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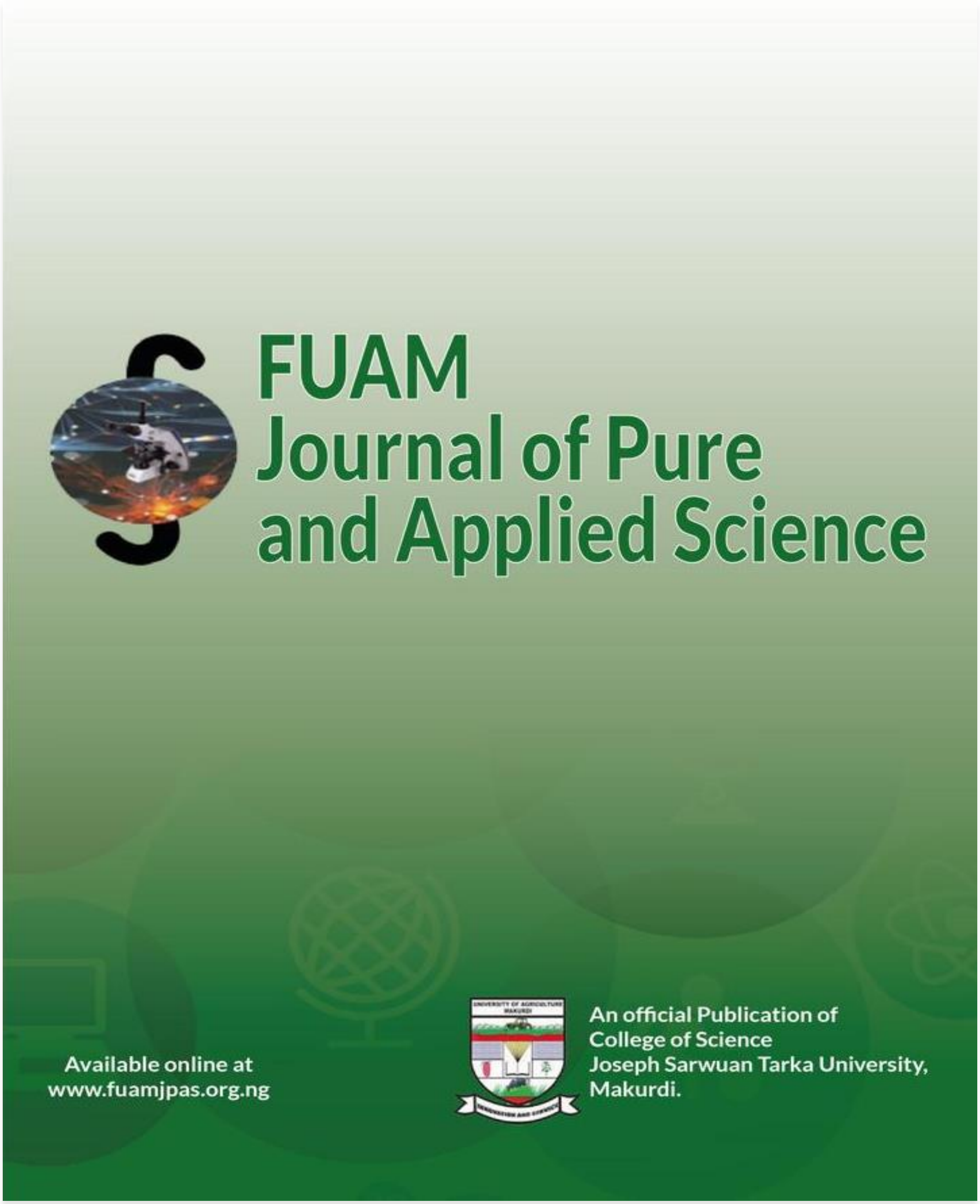

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Synthesis, Characterization and Nematicidal Properties of Triphenyltin (IV) Derivatives Of Butanedioic Acid and Hexandioic Acid

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Abstract

Two triphenyltin (IV) derivatives of butanedioic and hexanedioic acids with potassium (K) metal and two ligands were synthesized by reacting KOH separately with butanedioic acid and hexanedioic acid to give the ligands: **L_1** ($HCOO(CH_2)_2COOK$) and **L_2** ($HCOO(CH_2)_4COOK$) followed by grinding each ligand with triphenyltin (IV) hydroxide (Ph_3SnOH) in an agate mortar with a pestle. The purity of the complexes was checked by melting point determination. Characterization of the complexes was done using Fourier Transformed Infrared (FTIR) spectroscopy, solubility and nematicidal activity against the nematode *Meloidogyne javanica* was also investigated. Results revealed percentage yield of the compounds in the range 80.38 – 98.44 % and melting points (209.70 – 240.20 ^{0}C) indicate pure and fairly stable ligands and complexes. Both ligands and complexes were soluble in distilled water and ethanol indicating their fair polarity. FTIR data revealed complexation of the metals K and Sn with the ligands reflected by the V_{asym}(COO) and V_{sym}(COO) vibrational frequency shifts: **L_1** (1637.4-1409.9 cm^{-1}), **L_2** (1650.0-1403.7 cm^{-1}), **(1)** (1578.0-1427.7 cm^{-1}) and **(2)** (1642.0-1476.7 cm^{-1}). The modes of coordination were found to be bidendate in the complexes and monodentate in the ligands. The nematicidal studies result showed the highest mortality rate was exhibited by complex **(1)** and its ligand, L_1: 95.8 and 90.0 % respectively. The order of activity is thus: **(1) > L_1 > (2) > L_2.** This property increased with increase in concentration and contact time. These compounds are promising as nematicidal agents in the control and management of nematodes on the field thereby improving food security.

Keywords: Mechanochemical synthesis, Characterization, Nematicidal property, potassium triphenyltin (IV) Complexes.

Introduction

A significantly rising interest in the design of metal compounds as drugs and diagnostic agents is currently observed in the area of scientific inquiry [1]. Metals find relevance in medicinal applications due to the crucial roles they play in living systems. They lose electrons to generate positively charged species, which makes them soluble in biological systems. Their biological roles are exhibited in this ionic state [2]. Since biological molecules like DNA and proteins are electron-rich while metal ions are electron-poor perhaps, lack electrons, there is a greater tendency for these opposing charges of metal ions and biological molecules to interact and bind [2]. The interaction of metal ions with other small molecules and ions that are essential to life, like oxygen, is also subject to this tendency [1]. Organotin compounds have contributed immensely to the study and understanding of organometallic compounds. Tin, a member of group IV elements forms numerous inorganic and organometallic compounds. It has two 5*s* and two 5*p* electrons in its valence orbitals. It is able to lose the two electrons in the 5*p* orbital to form Sn^{2+} ion or share all four electrons with others by acquiring the stable electronic configuration of xenon. Sherman *et al.,* [3] opined that neither Sn^{4+} nor Sn^{2+} is found in aqueous solutions. The bonding of tin with carbon forms group of compounds known as organotin compounds which are a class of organometallic compounds.

Organotin compounds are primarily tetravalent in structure and have at least one covalent carbon-tin bond [4]. The biological activities of the organotin (IV) compounds have been observed to be significantly impacted by variation in the alkyl or aryl substituents. Organotin compounds are important because of their diverse structural makeup, aptitude for catalysis and redox, propensity to interchange ligands, and range of potential interactions with biologically beneficial properties. They also find application as anti-oxidant molecules (free radical scavengers), antihelminthics (poultry and animal husbandry) and insecticides (sheep and cattle). Dibutyltin dilaurate is a component of a commercial drug used for treatment of worm infestation in poultry. These potentials gave credence to organotin (IV) compounds usefulness in agriculture, industries, medicine and pharmaceuticals [5]. Organotin (IV) complexes are widely used in industries, and their biological characteristics against cancer cell lines, fungi, and bacteria [6, 7] have drawn significant attention to their investigation [7, 8]. They are used in agriculture as pesticides [9] and are well-known for their commercial and medicinal applications [8, 10]. They have been shown to have anti-tumor activity. Organotin (IV) carboxylates have been extensively researched because of their varied structures and medical uses as anticancer and anti-tuberculosis agents/drugs [6, 11 12]. The anionic groups' propensity to coordinate intra- or intermolecularly causes them to display a variety of intriguing structural characteristics [13, 14]. The production of polyesters and polyurethanes is one of the additional uses for organotin (IV) carboxylates [15].

Nematodes are reported to be tiny worms that are some of the most complex and numerous organisms in the world [16] causing damage to cultivated plant species. They are called plant-parasitic nematodes (PPNs). Plant-parasitic nematodes (PPNs) are one of the most destructive plant pathogens worldwide, and are extremely challenging controlling them [16-18].

Root-knot nematodes (RKNs) are a specific concern to global food security, as plant-parasitic nematodes (PPNs) greatly hinder crop productivity [19]. Among the group of plant-parasitic nematodes, the *Meloidogyne* genus is well known for its substantial economic and scientific significance [20]. *Meloidogyne javanica* is a highly destructive nematode of *Meloidogyne* spp. Pradhan [21], Rumbos [22] and Volvas, [23] reported *Meloidogyne javanica* to be the major pathogen for many crop plants like tomatoes, potatoes, peanuts, papayas, and rootstocks. Furthermore, *Meloidogyne species* have the ability to alter plant defense mechanisms, making plants more vulnerable perhaps susceptible to other diseases like fungus and bacteria. This leads to increased yield losses [24].

The global production of numerous economically significant crops is seriously threatened by plant-parasitic nematodes (PPNs), both in terms of quantity and quality. Report has shown that Plant parasitic nematodes are responsible for 12.3 % of agricultural losses, or USD 157 billion, annually [25]. As such, they are considered the most important Nematode worldwide [26]. There is therefore need in finding remedy in mitigating these gross damaged causing pathogens. Thus, the need for continuous research globally to improve food security.

In an attempt to further explore the interesting features of organotin (IV) compounds, we report here the nematicidal property of two mechanochemically synthesized organotin (IV) derivatives of butanedioic acid and hexanedioic acid against *Meloidogyne javanica*.

Materials and Methods

Reagents

The reagents used for the preparation of the ligands and complexes were of analytical grade, purchased from Sigma-Aldrich with purity ranging from 98- 99.8 % and used without further purification.

Synthesis of Ligands; L_1: $HCOO(CH_2)_2COOK$ (potassium butanedioate) and **L_2: $HCOO(CH_2)_4COOK$ (**potassium hexanedioate) **L_1** was prepared by neutralization reaction according to the method reported by Iornumbe, *et al* [27]. Butanedioic acid: $HCOO(CH_2)_2COOH$ (6.2000g, 0.05 mol) and Potassium Hydroxide: KOH (3.2910g, 0.05 mol) were ground in an agate mortar with a pestle for 15 min yielding white solid crystals as product. They were collected and stored in a desiccator for further analysis. Similarly, **L_2** was prepared by grinding hexanedioic acid: $HCOO(CH_2)_4COOH$ (6.5000 g, 0.05 mol) and KOH (1.9932 g, 0.05 mol) in an agate mortar with pestle for 15 min. The product was obtained as a white crystal.

Synthesis of potassium triphenyltin(IV) butanedioate: $Ph_3SnCOO(CH_2)_2COOK$ (1) and potassium trphenyltin(IV)hexanedioate:$ph_3SnCOO(CH_2)_4COOK$, (2)

$Ph_3SnCOO(CH_2)_2COOK$: **(1)** was synthesized as earlier reported [27, 28]. The ligand, **L_1**, $HCOO(CH_2)_2COOK$ (2.5000 g, 0.05 mol) and Triphenyltin (IV) hydroxide: Ph_3SnOH (2.6740 g, 0.05 mol) were ground with a pestle in an agate mortar for 15 min. White solid crystals were collected as product and kept in a desiccator for further analysis. Similarly, $Ph_3SnCOO(CH_2)_4COOK$, **(2)** was synthesized by grinding $HCOO(CH_2)_4$ COOK, **L_2** (1.0002 g, 0.05 mol) and Ph_3SnOH (2.0020 g, 0.05 mol) in an agate mortar with a pestle for 15 min. White solid crystal was obtained as product and kept in a desiccator until further use.

Characterization

Solubility Test

L_1, **L_2** and their complexes **(1)** and **(2),** exactly 0.01 g each were poured into 10 mL portions of distilled water, methanol, ethanol, dimethylsulfoxide (DMSO) and diethyl ether in separate test tubes and shaken vigorously. When the entire solute dissolves to give a homogenous mixture after shaking, it is considered soluble (S) When some dissolved and some left undissolved, the samples were considered slightly soluble (SS). Complexes that remained as introduced were reported as insoluble (IS) [29, 30].

Melting point Determination

L_1, **L_2** and their complexes **(1)** and **(2)** were filled into separate capillary tubes 2 mm depth, tapped several times at the bottom to ensure close packing and inserted into the heating block of Fischer Johns melting point equipment and heated until the compounds melted. The temperature at which the ligand and complexes melted were recorded accordingly [7, 30].

FT-IR spectroscopy

Fourier Transformed Infrared spectra from 4000 to 400 cm^{-1} were recorded on FTIR-8400S spectrophotometer (SHIMADZU) using KBr pellets [7].

Nematicidal Activity

The nematicidal activity of the synthesized ligands and complexes was evaluated using *Meloidogyne javanica* as the test organism. The nematodes were harvested from the roots of heavily infected tomato: *Lycopersicon esculentum* collected from an Abattoir farm along the River Benue within Makurdi metropolis of Benue State, Nigeria, identified in the Department of Plant Science and Biotechnology, Joseph Sarwuan Tarka University Makurdi-Nigeria and cultured in an agar medium. The method according to Iorungwa *et al.*, [31] was adopted without modification for this study. Different concentrations: 125 ppm, 62.5 ppm, 31.3 ppm 15.6 ppm and 7.8 ppm of the ligands and complexes were prepared separately and 10mL of each transferred into 3 petri dishes containing soil with very high root nematode diseases and observed for 30 min. The control consists of 10 mL of distilled water. Report was made for day 1, 3, 5, and 7.

The nematodes were considered alive if they were mobile or appeared as a winding shape and considered dead if they did not move when probed with a needle. To check if nematodes regain mortality or not they were transferred into distilled water for 12 h.

The corrected percentage mortality was calculated using the formula:

$$\%\ \text{mortality} = \frac{(mortality\ of\ treatment - mortality\ of\ Co.}{(1 - mortality\ of\ Co)}\ x\ 100 \quad (1)$$

Results and Discussion

Synthesis

The ligands: **L_1:** $HCOO(CH_2)_2COOK$ and **L_2:** $HCOO(CH_2)_4COOK$ and complexes: $Ph_3SnCOO(CH_2)_2COOK$ **(1)** and $Ph_3SnCOO(CH_2)_4COOK$ **(2)** were synthesized as white crystal with Percentage yields ranging from 80.38 – 98. %, (Tables 1 and 2). The reaction proceeds in two steps: KOH reacted with $HOCOO(CH_2)_2COOH$ and $HCOO(CH_2)_4COOH$ separately by neutralization yielding the corresponding ligands (Equations 2 and 3). Complexation reaction was followed by the reactions between **L_1** and **L_2** separately with Ph_3SnOH (Equations 4 and 5) and deprotonation yielding 91.86 % and 88.38 % of the corresponding products: $Ph_3SnCOO(CH_2)_2COOK$ **(1)** and $Ph_3SnCOO(CH_2)_4COOK$ **(2)** This agreed with our earlier reports [7, 32].

Table 1: Yield and Description of Prepared Ligands

Dicarboxylic acid	Mass of acid(g)	Mass of KOH(g)	Actual yield (g)	Theoretical Yield(g)	Percentage Yield (%)	Description of Products
Butanedioic Acid	6.2000	3.2910	7.8340(**L_1**)	7.9500	98.44	White Crystals
Hexanedioic Acid	6.5909	1.9932	7.5460(**L_2**)	9.3877	80.38	White Crystals

Key: L_1 = $HCOO(CH_2)_2COOK$, **L_2** = $HCOO(CH_2)_4COOK$

Table 2: Yields and Description of the Synthesized Complexes

Complexes	**Mass of Ligand (g)**	**Mass of Ph_3SnOH(g)**	**Theoretical Yield (g)**	**Actual Yield (g)**	**Percentage Yield (%)**	**Colour of compounds**
Ph_3SnL_1 **(1)**	2.5000(**L_1**)	2.6740 **(1)**	2.0233**(1)**	1.8587 **(1)**	91.86 **(1)**	White Crystals
$Ph_3Sn\ L_2$ **(2)**	1.0020(**L_2**)	2.0020 **(2)**	2.9045**(2)**	2.5710 **(2)**	88.52 **(2)**	White Crystals

Key: L_1 = $HCOO(CH_2)_2COOK$, **L_2** = $HCOO(CH_2)_4COOK$, **(1)** = $Ph_3SnCOO(CH_2)_2COOK$ and **(2)** =$Ph_3SnCOO(CH_2)_4COOK$

$$KOH + HCOO(CH_2)_2COOH \xrightarrow{grinding} HCOO(CH_2)_2COOK + H_2O \quad (2)$$

$$KOH + HCOO(CH_2)_4COOH \xrightarrow{grinding} HCOO(CH_2)_4COOK + H_2O \quad (3)$$

$$Ph_3SnOH + HCOO(CH_2)_2COOK \xrightarrow{grinding} Ph_3SnCOO(CH_2)_2COOK + H_2O \quad (4)$$

$$Ph_3SnOH + HCOO(CH_2)_4COOK \xrightarrow{grinding} Ph_3SnCOO(CH_2)_4COOK + H_2O \quad (5)$$

Where Ph is C_6H_5

Solubility Test

The polarity of the synthesized compounds was determined using solubility test. **L_1**, **L_2** and complex **(1)** were soluble in distilled water while complex **(2)** was slightly soluble forming a cloudy solution (Table 3). This solubility is enough for application since formulations of compounds as fungicides, pesticides, insecticides and drugs are applied as suspensions as well [33]. **L_1** and its complex **(1)** formed suspension in ethanol but were insoluble in methanol, hexane, DMSO and diethyl ether. **L₂** on the other hand, formed suspension in DMSO, ethanol and diethyl ether and was insoluble in methanol while its corresponding complex **(2)** was soluble in ethanol, formed suspensions in DMSO and methanol but was insoluble in hexane. The solubility of ligands and complexes in distilled water and polar solvents like methanol, ethanol and DMSO, could be attributed to the presence of K^+ ions in their structures (Equations 2 - 5) which immensely improved their solubility [9, 32]. This solubility indicates the slight polarities of the ligands and complexes and agrees with the principle of like dissolves like: organic solvents dissolve organic solutes/compounds [13]. The solubility in organic solvents could be due to the presence of OH and CO substituents [30, 34] in the compounds.

Table 3: Solubility, Melting Points of Ligands and Synthesized Complexes

Compounds	Distilled H_2O	Methanol	Ethanol	DMSO	Diethyl ether	Hexane	Melting Point (⁰C)
L_1	S	IS	SS	IS	IS	IS	240.20
L_2	S	IS	SS	SS	SS	IS	212.40
(1)	S	IS	SS	S	IS	IS	226.30-226.80
(2)	SS	SS	S	SS	IS	IS	209.30-209.70

Key: **L_1** = $HCOO(CH_2)_2COOK$, **L_2** = $HCOO(CH_2)_4COOK$, **(1)** = $Ph_3SnCOO(CH_2)_2COOK$ and **(2)** = $Ph_3SnCOO(CH_2)_4COOK$, **S** = Soluble, **SS** = Slightly Soluble, **IS**= Insoluble

Melting point determination

Melting points of **L_1** and **L_2** occurred at the temperatures of 240.20 ⁰C and 212. 40 ⁰C respectively, while that of complexes **(1)** and **(2)** occurred in the range 226.30 - 226.80 ⁰C and 209.30 - 209.70 ⁰C respectively (Table 3**).** The range recorded was not wide indicating the fairly stable nature and purity of the compounds [31, 35, and 36]. Pure substances are known to melt at a sharp, highly-defined temperature (0.5 – 1 ⁰C) whereas impure or contaminated substances generally exhibit a wider melting range. A melting point range is very narrow for pure solids (usually just 1 – 2 ⁰C). Young (2013) opined that if a solid substance is pure, it should melt over a very narrow range of about 1 ⁰C [36].

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

Assignments of vibrational frequencies for synthesized compounds were done by comparing with data reported from organotin compounds formed with oxygen donor atoms containing anionic groups. The asymmetrical carbonyl stretching V_{sym}(COO) for **L_1** and **L_2** were observed at 1637.4 cm^{-1} and 1650.0 cm^{-1} which shifted in their complexes to 1578.0 cm^{-1} in complex **(1)** and 1642.0 cm^{-1} in complex **(2)**, respectively (Table 4). Their symmetrical vibrations: V_{sym}(COO) were observed at 1409.9 cm^{-1} and 1403.7 cm^{-1} in **L_1** and **L_2** respectively which also shifted to 1427.7 cm^{-1} in complex **(1)** and 1476.7 cm^{-1} in complex **(2)**, respectively. These shifts occurred upon complexation of the ligands with Ph_3SnOH (Equations 4 and 5) and the deprotonation of butanedioic acid and hexanedioic acid when they reacted with KOH (Equations 2 and 3). This agrees with our earlier report [27].

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

Compounds	V_{asym}(COO)	V_m(COO)	ΔV	Sn-Ph	Sn-O-C	O-H	K-O-C
L_1	1637.4	1409.9	227.2	-	-	3030.6	1330.8
L_2	1650.0	1403.7	246.3	-	-	3044.6	1365.4
(1)	1578.0	1427.7	150.3	723.1	1077.2	-	1331.7
(2)	1642.0	1476.7	165.3	721.1	1077.2	-	1340.7

Key: **L_1** = $HCOO(CH_2)_2COOK$, **L_2** = $HCOO(CH_2)_4COOK$, **(1)** = $Ph_3SnCOO(CH_2)_2COOK$ and **(2)** = $Ph_3SnCOO(CH_2)_4COOK$

The differences between V_{asym}(COO) and V_{sym}(COO) that is, $\Delta v_{(COO)}$, gives the binding modes in the ligands and complexes. Values less than 200 cm^{-1} indicate bidendate mode of coordination while above 200 cm^{-1} indicates monodentate. The values in this study were 227.2 cm^{-1} and 246.3 cm^{-1} for **L_1** and **L_2**, respectively, and 150.3 cm^{-1} and 165.3 cm^{-1} for complex **(1)** and complex **(2)**, respectively (Table 4). These values indicate that the binding mode of coordination in the ligands is monodentate while that in the complexes is bidentate [27].

Sn-Ph bond vibrational frequency was observed at 723.1 cm^{-1} and 721.1 cm^{-1} in the complexes **(1)** and **(2)**, respectively. The formation of Sn-O-C bond was evident by the observed frequency at 1077.2 cm^{-1} in the complexes indicating the occurrence of complexation in the process during synthesis. The vibrational frequencies in the range 1330.8 cm^{-1} - 1365.4cm^{-1} were assigned to K-O-C bond in the ligands and complexes respectively (Table 4). The emergence of vibrational frequencies at 1077.2 cm-

^{1} due to Sn-C-O and 721.1 – 723.1 cm^{-1} due to Sn-Ph confirmed formation of the compounds [9, 35, 37].

Nematicidal Studies

The nematicidal studies of the ligands and complexes revealed their nematicidal property. The mortality values of the ligands and complexes against the test nematode: *Meloidogyne javanica* on day 1, 3, 5 and 7 at concentrations: 7.8 ppm – 125.0 ppm were investigated (Tabl 110 6). **L_1** recorded the highest percentage mortality rate which ranged from 81.0 – 90.0 % at the concentration of 125.0 ppm and the lowest from 19.3 – 19.7 % at the concentration of 7.8 ppm from day 1 to day 7 (Table 5).

Its complex: **(1)** showed increased nematicidal property with increased mortality rate at the same concentrations ranging from 90.3 – 95.8 % at 125.0 ppm and the least ranging from 37.6 – 39.9 % at 7.8 ppm. H_2O was used as the control and did not show any activity against the nematodes.

Table 5: Nematicidal studies of Ligand (L_1) and Complex (1) *against Meloidogyne javanica*

Synthesized compounds	No of days	Corrected percentage mortality at various concentration				
		125.0 ppm	62.5 ppm	31.3 ppm	15.3 ppm	7.8ppm
L_1	1	81.0	76.7	62.5	38.6	19.3
	3	83.9	78.0	63.3	40.9	19.5
	5	87.0	80.8	65.0	42.3	19.0
	7	90.0	85.3	65.3	43.3	19.7
Control	1	0	0	0	0	0
	3	0	0	0	0	0
	5	0	0	0	0	0
	7	0	0	0	0	0
(1)	1	90.3	79.3	74.2	43.4	37.6
	3	92.4	80.3	70.6	44.0	39.2
	5	94.3	83.0	73.5	49.7	39.8
	7	95.8	86.7	75.2	50.3	39.9

Key: L_1 = $HCOO(CH_2)_2COOK$ **(1)** = $Ph_3SnCOO(CH_2)_2COOK$

Table 6: Nematicidal studies of Ligand (L_2) and Complex (2) *against Meloidogyne javanica*

Synthesized compounds	No of days	Corrected percentage mortality at various concentration				
		125.0 ppm	62.5 ppm	31.3 ppm	15.3 ppm	7.8ppm
L_2	1	77.0	69.7	49.5	41.7	13.3
	3	77.9	70.0	53.3	41.9	14.5
	5	78.0	70.8	58.0	42.3	16.0
	7	79.0	71.3	64.3	43.3	16.3
Control	1	0	0	0	0	0
	3	0	0	0	0	0
	5	0	0	0	0	0
	7	0	0	0	0	0
(2)	1	80.3	79.3	69.3	43.4	23.5
	3	80.4	80.3	70.7	44.0	26.0
	5	81.3	84.0	73.3	49.7	32.7
	7	89.4	85.5	74.3	50.3	32.6

Key: L_2 = $HCOO(CH_2)_4COOK$ and **(2)** = $Ph_3SnCOO(CH_2)_4COOK$

The mortality rate of **L_2** against *Meloidogyne javanica* on day 1 to day 7 at the same concentrations ranged from 77.0 - 79.0 % as the highest at 125.0 ppm and 13.3 – 16.3 % as the lowest at 7.8 ppm respectively (Table 6). Its complex, (**2**), also showed increased nematicidal property with increased mortality rate at the same concentrations: ranged from 80.3 – 89.4 % at 125.0 ppm as the highest and 23.5 – 32.6 % at 7.8 ppm as the lowest (Table 6). The control: H_2O, did not show any activity against the nematodes. This nematicidal property exhibited by the complexes with a higher mortality rate than that of the ligands agrees with the work reported by Iorungwa *et al.,* [31]. The mortality rate increases with increase in concentration and contact time of the nematodes with the compounds. **L_1** and **L_2** and their corresponding complexes **(1)** and **(2)** respectively, are compounds with promising nematicidal properties that can be harnessed for application in agriculture for the control of nematodes on the field. **L_1** and complex **(1)** exhibited higher Nematicidal property than **L_2** and complex **(2).** In both cases, the complexes exhibited higher nematicidal property than their corresponding ligands. The order of activity is thus: **(1) > L_1 > (2) > L_2.**

Conclusion

The compounds; **L_1:** $HCOO(CH_2)_2COOK$, **L_2** :$HCOO(CH_2)_4COOK$, **(1)**: $Ph_3SnCOO(CH_2)_2COOK$ and **(2)**: $Ph_3SnCOO(CH_2)_4COOK$ were synthesized mechanochemically with high yields (80.44 – 98.44 %). They were all soluble in distilled water and ethanol indicating the slight polarity of the covalent bonds in the compounds. Melting points of the compounds revealed their high purity and stability. FTIR data revealed bidendate binding mode of coordination in complexes and monodentate mode in the ligands. The nematicidal studies revealed that both ligands and complexes exhibited nematicidal properties at all the concentrations and days investigated. The property was seen to increase with concentration and contact time of the nematodes with the compounds. These nematicidal properties can be harnessed for application in agriculture for the control and management of nematodes on the field thereby improving food security.

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