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The Importance of Palladium Metal Complexes Study in Biological System

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Abstract: Schiff base metal complexes made of palladium are presented in this paper. Palladium (II) nitrate and Schiff base ligand are used to create metal complexes. The synthesized metal complexes were characterized, and analyses using several spectroscopic methods, including elemental analysis, magnetic and molar conductivity studies, produced the expected results. Metal complexes have an octahedral shape and are non-electrolytic in nature. Using the Kirby-Bauer disc diffusion method, they were evaluated in vitro against the various bacterial strains.

Keywords: Metal-Ligand complexes, Schiff base, Antibacterial activity

I. INTRODUCTION

Coordination compounds has a 150-year history. *Alfred Werner¹* described the nature and production of coordination compounds in 1893. Werner was the first person to suggest the right structures for coordination compounds with metal ions in which a transition metal atom is in the centre which is surrounded by ligands. *Jannik Bjerrum* published his findings on determining the stability constant of metal amine complexes formation in aqueous solution in the year 1941.²

Complexes plays an essential role in the industry ranging from anti-corrosion and soil treatment agents to medicinal agents, which testify to their significance in contemporary life.³ The complex formation has played a vital role in the field of therapeutic and biological sciences.⁴ The bonds between the ligand and the metal ion are attributable to secondary metal ion valencies. The creation of Kossel, Sidgewick, Lewis, Langmuir, Fajan and others of electronic valency theory⁴ cleared the concepts of primary (oxidation state) and secondary (coordination number) valencies in complexes. In 1931, Pauling introduced the theory of valence bond theory based on the electronic structure of the central metal ion in its ground state, type of bonding, geometry (i.e. shape) and complex magnetic properties.⁵ The study of coordination compounds present in the living system is the focus of rapid development in the field of bio-inorganic chemistry.⁶ Therefore, it is not surprising that the use of coordination compounds such as metal-based drugs has recently become a very active research area.^{7,8,910}

Present study the synthesis and characterization of novel Pd (II) Metal Complexes from heterocyclic ligand 2,4dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl) imino) methyl) phenol. Moreover, the preliminary in vitro antibacterial and antifungal screening activities of the ligand arecarried out and the results are reported herein.

II. MATERIALS AND METHODOLOGY

2.1 Experimental Procedure

The salts are metal nitrates i.e. Pd $(NO_3)_2$.2H2O (Sigma- Aldrich) were purchased from Badar chemicals and used without further purification. 3,5-dichloro-2- hydroxybenzaldehyde and 5-amino-1,3,4-thiadiazole-2-thiol from same used without further purification. Distilled Ethanol used for synthesis of metal complexes and ligand diethyl ether. All the chemicals of analytical grade.

Bioactivity

Schiff Base and their metal complexes evaluated in vitro their antibacterial activity against two Gram-Positive bacteria, viz, B. Subtilis; S. aureus, two fungal strains A. niger and F. oxysporum by Kirby-Bauer disc diffusion method. The experimental value compare with standard drug value Miconazole for the Antifungal activity and Ciprofloxacin for the

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antibacterial activity.¹¹

Preparation of Bases

The ligand is prepared by reported method. The mixture of 1:1 3,5-dichloro-2- hydroxybenzaldehyde (2.1 g,0.02mol) with 5-amino-1,3,4-thiadiazole-2-thiol (2.1gm, 0.02 mol) dissolved in ethanol. Then add Few drops of glacial acetic acid was added. The resultant mixture stirred for 3-4 hrs the colored precipitate of Ligands was obtained. Then wash with Ethanol recrystallized with Ethanol and Ether then dried in air. The purity of compound was checked by TLC using Silica Gel method.¹²

Preparation of M-L Metal Complexes

The metal complexes were prepared by mixing Nitrates of Palladium solution with (50 ml) ethanolic solution of Ligand 2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl) imino)methyl)phenol in (metal: ligand) 1.5 : 4.5 ratio. The resulting mixture refluxed on water bath for 6-7 hr. A colored product obtains washed with ethanol, filtered, and recrystallized with ethanol.



Scheme 1. Schiff Base and its Metal Complex Synthesis

Products	Time(h)	Yield(%)	M.P(°C)
HS S N CI CI H20 Pd O CI CI N S SH N-N	6-7	81	>250

III. RESULTS AND DISCUSSION

Table 1: Proposed Structures of metal complexes M: Pd (II).

The ligand and its transition metal complex (Table.1) of 2,4-dichloro-6-(((5-mercapto-1,3,4- thiadiazol-2-yl) imino) methyl) phenol are stable at room temperature in solid state. The ligand is soluble in complexes have 1:2 metal to ligand stoichiometric ratio. The structure data of metal complexes as shown in the table above.

Characterization of Metal Complexes

The complex Bis(2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-l)imino)methyl)phenoxy)palladium dihydrate:Bis(2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-l)imino)methyl)phenoxy)palladium dihydrate: the color of Orange Red; M.F. $C_{18}H_{12}C_{14}N_6O_4PdS_4$; Yield :81%; M.P.>250°C

The bis(2,4-dichloro-6-(((5-mercapto-1,3,4-thiadiazol-2-yl) imino) methyl) phenoxy) palladium dehydrate Schiff base metal complex having IR data 3441 v O-H str. Water molecule that shows it does not donate electron to metal in MC.1622 v for C=N Azomethine group confirms formation of Schiff base MC it changes in MC as compare to free ligand confirms donation of electron from nitrogen of Azomethine group to metal in metal complexes. 1470 v for -C=N-N=C- str.in Thiadiazole ring,1230 v C-O Phenolic group,1020 is frequency for N-N Thiadiazole ring,740 v for C-S-C

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str.in thiadiazole ring, the newly formed bond in MC the metal to Oxygen IR frequency shows at v 552 for M-O bond for metal Nitrogen bond shows at v 450 for M-N bond 13

Molar conductance of Heterocyclic ligand were observed at room temperature at 1×10^{-3} M ethyl Acetate Solution. The studies show imperceptible molar conductance value is 12.1 ohm⁻¹cm²mol⁻¹.it is observed that all ligand was nonelectrolytic in nature. The electronic absorption spectral data of the Heterocyclic ligands is taken in ethyl acetate as a solvent. The band appearing at 231-333 is due to transition of benzene ring of the ligand. The other band due to free ligands 321-375 nm due to transition for azomethine groups and phenolic–OH. These band shifts longer due to formation of Schiff base metal complexes.¹⁴

Antimicrobial activity

The antimicrobial activity in vitro on selected two gram positive bacteria S. aureus and B. Subtlis two fungi A. niger and F. Oxysporum was carried out shown in table.2.All of the tested synthesized heterocyclic metal Complexes shows better antimicrobial activity as compare to parent ligand due to chelation increase in delocalization of π electron on chelating ring that enhance the penetration of complexes in lipid membrane and blocks the binding site enzymes of microorganism there are other factors i. e, solubility, lipophilicity/hydrophilicity, Conductivity and M-L bond length that increases the activity of complexes^{15,16,17,18,19}

IV. CONCLUSION

In this Research work, synthesis of Schiff base metal complexes was prepared and characterized. Metal complexes show better antibacterial and antifungal activity as compared to free ligands. These types of study in future is beneficial for multidrug resistance research.

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