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SYNTHESIS, SPECTRAL STUDY, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF COPPER (II) COMPLEX OF CHALCONE OF 5-NITROFURFURAL

Chemistry

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ABSTRACT

A metal complex of Cu(II) has been synthesized with newly prepared biologically active ligand. This ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and 5-Nitrofurfural. The structure of the complex has been proposed by the analytical data, conductivity measurement, magnetic moment, electronic absorption spectrum, thermal studies and XRD analysis. Analytical data confirmed 1:2 stoichiometry and the magnetic moment, TG-DTA suggests that Cu(II) complex has octahedral geometry. Presence of coordinated water molecules in Cu(II) complex is confirmed by TG-DTA studies. The conductivity data show that the complex was non electrolyte. Antimicrobial activities of complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards. The metal complex exhibit impressible potent biocidal activity than ligand.

KEYWORDS

TG-DTA, XRD study, Antimicrobial activities, Physico-chemical property, Magnetic susceptibility and Conductivity.

1. INTRODUCTION

Chalcones constitute an important group of natural products, chemically they consist of open chain flavonoid in which the two aromatic rings are joined by α, β unsaturated carbonyl system. The name chalcone is given by Kostanecki and Tambar [1]. Many complexes of chalcones are synthesized and studied in the literature[2]. It is believed that the (>CO–C=C<), moiety imparts biological characteristics to this class of compounds. Such *α*, β-unsaturated carbonyl compounds and their metal complexes possess interesting biochemical properties, such as antitumor, antioxidant, anti-fungal and antimicrobial activities [3]. The magnetic moment, TG-DTA supports the octahedral geometry of the metal complex of chalcone. All crystals of a substance possess the same elements of symmetry. The computer program, used for indexing data was powder-X. In this program all the essential features of X-ray program are presented and in addition it calculates the deviation in lattice parameter a, b, c in Å and α, β and γ in degree and minutes with better combination of h, k, l values until the final deviation is within the permissible limit of 2%. The refined values also give the volume of unit cell. [4].The X-ray powder diffractogram of the metal complexes were used for the structural characterization and determination of lattice dimensions.

2. MATERIALS AND METHODS

2.1 Synthesis of chalcone of 5-Nitrofurfural

The reagents used for preparation of chalcone of 5-Nitrofurfural are of A.R. grade. Concentrated Sulphuric acid (1.4 mL) was added dropwise to 5-Nitrofurfural (0.01) dissolved in glacial acetic acid (30mL) then 2,6-dihydroxy acetophenone (0.01 mol) was added and the reaction mixture was stirred for 10 hours at room temperature. The progress of the reaction was monitored by TL, ice cold water (100 mL) was added and the precipitate was collected by filtration and washed carefully with water and cold ethanol. The pure product was obtained by recrystallization in anhydrous ethanol [5].

2.2 Synthesis of metal complex

The solution of 0.02 mole of chalcone of 5-Nitrofurfural was taken in round bottom flask containing 30 ml of anhydrous methanolic solution and boiled for10 minutes. A hot solution of 0.01 mole, of Copper Sulphate in 20ml of methanol was added drop wise to the solution of the chalcone of 5-Nitrofurfural. To this reaction mixture, 10% alcoholic ammonia was added up to slightly alkaline pH. The complex was precipitated at 8 pH range. The pH 8-10 range was definite for these complexes [6].

The content was stirred on magnetic stirrer for one hour. The solid metal complex separated out and washed with methanol three to four times. Dried in vacuum desiccators over anhydrous granular calcium chloride. The melting point/decomposition temperature of the complex was determined by Thiele's melting apparatus. The reactions of formation of Cu(II) complex is shown in figure (1).

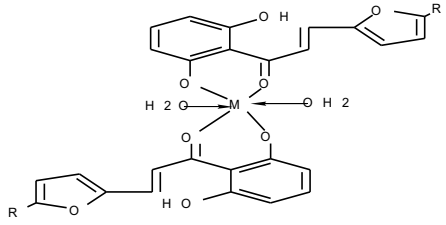

Fig.(1): Metal complex of Copper (II) with chalcone of 5-Nitrofurfural R= -NO2, M= Cu(II)

3. RESULTS AND DISCUSSION

3.1 Physical parameters

Metal complex of Copper(II) with chalcone of 5-Nitrofurfural was reddish brown in color. The complex was precipitated at 8 pH range, having Melting point 290^{0}C. The complex is insoluble in water and soluble in DMSO, DMF [7].

3.2 CHO analysis

The carbon, hydrogen, oxygen, and copper metals percentage in Cu(II) complex of chalcone measured at SAIF Cochin, Kerala. The calculated and measured values of CHO analysis are matching and are given in the table no.(1).

Table no. (1): CHO analysis

Metal complexes	Chemical formula	Mol. Wt.	Elemental analysis : % found (calculated)						
			C	H	N	O	S	X(Br)	M
Cu (II)complex	$[C_{26}H_{20}O_{14}N_2Cu]$	647	48.23 (48.19)	3.14 (3.11)	4.37(4.32)	34.60 (34.56)	-	-	10.00 (9.80)

3.3 Magnetic susceptibility, solution conductivity and electronic absorption spectral data

Magnetic susceptibility

The observed magnetic moment values of octahedral Cu(II) complexes fall in the range 1.8 to 2.1 B.M. These values are in good agreement with the moment reported for mononuclear high spin octahedral Cu(II) complexes by earlier workers[8].

Solution conductivity and electronic absorption spectral data

The solution conductivities of 10^{-3} M solution of metal complex in DMSO was measured on EQUIPTRONICS digital conductivity meter EQ-660 with 20 μΩ to 200 μΩ at 298K temperature. The complex was insoluble in water and soluble in DMSO, DMF. The low conductivity values in DMSO solution (10^{-3} M) which indicates non-electrolytic nature.

The Solution conductivity Cu(II) complex was (4.81), The magnetic moment of Cu(II) complex was(1.93 BM) and The electronic absorption spectra of Cu(II) complex was showed two bands at 25,906(386) $^2B_{1g} \rightarrow {}^2E_g$ and 38759 (258) cm^{-1}(nm) Charge Transfer band.

In the present investigation, Cu(II) show UV transition band in the range 24154 to 26560 cm^{-1} which is attributed to $^2B_{1g} \rightarrow {}^2E_g$ and charge transfer band observed in the range 35026-39370 cm^{-1} indicating distorted octahedral geometry around the Cu(II) ion[9-10].

3.4 Thermal analysis Cu(II) complex chalcone of 5-Nitrofurfural:
The simultaneous thermogravimetric, differential thermal analysis of Cu(II) complex chalcone of 5-Nitrofurfural was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000 at SAIF, Cochin, Kerala. The heating rate was 10˚/min and flow rate of nitrogen 50 ml/min. The reference substance used was α Al_2O_3 in platinum crucible and sample weighed in the range of 4-12 mg. The thermogram of Cu(II) complex chalcone of 5-Nitrofurfural is presented in figure(2). This curve reveals that there is presence of lattice as well as coordinated water in the complex.

The thermogravimetric analysis shows that, the first weight loss at 89.23˚C indicating the presence of lattice water with weight reduction 7.69 % (calc. wt. loss 6.50%). The second loss due to coordinated water molecule in the complex takes place at 136.93˚C. In the third step loss of 10.51% (calc. wt. loss 9.00%) to the temperature in the range 200-300˚C. This is supported by an endothermic peak in DTA at 246.98˚C due to the two -NO_2 groups in the ligand. The fourth step covering the reaction interval 350 to 370˚C with a considerable weight loss of 26.35% (calc. wt. loss 26%) supported by a sharp endothermic peak at 355.41˚C in DTA plot may be due to the decomposition of coordinated part of the complex. The fifth step covering the reaction interval 360-420˚C with 10% mass reduction corresponds to a loss of coordinated part of the complex. Beyond that residue attained almost constant weight loss of 12.29% corresponding to formation of CuO as a residue, final product.

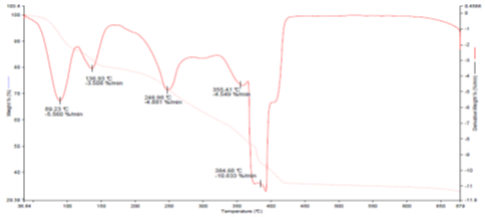

Fig. (2): TG-DTA curve of Cu(II) complex of chalcone of 5-Nitrofurfural

3.5 X-ray diffraction spectral studies of metal complex of Cu(II) complex of chalcone of 5-Nitrofurfural
The XRD spectral study has been done at SAIF, Cochin, Kerala. The standard deviation observed was within the permissible range. The observed density was 1.2919 gcm^{-3} and calculated densities was 1.3567 gcm^{-3} respectively. The Cu(II) complex of was triclinic lattice type P. For these complex lattice parameters are and a=7.9168 Å, b=8.9168 Å, c=10.8432 Å, α=82˚, β=79˚, γ= 89˚, V=738 $Å^3$ respectively, and Standard deviation was found to be 0.14%.

Unit cell data and crystal lattice parameters for Cu(II):
Unit cell data and crystal lattice parameters a (Å) = 7.9168 b (Å) = 8.9168 c (Å) = 10.8432 α = 82˚ β = 79˚ γ= 89˚ Standard deviation (%) = 0.14 Volume (V) = 738.78 $Å^3$ Density (obs.) = 1.2919 gcm^{-3} Density (cal.) = 1.3567 gcm^{-3} Z = 2 Crystal system= Triclinic Space group = P2/m

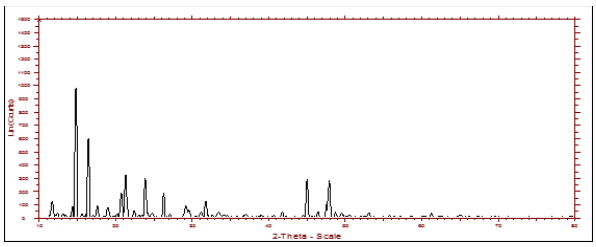

Fig. (3): X-ray diffractogram of Cu(II) complex of chalcone of 5-Nitrofurfural

3.6 Antimicrobial activity
Antimicrobial activity was assayed by cup plate agar diffusion method by measuring inhibition zones in mm [11]. In vitro antimicrobial activity of all synthesized compounds and standard have been evaluated against strains of the fungal toxicity of Cu(II) complex was studied *in vitro* against *Aspergillus niger* ATCC16404, *Saccharomyces cerevisiae* ATCC9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration.

The antibacterial activity of Cu(II) complex was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus* ATCC 6538, *Bacillus megaterium* ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739, *Salmonella typhi* ATCC9207, *Shigella boydii* ATCC 12034, *Enterobacter aerogenes* ATCC13048, *Pseudomonas aerogenosa* ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this study.

From the results of antimicrobial activity of ligands and complex it is clear that the complex shows enhanced activity than ligand[12]. The increase in antimicrobial activity is due to faster diffusion of metal complex as a whole through the cell membrane or due to the combined activity of the metal and ligand[13].

CONCLUSION
The Cu(II) complex was reddish brown in color, insoluble in most of the organic solvent but soluble in organic solvent. The stoichiometry ratio of the metal complex obtained has been found to be 1:2.Solution conductivity of this metal complex reveals nonelectrolytic nature. The electronic spectral data, magnetic moment, TG-DTA suggests that Cu(II) has Octahedral geometry. The CHO analysis gives C, H, and O percentage in the metal complex. The XRD parameters shows that the structure of Cu (II) is triclinic and has space group = P2/m.

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REFERENCES
[1] S.V.Kostanecki, and Tambar, J.Chem.Ber., 32:1921(1899).
[2] N.V.Damme,V.Zaliskyy,A.J.Lough,M.T. Lemaire, Polyhedron, 89, (2015) 155-159.
[3] V. N. Patange ,B. R. Arbad, Synthesis, spectral, thermal and biological studies of transition metal complexes of 4-hydroxy-3-[3-(4-hydroxyphenyl)-acryloyl]-6-methyl-2H-pyran-2-one, J. Serb. Chem. Soc. 76 (9) (2011) 1237–1246.
[4] A. J. Deshmukh, R. M. Patil, R. S. Lokhande and D. H. Ner, Synthesis, characterization and antimicrobial activity of copper (II) complexes of some 2-hydroxy-4, 5-dimethyl substituted chalcones, International Journal of Advanced Research and Review, 3(1), 2017, 27-31.
[5] Hong jin, Yucong Geng, Zhiyi Y.C, Ke Jao, Jaiping Hou. (2009). Pesticide Biochemistry and Physiology, 93133-137.
[6] Habib, S.I., Kulkarni, P.A.(2015). Synthesis and antimicrobial activity of chalcone derivatives of Ni(II) and Co(II) complexes-an in vitro evaluation, ICAIJ,10(4), [152-156].
[7] S.N. Ipper, N.K. Raut and G.K. Kakde, Physico-chemical properties of furan chalcone. Indian Journal of Applied Research. 2017; 7(6):683-684.
[8] Sirsat, S.B., Halikar, N.K. Pund, M.M. and Vartale, S.P. (April/June2012). Synthesis and biological screening of some novel hetero-aryl-chalcone and their Complexes.Research Journal of Pharmaceutical Biological and Chemical Sciences. Vol.3 issue 2, 240-248.
[9] Hong-Yun, Z., Dong-Li, C., Pei-Kun, C., De-Ji, C., Guang-Xia, C., Hong-Quan, Z., (1992). Polyhedron, 11, 2313.
[10] Reddy, Vidyavati. Patil, N. and Angadi S.D. (2008 July). Synthesis, characterization and antimicrobial activity of Cu(II), Co(II) and Ni(II) complexes with O,N and S doner ligands; E-Journal of Chemistry Vol. 5, No.3, 577-583.
[11] A. L. Barry, Procedure and therotical consideration for testing antimicrobial agents in agar media, 5th Edition, William Wilkiins Baltimore (1991).
[12] S. I. Habib, S. G. Konda, M. A . Basser, P. A. Kulkarni,Asian J. Research Chem., Oct-Dec, 2(4), (2009).
[13] Sengupta, S. K., Pandey, B. K., Shrivastava, B. K., and Sharma, V. K.,(1998). Transition Metal Chem., 23, 4, 349-353.