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Research Article

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Organotin(IV) complexes with ONS donor Schiff base ligand: Synthesis, characterization and antimicrobial evaluation

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ABSTRACT

Organotin(IV) complexes [R_2SnL^1, R_2SnL^2); R= Me, Et, Bu and Ph] have been synthesized by reaction of diorganodichlorotin(IV) with biological potent Schiff base ligands 5- hydroxymethyl-4-[(2-mercapto-phenylimino)-methyl]-2-methyl-pyridin-3-ol (H_2L^1), 4-[(5- chloro-2-mercapto-phenylimino)-methyl]-5-hydroxymethyl-2-methyl-pyridin-3-ol(H_2L^2) derived from pyridoxal hydrochloride with 2-aminothiophenol and 2-amino-4-chlorothiophenol respectively. The geometry of these compounds has been proposed on basis of elemental analyses, molar conductance and spectroscopic techniques (IR, electronic, ^1H, ^{13}C and ^{119}Sn NMR). On the basis of these studies it revealed that Schiff base ligands acted as ONS donor system and coordinated to tin atom in tridentate fashion with trigonal pyramidal geometry around tin atom. To compare the biopotency of these complexes, Schiff base ligands and their complexes were also tested for in vitro antimicrobial evaluation against some pathogenic fungi and bacteria. Enrichment was observed in biological activity of ligands on coordination with tin atom.

Key words: Organotin(IV), Spectroscopic, Biopotency, Pathogenic, geometry

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INTRODUCTION

Among metallic compounds of p-block elements that have been studied are organotin compounds. The chemistry of organotin(IV) complexes of Schiff bases has engrossed more intension due to their pharmacological activities like antibacterial, antifungal, antitumour [1,2], teratogenicity and neurotoxicity in animals and humans [3,4] industrial applications like pesticides, antifouling paints and fire retardants [5]. In last few years, large number of metal complexes of Schiff bases containing NS, NO and ONS donor atoms have been studied [6,7] attributed due to their structural features and the reported antimicrobial, carcinostatic and antiviral activity of nitrogen, oxygen, sulfur donor ligandss and their metal complexes [8.9]. Chelates of organotin(IV) moieties with these donor ligandss show significant activity [10-12]. Organotin compounds having formulae R_nSnX_{4-n} are biologically active in which alkyl group is important for measuring toxicity towards living species [13,14] this activity enhanced on coordination with Schiff base ligands [15,16]. Keeping these findings in mind and our interest in field of organotin complexes, this paper reports synthesis, spectral characterization and biological evaluation of organotin(IV) complexes of Schiff base ligands in order to investigate the effect of organic group on antimicrobial evaluation of organotin(IV) .

EXPERIMENTAL SECTION

Dimethyltindichloride, diethyltindichloride, dibutyltindichloride, and diphenyltindichloride, 2-aminothiophenol, 2-amino-4-chlorothiophenol were obtained through Aldrich and were used as such without any further purification. All the reagents and solvents used were dried, distilled and purified by the standard methods and purity was checked by thin layer chromatography. The reactions were carried out under strict anhydrous conditions and sufficient care has been taken to keep the organotin(IV) complexes, chemicals and glass apparatus free from moisture. Tin content was estimated gravimetrically as SnO_2. Elements (C, H, and N) were analyzed on Perkin-Elmer 2400 instrument (Waltham, Massachusetts) IR spectra were recorded on Shimadzu IR affinity-I 8000 FT-IR spectrometer using KBr disc having wavelength range 400-4000 cm^{-1}. NMR spectra were recorded on Bruker Avance II 400 MHz NMR spectrometer and all chemical shifts were reported in parts per million relative to TMS as internal standard. Electronic spectra were recorded on UV-VIS-NIR Varian Cary-5000 spectrometer in DMF. Molar conductance measurements of a 10^{-3} M solution of metal complexes in DMF at room temperature were carried out using a model-306 Systronics.

Synthesis of Schiff base ligands (H_2L^{1-2})

Pyridoxal hydrochloride (2.03 g, 10 mmol) was dissolved in methanol (25 mL) and added to a solution of sodium methoxide (0.54 g, 10 mmol) in the same solvent. A methanolic solution of 2-aminothiophenol (1.25 g, 10 mmol) / 2-amino-4-chlorothiophenol (1.59 g, 10 mmol) was added dropwise to reaction mixture. The reaction mixture was refluxed for 4h and the mixture was kept for overnight at room temperature. The solid formed during reaction was isolated and filtered, washed with small amount of water to remove sodium chloride, followed by diethyl ether, and then dried, similar procedure was adopted to isolate other ligands.

H_2L^1: mpt. 195°C, yield: 75%, (found: C, 61.60; H, 5.31; N, 10.05; S, 11.92. Calcd. For $C_{14}H_{14}N_2O_2S$: C, 61.29: H, 5.14; N, 10.21; S, 11.69)

H_2L^2: mpt. 172°C, yield: 80%, (found: C, 54.85; H, 4.44; N, 9.28; S, 10.04. Calcd. For $C_{14}H_{13}N_2O_2SCl$: C, 54.46: H, 4.24; N, 9.07; S, 10.38)

Synthesis of Diorganotin(IV) Complexes of 5-hydroxymethyl-4-[(2-mercapto-phenylimino)-methyl]-2-methyl-pyridin-3-ol (H_2L^1)

Diorganotin(IV)dichloride complexes were prepared by the reaction of diorganotindichloride (5mmol) in dry tetrahydrofuran (20 mL) and 5-hydroxymethyl-4-[(2- mercapto-phenylimino)-methyl]-2-methyl-pyridin-3-ol (1.37g, 5 mmol) (H_2L^1) and dry triethylamine (10 mmol) taken in a same solvent, at the room temperature under dry nitrogen atmosphere. The reaction mixture was stirred for 5 h. Triethylamine hydrochloride formed was filtered off and excess of the solvent was removed under vacuum. The solid obtained was dried under reduced pressure and washed with n-hexane and finally dried over vacuum. Similar procedure was adopted for synthesis of diorganotin(IV) Complexes of 4-[(5-chloro-2-mercaptophenylimino)-methyl]-5-hydroxymethyl-2-methyl-pyridin-3-ol (H_2L^2).

Me_2SnL^1: yield: 60%, (found: C, 45.90; H, 4.84; N, 6.93; S, 7.94; Sn, 28.42. Calcd. For $C_{16}H_{18}N_2O_2SSn$: C, 45.64; H, 4.31; N, 6.65; S, 7.61; Sn, 28.19)

Et_2SnL^1: yield: 64%, (found: C, 48.54; H, 5.12; N, 6.01; S, 7.42; Sn, 26.85. Calcd. For $C_{18}H_{22}N_2O_2SSn$: C, 48.13; H, 4.94; N, 6.24; S, 7.14; Sn, 26.43)

Bu_2SnL^1: yield: 58%, (found: C, 52.67; H, 5.56; N, 5.80; S, 6.72; Sn, 23.10. Calcd. For $C_{22}H_{30}N_2O_2SSn$: C, 52.30; H, 5.98; N, 5.54; S, 6.35; Sn, 23.49)

Ph_2SnL^1: yield: 62%, (found: 57.49: H, 4.37; N, 4.80; S, 6.04; Sn, 21.96. Calcd. For $C_{26}H_{22}N_2O_2SSn$: C, 57.27; H, 4.07; N, 5.14; S, 5.88; Sn, 21.77)

Me_2SnL^2: yield: 61%, (found: C, 42.43; H, 3.45; N, 6.34; S, 6.85; Sn, 26.42. Calcd. For $C_{16}H_{17}ClN_2O_2SSn$: C, 42.18: H, 3.76; N, 6.15; S, 7.04; Sn, 26.06)

Et_2SnL^2: yield: 57%, (found: C, 44.45; H, 4.72; N, 5.54; S, 6.24; Sn, 24.55. Calcd. For $C_{18}H_{21}ClN_2O_2SSn$: C, 44.70: H, 4.38; N, 5.79; S, 6.63; Sn, 24.55)

Bu_2SnL^2: yield: 59%, (found: C, 48.63; H, 5.65; N, 5.46; S, 5.45; Sn, 21.74. Calcd. For $C_{22}H_{29}ClN_2O_2SSn$: C, 48.96; H, 5.42; N, 5.19; S, 5.94; Sn, 22.00)

Ph_2SnL^2: yield: 63%, (found: C, 53.32; H, 3.93; N, 4.45; S, 5.16; Sn, 20.85. Calcd. For $C_{26}H_{21}ClN_2O_2SSn$: C, 53.87; H, 3.65; N, 4.83; S, 5.53; Sn, 20.48)

RESULTS AND DISCUSSION

Schiff base ligands H_2L^{1-2} reacted with R_2SnCl_2 in 1:1 molar ratio in presence of tetrahydrofuran (THF) and triethylamine as base to form addition products R_2SnL^{1-2}. All these metal complexes obtained as solids and found to be mostly insoluble in organic solvents except DMF and DMSO. These complexes were found to be monomeric and have low conductivity in range 2.5- 8.9 $\Omega^{-1}cm^2mol^{-1}$ in DMF showed non electrolytic nature of complexes.

$$R_2SnCl_2 + H_2L \xrightarrow[Et_3N\cdot\cdot]{THF} R_2Sn(L) + Et_3NH^+Cl^-$$

R = Me, Et, Bu and Ph

IR Spectra

IR spectra of complexes were compared with that of respective ligands (Table-1). Disappearance of strong bands at 2725-2734 cm^{-1}, 3236-3240 cm^{-1} due to ν (S-H) and ν (O-H) of Schiff base ligands the appearance of a new band around 740-748 cm^{-1}, 1015-1023 cm^{-1} due to ν (C–S) and ν (C–O) showed the deprotonation of thiol and hydroxyl group and involvement of sulfur and oxygen in coordination with tin, that was further supported by presence of new bands in region 410-425 cm^{-1} and 510-545 cm^{-1} due to ν (Sn-S) and ν (Sn-O) respectively [17,18]. In metal complexes ν (C=N) band observed at 1590-1615 cm^{-1} where as for free ligands it was observed at 1635-1638 cm^{-1}, this shifting of band showed coordination of azomethine nitrogen in complexation that was confirmed by presence of new bands at 445-470 cm^{-1} due to ν (Sn-N) [19].

Electronic Spectra

In electronic spectra of ligands, phenyl ring showed a band at 220 nm which shifted to higher wavelength 245-258 nm on complexation. An azomethine C=N group absorbs at 295 nm which shifted to higher wavelength in complexes. Sharp bands observed in range of 230-285nm which may be due to charge transfer and dπ-pπ bonds between oxygen, nitrogen, sulfur of ligands and vacant 5d orbitals of tin [20].

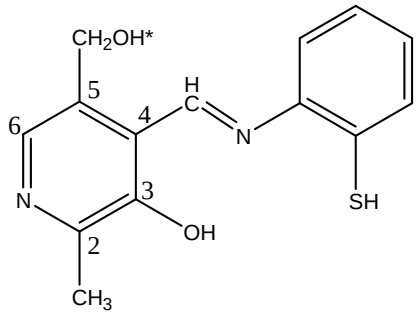
Table-1: IR spectral characteristics (cm^{-1}) of Schiff base ligands and their diorganotin(IV) Complexes

Ligands/ complexes	ν (S-H)	ν (O-H)	ν (C=N)	ν (C-S)	ν (C-O)	ν (Sn-O)	ν (Sn-O)	ν (Sn-S)
H_2L^1	2725	3236	1635	--	--	--	--	--
H_2L^2	2734	3240	1638	--	--	--	--	--
Me_2SnL^1	--	--	1598	743	1015	525	448	410
Et_2SnL^1	--	--	1603	745	1016	536	461	420
Bu_2SnL^1	--	--	1590	740	1023	515	445	416
Ph_2SnL^1	--	--	1610	745	1016	545	458	425
Me_2SnL^2	--	--	1594	742	1020	510	462	418
Et_2SnL^2	--	--	1603	742	1022	518	450	424
Bu_2SnL^2	--	--	1599	748	1018	527	465	417
Ph_2SnL^2	--	--	1615	746	1020	542	470	419

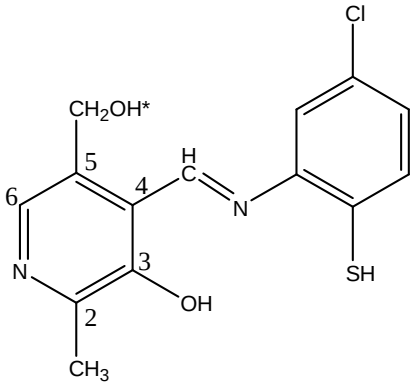
^{1}H NMR Spectra

^{1}H NMR spectra of ligands and their organotin (IV) complexes were recorded in DMSO-d_6 (Table-2). Ligands (H_2L^1, H_2L^2) showed signal at δ 5.80, 5.82 and δ 11.35, 11.37 due to thiol (SH) proton and phenolic (OH) proton respectively. On comparison of ^{1}H NMR of ligands and their organotin(IV) complexes, absence of these proton signals in complexes showed deprotonation when attached to tin atom. Signals at δ 9.35 and 9.39 was observed due to azomethine proton in the spectra of free ligands H_2L^1 and H_2L^2 respectively, which shifted downfield δ 9.56-9.65 in case of metal complexes supported involvement of azomethine nitrogen in coordination. Alcoholic proton of

pyridoxal moiety as a singlet of (CH_2-OH*) group was observed at δ 5.34 while methylene, methyl proton and another atomic proton appeared at δ 4.58, δ 2.43 and δ 7.28-7.59 respectively in ligands which almost unaltered on coordination with metal atom. Chemical shift of methyl, ethyl, butyl and phenyl group attached to tin appeared at singlet δ 1.13-1.15, multiplet in region at δ 1.04-1.26, multiplet in range at δ 0.94-1.98 and multiplet in range at δ 6.93-7.98 respectively.



H_2L^1



H_2L^2

^{13}C NMR Spectra

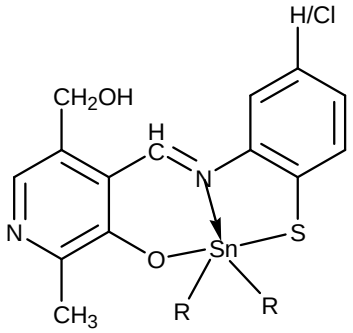
^{13}C NMR spectra of ligands and their organotin (IV) complexes were recorded in DMSO-d_6 (Table-2). The signals due to carbon attached to hydroxy group, azomethine carbon atom and carbon attached to thiol group in ligandss observed at δ 189.34-189.40, δ 164.03-164.20 and δ 197.42-197.65 shifted to δ 192.45-194.84, δ 170.45- 171.34 and δ 193.78-195.30 in metal complexes. Deshielding of these signals of carbon atom attached to oxygen, nitrogen and sulfur observed may be due to coordination of phenolic oxygen, azomethine nitrogen and thiol sulfur to tin. Small shift was observed in signal of methylene, methyl and aromatic carbon atom signal. The signals due to methyl, ethyl, butyl group attached to the tin atom appeared in range δ 9.41–30.45 and phenyl group attached to tin appeared in range δ 147.50-126.15.

Table-2: ^{1}H NMR and ^{13}C NMR spectral data (δ, ppm) of Schiff base ligands and their diorganotin(IV) complexes

Ligands/ complexes	^{1}H NMR (δ, ppm)	^{13}C NMR (δ, ppm)
H_2L^1	5.80(s, 1H, SH), 11.35 (s, 1H, OH), 9.35 (s,1H, CH=N), 5.34 (s, 1H, OH*), 4.58 (s, 2H,CH_2), 2.43 (s, 3H, CH_3), 7.28-7.56 (m, Ar-H)	189.34 (C-OH), 164.03 (>C=N), 197.65 (C_5), 61.54 (CH_2), 12.32 (CH_3), 128.34-148.34 (Ar-C)
H_2L^2	5.82(s, 1H, SH), 11.37 (s, 1H, OH), 9.39 (s, 1H, CH=N), 5.34 (s, 1H, OH*), 4.58 (s, 2H, CH_2), 2.43 (s, 3H, CH_3), 7.32-7.59 (m, Ar-H)	189.40 (C-OH), 164.20 (>C=N), 197.42 (C_5), 60.65 (CH_2), 12.24 (CH_3), 129.09-147.82 (Ar-C)
Me_2SnL^1	9.57 (s, 1H, CH=N), 5.38 (s, 1H, OH*), 4.59 (s, 2H, CH_2), 2.43 (s, 3H, CH_3), 7.26-7.59 (m, Ar-H), 1.13 (s, Sn-Me)	192.52 (C-OH), 170.65 (>C=N), 193.78 (C_5), 61.85 (CH_2), 12.02 (CH_3), 128.88-148.89 (Ar-C), 10.54 (Sn-Me)
Et_2SnL^1	9.56 (s, 1H, CH=N), 5.34 (s, 1H, OH*), 4.58 (s, 2H, CH_2), 2.43 (s, 3H, CH_3), 7.27-7.58 (m, Ar-H), 1.04-1.23 (m, Sn-Et)	194.84 (C-OH), 171.08 (>C=N), 194.54 (C_5), 61.60 (CH_2), 12.38 (CH_3), 128.34-148.34 (Ar-C), 11.25-18.34 (Sn-Et)
Bu_2SnL^1	9.60 (s, 1H, CH=N), 5.36 (s, 1H, OH*), 4.57 (s, 2H, CH_2), 2.40 (s, 3H, CH_3), 7.28-7.56 (m, Ar-H), 0.94-1.90 (m, Sn-Bu)	193.20 (C-OH), 170.45 (>C=N), 193.98 (C_5), 60.98 (CH_2), 12.47 (CH_3), 127.98-148.87 (Ar-C), 12.24-30.28 (Sn-Bu)
Ph_2SnL^1	9.65 (s, 1H, CH=N), 5.32 (s, 1H, OH*), 4.57 (s, 2H, CH_2), 2.38 (s, 3H, CH_3), 7.30-7.57 (m, Ar-H), 6.93-7.80 (m , Sn-Ph)	193.06 (C-OH), 170.86 (>C=N), 195.30 (C_5), 61.85 (CH_2), 12.02 (CH_3), 128.06-148.47 (Ar-C), 126.80-146.89 (Sn-Ph)
Me_2SnL^2	9.59 (s, 1H, CH=N), 5.34 (s, 1H, OH*), 4.54 (s, 2H, CH_2), 2.39 (s, 3H, CH_3), 7.29-7.55 (m, Ar-H), 1.15 (s, Sn-Me)	194.24 (C-OH), 171.18 (>C=N), 194.24 (C_5), 61.34 (CH_2), 12.18 (CH_3), 128.87-148.04 (Ar-C), 9.41 (Sn-Me)
Et_2SnL^2	9.59 (s, 1H, CH=N), 5.34 (s, 1H, OH*), 4.54 (s, 2H, CH_2), 2.39 (s, 3H, CH_3), 7.29-7.55 (m, Ar-H), 1.15 (s, Sn-Me)	193.64 (C-OH), 170.83 (>C=N), 193.89 (C_5), 60.94 (CH_2), 12.49 (CH_3), 128.91-148.04 (Ar-C), 12.04-19.87 (Sn-Et)
Bu_2SnL^2	9.59 (s, 1H, CH=N), 5.36 (s, 1H, OH*), 4.59 (s, 2H, CH_2), 2.41 (s, 3H, CH_3), 7.28-7.56 (m, Ar-H), 0.99-1.98 (m, Sn-Bu)	194.01 (C-OH), 170.63 (>C=N), 195.01 (C_5), 61.04 (CH_2), 12.49 (CH_3), 129.18-148.09 (Ar-C), 13.12-30.45 (Sn-Bu)
Ph_2SnL^2	9.63 (s, 1H, CH=N), 5.39 (s, 1H, OH*), 4.56 (s, 2H, CH_2), 2.42 (s, 3H, CH_3), 7.28-7.60 (m, Ar-H), 6.96-7.98 (m , Sn-Ph)	193.98 (C-OH), 171.34 (>C=N), 194.06 (C5), 61.85 (CH_2), 12.46 (CH_3), 128.94-147.97 (Ar-C), 126.15-147.50 (Sn-Ph)

^{119}Sn NMR Spectra

^{119}Sn NMR chemical shifts are useful for determining coordination number around tin atom. One sharp singlet was observed in ^{119}Sn NMR spectra of organotin(IV) complexes showed formation of single tin species. Complexes gave signals at δ – 181.9 to – 193.5 for all complexes except diphenyltin(IV) complexes, diphenyltin(IV) complexes gave signal from δ – 303.4 to –303.8 respectively supported penta-coordinated environment around the central tin atoms in these complexes with probably trigonal bipyramidal geometry [21, 22] (Figure-1).



R = Me, Et, Bu and Ph

Figure-1: Probable structure for organotin(IV) complexes

Pharmacology

Test microorganisms

Bacillus subtilis (MTCC no. 2063), *Staphylococcus aureus* (MTCC no. 2901), gram negative bacteria *Escherichia coli* (MTCC no. 1652) and fungi *Candida albicans* (MTCC no.183), *Aspergillus niger* (MTCC no. 1344). The bacteria were subcultured on nutrient agar, whereas fungi on Sabouraud dextrose.

Antibacterial assay

Bacterial culture from slant was inoculated into the medium and incubated at 37°C (±1°C) for 24 hours and used as such for testing the compounds by applying the two fold serial dilution technique. A stock solution of test compound was prepared in dry dimethylsulfoxide concentration of 1.00 mg/ml. The stock solution (0.1 mL) was added to sterile nutrient broth for making first dilution (50 µg/mL) and then dilute it to form various concentrations 50, 25, 12.5, 6.25 and 3.12 µg/mL. The tubes were then inoculated with 100 µl of suspension of respective organisms (*B. subtilis*, *S. aureus* and *E. coli*) in sterile saline. The inoculated tubes were incubated at 37°C (±1°C) for 24 hours and minimum inhibitory concentrations (MIC) were determined.

Antifungal assay

The antifungal activity of the test compounds against the fungal species *C. albicans* and *A. niger* was determined by serial dilution method similar to antibacterial assay using Sabouraud dextrose broth following the incubation condition of 37°C (±1°C) for a period of 24 hours for *C. albicans* and 25°C (±2°C) for a period of 7 days for *A. niger*. The results of antimicrobial activity are given in Table-3 and graphical representation is given in Figure-2. The antimicrobial data suggested that:

(i) Diorganotin(IV) complexes were found to be more potent than respective ligands, This increase in the activity of organotin(IV) complexes consequence as a result of coordination with metal ion in the normal cell process explained on basis of chelation theory [23].

(ii) The antimicrobial activity of these ligands and their complexes can also be attributed due to hydrogen bond formation between bioreceptors in the cells of microorganisms and azomethine group (C=N) atom of compounds, which sequentially block the synthesis of protein by inhibiting the movement of ribosome along with RNA. These inhibit their synthesis of DNA in the cell nucleus. The greater toxicity of complexes than the bases can also be explained by the greater lipophilic character of the complexes in comparison of respective ligands.

Table-3: The in vitro antimicrobial activity of Schiff base ligands and their diorganotin(IV) complexes (MIC in µg/mL)

Sr. No	Ligands/ complexes	*B. Subtilis*	*S.Aureus*	*E.Coli*	*C. Albicans*	*A. Niger*
1	**H_2L^1**	50	25	50	25	50
2	**H_2L^2**	50	25	25	50	25
3	**Me_2SnL^1**	25	12.5	12.5	25	25
4	**Et_2SnL^1**	12.5	12.5	12.5	6.25	12.5
5	**Bu_2SnL^1**	12.5	6.25	12.5	6.25	12.5
6	**Ph_2SnL^1**	6.25	3.12	12.5	12.5	6.25
7	**Me_2SnL^2**	50	25	12.5	12.5	25
8	**Et_2SnL^2**	25	12.5	12.5	25	6.25
9	**Bu_2SnL^2**	6.25	6.25	12.5	25	12.5
10	**Ph_2SnL^2**	3.12	6.25	12.5	3.12	6.25

(iii) The order of inhibiting activity was Ph_2SnL^{1-2} > Bu_2SnL^{1-2} > Me_2SnL^{1-2} > Et_2SnL^{1-2} > H_2L^{1-2}, the toxicity depends on the properties of R group present around tin atom. The larger and the more lipophilic the R group, the more toxic is the organotin [24].

(iv) Phenyl Complexes were found to be more active against these strains, this can be explained on basis of electron releasing ability of phenyl group that increase delocalization of π-electrons in whole chelate ring and lipophilicity of the complexes was also enhanced. This improved lipophilicity enhances the penetration of the complexes into the lipid membranes and blocks the metal binding sites in the enzymes of microorganisms. These complexes also perturb the respiration process of the cell and thus block the synthesis of proteins, which restricts additional growth of the organism.

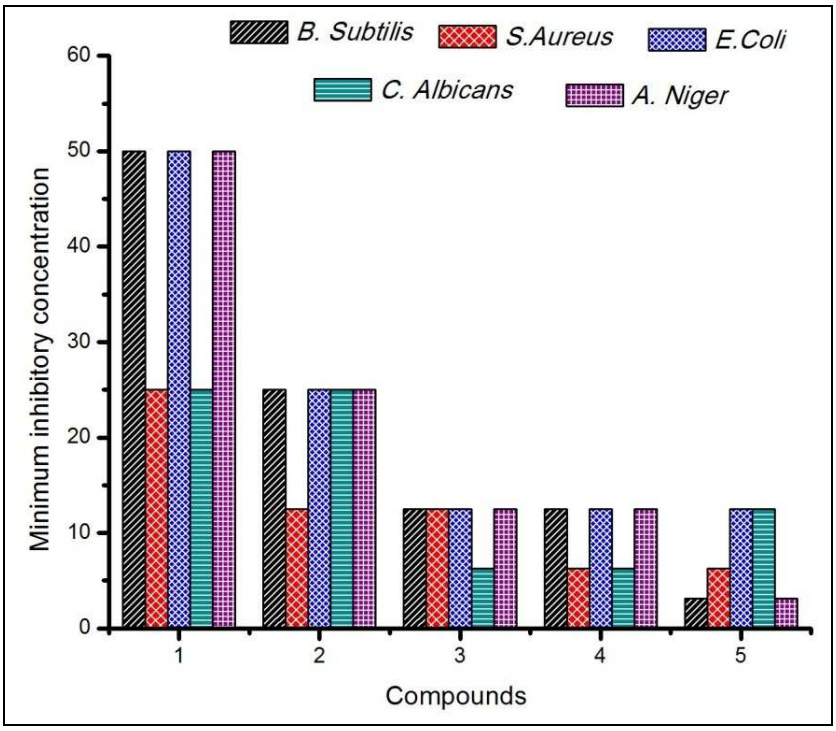


Figure-2: Graphical representation of Schiff base ligands and their organotin(IV) complexes (as in Table-3)

CONCLUSION

Schiff base ligands and their organotin(IV) complexes were synthesized and characterized by various spectroscopic techniques. On the basis of various spectroscopic techniques it revealed that Schiff base acted as tridentate ONS system and coordinated to tin atom through phenolic oxygen, azomethine nitrogen and sulfur of thiol group with trigonal bipyramidal geometry. Ligands and complexes were evaluated for *in vitro* antimicrobial evaluation and complexes were found to be more potent biocides.

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