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Synthesis,Characterization And In-Vitro Antimicrobial Studies Of Cu(Ii)Complex With 1-[5-(1,3-Benzodioxol-5-Yl)-1-Oxo-2,4-Pentadienyl]Piperidine Derived From Black Pepper

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ABSTRACT : *Piperine is used in many different ways to influence our lives.The piperine in that spicy food has a habit of stimulating perspiration, which in effect causes a cooling of the body. Thus very helpful in the south where summer, temperatures can reach into the 100's.While piperine may sound like a pleasant spice, it can also be a deadly killer. Piperine can be found in most insecticides, particularly those that killed the common housefly. But perhaps piperine's most important use is in the medical field. For thousand of years people have been using piperine to cure many small medical ailment.Instead of piperine,piperine complex has wide biological application and also it act as more bioactive complex.This paper discussed the,Isolation of (piperine) 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine compound from pepper using suitable solvent.Synthesis of Cu(II) complex with 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine as a ligand.Characterisation of prepared complex using UV, FT-IR, H^1-NMR Spectroscopy,Measurements molar conductance of the above ligand and complex and investigation of anti-microbial activity of Cu(II) complex by disc diffusion method.*

KEY WORDS*:Piperine,Spectroscopy,Molar conductance,Anti-microbial activity.*

I. INTRODUCTION

Medicinal plants are important substance for the study of their traditional uses through the verification of pharmacological effects and can be natural composite sources that act as new anti-infectious, anti-oxidant(*chien-chang et al.,1993*).In order to find out new sources of drugs, a number of medicinal plants have been screened for wide range of biological activities. About 3000 materials from 2,764 plant species have been screened for their pharmacological and chemotherapeutic properties (*Anon, 1988*). Traditionally used medicinal plants produce a veriety of compounds of known therapeutic properties(*Lyengar,1976; Harborne, 1989; Chopra et al.,1992*). Plants used in ethno medicine for the production of bioactive compounds are used and rationalize the use of these medicinal plants in health care (*Morales et al.,2008*). Most of their properties are due to secondary metabolites produced by medicinal plants. Transition metal complexes derived from herbal medicines have been among the most widely studied coordination compounds in recent years (*farrel et al., 1984; Hondrellist et al.,1988;Bontchev et al.,2001*), since they are becoming increasingly important as biochemical (*Apostolova et al., 1998*), analytical (*Golcu et al., 2001*) and antimicrobial (*Tumer,2000*).Dried ground pepper has been used since antiquity for both its flavor and as a medicine. Black pepper is the world's most traded spice. It is one of the most common spices added to European cuisine and its descendants. The spiciness of black pepper is due to the chemical piperine.This paper deals about synthesis,characterisation and antimicrobial studies of Copper(II)complexes of 1-[5-(1,3-Benzodioxol-5-yl)-1-oxo-2,4-pentadienyl] piperidine derived from black pepper.In piperine compound,active sites (N,O)donate electrons to copper metal to form complex which was characterised by UV,FT-IR,NMR studies.

II. EXPERIMENTAL SECTIONS

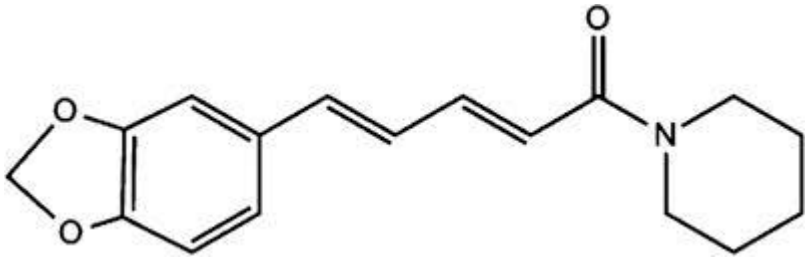
SYNTHESIS OF of 1-[5-(1,3-Benzodioxol-5-yl)-1-oxo-2,4-pentadienyl] piperidine

EXTRACTION : About 10gm of pure ground pepper was taken in 100ml Round bottom flask and added 20ml of Methylene dichloride with a magnetic stirrer. Attached a water condenser to the top of the flask and allowed water to run through it to condense the methylene dichloride vapours. While refluxing the solution for 20mins. After cooling the flask, used vaccum filtration with buchner funnel and filter paper to filtered out the pepper grounds. Washed the grounds with 10ml Methylene dichloride and saved two drops of the filtrate for TLC analysis.

ISOLATION : Transfered the filtrate to a 50ml RB flask and using a sand bath, removed the methylene dichloride until a dark brown oil is left. Cooled the oil in an ice bath and added 6ml of cold ether. After stirring for 5mins, removed the solvent again via sand bath heating. Cool the oil in an ice bath and added 6ml of cold ether. Allow the flask to sit for 15mins in an ice bath with an occasional stirring. 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine should precipitated out. Using the Buchner funnel, vaccum filter the yellow 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine crystal washed them with cold ether (2×4 ml).

III. PURIFICATION :

To crystallize, place the 1,3-benzodioxol-5-yl-1-oxo-2,4-pentadienyl in a test tube and dissolve it in 5ml of hot 3:2 acetone : hexane solution. stand for 15mins at room temperature and then 30mins in an ice bath, vaccum filter the crystal using the Buchner funnel and washed with 4ml of cold ether. It may be necessary to get a second crop of pale yellow crystals from the mother liquor to improve yield.
The structure of the 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine are given below



Yield = 0.160gm
Melting Point = 125^0C

SYNTHESIS OF Cu(II) COMPLEX OF 1-[5-(1,3BENZODIOXOL-5-YL)-1-OXO-2,4-PENTADIENYL]PIPERIDINE

About 0.285gm of 1-[5-(1,3-Benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine was taken in the 400ml beaker. Then it was dissolved with 15ml of methanol . About 0.121gm of the metal solution was added to the beaker. It was stirred with magnetic stirrer for 4hours, which was covered with filter paper, After the complete evaporation of methanol, the green crystal was appeared.
yield of the copper complex = 0.282gm
Melting point = 146^0C

FOURIER – TRANSFORMS INFRARED SPECTRASCOPIC STUDIES

Infrared spectroscopy gives information on molecular vibrations or more precisely on transitions between vibrational and rotational energy level in molecules.
The SCHMADZHU Fourier Transform infrared spectrometer was used to record the FT-IR spectra 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine investigated the spectrum which was recorded in the range from 400-4000 cm^{-1}.An Infrared spectrum is obtained when the frequency of molecule vibration correspond to the frequency of the infrared radiation absorbed.

NUCLEAR MAGNETIC RESONANCE SPECTRASCOPIC STUDIES : The H^1-NMR spectra for the samples were recorded in a Bruker 300MHZ NMR spectrometer using DMSO as the solvent. Two properties of nuclear particles pertinent to an understanding of Nuclear Magnetic Resonance Spectrascopy are the net spin associated with the protons and neutrons and the distribution of positive charge.

ULTRA-VIOLET SPECTROSCOPIC STUDIES : Ultra Violet absorption spectroscopy deals with the measurement of energy absorbed when electrons are promoted to higher energy levels. The ultra-violet spectrum is simply a plot of wavelength of light absorbed versus the absorption intensity absorbance or transmittance and conveniently recorded by plotting molar asborptivity (E) against wavelength (nm) since (E) values range in practice from a low as 10 to as high as 100000. It is convenient to use log ( ) as the abscissa of UV spectrum.

ANTIMICROBIAL SCREENING PROCEDURE : The anti-microbial study of the ligand and their complex were investigated using the procedure related to (Carr, et al 1972). 7gm of nutrient agar was measured and put into 250ml of sterilized H_2O. The mixture was heated for 15mins and placed in an autoclave at a temperature of 121^0C for about 24 hours. The prepared agar was poured into petri dish which was also sterilized. It was

allowed to sit in which holes were drilled by the use of sterilized cork borer. The organisms were rubbed on the surface of the petri dish containing the nutrient agar. Various concentration of complex was introduced into the hole with the use of the pipette . It was incubated for 24hours at a temperature of 37^0C. A well cleared zone around the nutrient agar indicates the antimicrobial effect of the complexes on the tested organisms.

IV. RESULT AND DISCUSSION

The functional group present in the molecule 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine and its Copper (II)complex was identified with the help of FT-IR Spectrum and NMR Spectrum and UV Spectrum.

FT-IR CHARACTERIZATION OF 1-[5-(1,3-BENZODIOXOL-5-YL)-1-OXO-2,4-PENTADIENYL]PIPERIDINE

FUNCTIONAL GROUP	**ABSORPTION BANDS (cm^{-1})**
C-H stretching vibration (Aromatic methylene dioxy compounds)	2856
C=O Sretching vibration	1703
C=C stretching vibration	1634
C-N stretching vibration	1364
C-O stretching vibration (Aromatic methylene dioxy compounds)	1252
Tri substituted benzene	1193

Table :1

The functional group present in 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine was identified with the help of the FT-IR spectrum.

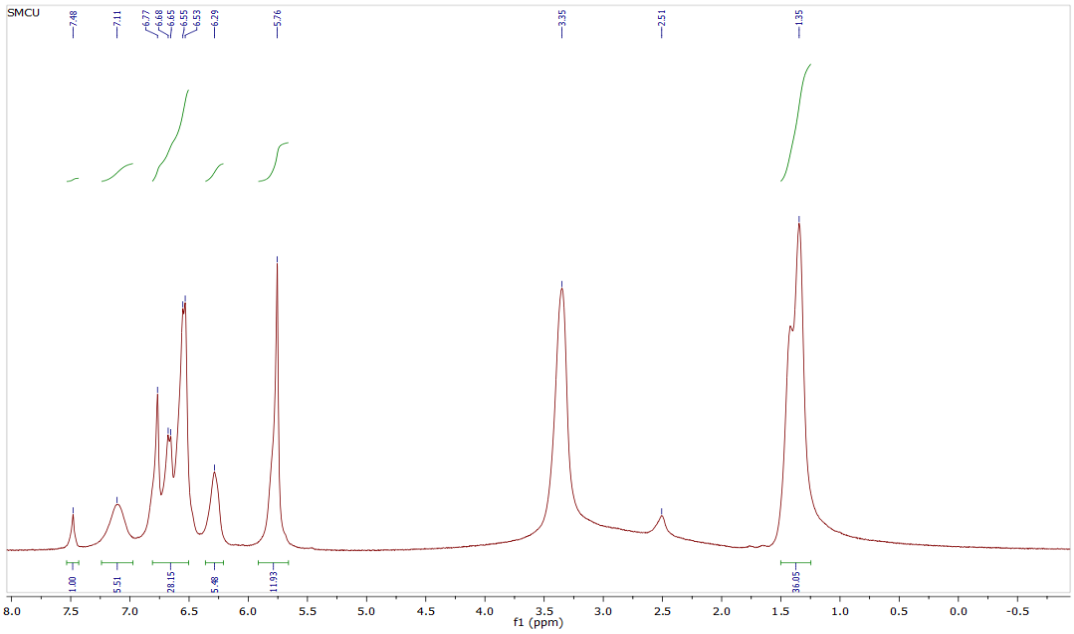
FT-IR CHARACTERIZATION OF 1-[5-(1,3-BENZODIOXOL-5-YL)-1-OXO-2,4-PENTADIENYL]PIPERIDINE Cu(II) COMPLEX

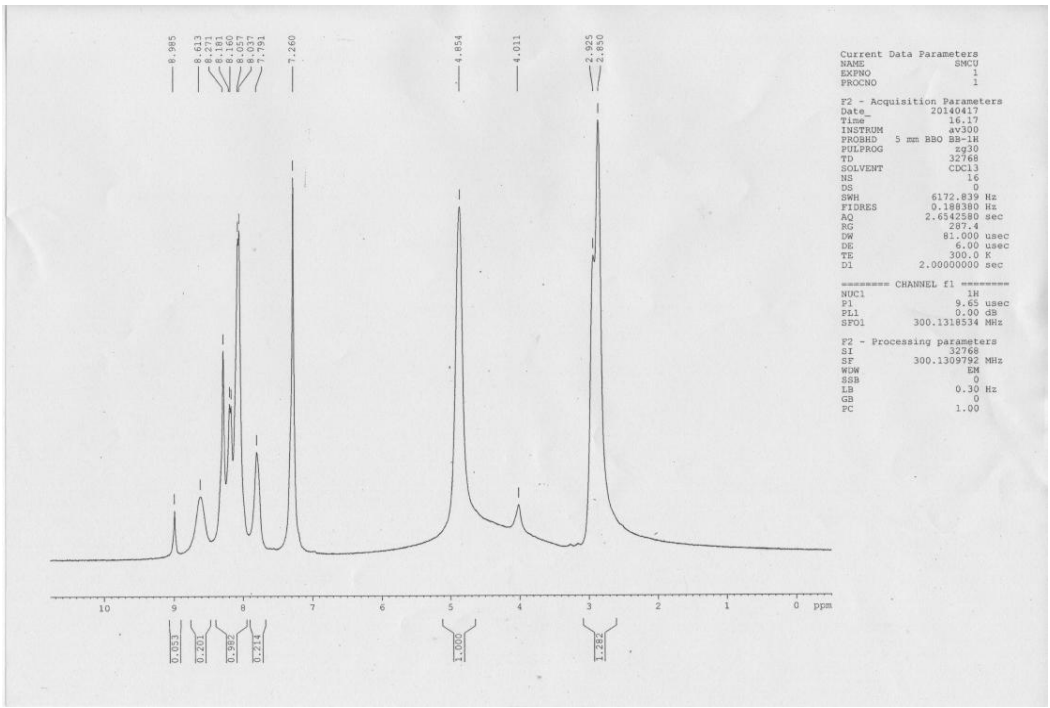
FUNCTIONAL GROUP	**ABSORPTION BANDS (cm^{-1})**
C-H stretching vibration (Aromatic methylene dioxy compounds)	2857
C=O Stretching vibration	1745
C=C stretching vibration	1631
C-N stretching vibration	1385
C-O stretching vibration (Aromatic methylene dioxy compounds)	1251
Tri substituted benzene	1192

Table :2

FT-IR absorption bands (cm^{-1}) of the synthesized complex shown higher than their parent ligand . This probably indicated the formation of complex UV Spectrum of 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine Cu(II) complex From UV spectra it is known that the absorption of UV corresponding to the copper complex was shown. The maximum absorption range is 342nm. This range is indicated copper present in this complex. From this evidence we can elucidate the geometry of the complex may be tetrahedral.

NMR SPECTRUM OF 1-[5-(1,3-BENZODIOXOL-5-YL)-1-OXO-2,4-PENTADIENYL]PIPERIDINE Cu (II) COMPLEX





From the H^1 NMR spectral data for the complex exhibits all the expected signal C_6H_6 protons appeared at the range 7.260ppm and heterocyclic ring appeared at the range 1.35ppm, aliphatic protons appeared at the range 4.854ppm and 3.35ppm, respectively.

MOLAR CONDUCTIVITY MEASUREMENT: Small quantity(0.01mol) of ligand / complex was taken in the 100ml beaker then it was dissolved in 10ml of DMSO. The conductivity cell was dipped into the solution and the conductance was measured by using systronics conductivity meter.Minimum Conductance(0.07mho cm^2 mol^{-1})showed that the complex might be non- electrolyte.

ANTI-MICROBIAL STUDIES OF 1-[5-(1,3-BENZODIOXOL-5-YL)-1-OXO-2,4-PENTADIENYL]PIPERIDINE AND ITS COMPLEX

Ligand/ Complex	ZONE OF INHIBITION (mm)		
	BACTERIA		FUNGI
	Bacillusceres	Stephylococcusaureus	Candida Albicans
SML	**R**	**R**	**12**
SMCU	**10**	**12**	**16**

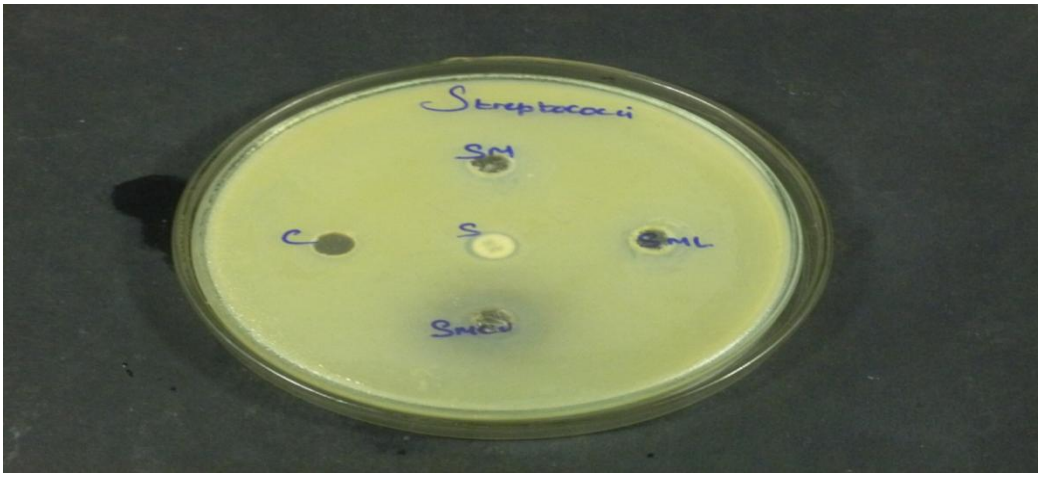
Table :3

Standard disc – Bacteria (Amikacin) Fungi (Ketokonazole)

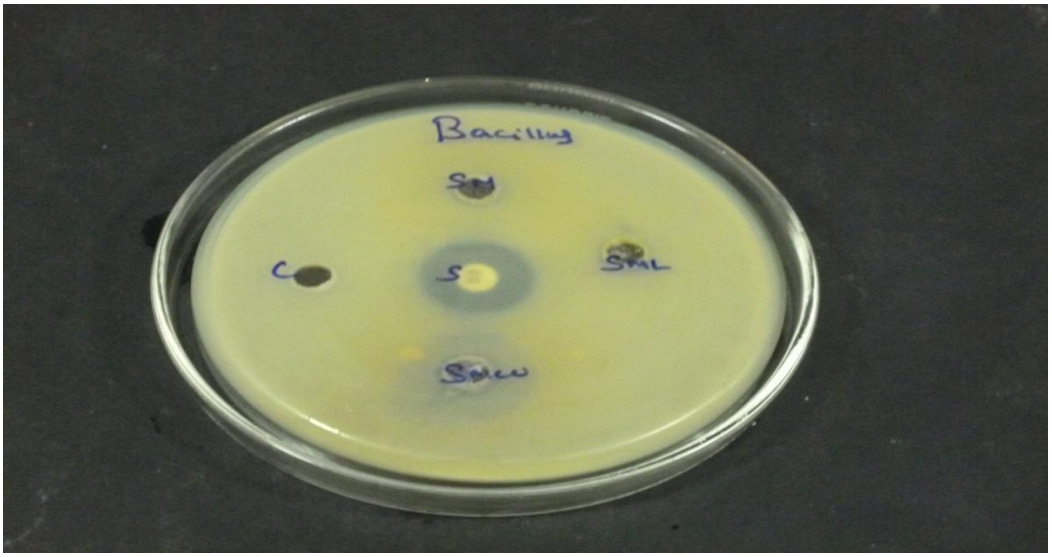
SML – Ligand , **SMCU –** Metal complex , **R –** Resistant.

BACTERIAL STUDIES

STAPHYLOCOCCUS AUREUS

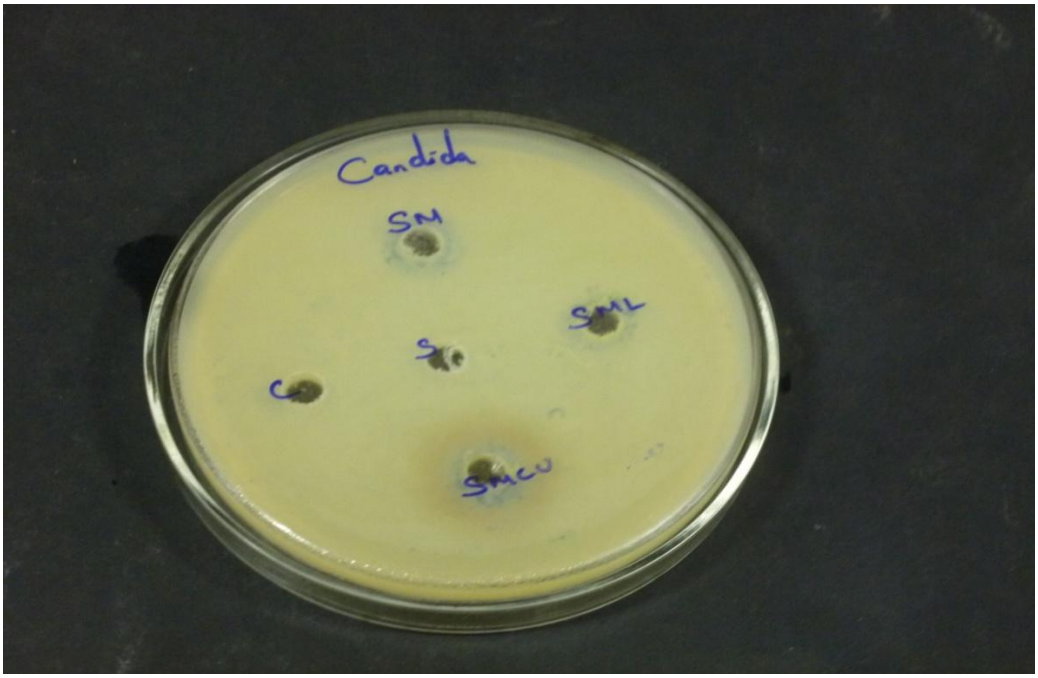


BACILLUS CEREUS



FUNGICIDAL ACTIVITY

CANDIDA ALBICANS



From **table 3**,We can infer that the complex would be more biologically active than the Parent ligand.

V. CONCLUSION

1-[5-(1,3-Benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine ligand was isolated from black pepper then it was binded with Cu(II) metal by using conventional method. The functional groups in the ligand and complex were confirmed by the Fourier – Transforms infrared spectroscopy (FT-IR), Nuclear Magnetic Resonance spectroscopy (NMR) , Ultra – Violet spectroscopy (UV) .The Infrared spectrum of both ligand and complex were presented in table1&2 in which comparisons were made between the ligand and complexes.The melting point of the synthesized complex shown higher than their parent ligand. This is probably indicates the formation of complex.The result obtained from conductivity measurement showed that the complex is non electrolyte.The antimicrobial activities were evaluated on both the ligand and the complexe by determining the measurements of the zone of inhibition using Bacilluscereus and Stephylococcusaureus, Candida Albicans which was indicated in table 3. From the result obtained, comparisons were made between the ligand and the complex, the complex had higher antimicrobial activities than their parent ligand.

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