Proceedings of the Pakistan Academy of Sciences: B
Life and Environmental Sciences 62(2): 167-175 (2025)
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ISSN (Print): 2518-4261; ISSN (Online): 2518-427X
http://doi.org/10.53560/PPASB(62-2)1075

Pakistan Academy of Sciences

Research Article

Synthesis, Spectroscopy, Antibacterial and Anti-inflammatory Studies of Homo and Hetero Bimetallic Complexes with Bifunctional (O, S) Ligand

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Abstract: Current studies were performed to synthesize homo- (Sn & Sn) and heteronuclear (Sn & Cd/Zn) complexes (1-**7**) of sarcosine dithiocarbamate and investigate their antibacterial and anti-inflammatory potential. Homobimetallic products, i.e., $Ph_2(Cl)SnSSCLSn(Cl)Me_2$ (1), $Ph_2(Cl)SnSSCLSn(Cl)Me_3$ (2), $Ph_2(Cl)SnSSCLSnBu_3$ (3) were produced by a reaction of sarcosine (HLH), CS_2 and Ph_2SnCl_2 and then with Me_2SnCl_2, Me_3SnCl and Bu_3SnCl, respectively. $Ph_3SnSSCLSn(Cl)Bu_2$ (4) and $Ph_3SnSSCLSnMe_3$ (5) were produced by reacting HLH with KOH, CS_2 and Ph_3SnCl firstly and then with Bu_2SnCl_2 and Me_3SnCl, respectively. The heteronuclear products, i.e., $(Ph_3SnSSCL)_2Cd$ (6) and $(Ph_3SnSSCL)_2Zn$ (7**)** were formed by reaction between HLH, KOH, CS_2 and Ph_3SnCl followed by treatment with $CdCl_2$ or $ZnCl_2$, respectively. Elemental analysis data was agreed well with the molecular composition of the products. Fourier transform infrared (FTIR) spectroscopy have shown the bidentate binding of carboxylate and dithiocarbamate donor sites of ligand. Proton nuclear magnetic resonance (^1H NMR) spectroscopy of 3 displayed the expected signals of ligand portion and organotin(IV) moieties. TGA data of product 7 verified its heterobimetallic (2Sn, ZnO) composition. All the products except 4 and 6 exhibited significant antimicrobial activities as compared to free ligand. The highest anti-inflammatory potential was displayed by product 3.

Keywords: Homobimetallic (Sn, Sn), Heterobimetallic (Sn, Cd/Zn), Spectroscopy, TGA, Antibacterial, Anti-inflammatory.

1. INTRODUCTION

A significant attention has been focused on the study of homo- [1] and hetero- [2] bimetallic complexes in the last few years. In such products, the cooperative effects of more than one metals can lead to new or increased optic, magnetic, or reactive functions. So, such kinds of products are especially important for designing the catalysts and functional materials [3]. Homobimetallic complexes have been reported for tandem organic transformations [4], as catalysts [5], antibacterial [1], anticancer

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Received: August 2024; Revised: May 2025; Accepted: June 2025
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and microbiocidal agents [6]. In heterobimetallic complexes, the bifunctionality of the two different metal atoms may result in important applications of the activating substrates. Such compounds display synergistic reactivity of both metal centers, which is different as compared to that observed for chemical species containing only a single type of metal [7]. The two metal atoms may adopt different roles and may act in a cooperative manner. Such a kind of reactivity is generally called as "cooperative reactivity [7, 8]. The heteronuclear complexes commonly find applications in photochemical and catalytic systems [9, 10]. Mixed metal clusters (in homogeneous or heterogeneous formulations) have been reported to exhibit unusual reactivity due to metal-metal interactions or act as "storehouses for the release of catalytically active fragments [11].

Dithiocarbamates are specifically important among various sulfur ligands due to their applications as high-pressure lubricants, molecular precursors, vulcanization accelerators, in CVD processes, as active ingredients in pesticides, fungicides and pharmaceutical products. Because of close similarities with important biomolecules such as vitamins and amino acids, complexes of metals with sulfur donating ligands (e.g., dithiocarbamates) also continues to increase [12].

Organotin compounds are famous for their numerous structural diversities [13, 14] and coordination with different oxygen [15, 16] and sulfur [17, 18] donor ligands. They have been used as antimicrobial, antioxidant, antileishmanial, anticancer [19], antitumor agents [20], insecticides, acaricides, wood preservatives, ceramic pacifiers, antifouling agents, food additives, textile additives, in coatings of electroconductive materials, in metal finishing operations and paints [21]. Many organotin polymers have been reported as antiviral agents [22]. Organotin(IV) derivatives of carboxylate ligands have been investigated as anticancer agents and find useful applications in the field of cancer chemotherapy [23]. They have been widely investigated for their potential agriculture, materials science, medicinal chemistry and catalysis [24]. Their catalytic activity has been reported in various fields, e.g., formation of polymeric olefins or polyurethanes [25]. Organotin compounds have also been found effective for the stabilization of PVC since they can neutralize the formation of HCl in PVC during high temperature conditions [26]. Some organotin compounds are used to inhibit corrosion in porous materials [27].

A lot of work has been reported on tin(IV) derivatives but heterometallic (Sn, Cd/Zn) derivatives of sarcosine and homometallic products having two different organotin moieties, were rarely reported earlier. In continuation of our previous work on organotin(IV) complexes [28], current studies were conducted to synthesize the homo- (Sn, Sn) and heterobimetallic (Sn, Cd/Zn) complexes. The synthesized products were characterized by elemental analysis (CHNS), FTIR, TGA and ^{1}H NMR studies. They have been tested for their antimicrobial potential by biofilm inhibition method and anti-inflammatory activities.

2. MATERIALS AND METHODS

Dimethyltin(IV) dichloride, dibutyltin(IV) dichloride, diphenyltin(IV) dichloride, trimethyltin(IV) chloride and carbon disulfide were purchased from Sigma-Aldrich-Germany. Sarcosine was procured from Merck, Germany. Remaining chemicals, i.e., potassium hydroxide, zinc chloride and cadmium chloride and solvents of analytical grade were used. All the solvents were used without any further purification.

FTIR spectra were recorded in the range of 4000 to 450 cm^{-1} using KBr discs by Perkin Elmer FTIR spectrometer (L1600301 Spectrum Two FT-IR APV). The ^{1}H NMR spectra were recorded at 300 Hz by a Bruker ARC FT-NMR spectrometer. Thermo-gravimetric analysis was performed by a TGA-7 Perkin-Elmer USA. Antimicrobial activities were performed by biofilm inhibition method using ciprofloxacin as a standard drug [29]. A reported method was used to assess the anti-inflammatory potential of the products while using diclofenac as a positive control (standard drug) and DMSO as a negative control [30].

2.1. Syntheses of Homobimetallic Complexes 1-5

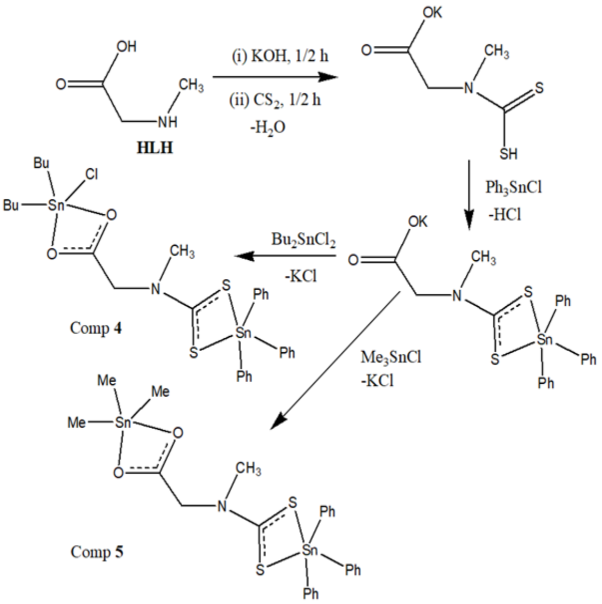
Sarcosine (HLH, 1 mmol, 0.089 g) was stirred with KOH (1 mmol, 0.056 g) for ½ hour in methanol solvent (30 ml) in a 250 ml two necked round bottom flask at room temperature. Then CS_2 (1 mmol, 0.06 ml) was added and the resultant mixture was stirred for half hr. Subsequently, Ph_2SnCl_2 (1 mmol, 0.344 g) was added and stirring was continued for more

1 hour. Finally, Me_2SnCl_2 (1 mmol, 0.220 g) was added and the solution was stirred for four hours. The precipitated KCl was removed after filtration. The filtrate was rotary evaporated to leave behind the solid product 1 which was recrystallized from methanol after addition of few drops of petroleum ether. Use of Me_3SnCl (1 mmol, 0.199 g) or *n*-Bu_3SnCl (1 mmol, 0.325 g) in place of Me_2SnCl_2 in the above reaction has resulted in the formation of products 2 or 3, respectively. The whole reaction route has been displayed in Scheme 1.

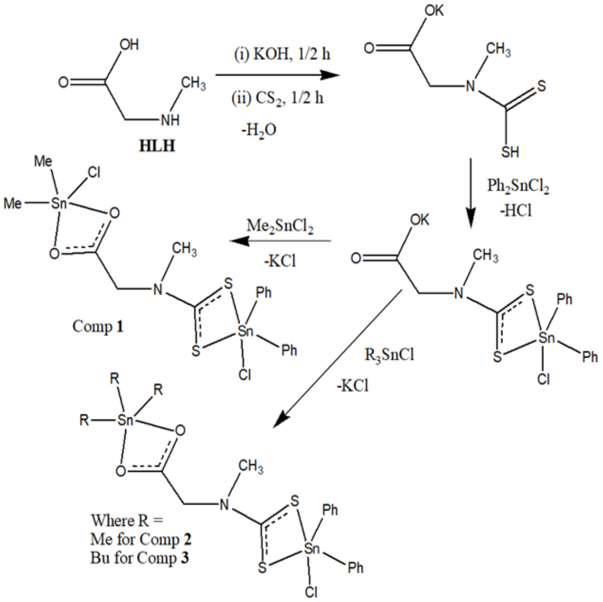
For the synthesis of products 4 and 5, sarcosine (HLH, 1 mmol, 0.089 g) was first stirred with KOH (1 mmol, 0.056 g) for ½ hour in methanol (30 ml) and then with CS_2 (1 mmol, 0.06 ml) for ½ hour followed by stirring with Ph_3SnCl (1 mmol, 0385 g) for 1 hour at room temperature. Finally, the reaction mixture was stirred for four hours with Bu_2SnCl_2 (1 mmol, 0.304 g) or Me_3SnCl (1 mmol, 0.199 g), filtered and the filtrate was rotary evaporated to leave behind the solid product 4 or 5, respectively which was recrystallized from methanol after addition of few drops of petroleum ether. The whole reaction route has been displayed in Scheme 2.

2.2. Syntheses of Heterobimetallic Complexes 6-7

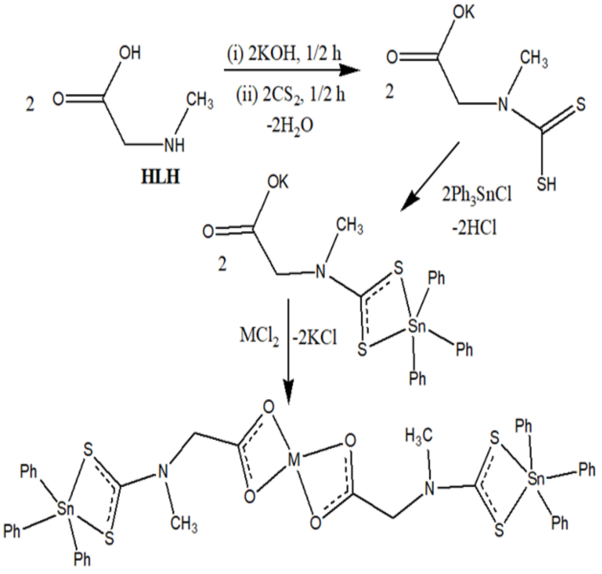
A mixture of sarcosine (2 mmol, 0.178 g) and KOH (2 mmol, 0.112 g) was stirred in methanol (20 ml) for 1/2 hour in a round bottom flask (250 ml) at room temperature; it was followed by addition of CS_2 (2 mmol, 0.12 ml) and subsequent stirring for more ½ hr. Then 2 mmol of Ph_3SnCl (0.770 g) was added and stirring was continued for 3 hours. Finally, there was addition of aqueous solution of a either $CdCl_2$ (1 mmol, 0.183 g) or $ZnCl_2$ (1 mmol, 0.136 g) with stirring for ½ hr for the formation of $(Ph_3SnSSCL)_2Cd$ (6) or $(Ph_3SnSSCL)_2Zn$ (7), respectively. The whole reaction path has been summarized in Scheme 3.



Scheme 2. Reaction scheme for **the** formation of complexes 4 and 5.



Scheme 1. Reaction scheme for the formation of complexes 1-3.



where M = Cd for Comp **6**; Zn for Comp **7**

Scheme 3. Reaction scheme for the formation of complexes 6 and 7.

3. RESULTS AND DISCUSSION

Sarcosine (HLH) was firstly treated with KOH, CS_2 and Ph_2SnCl_2 in equimolar ratio in methanol and then with dimethyltin dichloride, trimethyltin chloride or tri-*n*-butyltin chloride to produce the complexes 1, 2 or 3, respectively. The use of HLH, KOH, CS_2 and Ph_3SnCl in first step and further reaction with either Bu_2SnCl_2 or Me_3SnCl in second step most probably produces the product 4 or 5, respectively. If the reactants (Ph_2SnCl_2 or Ph_3SnCl) in the second step are replaced by $CdCl_2$ and $ZnCl_2$, then the result is the formation of $(Ph_3SnSSCL)_2Cd$ (6) and $(Ph_3SnSSCL)_2Zn$ (7), respectively. The synthesized compounds 1-7 have shown stability in air and have sharp melting points. They are in whitish crystalline solids and have shown good solubility in some organic solvents. The results of elemental analysis were agreed well with the molecular composition of the compounds. Their physical data have been given in Table 1.

3.1. FTIR Spectroscopy

FTIR spectroscopy is an important technique which is used to find the functional groups present in the compounds. The newly synthesized samples were characterized via FTIR spectroscopy. The spectra were recorded in the region of 4000-400 cm^{-1} by a FTIR spectrophotometer. The obtained data are summarized in Table 2. The spectra are shown in Figures S1-S7 (Supplementary Information). For the free ligand (HLH), COO asymmetric (υ_{asym}) and symmetric vibrations (υ_{sym}) are reported earlier in

Table 1. Physical data of complexes 1-7.

Comp. no.	Molecular formula	Melting point (°C)	Mol. wt (g/mol)	% Yield	Color	Elemental analysis %age (Calculated/Found)				Solubility
						C	H	N	S	
1	$C_{18}H_{21}NO_2S_2Sn_2Cl_2$	251	655.82	87	White crystalline	32.97/ 32.46	3.23/ 3.14	2.14/ 1.99	9.78/ 9.83	DMSO, Methanol, Ethanol
2	$C_{19}H_{24}NO_2S_2Sn_2Cl$	207	635.40	78	White crystalline	35.91/ 35.78	3.81/ 3.94	2.20/ 2.08	10.09/ 9.98	DMSO, Chloroform
3	$C_{28}H_{42}NO_2S_2Sn_2Cl$	210	761.64	74	White	44.15/ 44.23	5.56/ 5.43	1.84/ 1.73	8.42/ 8.35	DMSO
4	$C_{30}H_{38}NO_2S_2Sn_2Cl$	235	781.63	82	White crystalline	46.10/ 45.95	4.90/ 4.83	1.79/ 1.84	8.20/ 8.09	DMSO, Chloroform
5	$C_{25}H_{29}NO_2S_2Sn_2$	200	677.05	89	White	44.35/ 44.16	4.32/ 4.19	2.07/ 1.94	9.47/ 9.35	DMSO, Methanol, Ethanol
6	$C_{44}H_{40}N_2O_4S_4Sn_2Cd$	275	1138.89	73	White	46.40/ 46.23	3.54/ 3.47	2.46/ 2.59	11.26/ 10.96	Chloroform, Ethanol
7	$C_{44}H_{40}N_2O_4S_4Sn_2Zn$	266	1091.87	71	White	48.40/ 48.11	3.69/ 3.74	2.57/ 2.68	11.75/ 11.43	Methanol, Ethanol

Table 2. FTIR data (cm^{-1}) of complexes 1-7.

Comp. no.	COO			υC-N	υC-S	υSn-C	υM-O	υSn-S
	υ_{asym}	υ_{sym}	Δυ					
HLH	1621	1407	214	-	-	-	-	-
1	1590s	1430w	160	1504m	971s	730s	693m	517w
2	1591s	1479w	112	1508m	997s	729s	659m	519w
3	1610s	1459w	151	1479m	997s	692s	565m	454w
4	1624s	1429w	195	1462m	996s	694s	580m	526w
5	1621s	1463w	158	1479m	997s	642s	563m	453w
6	1655s	1430w	225	1509m	962s	726s	691m	551w
7	1653s	1480w	173	1509m	997s	728s	692m	549w

literature to be 1621 and 1407 cm^{-1}, respectively [31]. In the coordinated products 1-7, the value of υ_{asym} and υ_{sym} was appeared in the ranges of 1590-1655 and 1429-1480 cm^{-1}, respectively. Thus, there was a significant rise of υ_{sym} value to 1429-1480 cm^{-} in complexes^{1} as compared to that (1407 cm^{-1}) of free ligand precursor (HLH), this shift in υ_{sym} evidently verifies the ligand metal coordination. The value of $\Delta\upsilon$ (υ_{asym}- υ_{sym}) depends upon the mono-/bidentate coordinating nature of carboxylate ligand. $\Delta\upsilon$ was appeared in the range of 112-195 cm^{-1} indicating bidentate coordination mode in the complexes 1-5 and 7 [32]. However, $\Delta\upsilon$ value in product 6 was appeared at 225 cm^{-1} which indicates monodentate coordination of the carboxylate moiety for its binding with a metal [33]. FTIR data thus supports a penta-coordinated configuration of tin at -COO donor site in the solid state of all products except **7**. The υ(C–N) vibrations were observed at 1462-1509 cm^{-1} which were lied between the ranges of C=N double bonds (1640-1690 cm cm^{-1}) and C–N single bonds (1250–1360 cm^{-1}) indicating a bidentate coordination fashion of the dithiocarboxylate group [34]. Bidentate coordination mode of the dithiocarboxylate group was further verified by the appearance of a solitary υCS band at 962-997 cm^{-1} [35]. The obtained FTIR results thus demonstrate a penta- and tetra coordinated geometry of tin and Cd/Zn, respectively with the dithiocarbamate donor site of products 1-7 in the solid state. Metal ligand linkage was further verified due to the appearance of Sn-C, Sn-O and Sn-S vibrations in the ranges of 642-730, 563-693 and 453-526 cm^{-1}, respectively.

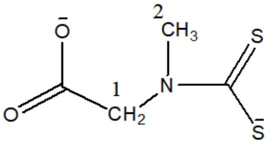
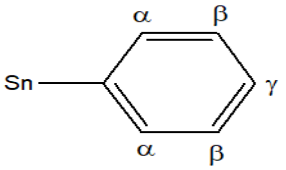
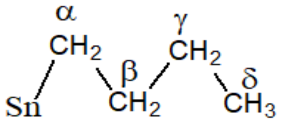
3.2. ^{1}H NMR Spectroscopy

Compound 3 was subjected to proton NMR spectroscopy in DMSO solvent. The chemical shifts are summarized in Table 3 while the spectrum has been displayed in Figure 1. The numbers of protons calculated by incremental method are in good agreement with the experimentally observed data. The product has shown the ^{1}H NMR chemical shifts for ligand portion as well as for both the organotin (phenyltin and butyltin) moieties, thus verifying homobimettalic (Sn, Sn) complexation in product 3.

3.3. Thermogravimetric Analysis (TGA)

Compound 7 was subjected to thermogravimetric analysis (TGA) up to a temperature of 800 °C. The obtained TGA curve is shown in Figure 2.

Table 3. H^{1} NMR data of complex 3.

Portion of complex	**Chemical shift** (ppm) **and protons**
	2.085-2.093t and 1.587-1.618t for protons 1 and 2, respectively.
	8.29d, 7.27-7.77m and 7.03-7.05m for α, β and γ-protons, respectively.
	1.096-1.127t for α protons; 1.29-1.33m for β and γ protons; 0.86-0.89m for δ-protons.

$^{\#}$t = triplet, d = doublet, m = multiplet

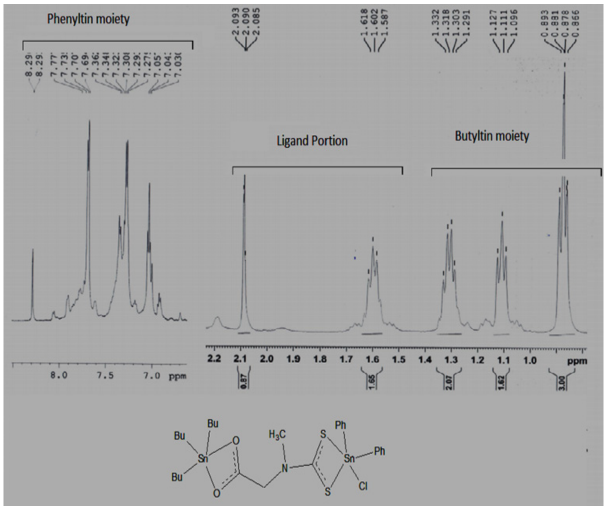


Fig. 1. ^{1}H NMR spectrum of compound 3.

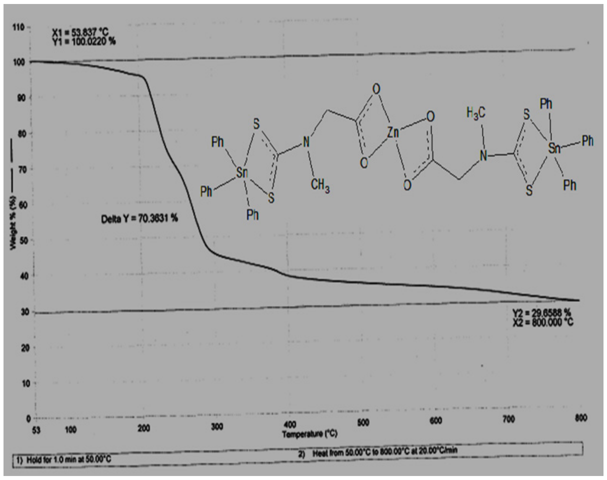


Fig. 2. Thermogram of Compound 7.

The product **7** has shown thermal stability up to a temperature of about 200 °C. However, when it was heated up to 800 °C, it lost 70.35% mass as organic components leaving behind 29.65% residue (Figure 2) in the form of two tin atoms and a zinc oxide molecule. The obtained TGA data thus strongly supports the heterobimetallic composition of the product 7.

3.4. Antibacterial Activities

The free ligand (HLH) and the coordinated products 1-7 were tested for their antimicrobial potential by biofilm inhibition method. Ciprofloxacin was used as a standard drug. The recommended concentration of 1mg/1ml in DMSO was used for each test. The obtained data are displayed in Table 4.

The free ligand has shown no inhibition against both the bacterial strains (*E. coli* and *Staphylococcus*). However, all the complexes except 4 & 6 displayed significant inhibitions of *E. coli* and *Staphylococcu*s biofilms. It is reported in literature that partial sharing of positive charge of a metal atom with the ligand, results in the lowering of its polarity which thus facilitates the permeation of the resultant metal complex through the lipid layer of a membrane [36, 37]. The coordination of the ligand with a metal increases the lipophilic character of consequent complexes, which is responsible for destruction of cell membranes of bacteria and their ultimate death and thus lipophilic character defines the extent of antibacterial activity [38]. The antibacterial potential of our investigated complexes was found to depend upon the nature of target bacterial strain, the substitution pattern at tin and homo- (Sn & Sn) or heteronuclear (Sn & Cd/Zn) nature of a complex.

Table 4. Bacterial biofilm inhibitions (%) by HL and complexes 1-7.

Comp. No	*E. Coli*	*S. Aureus*
HL	-	-
1	30.36	30.61
2	59.51	61.79
3	52.63	62.08
4	-	-
5	66.8	61.79
6	-	-
7	46.15	61.47
Ciprofloxacin	67	69

The highest activity (66.8% inhibition) against *E. coli* was exhibited by compound 5, which may be owed to its homobimetallic (Sn & Sn) nature as well as coordinated triorganotin (trimethyltin and triphenyltin) moieties since triorganotin(IV) complexes in general have been reported to be more active as compared to diorganotin(IV) complexes [39]. By changing the bacterial strain to *S. aureus*, the highest activity (62.08% inhibition) was shown by coordinated product 3 which is also homobiemtallic (Sn & Sn) and is comprised of tributyltin(IV) and chlorodiphenyltin(IV) moieties. The compound 1 containing chlorodimethyltin and chlorodiphenyltin moieties has shown moderate activity with 30.36 and 30.61 % inhibitions against *E. coli* and *S. aureus*, respectively. The coordination products 4 and 6 were totally inactive against each bacterial strain, i.e., *E. coli* and *S. aureus*. Among these products, the homobimetallic complex 4 contains chlorodibutyltin(IV) and triphenyltin(IV) moieties whereas heterobimetallic product 6 consists of two triphenyltin(IV) moieties and one cadmium metal. In going from a heterobimetallic product 6 to 7, there was replacement of Cd metal with zinc which resulted in significant antibacterial potential of 7 against both the tested bacterial strains.

3.5. Anti-inflammatory Activities

Inflammation is the body's defensive reaction to injury, marked by visible signs, e.g., pain, heat, redness and disruptions in normal functions of the body. It is triggered by numerous factors such as microbial invasion, physical trauma and chemical exposure. Its main goals are to facilitate tissue repair, eliminate irritant and neutralize invading pathogens. However, chronic inflammation is associated with numerous health diseases such as cancer, neurodegenerative conditions, autoimmune issues and cardiovascular disorders and is needed to be address. Organotin(IV) complexes have recently attracted the attention of the researchers to address such issues [40]. Rahim et al., 2024 reported the potent anti-inflammatory effects (comparable to indomethacin as a standard) of {$(n\text{-}C_4H_9)_3$SnL and $(CH_3)_3$SnL complexes derived from 4-bromophenoxyacetic acid (HL) [19].

The free ligand (HLH) and our investigated coordinated products 1-7 were tested for their anti-inflammatory activities. Diclofenac was used as a standard drug (positive control) while DMSO was used as a negative control. The recommended concentration of 1mg/1ml in DMSO was used for each test. The obtained data are summarized in Table 5.

The free ligand (HLH) has shown no anti-inflammatory. However, coordination of ligand with the metal atoms significantly induced the anti-inflammatory potential in all the complexes except 5. The anti-inflammatory potential of the investigated products 1-7 was found to depend upon the substitution pattern at tin and homo- (Sn & Sn) or heteronuclear (Sn & Cd/Zn) nature of a complex. The homobimetallic products 1 to 4 displayed 70.32-89.67% anti-inflammatory potential whereas heterobimetallic product 7 has shown 71.61% anti-inflammatory activity. The highest anti-inflammatory activity (89.67%) was displayed by the coordinated product 3 which is homobiemtallic (Sn & Sn) and contains tributyltin(IV) and chlorodiphenyltin(IV) moieties. The homobimetallic product 5 containing trimethyltin(IV) and triphenyltin(IV) was totally inactive whereas lowest anti-inflammatory activity was displayed by heterobimetallic (Sn & Cd) product 6.

Table 5. Anti-inflammatory activity data of ligand and products 1-7.

Comp. no.	Negative control (DMSO)	Positive control (Diclofenic)	Anti-inflammatory (%)
HLH	0.155	0.042	-
1	0.155	0.042	70.32
2	0.155	0.042	86.45
3	0.155	0.042	89.67
4	0.155	0.042	89.03
5	0.155	0.042	-
6	0.155	0.042	10.96
7	0.155	0.042	71.61

4. CONCLUSIONS

Bimetallic complexes were produced at room temperature by treating sarcosine, KOH and CS_2 in 1:1:1 molar ratio under stirring conditions and subsequent reaction with a di- or tri-phenyltin chloride and then with Me_2SnCl_2/Me_3SnCl/Bu_3SnCl/Bu_2SnCl_2/$CdCl_2$/$ZnCl_2$. The synthesized products were analyzed by elemental analysis, IR, ^1H NMR and thermo gravimetric analysis. Elemental analysis data was agreed well with the molecular composition of the products. It was concluded that carboxylate and di thiocarbamate donor sites of the ligand act in a bidentate fashion. The central tin atom exhibits trigonal bipyramidal geometry in solid state with both the oxygen and sulfur donor sites whereas a square planar geometry was assigned around cadmium and zinc ions. ^1H NMR data verified the the incorporation of two organotin (phenyltin and butyltin) moieties, thus verifying homobimettalic (Sn, Sn) complexation. Thermo gravimetric analysis verifies the hetero bimetallic nature of complex 7. The investigated complexes exhibit significant antimicrobial activities as compared to free ligand. Anti-inflammatory results show the compound 5 is inactive while the coordinated product 3 had displayed the highest anti-inflammatory activity.

5. SUPPLEMENTARY INFORMATION

FTIR spectra of compounds 1-7 are given in Figures S1-S7, respectively.

6. CONFLICT OF INTEREST

It is hereby declared that there is no conflict of interest among the authors.

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