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SYNTHESIS AND BIOLOGICAL SCREENING OF NOVEL ORGANOTIN (IV) COMPLEXES OF CARBOXYLATE BASED LIGANDS

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Keywords

Organotin (IV) complexes, Pathogenic strains, Antibacterial activitiy, Penicilin and Amoxicilin as references.

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Abstract

The purpose of this research was synthesis and characterization of different di and triorganotin (IV) complexes by combining organotin (IV) compounds of type R_3SnL and R_2SnL_2 (R=methyl, n-butyl, phenyl) with two newly prepared ligands - 2-((3,4-dichlorophenylcarbamoyl)benzoic acid and (E)-4-methyl-5-(4-methylthiazole-3(2H)-yl)5-oxopent-3-enoic acid in 1:1 and 1:2 depending on tin metal oxidation state. From FT-IR data, ligands and organotin (IV) complexes structures were ascertained. All organotin (IV) complexes exhibited absence of hydroxyl group broad band (deprotonation of carboxylate group), existence of sharp, medium intensity bands in lower wave number region indicating presence of Sn-C, Sn-O bond and revealed C-O-Sn bridge which confirmed complexation phenomena. Antibacterial activities were performed against two bacterial strains. Ligand-2 was more active than ligand-1 and organotin (IV) complexes with ligand-2 were more promising and biologically active than ligand-1 and its complexes

INTRODUCTION

The world's most prevalent cause of mortality is infectious illnesses, yet the antibiotics that have been invented to treat them are becoming less effective due to a dramatic increase in resistance to drugs[1].Currently, in the twenty-first century, antimicrobial resistance (AMR) is a significant worldwide health concern[2].The difficulties related to viral infections combined with the improper and overuse of antibiotics, also known as antimicrobials (sometimes known as "antibacterials"), reduce the effectiveness of traditional antibiotic therapy and cause a variety of other issues[3]. Anti-microbial resistance (AMR) is the most significant health concern confronting humanity, according to WHO (one of the top ten global public health organizations). It threatens the efficient diagnosis and treatment of an ever-widening variety of illnesses caused by fungi and bacteria[4]. Antibiotic-resistant microbes are predicted to kill 10 million people globally by the year 2050[5]. New compounds with improved binding affinities for the drug-target are therefore desperately demanded[6].

Scientists are interested in the flexible molecular structures and a variety of biochemical devours of organometallic compounds. Of these, organotin compounds are of particular significance due to their substantial biological activity and molecular structure[7].In addition to their wide range of industrial and material science uses, organometallic compounds exhibit significant biological activity. Because of their distinct biological target ability, They have potential uses in drug development, diagnosis, and treatment[8]. Because of differences in coordination geometry and structure, organometallic compounds have the potential to function as both biocides and antibacterial agents[9-11].

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Organotin, a class of organometallic compounds, is receiving a lot of importance since it may be utilized as a reagent or catalyst in the synthesis of organic molecules[12]. Because of its unique method in the synthesis of many different kinds of chemical compounds, organotin compounds are regarded as one of the well-known organometallic categories[13]. At least one C-Sn covalent bond is required to recognize organotin compounds[14].Their general formula is R_nSnX_{4-n}, where n can range from 1 to 4[15]. Mono-, di-, tri-, and tetra organotin(iv) compounds with tetravalent tin centre capacity are called ($RSnX_3$, R_2SnX_2, R_3SnX, R_4Sn), where R part can be an alkyl or aryl group and X part is of anionic type, depending on the quantity of alkyl or aryl part[16]. Oxides, hydroxides, fluorides, chlorides, carboxylates, and thiolates are among the frequently employed anions, and it appears that these groups have a significant role in biological activity[17].

Organotin(IV) carboxylates are the most researched of the several types of organotin compounds due to the unique benefits of the carboxylate moiety[7]. Organotin(IV) complexes consisting of relatively small semi-rigid and flexible benzene core carboxylate ligands show significant biological activities[18].The stereochemistry of organotin(IV) and the oxidation number of the tin atom plays an important role in the antipathogenic actions of some organotin(IV) carboxylates that have been reported[19].

Because organotin(IV) carboxylate complexes adopt a variety of geometries, including octahedral, trigonal bipyramidal, and tetrahedral, researchers have focused their attention on developing structurally modified new carboxylate and their organotin(IV) derivatives in order to better understand the coordinating ability of carboxylate ligands toward organotin compounds[7]. The composition of carboxylate ligand plays a significant role in determining the bioactivity of the organotin(IV) complexesbecause it can affect the hydro/lipophilicity of the complex and aid in transporting it across the cell membrane [20].

Organotin (IV) compounds have been the subject of research attention due to their variety of biological roles in addition to their unique structural characteristics[21, 22].The Primary factors that influence their activities include the quantity and type of functional organic ligand present, which are linked to the Sn metal core, hence a large variety of derivatives, including those with antifungal, anticancer and antitumor, antiviral, antibacterial, antimalarial, and corrosion-inhibiting properties, have been produced and their biological activity examine[22-25]A special focus must be paid to tackle the issue of bacterial resistance to antibacterial medications, which has grown to be a major concern in a number of fields[26, 27].Due to this, lots of efforts have been made to discover novel antibacterial medications[28].

On the basis ofpreviously described potential uses of the organotin (IV) carboxylate complexes, in the current study, we present the synthesis and comparative study on the antibacterial activity of Organotin (IV) complexes of ligands specifically -2-((3,4-dichlorophenylcarbamoyl) benzoic acid and (E)-4-methyl-5-(4-methylthiazole-3(2H)-yl)5-oxopent-3-enoic acid against two bacterial strains, Gram-positive Staphylococcus aureus and Gram-negative Pseudomonas aeruginosa.

2. Materials and Methods

2.1. Chemicals Used

Analytical grade organic solvents were used and dried prior to use. The ligand was used as such synthesized. Trimethyl tin (IV) chloride, Bis-tributyltin-oxide, Amino thiazole, Phthalic anhydride, 3,4-dichloro-aniline, Maleic anhydride, Acetone, Chloroform, DMSO, Methanol, Toluene, Triethylamine, Distilled water and Organotin (IV) salts were purchased from Sigma Aldrich.

2.2. Procedure for the synthesis of ligands L_1 and $L_2$and their complexes

2.2.1. Synthesis of Ligand-1 (L_1) (-2-(3,4-dichlorophenylcarbamoyl) benzoic acid)

0.44 mmoles of 2-methyl-4-aminothiazole and 2-methyl maleic anhydride were prepared in a round-bottom flask with glacial acetic acid by stirring for about 12-hour period at room temperature. The precipitates were observed, filtered, washed with ice-cold distilled water and then allowed to dry at room temperature. TLC verified the product formation[29].

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Reaction scheme for synthesis of Ligand-1

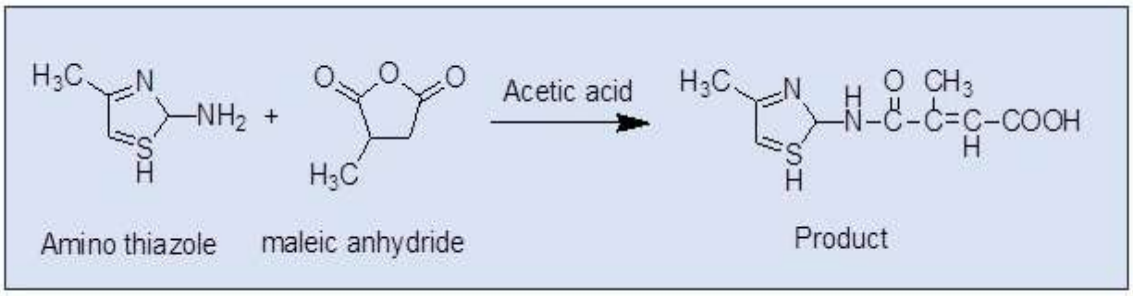

Figure 1: Reaction for Synthesis of Ligand-(-2-(3,4-dichlorophenylcarbamoyl) benzoic acid)

2.2.2. Synthesis of complex L_1R_1:

120 ml of dried toluene,0.3 g of ligand and 0.18 ml of triethylamine (Et_3N) were added in a round bottom flask assisted with magnetic stirrer and condenser.In order to completely incorporate the ligand into the dry toluene, the reaction mixture was allowed to stand for 30 minutes. The reaction flask was allowed to reach room temperature for 15 to 20 minutes. Then,0.3g of trimethyl tin chloride $(CH_3)_3SnCl$ was added. Ligand-metal ratio was 1:1 and average 6 hours were given for refluxing. A yellow-colored gummy product was obtained after the solution was filtered and the solvent was removed using a rotary evaporator. It was re-crystallized by using distilled $CHCl_3$, a yellow-colored solid product that was insoluble in water and soluble in acetone and chloroform but sparingly soluble in methanol was obtained. At 117°C,the product was decomposed. The final product of organotin complex L_1R_1 is shown in Figure 7 (a).

2.2.2.1. General reaction scheme for synthesis of complex L_1R_1

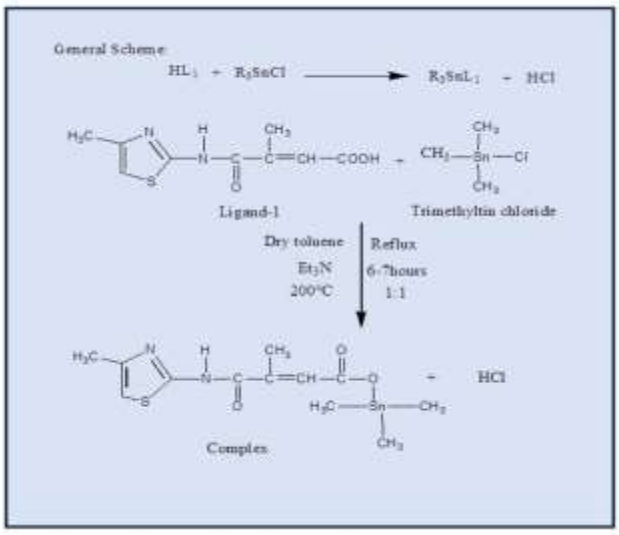

Figure 2: Reaction scheme for synthesis of complex L_1R_1

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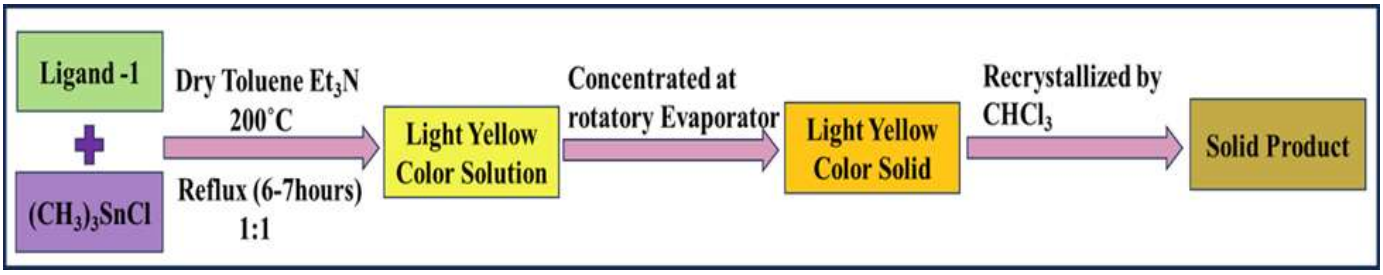

Figure 3: Schematic illustration for the synthesis of complex L_1R_1

2.3.1. Synthesis of Ligand-2 (L_2) ((E)-4-methyl-5-(4-methylthiazole-3(2H)-yl)5-oxopent-3-enoic acid)

0.44 mmoles of 3,4-dichloroaniline and phthalic anhydride were prepared in a round-bottom flask with glacial acetic acid by stirring for about 12-hour period at room temperature. The precipitates were observed, filtered, washed with ice-cold distilled water and then allowed to dry at room temperature. TLC verified the product formation[29].

Reaction scheme for synthesis of Ligand-2

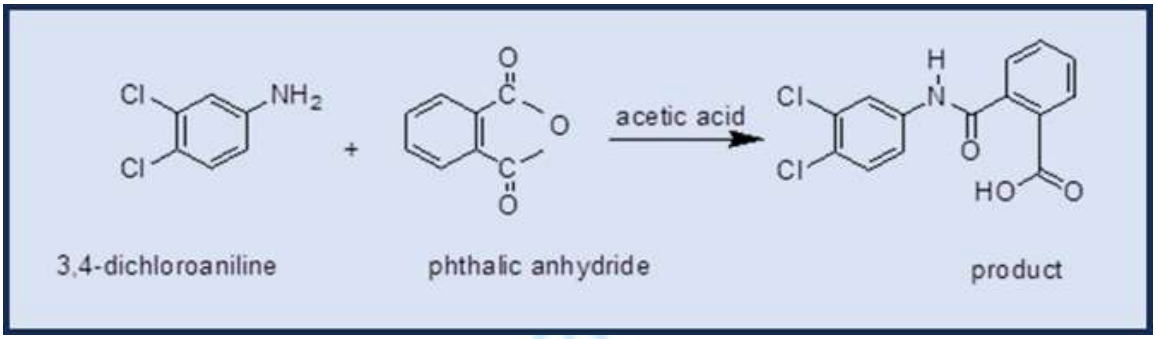

Figure 4: Reaction for Synthesis of Ligand-((E)-4-methyl-5-(4-methylthiazole-3(2H)-yl)5-oxopent-3-enoic acid)

2.3.2. Synthesis of complex L_2R_3

150 ml of dried toluene,0.3 g of ligand and 0.27 ml of bis-tributyltin oxide were added in a round bottom flask assisted with magnetic stirrer and condenser. Ligand-metal ratio was 1:2 and average 8-10 hours were given for refluxing. A light-yellow color sticky product was obtained after the solution was filtered and the solvent was removed using a rotary evaporator. It was re-crystallized by using $CHCl_3$ and the final gummy product was obtained which is soluble in acetone, chloroform, and methanol but insoluble in water. The final product of organotin complex L_2R_3 is shown in Figure 7 (b).

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2.3.2.1General reaction mechanism for synthesis of complex L_2R_3

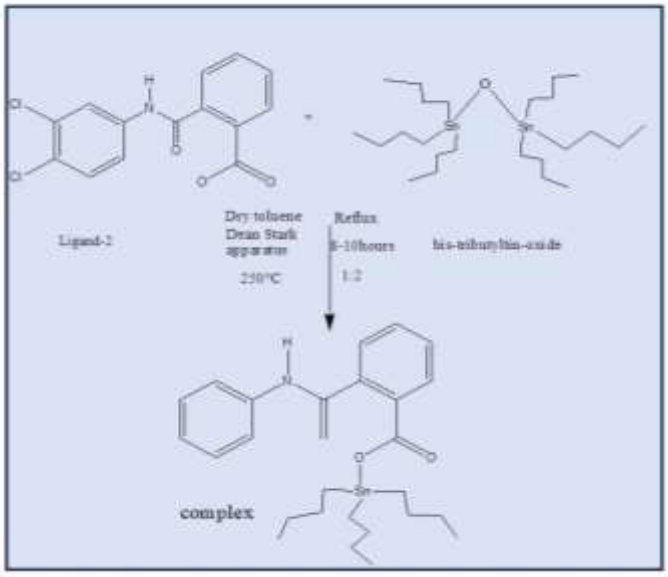

Figure 5: Reaction mechanism for synthesis of complex L_2R_3

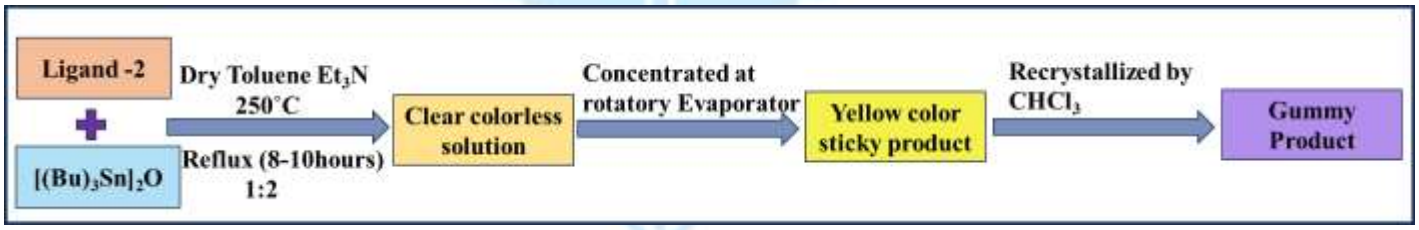

Figure 6: Schematic illustration for the synthesis of complex L_2R_3

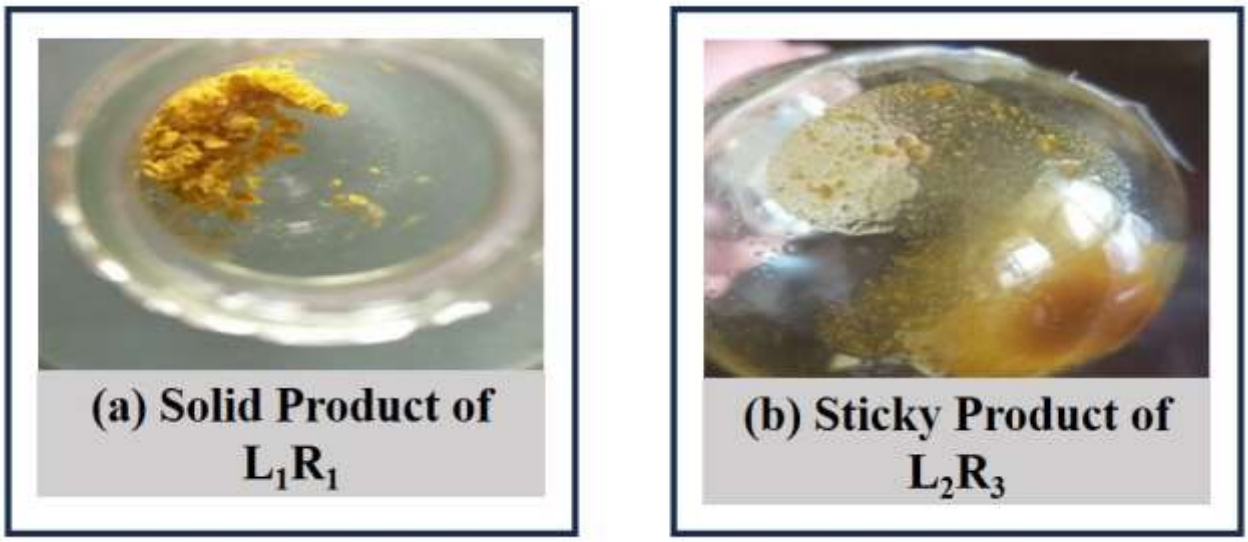

Figure 7: Final products of Organotin (IV) complexes (a) L_1R_1 (b) L_2R_3

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2.3. Characterization

A Perkin Elmer spectrophotometer Model-577 equipped with a KBr disc was used to characterize the ligands using an infrared spectrophotometer. The ligand's infrared spectrum showed all of the peaks in accordance with the published literature.

2.4. Antibacterial activity test by Diffusion method:

The well-known Agar disc diffusion method is frequently employed to evaluate the antibacterial activity of plants. The process of occulting the agar surface involves covering the entire surface with a volume of microbial occulum. Agar powder was dissolved and used to make nutrient agar medium in distilled water. To achieve sterility, the medium was autoclaved. After autoclaving, media was poured onto petri dishes. Disc cutter was used to cut 6 mm discs out of Whattman paper no. 1. After the bacterial suspension from aliquots was uniformly distributed, the test compound-impregnated discs were placed on the agar surface under aseptic conditions. Using a sterile glass rod spreader, the remaining 0.2 milliliter was evenly separated after the 1 milliliter aliquots were centrifuged for 5 minutes at 6000 rpm and the supernatant was removed. Ampicillin solution (1 mg/ml) was utilized as a control on discs that had been washed in diluted DMSO solutions. In order to get reliable growth of bacteria in an upside-down position, the plates were incubated for a duration of 24 to 48 hours at an ideal temperature of 37 °C. After incubation, the zone of inhibition was measured and noted in millimeters. The compound's antibacterial activity is indicated by the amount of space surrounding the disc (zone of inhibition). Compounds that exhibited no zone of inhibition were considered ineffective against the bacterial strains that were administered. Since serial dilution was used to measure the minimum inhibitory concentration (MIC), it is commonly understood that a broad zone of inhibition indicates a modest minimum inhibitory concentration[30, 31].

3. Results and Discussions

3.1. FTIR

By using FT-IR spectroscopy, the various functional groups found in the ligands and complexes were identified. Infrared frequencies of Organotin (IV) complexes with Ligand-1 and Ligand-2 are shown in table 1. Organotin (IV) complex L_1R_1 showed absence of broad band of hydroxyl group which demonstrated deprotonation of carboxylate group of ligand and appearance of new bands for Sn-C and Sn-O bond absent in ligand confirmed coordination of ligand to tin center and phenomena of complexation. Some peaks observed at 900-700cm^{-1} region while in Organotin (IV) complex L_2R_3 Two medium intensity peaks with broad bands at 1623cm^{-1} and 1329$cm^{-1}$are observed due to C=C stretching and –OH bending of carboxylic acid[32].

Table 1.IR Spectral Analysis of Organotin (IV) complex with Ligand-1 and Ligand-2

Compound code	Δv $(COO)_{asym}$	Δv $(COO)_{sym}$	Δv	v(Sn-C)	v(Sn-O)	v(NH)	C=O
L_1R_1	1630.00	1474.01	155.99	583	440	2983.70	1725
L_2R_3	1623.42	1477.31	146	581	501	2955.32	1716

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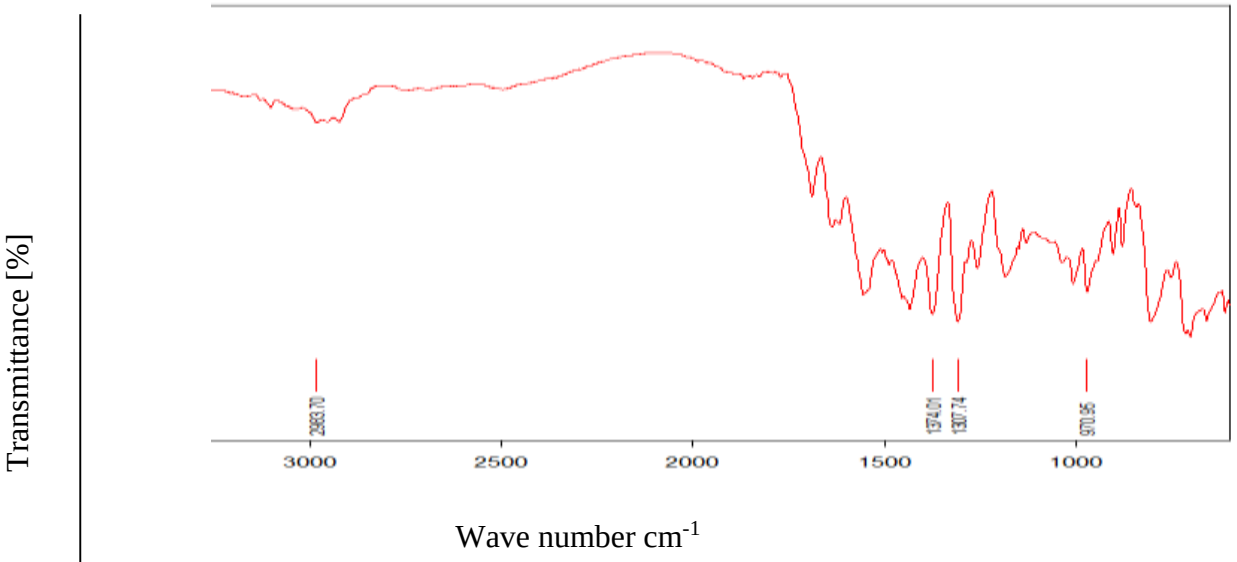

Figure 8 :FTIR Result of L_1R_1

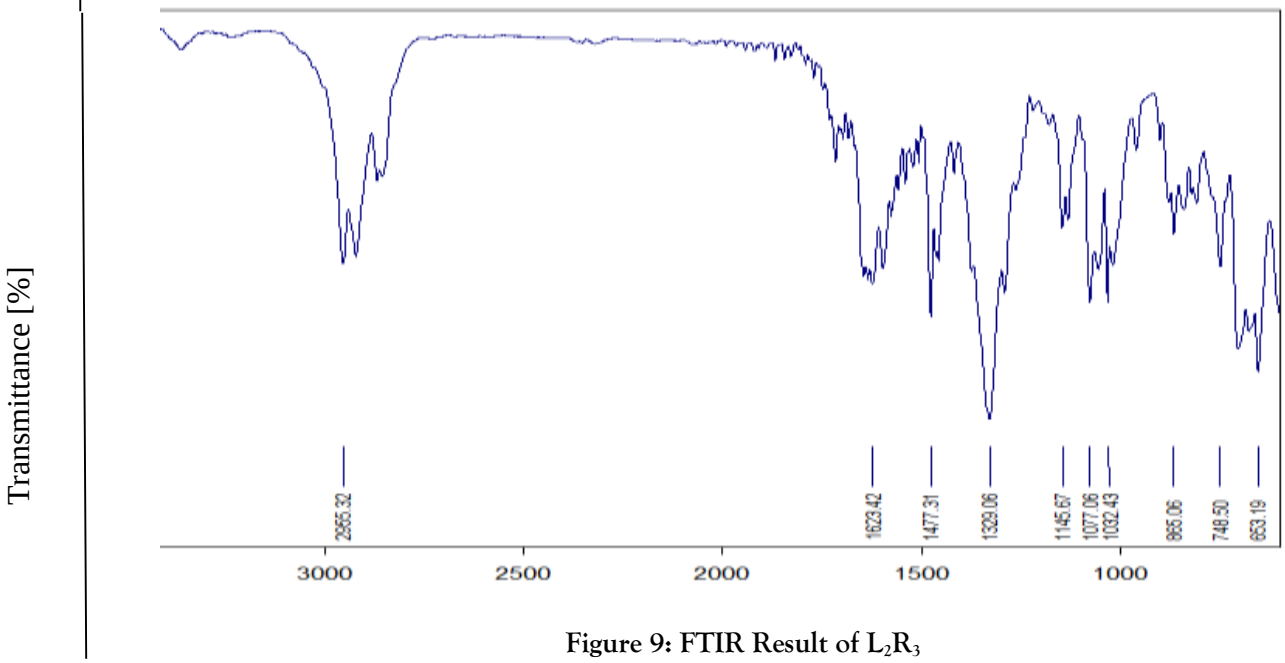

Figure 9: FTIR Result of L_2R_3

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3.2. Antibacterial Activity

Ligand-1, Ligand-2 and their organotin (IV) complexes were tested; their antibacterial activity is shown in figure 10 and table 2 against two bacterial strains staphylococcus aureus and pseudomonas aeruginosa. L_1R_1 showed maximum antibacterial activity with 12mm and 16mm zone of inhibition against pseudomonas aeruginosa and staphylococcus aureus respectively while L_2R_3 was good antibacterial agent with 8mm inhibition zone against both pathogenic strains.Results indicated that ligand-2 was more potent and active than ligand-1, and that the organotin (IV) complexes that formed with the corresponding ligand were likewise more potent and active than those prepared with ligand-1 and its complexes.

Figure 10: Antibacterial activity of di and tri organotin complexes

Table 2. Antibacterial activity of di and Tri organotin derivatives of carboxylic acids against Pseudomonas aeruginosa and Staphylococcus aureus

Sr.no	Comp. code	Pseudomonas aeruginosa		Staphylococcus aureus	
		100µg	200µg	100µg	200µg
1	L_1	1	~	~	~
2	L_1R_1	12	~	16	~
3	L_2	12	~	13.5	~
4	L_2R_3	08	10	08	10
5	Penicillin (10µg)	24		~	
6	Amoxicillin(30µg)	17		~	

All the zones of inhibition were measured in millimeters (mm)

3.3 Comparison between past and present work

Antibacterial Activity data of Organotin (IV) derivatives	Pseudomonas aeruginosa Zone of inhibition (mm)	Staphylococcus aureus Zone of inhibition (mm)	References
Antibacterial activity data of organotin(IV)	10	10	[33]

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derivatives of (E)-3-(4-methoxyphenyl)-2-(4-chlorophenyl-2-propenoic acid			
Antibacterial Activity of Organotin(IV) Derivatives of (*E*)-3-(4-Hydroxy-3-methoxyphenyl)-2-phenylpropenoic Acid	10	5	[34]
Antibacterial activity of di and Tri organotin derivatives of carboxylic acids (L_1R_1)	12	16	Present work
Antibacterial activity of di and Tri organotin derivatives of carboxylic acids (L_2R_3)	8	8	Present work

Conclusion

Organotin (IV) complexes of ligands i.e. -2-((3,4-dichlorophenylcarbamoyl)benzoic acid and (E)-4-methyl-5-(4-methylthiazole-3(2H)-yl)5-oxopent-3-enoic acid were synthesized in different stichometric ratios (1:1 and 1:2) by refluxing for six hours using dry toulene as solvent and triethylamine as base.Organotin (IV) complexes were characterized by using FT-IR spectroscopy.The various functional groups found in the ligands and complexes were identified. All organotin (IV) complexes showed deprotonation of carboxylate group and existence of C-Sn-O bond ascertaining complexation phenomena. Multiple peaks of distinct sharp and medium intensity bands at different frequencies were appeared and confirmed organotin (IV) compounds coordination with ligand. Using Penicilin and Amoxicilin as references, ligands and organotin (IV) complexes were investigated and their antibacterial efficacy against two pathogenic strains (Pseudomonas aureginosa and Staphylococcus aureus) was evaluated. Ligand-2 was more active than ligand-1 and organotin (IV) complexes with ligand-2 were more promising and biologically active than ligand-1 and its complexes.

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