Bio-Active Phenylacetic Acid Complexes: Synthesis, Structure
And Antimicrobial Activities

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ABSTRACT

Cu(II) complexes due to their coordination properties and their biological activity these act as good chelating agents and have high pharmacological potential. Cu (II) with phenyl acetic acid and azide have been synthesized and characterized by IR, LC-MS, TG-DTA and UV confirms the coordination of ligands by oxygens of phenyl acetic acid and nitrogens of azide ligands. Complex is screened for anti microbial activity.

KEY WORDS: Phenyl acetic acid, Antimicrobial Activity.

1. INTRODUCTION

Coordination of metal with organic compounds causes drastic change in the properties of metal and ligand. Phenyl acetic acid and its derivatives are of much interest because of their biological activities. Phenyl acetic acid a type of plant hormone, and an active auxin molecule which plays a vital role in coordination of many growth and behavioural processes in the life cycle of plants. They have antibacterial activity against micro organisms. Pseudo halide ions like azides, thio cyanates, isocyanates are versatile ligands that can bind divalent metal ions (Cu2+, Mn2+, Co2+ and Ni2+) in a variety of ways of connecting transition metals in the solid state. Coordination of organic compounds with metal causes drastic change in the biological property of the ligand and also the metal moiety.

2. MATERIAL AND METHODS

IR spectra are obtained with a Shimadzu IR Prestige 21 FT-IR spectrophotometer. Electronic spectra are recorded on LABINDIA UV3000 UV/VIS spectrophotometer. LC-MS spectra are recorded on AGILANT QQQ (ESI-MS). Mass spectrometer. TG-DSC spectra are obtained using SDT Q600 V20.9 BUILD 20.

Synthesis of [Cu(PAA)2(N3)2] (1): An aqueous (5 ml) solution of Copper perchlorate hexahydrate (0.185g, 0.5 mmol) is added to an methanolic solution (10ml) of Phenyl acetic acid (0.068g, 0.5 mmol) under stirring conditions at 60°C, blue solution is formed and then aqueous solution (5 ml) of NaN3 (0.03 g, 1.0 mmol) is added which turned to parrot green solution. After constant stirring at 60°C temperature for 30 minutes, the solution turned to greenish blue. The solution is filtered off, greenish blue precipitate is formed. The precipitate was washed with methanol to remove uncoordinated ligands. Yield is 0.168 g (59.4%). Anal.expt C26H32CuN6O6, Mol.Wt. 580.05, C 53.84, H 4.17, N 14.49 (observed) C 53.62, H 4.12, N 14.20 (calculated). Important IR bands (KBR disc cm⁻¹) 3569, 2145, 1634, 1298 cm⁻¹. Mass Peak (m/z): 416, 485, 551, 617.

IR Spectrum of [Cu(PAA)2(N3)2]: The Infrared spectrum exhibited bands in regions 3344, 3450 cm⁻¹ due to C=O of the free ligand. These bands are shifted to higher frequency 3569 cm⁻¹ on complexation with Cu(II). The C=O stretching in the infrared spectrum of Phenylacetic acid is assigned to the very intense infrared band observed at 1634 cm⁻¹ another intense band observed at 660 cm⁻¹ in the IR spectrum of Phenylacetic acid is assigned to a ring stretching deformation. However the νas(N3) modes appeared as strong peaks at 2051 cm⁻¹ respectively. The shift to higher frequencies of νas(N3) band at 2145 cm⁻¹ indicates the end-on bridging mode of azide. In addition, the νe(N3) modes appeared as a weak band at 1298cm⁻¹ also indicates the terminal nature of azido group. Important peaks reported in table 1.

| Complex | ν C=O | νas(N3) | Terminalazalido group |
|---------|------|---------|---------------------|
|         | 3344, 3450 cm⁻¹ | 2051 cm⁻¹ | 1298 cm⁻¹ |

LC-MS Spectrum of [Cu (PAA)2(N3)2]: Peak at 416 (m/z) is complex bound to two Phenylacetic acid and two azide ions refer to [Cu(PAA)2(N3)2]. Peak at 485 (m/z) at complex bound to fragments of two Phenylacetic acid and two azideions. Peak at 551 (m/z) is complex bounded to three Phenylacetic acid and two azide ions refer to [Cu(PAA)2(N3)2]. Peak at 617(m/z) corresponds to Cu bound to fragment of four PAA fragment and azide ions.

Electronic Spectrum of [Cu (PAA)2(N3)2]: The UV-VIS spectrum of the metal complexes is recorded in DMSO solution in the wavelength range 200–800 nm. The UV-VIS spectrum of Cu(II) complex displays a broad band at 360 nm attributed to d-d transition, which is compatible with complexes having square pyramidal structure. Important absorption band reported in table 2.

| Complex | Absorbance | μ cm⁻¹ | Assignment |
|---------|------------|--------|------------|
|         | 360        | 320    | d-d        |

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Thermo gravimetric Analysis of [Cu(PAA)\textsubscript{3}(N\textsubscript{3})\textsubscript{2}]: The thermal decomposition of the complex [Cu(PAA)\textsubscript{3}(N\textsubscript{3})\textsubscript{2}] takes place in two stages Cu(II) complex exhibits thermal stability up to 200\degree C, which confirms that this complex is free from any types of water molecules. In the first stage loss of three PAA ligands from the complex takes place in between 50-220\degree C with mass loss of 57.2\% (obs 57.9\%). This appeared as an exothermic peak in the DTA curve within the temperature range 200-250\degree C. In the second stage which occurs in the temperature range 280-400\degree C corresponds to the decomposition of azide ligand takes place with mass loss is recorded as 10.09\% (obs 10.63\%). This appeared as an exothermic peak within the temperature range maximum rate of mass loss is indicated by DTA peak at 280-400\degree C. Above this temperature, a weight loss has been occurred upto 500\degree C. This corresponds to Copper Oxide formation.

TG-DTA graphs mentioned in Figs.4, 5.

Proposed Structure of [Cu(PAA)\textsubscript{3}(N\textsubscript{3})\textsubscript{2}]: In the title complex [Cu(PAA)\textsubscript{3}(N\textsubscript{3})\textsubscript{2}] Cu\textsuperscript{2+} was coordinated by three oxygens from three phenylacetic acid ligands, two N atoms from the azide ligands. Hence Cu\textsuperscript{2+} in the complex adopt perfect square pyramidal structure shows Fig.6.

Antimicrobial Screening of [Cu(PAA)\textsubscript{3}(N\textsubscript{3})\textsubscript{2}]: The complex is screened in vitro for Antibacterial activity against E.coli, S.aureus and Antifungal activity against R.Oligospores by disc diffusion method. The in vitro Antimicrobial properties of the complex is tested against these gram-positive and gram- negative bacteria- S. aureus, E.coli, P.aeruginosa and fungi like R.Oligospores, complex presents very good results of Antibacterial activity and no Antifungal activity. The diameters of the inhibition zone are 3 and 2.5 mm for respectively for S.aureus & E.coli while no inhibition zone is found for R.Oligospore. The Antimicrobial activity of all the complexes are listed in table.3 and zone of inhibitions showed in figs.7,8.
3. RESULTS AND DISCUSSIONS

Transition metal complex of copper involving phenyl acetic acid as primary ligand and pseudo halide as secondary ligand is synthesized by self assembled method which were analysed by means of IR, LC-MS, UV and Thermo gravimetric analysis. The IR spectra show that Phenylacetic acid act as unidentate ligand coordinating with carboxylate ion. Electronic spectra confirm square pyramid geometry. The metal complex was screened for antimicrobial activities and evaluated against three different bacteria (E. coli, S. aureus & P. aeruginosa) and also fungi (R.Oligospores) and the results showed that complex exhibit good antibacterial activity in comparison with standard drug streptomycin and totally inactive against fungi. Hence this new Bio Active Phenylacetic Acid complex endowed with potent antimicrobial activity which can be as drugs on further study.

4. CONCLUSION

In this article we are reported the synthesis of copper,Phenyl acetic acid Azide complex and their antimicrobial studies. The results for complex revealed that they are good in microbial activity.

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