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Mechanochemical Synthesis, Characterization and Antimicrobial Activity of Some Organotin (IV) Derivatives of Propan-1,3-Dioic Acid

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Abstract

Three organotin (IV) derivatives of propan-1,3-dioic acid with potassium (K) metal; potassium dibutyltin(IV)propan-1;3-dioate **(1),** potassium diphenltin(IV)propan-1,3-dioate **(2),** and potassium triphenltin(IV)propan-1,3-dioate **(3)** were mechanochemically synthesized by grinding KOH with propan-1,3-dioic acid to give the ligand **L:** potassium propan-1,3-dioate, followed by reacting the ligand, **L** with Bu_2SnO, Ph_2SnO and Ph_3SnOH separately. The complexes were purified by recrystallization and purity checked by melting point determination. Characterization of the complexes was done using Fourier Transformed Infrared Spectroscopy (FTIR) and solubility studies. The complexes were screened for antimicrobial activity against four strains of bacteria: *Escherichia coli, Klebstella Pneumonia, Salmonella tyhi* and *Pseudomonas aeruginosa* and four fungi strains: *Conioplora puteana, Rhizopus spp, Scleratium rofsii* and *Serpula lacrymans*. Ciproflxacin and Fulcin were used as controlled drugs. FTIR spectrum of the ligand showed $V_{asym}(COO)$ absorption bands which shifted from 1699.7 cm^{-1} to 1479.8 cm^{-1}, 1640.8 cm^{-1} and 1703.4 cm^{-1} in complexes **(1), (2)** and **(3),** respectively. These shifts indicated that the carbonyl oxygen were in coordination of the metal ions. Their modes of coordination were found to be bidendate since their $\Delta\upsilon$ values obtained were < 200 cm^{-1}. The antimicrobial result showed significant activity (20-27 mm) against the microbes at the minimum inhibition concentrations (MIC) in the range 5-20 µg/mL and minimum bactericidal/fungicidal concentrations (MBC/MFC) of 10 and 20 µg/mL. The order of activity showed by the ligand and complexes is **(2)>(1)>(3)>L** against bacteria and **(3)>(2)>L>(1)** against fungi which competed favourably with the control drugs: Ciproflxacin and Fulcin.

Keywords: Mechanochemical, synthesis, Antibacterial, Antifungal Activities, Complexes.

Introduction

Organometallic compounds are a class of compounds which contain at least one metal-carbon bond of which the carbon is a part of an organic group [1, 2]. They have been studied extensively due to their diverse applications in catalysis [1, 2], materials science, Medicine [3], chemical synthesis, agriculture [4-6] industry and pharmacological biochemistry as catalysts, intermediates [2], pharmaceuticals [7], anticancer agents [3] pesticides [8] alkylating agents, antifouling agents [9], semiconductors, additives and stoichiometric reagents. Organometallic compounds are attractive candidates for medical applications as their mechanisms of action are often multi-modal, and thus not commonly accessible with purely organic pharmacophores [10]. Their unique electronic and structural properties make them attractive candidates for drug development and other applications [11]. Organotin (IV) carboxylates are examples of organometallic compounds that have found application in agriculture [12], industry, medicine, pharmaceuticals and as intermediates for the synthesis of other compounds [2], water repellant agents in textiles, paper and wood [12], stabilizers for polyvinyl chloride(s), PVCs [13]. This wide application of organotin (IV) carboxylates has made these class of compounds of interest and great importance and likely, new applications will emerge in the future [14]. They are used in paints as antifouling agents, in agriculture as active ingredients in pesticides and fungicides, in industries as catalysts. They also find application as fire retardants [15]. Organotin compounds exhibit promising invitro antitumour activities against human tumour cell lines [15, 16]. However, reports have shown that microorganisms have grown resistance [5] to available chemical substances, hence, this work seeks to explore the synthesis of new

metal based substances/compounds using propan-1,3-dioic acid and two parent organotin (IV) compounds with biologically active centers. This will aid the antimicrobial property of the compounds. Conventionally, Organotin (IV) compounds have been synthesized using organic solvents like methanol and propanol using dean and stark apparatus, which is expensive. This process often takes a longer time to yield the required product with a lower percentage yield and exposure of the researcher(s) to harmful effects of organic solvents and contamination of the environment [18]. The use of large volume of solvents, their toxicity, and time-consuming synthesis make this conventional method less effective [17, 18]. This work has used an environmentally friendly method: mechanochemical method, to synthesize metal based compounds within few minutes, with higher percentage yield and no exposure to organic solvents with no consequent health effect (cancer or even death) or pollution on man and his environment [19].

Mechanochemical synthesis is an emerging way of synthesizing compounds by performing chemical transformations using mechanical forces like compression, continuous deformation, fractures, shear, or friction [17, 19]. This method involves the use of ball mill or agate mortar for grinding the reactants to yield products. It has been used for the synthesis of various types of materials such as metallics, metal oxides, metal-organic frameworks, organics, carbons, and related nanomaterials [17] This environmentally benign approach is pollution-free, eco-friendly, low-cost, high yield, and simple [18, 20, 21]. Grinding synthesis has been reported to demonstrate efficiency in shortening time and increasing yield [18, 22, 23]. Employing this synthetic approach, will overcome the limitations of conventional methods and explore the synthesis of new compounds that could exhibit improved efficacy against microbes. Characterization of the synthesized compounds will provide valuable insights into their structural features and properties [24].

Materials and Method

Reagents and solvents

Reagents include KOH, $HOCOCH_2COOH$, Bu_2SnO, Ph_2SnO and Ph_3SnOH. Solvents used are Methanol, ethanol, acetone, DMSO and Hexane. All the reagents and solvents were of analytical grade, bought form Zigma-Aldrich and used without further purification. Their purity ranged from 95-99 %

Preparation of Ligand: potassium propan-1,3-dioate, ($KCOOCH_2COOH$)

The method according to Iorungwa *et al.,* (2019) was adopted with little modification: No catalyst was used [25]. Ligand, $HCOOCH_2COOK$ was prepared by the neutralization reaction between KOH (0.005 mol, 2.8330 g) and propan-1,3-dioic acid (0.005 mol, 5.2530 g) through grinding using an agate mortar and pestle for 10 minutes yielding 91 % of a white solid crystal as the product. The white crystals formed were collected and stored in a desiccator until further use.

Synthesis of Potassium dibutyltin(IV)propan-1,3-dioate: $Bu_2Sn(OCOCH_2COOK)_2$: (1) and Potassium diphenyltin(IV)propan-1,3-dioate, $Ph_2Sn(OCOCH_2COOK)_2$ (2)

Dibutyltin(IV) oxide, Bu_2SnO (0.1162g 0.0030mol) was weighed and transferred into an agate mortar containing already weighed Ligand, $KCOOCH_2COOH$ (0.2388 g, 0.0030mol) ground for 15 minutes to obtain a white powdered crystal as the product: potassium dibutyltin (IV) propan-1,3-dioate: $Bu_2Sn(OCOCH_2COOK)_2$ **(1)** . Similarly, 0.8762 g of Ph_2SnO was weighed, transferred into a mortar containing potassium propan-1,3-dioate and ground for 15 minutes to yield the product: potassium diphenyltin (IV) propan-1,3-dioate: $Ph_2Sn(OCOCH_2COOK)_2$ **(2)** [25, 26].

Synthesis of Potassium triphenylyltin (IV) propan-1,3-dioate: $Ph_3SnOCOCH_2COOK$ (3)

Triphenyltin (IV) hydroxide (0.0014 mol, 1.2917 g) and potassium propan-1,3-dioate (0.0014mol, 0.5000 g) were weighed, transferred into an agate mortar and ground for 15 minutes to obtain a white crystals $Ph_3SnOCOCH_2COOK$ **(3)** as the product [26].

Characterization

Solubility test

Exactly 0.01 g of the prepared ligand and its complexes each was added to 10 mL portions of distilled water, methanol, ethanol, dimethylsulfoxide (DMSO), ethyl acetate and acetone in separate test tubes and shaken vigorously. The sample was considered soluble (S) when the entire solute dissolved to give a homogenous mixture after shaking. When some dissolved and some are left, the samples were considered slightly soluble (SS). Complexes that remained as introduced were identified insoluble (IS) [27, 28, 29].

Melting point determination

Ligand and complexes were filled in separate capillary tubes to a depth of 2 mm and tapped several times at the bottom to ensure close packing. The tubes were inserted into the heating block of Fischer John's melting point equipment and heated. The temperature at which the ligand and complexes melted was read from the digital screen and recorded appropriately [6, 28, 30].

FT-IR spectroscopy

Infrared spectra from 4000 to 400 cm^{-1} were recorded on FTIR-8400S spectrophotometer (SHIMADZU) using KBr pellets [6].

Antimicrobial screening

The antimicrobial activities of ligand and complexes were determined using four bacteria strains: *Escherichia coli*, *Klebstella Pneumonia, Salmonella tyhi* and *Pseudomonas aeruginosa* and four fungi strains: *Conioplora puteana, Rhizopus spp, Scleratium rofsii* and *Serpula lacrymans.* Obtained from the Department of Medical Microbiology, Ahmadu Bello University Teaching Hospital, Zaria. Agar

well diffusion technique and dilution methods were employed for the screening.

Agar well diffusion technique

Agar well diffusion technique was adopted for determination of antimicrobial activity using Sabouraud Dextrose Agar (SDA) as culture medium. This was prepared according to manufacturer's instructions, sterilized at 121 ^{0}C for 15 minutes, poured into sterile petric dishes under an aseptic hood, allowed to cool and solidified. The sterile medium was seeded with 0.1 mL standard inoculums of test microbes and spread evenly over the surface of the medium using a sterile swab. To wells made at the center of each inoculated medium using a standard cork borer of 6 mm diameter and 200 µg/mL of test propan-1,3-dioic acd, **D**, Ligand, **L** and complexes **(1), (2)** and **(3)** dissolved in DMSO were introduced into their respective wells. Other wells supplemented with standard antibacterial and antifungal drugs; ciprofloxacin and fulcin were used as controls. After allowing for diffusion, the media were incubated immediately at 37 ^{0}C for 24 hours for bacteria and 30 ^{0}C for 7 days for the fungi. They were checked daily for inhibition zones: area where the microbes were unable to grow were recorded as antibacterial and antifungal activity. Where inhibition zones were not observed, the compound used was inactive or concentration used may be less than required [6, 21].

Broth dilution method

The broth dilution method as reported by Iornumbe, et al., (2015) was adopted for the determination of the minimum inhibition concentration (MIC), Minimum bactericidal (MBC) and minimum fungicidal concentrations (MFC) of the ligand and complexes against test bacteria and fungi.

Results and Discussion

Synthesis

The ligand: $KOCOCH_2COOH$ (**L**) and complexes: $Bu_2Sn(COOCH_2COOK)_2$, **(!)** $Ph_2Sn(COOCH_2COOK)_2$ **(2)** and $Ph_2SnOCOCH_2COOK$ **(3)** were synthesized as white crystal with Percentage yields ranging from 89 - 99 %, (Tables 1 and 2). The mechanochemical reaction proceeds basically in two steps: KOH reacted with $HOCOCH_2COOH$ by neutralization to yield the ligand $HOCOCH_2COOK$ (Equation 1). This was followed by the complexation reaction between **L** and each parent organotin compound: Bu_2SnO, Ph_2SnO and Ph_3SnOH (Equations 2-4) and deprotonation yielding of the products. This is in agreement with our earlier reports [6, 29].

Table 1: Yield and Description of Prepared Potassium Salt

Dicarboxylic acid	Mass of acid(g)	Mass of KOH(g)	Mass of Potassium Salts(g)	Theoritical Yield(g)	Percentage Yield (%)	Description of Products
Propan-1.3-dioic Acid	5.2530	2.8330	6.5200	7.1724	90.94	White Crystals

Table 2: Yields and Description of the Synthesized Complexes

Compounds	Mass of Salt (g)	Mass of Organotin(g)	Theoretical Yield (g)	Actual Yield (g)	Percentage Yield (%)	Colour of compound
Bu_2SnL_2 **(1)**	0.2388	0.1162**(A)**	2.7193	2.6300 **(1)**	98.55 **(1)**	White Crystals
Ph_2SnL_2 **(2)**	1.0000	0.8762 **(B)**	1.8762	1.7675 **(2)**	94.21 **(2)**	White Crystals
Ph_3SnL **(3)**	0.5000	1.2917 **(C)**	1.7917	1.6000 **(3)**	89.00 **(2)**	White Crystals

Key: **L** = $HOCOCH_2COOK$, **(1)** = $Bu_2Sn(OCOCH_2COOK)_2$, **(2)** = $Ph_2Sn(OCOCH_2COOK)_2$, **(3)** = $Ph_3SnOCOCH_2COOK$, **(A)** = Bu_2SnO, **(B)** = Ph_2SnO **(C)** = Ph_3SnOH,

$$KOH + HCOOCH_2COOH \xrightarrow{\text{Grinding}} HCOOCH_2COOK + H_2O \quad (1)$$

$$Bu_2SnO + 2HCOOCH_2COOK \xrightarrow{\text{Grinding}} Bu_2Sn(COOCH_2COOK)_2 + H_2O \quad (2)$$

$$Ph_2SnO + HCOOCH_2COOK \xrightarrow{\text{Grinding}} Ph_2Sn(COOCH_2COOK)_2 + H_2O \quad (3)$$

$$Ph_3SnOH + HCOOCH_2COOK \xrightarrow{\text{Grinding}} Ph_2SnCOOCH_2COOK + H_2O \quad (4)$$

Where Bu is C_4H_9 and Ph is C_6H_5

Solubility test

Solubility of compounds was used to determine the polarity of the synthesized compounds. Generally, the compounds were more soluble in organic solvents than water. This is in agreement with the principle of dissolution which states that like dissolves like [28]. The Ligand and Complexes were all slightly soluble in water, but soluble in methanol and DMSO. However, **L** and compound (**2**) were insoluble in hexane and ethanol. Compound **(1)** was only slightly soluble in all solvents

(Table 3). The solubility of ligand and complexes in distilled water and polar solvents like methanol, DMSO, could be as a result of the presence of K^+ ions in the structures which improved their solubility [8,29]. The solubility indicated that the complexes and ligand synthesized were slightly polar. The solubility in organic solvents could be due to the presence of substituents group OH and CO [28, 31].

Melting Point Determination

Melting points of the ligand and complexes occur at the temperatures of 203.2 ^{0}C for the ligand, 210-211^{0}C for complexes **(1),** 148 – 152 ^{0}C for **(2)** and 188 – 190 ^{0}C **(3).** This result indicated that they are fairly stable [28] and pure since the range is not wide [26]

Table 3: Solubility and Melting Points of Ligand Synthesized Complexes

Compounds	Distilled H_2O	Methanol	Ethanol	Acetone	DMSO	Hexane	Melting Point (^{0}C)
L	SS	S	IS	S	S	IS	203.2
(1)	SS	SS	SS	SS	SS	SS	210-211
(2)	SS	S	IS	IS	S	IS	148-152
(3)	SS	S	SS	SS	S	SS	188-190

Key: **L** = $HOCOCH_2COOK$ **(1)** = $Bu_2Sn(OCOCH_2COOK)_2$, **(2)** = $Ph_2Sn(OCOCH_2COOK)_2$, **(3)** =$Ph_3SnOCOCH_2COOK$, **S** = Soluble, **SS** = Slightly Soluble, **IS**= Insoluble

Fourier transformed infrared (FTIR) spectra studies of ligand and complexes

The IR spectra of these compounds were recorded in the range of 4000-400 cm^{-1}. The peculiar feature of the IR spectra of the complexes is the absence of νOH stretching vibration of propan-1,3-dioic acid **(D**) at 3500-3100 cm^{-1} due to deprotonation for coordination with tin(IV). Important tentative infrared vibration band assignments have been made on the basis of publications preceding this workand the important data are listed in Table 4. The absorptions of most interest in the spectra of the complexes are ν(COO), ν(Sn-O-C), ν(Bu-Sn) and ν(Ph-Sn).

Table 4: FTIR band (cm-1) of Functional groups for Ligand Synthesized Complexes

Compounds	V_{asym}(COO)	V_m(COO)	ΔV	Sn-Bu/Ph	Sn-O-C	O-H
D	1692.2	1435.0	257.2	-	-	3049.0
L	1699.7	1595.3	104.4	-	-	-
(1)	1479.8	1375.4	103.4	723.1	1077.2	-
(2)	1640.8	1475.1	165.7	720.8	1073.5	-
(3)	1703.4	1540.4	163.0	723.31	1077.2	-

Key: D = Propan-1,3-dioic acid, **L** = $HOCOCH_2COOK$ **(1)** = $Bu_2Sn(OCOCH_2COOK)_2$, **(2)** =$Ph_2Sn(OCOCH_2COOK)_2$, **(3)** =$Ph_3SnOCOCH_2COOK$

Generally, the $\Delta\nu = [\nu(COO)_{as} - \nu(COO)_s]$ value is used to determine the bonding properties of carboxylate anion to tin atom in organotin(IV) carboxylate complexes [32] and the result showed Δν (COO) < 200 cm^{-1} for the three complexes synthesized (Bu_2SnL_2: **(1),** Ph_2SnL_2: **(2)** and Ph_3SnL: **(3)**) which have values 103.4 cm^{-1}, 165.7 cm^{-1} and 163.0 cm^{-1} respectively (Table 4). $\Delta\nu_{(COO)}$ values ≤ 200 cm^{-1} indicate bidentate mode of bonding while values ≥ 200 cm^{-1} indicate monodentate binding. The binding modes in the complexes: **1**, **2** and **3** and ligand **L,** indicate bidentate while the acid (**D**) indicate monodendate: 257.2 cm^{-1} [33]. Abdellah, *et al*., [34] supported a distorted octahedral geometry for diorganotin derivatives in solid state and trigonal bipyramidal structure for triorganotin compound. The emergence of medium intensity bands in the range 1073.5 -1077.2 cm^{-1} due to Sn-C-O and 720.8 – 723.1 cm^{-1} due to Sn-Bu and Sn-Ph further confirmed formation of the compounds [8, 26].

Antimicrobial activity

Antibacterial activity

The synthesized complexes; $Bu_2Sn(OCOCH_2COOK)_2$ **(1),** $Ph_2Sn(COCOH_2COOK)_2$ **(2),** $Ph_3SnOCOCH_2COOK$ **(3)**, ligand $HOCOCH_2COOK$, **L** and propan-1,3-dioic acid $HOCOCH_2COOH$ **(D)** were tested against four bacterial strains: *Escherichia coli*, *Klebstella Pneumonia, Salmonella tyhi* and *Pseudomonas aeruginosa* and four fungi strains: *Conioplora puteana, Rhizopus spp, Scleratium rofsii* and *Serpula lacrymans.* Results obtained were compared with stand drugs: ciprofloxacin for bacteria and fulcin for fungi. Tables 5 – 10 present the zones of inhibition, mininmum inhibition concentration (MIC) and minimum bactericidal/fungicidal concentration MBC/MFC of the ligand, complexes, propan-1,3-dioic acid and control drugs. Zones of inhibition showed antimicrobial activity ranging from 20 mm to 38 mm. Only $Ph_2Sn(COCOH_2COOK)_2$ **(2)** could inhibit the growth of *Escherichia coli*, with the inhibition zone of 23 mm at MIC and MBC 10 µg/mL (Table 8) which is not too far from that exhibited by the control drug: ciprofloxacin: 38 mm at the same MIC and MBC (Table 10). The ligand inhibited the growth of *Klebstella Pneumonia* with the inhibition zone of 24 mm at the MIC and MBC of 10 µg/mL (Table 6) which is higher

than the activity of compound **(2)** with inhibition zone of 21 mm at MIC and MBC of 10 µg/mL and 20 µg/mL (Table 8), respectively against the same organism. Compound **(3)** however, exhibited a slightly higher activity against similar organism than the ligand with inhibition zone of 25 mm at MIC and MBC of 10 µg/mL and 20 µg/mL (Table 9), respectively. Ciprofloxacin, the control drug, could not inhibit the growth of *Klebstella Pneumonia* at the same MIC and MBC. *Escherichia coli*, *Salmonella tyhi* and *Pseudomonas aeruginosa* showed resistant to the ligand and compound **(3)**. The results indicated that all the synthesized compounds (**1), (2)** and (**3)** as well as the ligand showed significant activity against tested bacterial strains with zones of inhibition ranging from 23-27mm (tables 5-9). Compound **D** showed activity against *Klebstella Pneumonia* (21 mm at MIC and MBC of 10 and 20 µg/mL) and *Pseudomonas aeruginosa* 24 mm at MIC and *MBC:* 10 µg/mL (table T). Compound (**1)** inhibited the growth of *Salmonella tyhi* and *Pseudomonas aeruginosa* with inhibition zones of 26 mm and 25 mm (table 7) similar to that exhibited by ciprofloxacin (Table 10) Compound **(2),** inhibited the growth of *Escherichia coli*, *Klebstella Pneumonia* and *Pseudomonas aeruginosa* with inhibition zones of 23 mm, 21 mm and 27 mm respectively at the MIC of 5 and 10 µg/mL and MBC of 20 µg/mL (Table 8). Complex **(3)** showed significant activity: 25 mm zone of inhibition against *Klebstella Pneumonia* at MIC and MBC of 10 µg/mL and 20 µg/mL respectively (Table 9). However, there were cases where the synthesized compounds and ligand did not exhibit activity against the test bacteria. Minimum Bacterial concentration (MBC) revealed that all the compounds synthesized in this work sacrificed the test bacteria at low concentrations, indicating higher activity at higher concentrations. This display of the enhanced inhibition property/activity of the synthesized ligand and complexes could be as a result of the complexation of metals to the parent organotin (IV) compounds during the synthetic process which facilitated the penetration of the complexes and ligand into the membrane and DNA of the microbes leading to the perturbation of their respiratory processes, thereby blocking protein synthesis consequently resulting to death of the microbes [28, 35]. The order of antibacterial activity of the synthesized ligand and complexes is thus: **(2)**> **(**1) >**(3) >L**

Antifungal activity

Compounds (**1), (2)** and (**3)** were active against *Rhizopus spp* with the respective inhibition zones: 23 mm, 23 mm and 27 mm ((Tables 7-9). MIC and MFC were obtained at the concentrations of 5 – 10µg/mL and 10 – 20 µg/mL similar to the concentration of standard/control drug: fulcin. Compounds (**2)** and (**3)** inhibited the growth of *Conioplora puteana and Scleratium rofsii* (Tables 7 and 8) with inhibition zones of 25 and 23 mm and 20 mm and 24 mm respectively. Only compound **(1)** inhibited the growth of *Serpula lacrymans* with inhibition zone of 20 mm (table 7) at MIC and MFC of 10µg/mL (Table 7). This result is in agreement with our earlier work reported [28, 36].which showed significant activity of synthesized complexes and ligands against fungi too. The order of the antifungal activity shown by the ligand and complexes is **(3)** > **(2**) > **L> (1).**

Table 5: Antibacterial Activity of $HCOOCH_2COOH$ (D)

Test Organisms	Effects	Inhibition Zone (mm)	MIC (µg/mL)	MBC/MFC (µg/mL)
Escherichia coli,	R	0	-	
Klebstella Pneumonia	S	21	10	20
Salmonella tyhi	R	0	-	
Pseudomonas aeruginosa	S	24	10	10
Conioplora puteana	S	23	10	20
Rhizopus spp,	R	0	-	
Scleratium rofsii	S	23	10	20
Serpula lacrymans.	S	21	20	20

Key: S = sensitive, R = Resistant

Table 6: Antimicrobial Activity of Ligand: L, $HCOOCH_2COOK$

Test Organisms	Effects	Inhibition Zone (mm)	MIC (µg/mL)	MBC/MFC (µg/mL)
Escherichia coli,	R	0	-	
Klebstella Pneumonia	S	24	10	20
Salmonella tyhi	R	0	=	
Pseudomonas aeruginosa	R	0	-	10
Conioplora puteana	S	22	10	10
Rhizopus spp,	S	23	10	20
Scleratium rofsii	S	20	20	20
Serpula lacrymans.	R	0	-	-

Key: S = sensitive, R = Resistant

Table 7: Antimicrobial Activity of (1): $Bu_2Sn(OCOCH_2COOK)_2$

Test Organisms	Effects	Inhibition Zone (mm)	MIC (µg/mL)	MBC/MFC (µg/mL)
Escherichia coli,	R	0	-	-
Klebstella Pneumonia	R	0	-	-
Salmonella tyhi	S	26	5	20
Pseudomonas aeruginosa	S	25	10	20
Conioplora puteana	R	0	-	-
Rhizopus spp,	S	23	10	10
Scleratium rofsii	S	21	20	20
Serpula lacrymans.	R	0	-	-

Key: S = sensitive, R = Resistant

Table 8: Antimicrobial Activity of (2): $Ph_2Sn(COOCH_2COOK)_2$

Test Organisms	Effects	Inhibition Zone (mm)	MIC (µg/mL)	MBC/MFC (µg/mL)
Escherichia coli,	S	23	10	10
Klebstella Pneumonia	S	21	10	20
Salmonella tyhi	R	0	-	-
Pseudomonas aeruginosa	S	27	5	10
Conioplora puteana	S	25	10	10
Rhizopus spp,	S	23	10	20
Scleratium rofsii	S	20	20	20
Serpula lacrymans.	R	0	-	-

Key: S = sensitive, R = Resistant

Table 9: Antimicrobial Activity of (3): $Ph_2SnOCOCH_2COOK$

Test Organisms	Effects	Inhibition Zone (mm)	MIC (µg/mL)	MBC/MFC (µg/mL)
Escherichia coli,	R	0	-	-
Klebstella Pneumonia	S	25	10	20
Salmonella tyhi	R	0	-	-
Pseudomonas aeruginosa	R	0	-	-
Conioplora puteana	S	25	10	20
Rhizopus spp,	S	27	10	10
Scleratium rofsii	S	24	10	20
Serpula lacrymans.	R	0	-	-

Key: S = sensitive, R = Resistant

Table 10: Antimicrobial Activity of Control Drugs

Test Organisms	Ciprofloxacin	Fulcin
Escherichia coli,	38	R
Klebstella Pneumonia	-	R
Salmonella tyhi	25	R
Pseudomonas aeruginosa	25	R
Conioplora puteana	R	28
Rhizopus spp,	R	R
Scleratium rofsii	R	R
Serpula lacrymans.	R	26

Key: S = sensitive, R = Resistant

Conclusion

$HCOOCH_2COOH$, **L** $Bu_2Sn(OCOCH_2COOK)_2$, **(1)** $Ph_2Sn(COOCH_2COOK)_2$ **(2)** and $Ph_2Sn(OCOCH_2COOK)_2$ **(3)** were mechanochemically synthesized with 89 – 99 percentage yield. The method demonstrated high yields, indicating the efficiency of the chosen synthetic method/route. The solubility in distilled water, methanol, ethanol, DMSO and acetone indicated slight polarity of the covalent bonds in the compounds. Melting points of the compounds revealed their stability and purity. FTIR data revealed that all the complexes and ligand exhibited bidendate binding mode with their $\Delta\nu(COO) \leq 200\ cm^{-1}$. The antimicrobial studies revealed that the metal complexes exhibited

more activity against test microbes. Both antibacterial activity and antifungal activities were significant with zones of inhibition ranging from 20 mm to 27 mm at the MIC and MBC/MFC ranging from 10-20 µg/mL and 5 – 20 µg/mL, respectively. The compounds exhibited significant activity at lower concentrations. The synthesized complexes and their ligand are promising in the design and manufacture of metal-based drugs and formulations as well as being potential bactericide and fungicides. The order of their antibacterial activity is: **(2)** > **(1)** > **(3) > L**, while that of antifungal activity is : **(3)** > **(2)** > **L >(1).**

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