Research Article

Preparation, Characterization, and Antimicrobial Activities of Bimetallic Complexes of Sarcosine with Zn(II) and Sn(IV)

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Received 29 April 2013; Accepted 30 August 2013

Academic Editor: Claudio Pettinari

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Heterobimetallic complexes of Zn(II) and Sn(IV) with sarcosine have been synthesized at room temperature under stirring conditions by the reaction of sarcosine and zinc acetate in 2:1 molar ratio followed by the stepwise addition of CS₂ and organotin(IV) halides, where R = Me, n-Bu, and Ph. The complexes were characterized by elemental analysis, FT-IR and NMR (¹H, ¹³C) spectroscopy. IR data showed that the ligand acts in a bidentate manner. NMR data revealed the four coordinate geometry in solution state. In vitro antimicrobial activities data showed that complexes (3) and (4) were effective against bacterial and fungal strains with few exceptions.

1. Introduction

Organotin compounds are amongst the most widely used organometallic compounds. Over the last several decades, they have been utilized for a variety of industrial and agricultural applications including pesticides, fungicide, and antifouling agents [1]. In general, the biochemical activity of organotin(IV) carboxylates is greatly influenced by the structure of the molecule and the coordination number of the tin atom [2, 3]. Therefore, the recognition of the importance between the biological properties and the structure of organotin(IV) carboxylates [4] has stimulated the study of carboxylates of tin.

The diverse structural motifs are known in organotin compounds and attributed to the ambidentate character of the carboxylate ligands [5]. Steric and electronic attributes of organic substituents on tin and/or the carboxylate moiety impart significant influence on the structural characteristics in tin carboxylates. Therefore, synthesis of new organotin carboxylates with different structural features will be beneficial in the development of pharmaceutical organotin and in other properties and applications.

Meanwhile, dithiocarbamate (DTC) is the ligand which strongly bonds with metal ions and can stabilize metal complexes with high oxidation number [6]. Organotin(IV) dithiocarbamate complexes have been widely studied due to Sn-S bond in their structure and the effects of the bonding on diversified applications basically in biological field [7, 8].

Zinc chloride is chiefly used as a catalyst for the Fischer indole synthesis and Friedel-Craft acylation reaction that was involved in the synthesis of organic compounds used in the laboratory [9]. The medical applications of Zn(II) compounds include treatments of parasitic diseases (eczema, ringworm, fungus, and athletes foot), and in many biological processes zinc plays an important role as metalloenzymes in which Zn(II) is coordinated by a ligand that leads to a structural as well as functional model [10].

In our previous work, we reported several organotin complexes with oxygen and sulfur donor atoms [11–15]. As an extension of this research program, we report here the complexes of sarcosine (Figure 1) containing Zn(II) and Sn(IV) to study the effect of Zn along with tin on biological activities and compare these results with already reported organotin complexes of sarcosine [16–18].
2. Experimental

2.1. Materials and Methods. N-Methyl glycine (sarcosine), zinc acetate, carbon disulphide, and organotin(IV) halides were of Sigma-Aldrich (UK) origin. The organic solvents (chloroform, n-hexane, ethanol, methanol, DMSO, and acetone) were procured from Merck (Germany). The solvents were dried [19] prior to use. Nutrient broth, nutrient agar, and potato dextrose agar were of Oxoid (UK) origin. Autoclave and laminar air flow were purchased from Omron and Dalton companies of Japan, respectively. Micropipettes were purchased from Gilson (France). The melting points were determined on an electrothermal melting point apparatus, model Staurt SMP3 by using capillary tubes. Elemental analysis was done for carbon, hydrogen, nitrogen, and sulphur on a CHNS-932 elemental analyzer Leco Corporation (USA). The infrared spectra were recorded as KBr pellets on Perkin Elmer 1000 spectrometer in the frequency range of 4000–250 cm$^{-1}$. NMR ($^1$H and $^{13}$C) spectra were recorded on Bruker AM-300 MHz FT-NMR spectrometer (Germany) using CDCl$_3$ as an internal reference.

2.2. General Procedure for the Synthesis of Complexes. Sarcoine (0.178 g, 2 mmol) was dissolved in methanol (25 mL) in a round bottom flask with continuous stirring at room temperature. Then solution of zinc acetate (0.22 g, 1 mmol) in methanol (3 mL) was added dropwise to the above solution, and mixture was stirred continuously for 20 minutes. After that CS$_2$ (0.120 mL, 2.0 mmol) in methanol (5 mL) was added dropwise to the reaction mixture and stirred for 0.5 hr at room temperature. Subsequently, the solution of diorganotin dichloride/triorganotin chloride (2 mmol) in methanol (50 mL) was added and the mixture was continuously stirred for 4 hr. Solvent was slowly evaporated at room temperature and product obtained was dried in air. Purity was checked by TLC and recrystallization was done in methanol: n-hexane (1:1) (see Scheme 1).

3. Results and Discussion

The ligand and synthesized complexes are solid and stable in air. They have sharp melting points. Elemental analysis was done to compare the observed values with predicted values of percentage of the carbon, hydrogen, nitrogen, and sulfur.

The physical data of ligand and synthesized complexes is summarized in Table 1.

3.1. Infrared Spectroscopy. IR spectra of the ligand and its newly synthesized complexes (1)-(6) were recorded as KBr pellets in the range of 4000–250 cm$^{-1}$. The characteristic IR absorption frequencies (cm$^{-1}$) have been listed in Table 2.

The IR spectrum of the ligand exhibited a band at 3365 cm$^{-1}$, which was attributed to the NH stretching mode. Deprotonation of carboxylic group is confirmed by the absence of OH band in the spectra of the complexes. Regarding the carboxyl vibrational modes, there are different binding possibilities [20, 21]:

(1) bridged structure, $\nu_{\text{C} = \text{O}} = 1560–1540$ cm$^{-1}$,
(2) dimer and monomer chelate structures, $\nu_{\text{C} = \text{O}} = 1640–1650$ cm$^{-1}$,
(3) “free ester” monodentate structure, $\nu_{\text{C} = \text{O}}$ of complexes ($\text{C} = \text{O}$) = 1680–1640 cm$^{-1}$ and $\nu_{\text{C} = \text{O}}$ of complexes ($\text{C} = \text{S}$) = 1770–1700 cm$^{-1}$.

The carboxylate group gives strong asymmetric and symmetric stretching bands in the range of 1602–1650 cm$^{-1}$ and 1433–1470 cm$^{-1}$, respectively, and it is assignable to a chelate or monodentate structure; however, the absence of $\nu_{\text{C} = \text{O}}$ band in expected range is indicative of the presence of the chelate form. Bidentate nature of ligand is also confirmed by $\Delta\nu_{\text{COO}}$ value that is less than 200 cm$^{-1}$ in all complexes [22]. Presence of $\nu_{\text{Zn–O}}$ vibrational bands in the range of 310–385 cm$^{-1}$ in all the complexes [23] confirms the zinc carboxylate interaction. It has been reported [15] that the observation of a single $\nu_{\text{C} = \text{S}}$ absorption in the region around 1000 cm$^{-1}$ is indicative of dithiocarbamate groups that are bonded symmetrically or bidentate in nature. A strong absorption band of $\nu_{\text{C} = \text{S}}$ lies in the range of 1013–1043 cm$^{-1}$, while $\nu_{\text{C} = \text{S}}$ band appears in the lower frequency range, 942–988 cm$^{-1}$ [24]. The Sn–S bond formation is confirmed by the appearance of a new band in the range of 456–480 cm$^{-1}$ [25]. The strong band at 302–318 cm$^{-1}$ was assigned to $\nu_{\text{Sn–Cl}}$ absorption. The Sn–C bond was observed in the range of 530–547 cm$^{-1}$ for complexes (1) and (2)–(5) but at 272 and 280 cm$^{-1}$ for complexes (3) and (4), respectively, which are di- and triphenyltin derivatives. The absence of the band in the range 440–420 cm$^{-1}$ which is commonly assigned to the $\nu_{\text{Sn–N}}$ mode in analogous compounds [26, 27] rules out amino coordination to tin, thus leading support to the proposed carboxylate carboxyl atom [16] and dithiolate sulphur atom coordination in all synthesized complexes.

3.2. $^1$H NMR Spectroscopy. $^1$H NMR spectra of the ligand and complexes (1)-(6) were recorded in deuterated DMSO to find the behaviour of magnetically nonequivalent protons. The data is presented in Table 3. The number of protons calculated by integration of peaks is in excellent agreement with those theoretically calculated by incremental method [28]. The absence of OH signal in the complex suggested the deprotonation of carboxylic acid group for O→Zn coordination through COO$^{-}$ anions.
Scheme 1

Table 1: Physical data of heterobimetallic complexes with sarcosine.

| Comp. no. | Molecular formula | Mol. weight | % yield | m.p. (°C) | % C Calcd. (found) | % H Calcd. (found) | % N Calcd. (found) | % S Calcd. (found) |
|-----------|------------------|-------------|---------|---------|-------------------|-------------------|-------------------|-------------------|
| HL        | C₃H₇O₂N          | 89.09       | —       | 208     | 40.45 (40.42)     | 7.92 (7.95)       | 15.71 (15.68)     | —                 |
| (1)       | C₁₂H₂₂O₄N₂S₄Cl₂ZnSn₂ | 760.31     | 75      | 121-122 | 18.95 (18.99)     | 2.91 (2.95)       | 3.68 (3.64)       | 16.87 (16.89)     |
| (2)       | C₂₄H₄₆O₄N₂S₄Cl₂ZnSn₂ | 928.63     | 68      | 87-91   | 31.04 (31.07)     | 4.99 (4.96)       | 3.01 (3.04)       | 13.81 (13.78)     |
| (3)       | C₃₂H₃₀O₄N₂S₄Cl₂ZnSn₂ | 1008.53    | 57      | 107-109 | 38.10 (39.12)     | 2.99 (2.96)       | 2.77 (2.74)       | 12.71 (12.74)     |
| (4)       | C₁₄H₂₈O₄N₂S₂ZnSn₂  | 719.61      | 67      | 145-146 | 23.36 (23.39)     | 3.92 (3.96)       | 3.89 (3.92)       | 17.82 (17.78)     |
| (5)       | C₃₂H₆₄O₄N₂S₂ZnSn₂  | 971.53      | 51      | 224-225 | 39.55 (39.50)     | 6.64 (6.68)       | 2.88 (2.85)       | 13.20 (13.17)     |
| (6)       | C₄₄H₄₀O₄N₂S₂ZnSn₂  | 1091.89     | 73      | 86-88   | 48.39 (48.36)     | 3.69 (3.65)       | 2.56 (2.59)       | 11.74 (11.78)     |
at 1.27ppm with tin satellites having formethylene and methyl protons, respectively.

The CH$_3$ protons in compound (1) appear as a singlet at 1.27 ppm with tin satellites having $J$[119Sn–1H] = 96 Hz. In compound (2), the protons of butyl group appear as a multiplet in the region 0.88–0.93 ppm. The terminal CH$_3$ group of butyl gives triplet at 0.23 ppm with $J$[H–H] value of 7.2 Hz. The proton chemical shift of the methyl group in compound (3), attached to the Sn, gives a singlet at 0.51 ppm.

Two resonance signals appeared as a singlet at 3.15 ppm and 2.31 ppm for the methyl and methylene protons, respectively. These signals exhibited downfield shift to 3.48 ppm for the methylene and methyl protons, respectively, in the complexes (1)–(6), which is indicative of four coordinate geometry [30] in solution state.

### 3.3. $^{13}$C NMR

The assignments of –CSS group in investigated compounds is straightforward which are observed in the range of 202.5–202.8 ppm indicating the coordination of sulfur to the tin atom.

Table 4 lists the chemical shifts of $^{13}$C and tin-carbon coupling constants for the reported complexes (1)–(6). The $^{13}$C NMR chemical shifts due to the phenyl groups are observed at positions comparable to other similar compounds [30, 31]. The $^{13}$C NMR chemical shift due to –COO group for complexes (1)–(6) is 202.5–202.8 ppm. Coordination of the tin atom in di- and triphenyltin(IV) derivatives show a complex pattern and were assigned according to the literature [29]. The $^2J$[119Sn–1H] value for di- and triphenyltin(IV) derivatives is 82 Hz, in the range expected for pentacoordinated tin atom, and consistent with C–Sn–C angle of 126.5°.

### 3.4. Antibacterial Activity

Sarcosine and synthesized complexes (1)–(6) were tested in vitro for their antibacterial activity against gram positive and gram negative bacterial...
strains including *B. subtilis* and *S. aureus* and *P. multocida* and *E. coli*, respectively, and the results are given in Table 5. Ampicillin was used as a standard drug. The results revealed that all the newly synthesized complexes show higher activity than the ligand but markedly lower than the standard drug with few exceptions.

Complex (5) showed maximum inhibitory action against *B. subtilis* (54.5 ± 0.86) and *E. coli* (51.0 ± 1.00), probably more than standard drug (40.00 ± 1.41) and (39.5 ± 0.86), respectively. Complex (2) displayed lowest inhibition zone in case of *B. subtilis* (20.5 ± 0.86) and *S. aureus* (21.0 ± 1.00). The ligand exhibited low activities, while the complexes exhibited moderate activities as compared to standard drug towards all the bacterial strains. Complex (6) showed moderate antibacterial activity against all the bacterial strains. Among all the complexes, the tributyltin complex (5) showed significant antibacterial activity. It was clear from the data that antimicrobial activities varied according to substitution (increase in substitution on the Sn(IV) enhances the antimicrobial activity) [32].

Data revealed that the synthesized complexes showed more activity against gram positive bacterial strain than gram negative strains. It was due to the difference in the composition of cell wall of both the gram positive and gram negative strains [33]. It was also suggested that the antimicrobial activity of the complexes was either due to killing of microbes or inhibiting their reproduction by blocking their active sites [34].

The results show that all compounds exhibit antibacterial activity, and in many cases, complexes are more potent in their inhibition properties than the free ligand. This can be explained in terms of the greater lipid solubility and cellular penetration of the complexes [35]. It is clear that the coordination enhances the antibacterial activity and clearly indicates that the newly synthesized complexes in the present studies are more active against gram positive than gram negative bacteria. The preliminary results achieved have led us to conclude that these types of complexes should be studied in detail for their applications in diverse area.

The screening data of a particular ligand and its metal complexes show that the former has greater activity than...
the latter from the biochemical point of view. On comparing the results in general, it may be concluded that complexes containing Zn(II) and Sn(IV) have greater inhibiting power than the free ligands as compared to organotin complexes with sarcosine [16, 17] against all the microbes.

3.5. Antifungal Activity. The synthesized compounds (1)–(6) and sarcosine were tested for antifungal activity by using fluconazole as standard drug against four fungal strains including A. flavus, A. niger, A. alternate, and G. lucidum by disc diffusion method [36]. The results are summarized in Table 6. It was evident from tabulated data that the sarcosine as well as the synthesized complexes exhibited, variety of fungicidal activity. Sarcosine exhibited maximum activity against A. flavus (37.5 ± 0.86) while complex (5) exhibited maximum activity (39.5 ± 1.65) against G. lucidum. Complex (2) showed the lowest inhibition zone (19.0 ± 1.00) against G. lucidum. It is noted that tributyltin complexes showed the greatest inhibitory effect against fungi as compared to other alkyl groups. Thus the presence of butyl groups in compound (5) bonded with tin atom was responsible for the rise of antifungal activity [37]. The increased activity of complexes might be due to the coordination of zinc and tin with oxygen and sulfur, respectively [38]. From the data it might be concluded that triorganotin compounds were usually more effective against the fungi than diorganotin compounds [39] in contrast to earlier reported compounds in the literature [16]. This might be due to the presence of Zn(II) along with Sn(IV) which enhances the fungicidal activity of triorganotin complexes.

3.6. Structure Activity Relationship. Although it is difficult to make out an exact structure-activity relationship between the antimicrobial activity and the structure of these complexes, it can possibly be concluded that the chelation as well as the addition of a substrate enhances the activity of the complexes. The variation in the toxicity of different antibacterial agents against various organisms depends on either the impermeability of the cell or differences in site of action or ability to cause mutations in the microorganism. Though the results suggest that the ligand has a remarkable toxic property, their complexes of tin inhibit the growth of microorganisms to a greater extent. This is in accordance with earlier reports [40]. Further, the greater activity of the complexes can also be explained on the basis of their higher solubility of the particles.

4. Conclusion

The FT-IR data of synthesized complexes clearly demonstrate that the zinc and tin become attached with the oxygen and sulphur of the ligand in a bidentate mode. In solid state, chlorodiorganotin complexes exhibit the penta/hexacoordinated geometry, whereas the triorganotin(IV) complexes show the five-coordinate geometry. Biological activity data showed that all the complexes were biologically active with few exceptions. These complexes were found to be more potent inhibitors toward fungal culture as compared to bacterial strains.

Acknowledgment

Higher Education Commission of Pakistan (HEC) is acknowledged for support under the Project no. 20-1623/R&D/10/4402.

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