, 8.47; S, 4.85; Cl, 10.72; Bi, 31.60 (%), Found: N, 8.08; S, 4.56; Cl, 10.67; Bi, 31.12 (%); Mol. wt. Calcd.:661.35, Found:659.78.
Bi[$C_{19}H_{17}Cl_2N_4O_3S$]**(b(vi))**: Brownsolid; yield 74%; m.p. 195 ºC; Calcd. : N, 8.47; S, 4.85; Cl, 10.72; Bi, 31.60 (%), Found: N, 8.16; S, 4.36; Cl, 10.56; Bi, 31.22 (%); Mol. wt. Calcd. : 661.35, Found:660.16.

3. Bioassay

3.1. Antibacterial Activity

In vitro, the antibacterial activity of the ligands and their corresponding complexes were evaluated against *Escherichia coli* (-) and *Staphylococcus aureus* (+) by using the paper disc plate method [18-22]. The nutrient agar medium was used as a culture medium for bacterial growth. The compounds under investigation were dissolved in DMSO to get final concentrations of 500 and 1000 ppm. The paper disc (Whatman no.1) having a diameter of 5 mm was soaked in these solutions and placed in an appropriate medium previously seeded with the tested organism in Petri dishes. The plate was incubated for 24 h at 30±2°. After inoculation, the diameter of the clear zone of inhibition surrounding each sample is taken as a measure against the particular test organism [23-25]. Streptomycin was used as a reference compound for antibacterial activities. The antibacterial activities displayed by ligands and their complexes are shown in Table 3.

3.2. Antifungal Activity

Antifungal activity of the ligands and their corresponding complexes is found in vitro against *Aspergillus niger* and *Rhizopus phaseoli* by the agar plate technique [26]. The solutions of the compounds in different concentrations (100 and 200 ppm) in DMF were then mixed with the medium. The linear growth of the fungus was recorded by measuring the diameter of the colony and the percent inhibition was calculated. Micostatin was used as a reference compound for antifungal activities (Table 4).

4. Results and discussion

The reactions that led to the formation of the $[Cl_2Bi(N\frown O)]$ and by the reaction of $BiCl_3$ with sulpha drugs Schiff base ligands in a 1:1 molar ratio in methanol as shown below-

$$BiCl_3 + NaN\frown O \xrightarrow[\text{methanol}]{1:1} [Cl_2Bi(N\frown O)] + NaCl$$

The above reaction was feasible with the liberation of NaCl, which was removed. The reaction was completed in ~ 4-5 h of refluxing. After removing the solvent under reduced pressure, colored solid compounds were obtained, which were found to be soluble in MeOH and DMSO. Molecular weight determinations show them to be monomeric. The molar conductances of 10^{-3} mol L^{-1} solutions of the complexes in dry DMF lie in the 16–18 $\Omega^{-1}cm^2$ mol^{-1} range, indicating that they are non-electrolytes. The products were isolated in virtually qualitative yield and satisfactorily characterized by elemental analyses and various spectroscopic techniques.

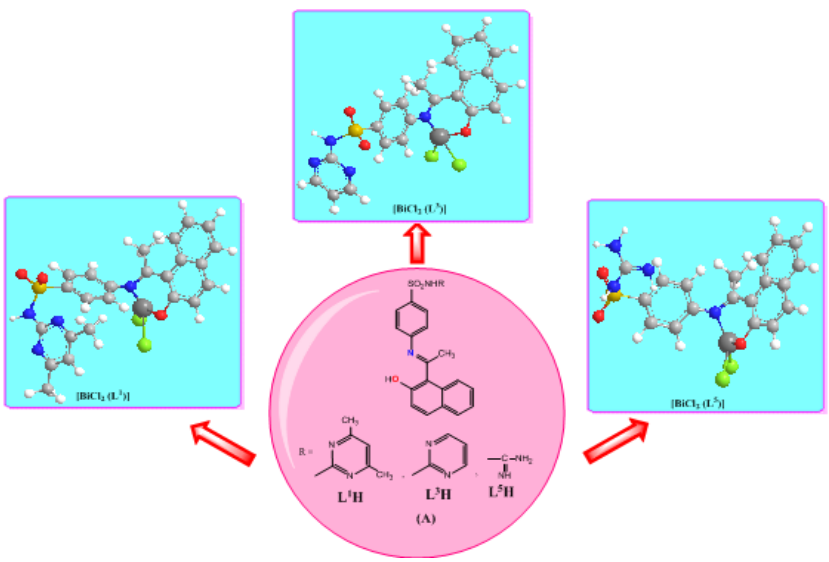

Figure 3. Structure of the complexes (1:1) [a- i, iii & v]

4.1. Infrared Spectra

The relevant IR spectral bands and their assignments of the synthesized Schiff base and its metal complexes are represented in Table 1. IR spectra of the ligands two broad bands in the region 3450-3150 cm^{-1} are observed which can be assigned to ν(NH) and ν(OH) phenolic vibrations. No significant change in ν (NH) band in the spectra of complexes indicates non-involvement of the NH group in coordination [27-28].

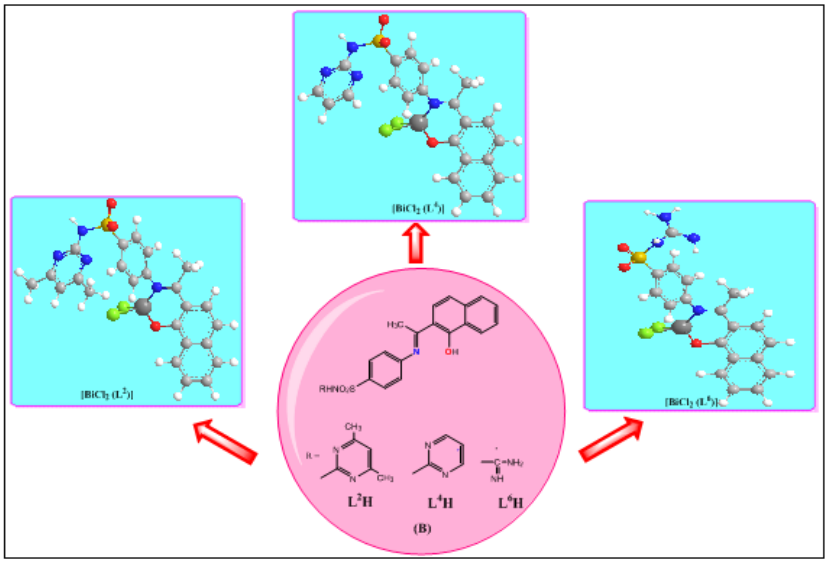

Figure 4. Structure of complexes (1:1) [b- ii, iv & vi]

However, the band due to the –OH group disappears in the complexes showing the coordination of bismuth through oxygen after deprotonation. A medium-intensity band appears in the ligand at ~1280 cm^{-1} due to the phenolic C-O stretching vibrations. The chelation of the phenolic oxygen to the bismuth atom is indicated by the shifting of this band's higher frequency side (~1300 cm^{-1}), indicating the chelation through the phenolic oxygen to the bismuth atom. The azomethine ν(>C=N-) band at 1610-1620 cm^{-1} in Schiff base is shifted to a lower frequency in ~18-22 cm^{-1} indicating the co-ordination of azomethine nitrogen on complexation. The linkage with the oxygen atom is further supported by the appearance of a band in the region around 420-435 cm^{-1} which may be assigned to ν (Bi-O) [29-30]. Further evidence of the coordination of the N atom of the Schiff base with the metal atom is shown by the appearance of a new weak frequency band at 347-365 cm^{-1} assigned to the metal nitrogen ν(Bi← N) [31-32]. These new bands were observed only in the spectra of the metal complexes and not in the Schiff base which confirmed the participation of the donor groups. The bands in the ligand due to ν(SO_2) and ν(SO_2) appear at 1155 $cm^{-1}$and 1332 cm^{-1}respectively. These bands almost remain unchanged in the complexes indicating that this –SO_2 group is not participating in coordination. This is confirmed by the unchanged ν(S-N) and ν(C-S) modes appearing around 975 $cm^{-1}$and 869 cm^{-1}respectively.

Table 1. IR spectral data (in cm-1) of the ligands and their bismuth complexes

Compound	ν (O-H)	ν (C-O)	ν (>C=N-)	ν (Bi-N)	ν (Bi-O)
($C_{24}H_{22}N_4O_3S$) **(L^1H)**	3150-3450	1275	1615	-	-
($C_{24}H_{22}N_4O_3S$) **(L^2H)**	3145-3415	1280	1620	-	-
($C_{22}H_{18}N_4O_3S$) **(L^3H)**	3140-3425	1200	1615	-	-
Bi [$C_{24}H_{21}Cl_2N_4O_3S$]**(a(i))**	-	1310	1605	360	420
Bi [$C_{24}H_{21}Cl_2N_4O_3S$]**(b(ii))**	-	1300	1605	355	435
Bi [$C_{22}H_{17}Cl_2N_4O_3S$]**(a(iii))**	-	1310	1600	365	422

4.2. Electronic spectra

Electronic spectra of the ligands in methanol display maxima at ~264 and ~326 nm, which are due to *л-л** electronic transitions and remain almost unchanged in the spectra of the metal complexes. The band at 370nm is due to n–*л** transitions of the >C=N- chromophore and shows a bathochromic shift of 20–30nm after coordination of azomethine nitrogen to the metal, indicating delocalization of the electronic charge within the chelate ring [33].

4.3. ^{1}H NMR spectra

The ^{1}H spectra of ligands display signal at δ 12.10-12.20 ppm due to –OH proton which disappears in the spectra of the complexes showing chelation through oxygen. The ligands show NH proton signals at δ 10.35-10.50, which remain unchanged in the compounds showing non-participation of the NH group [34] in the complex formation. Proton signals at 1.82-1.85 due to methyl proton of [-C(CH_3)=N-] [35] group shows a downfield shift, thereby indicating coordination through the nitrogen of >C=N- group. This is probably due to the donation of the lone pair of electrons by the nitrogen to the metal atom resulting from the formation of the lone pair of electrons by the nitrogen to the metal atom resulting in the formation of the coordination linkage (Bi←N). The ligands show a complex multiplet in the region δ 7.80-6.90 ppm for the aromatics protons and remain almost at the same position in the spectra of the bismuth complexes (Table 2).

Table 2. ^{1}H NMR spectral data (δ, ppm) of the ligands and their bismuth complexes

Compound	-CH_3	-SO_2NH	Aromatic protons	-OH
($C_{24}H_{22}N_4O_3S$) **(L^1H)**	1.86	10.42s	6.92-7.50m	12.16bs
($C_{24}H_{22}N_4O_3S$) **(L^2H)**	1.84	10.35s	6.95-7.62m	12.10bs
($C_{22}H_{18}N_4O_3S$) **(L^3H)**	1.82	10.40s	7.00-7.85m	12.20bs
Bi [$C_{24}H_{21}Cl_2N_4O_3S$] **(a(i))**	1.95	10.50s	6.95-7.62m	-
Bi [$C_{24}H_{21}Cl_2N_4O_3S$] **(b(ii))**	1.98	10.45s	6.95-7.85m	-
Bi [$C_{22}H_{17}Cl_2N_4O_3S$] **(a(iii))**	2.03	10.45s	7.02-7.85m	-

4.4. Antimicrobial results

The results of the antimicrobial screening of the Schiff bases and their complexes against Gram-negative (*Escherichia coli*) & Gram-positive (*Staphylococcus aureus*) bacteria and some selected fungi (*Aspergillus niger* and *Rhizopus phaseoli)* have been found. The inhibition zones were measured in mm and results have been recorded in Table 3 &Table 4.

The experimental data indicate that the metal complexes have more potent activity in inhibiting the growth of microorganisms than the ligands. The result further shows that the antimicrobial activity of the complexes enhanced as compared to its starting material [36-37] which indicates that chelation increases the activity.

Table 3. Antibacterial screening data of the ligands and Bi(III) complexes

Diameter of inhibition zone (mm)after 24 h (Conc. in ppm)				
Compd.	*Escherichia coli (–)*		*Staphylococus aureus (+)*	
$(C_{24}H_{22}N_4O_3S)$ **(L^1H)**	7	9	11	15
$(C_{24}H_{22}N_4O_3S)$ **(L^2H)**	8	1	13	16
$(C_{22}H_{18}N_4O_3S)$ **(L^3H)**	10	12	11	14
Bi $[C_{24}H_{21}Cl_2N_4O_3S]$ **(a(i))**	10	14	16	19
Bi $[C_{24}H_{21}Cl_2N_4O_3S]$ **(b(ii))**	12	17	18	18
Bi $[C_{22}H_{17}Cl_2N_4O_3S]$ **(a(iii))**	14	19	20	22
Streptomycin	16	21	19	20

Table 4. Antifungal activity of the ligands and Bi (III) complexes

Micostatin		$(C_{24}H_{22}N_4O_3S)$ **(L^2H)**		Bi $[C_{24}H_{21}Cl_2N_4O_3S]$ **(b(ii))**		$(C_{22}H_{18}N_4O_3S)$ **(L^3H)**		Bi $[C_{22}H_{17}Cl_2N_4O_3S]$ **(a(iii))**	
		100 ppm	200 ppm	100 ppm	200 ppm	100 ppm	200 ppm	100 ppm	200 ppm
A. niger	IZ	25	24	18	19	24	23	14	18
	(AI)	1.80	1.75	1.30	1.35	1.72	1.65	1.00	1.25
R. phaseoli	IZ	14	27	21	22	14	16	17	18
	(AI)	1.93	1.57	1.50	1.54	0.98	1.86	1.18	1.27

IZ = Inhibition zone (diameter in ppm); AI = Activity index (inhibition zone of tasted compounds), Inhibition (%) after 96 hrs at 25 ± 2°C.

5. Conclusion

Our present studies describe the synthesis, characterization, and biological activity of sulpha drug Schiff bases ligands and Bi (III) complexes. Based on the above analytical and spectral evidence the ligands coordinate to metal monobasic bidentate giving four and five-coordinate geometries around the metal. The complexes showed better antimicrobial activities than parent ligands. The compounds showed toxicity against all species of fungi and inhibition zone growth of fungi depends on the concentration of the compounds. In the present case, we have used Escherichia coli for antibacterial activity and Aspergillus niger for antifungal activity against tested microorganisms of the studied complexes. The results showed that the compounds are more active than the ligands but less active than these standard drugs.

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