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Metal Complexation Studies of Novel Ligand Containing 8-Hydroxyquinoline with Chalcone Group

Vishvajitsinh Raj, R. I. Patel, P. J. Vyas
Sheth M. N. Patel Science College, Patan, Gujarat, India

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ABSTRACT

The novel ligand 1-(2naphthalenyl)-3-(8-hydroxyquinolin-5-yl)prop-2-en-1-one (3) was prepared by aldol condensation of 5-formyl-8-hydroxyquinoline(1) with 2-acetyl naphthalene(2).The transition metal chelates of ligand (3) were prepared by using Cu2+, Co2+, Ni2+, Mn2+ and Zn2+ metal ions. All the ligand and its metal chelates were characterized by elemental content, IR spectroscopic, metal: ligand ratio and magnetic properties. The samples also were monitored for antifungal activities.

Keywords : Chalcone, Metal Chelates, Spectral studies and Antifungal activity.

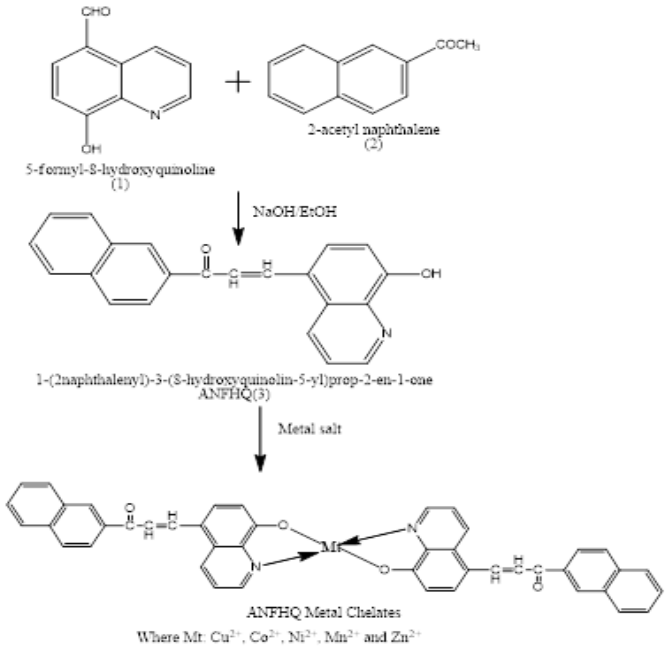
I. INTRODUCTION

In recent years, the research on synthesis and study of metal-containing compounds is become an interesting field of chemistry. [1] Organic ligands and their metal complexes played an important role in the development of coordination chemistry, they shows a wide range of applications like, physicochemical as well as biochemically.[2-4] 8-Hydroxyquinoline is well known as an analytical reagent[5,6]. It's various derivatives [7] are also useful in pharmaceuticals. One of the derivative say 5- formyl -8- hyroxyquinolinol (AHQ) can be synthesize easily and studied extensively for number of derivatives [8]. Some of the ions exchanging resins are also reported with good potentiality [9-13].

The approach to the preparation of potential biologically active compounds and in continuation of over ongoing studies on novel biologically active molecules, the product work designed the synthetic route of some novel Metal Chelates shown in scheme-1. The synthesized compounds were evaluated for its antifungal activity.

II. EXPERIMENTAL

The entire chemical used were of laboratory grade. 2-acetyl naphthalene and 5-formyl-8-hydroxyquinoline be were prepared by reported method.[14,15]



Synthesis of 1-(2naphthalenyl)-3-(8-hydroxyquinolin-5-yl) prop-2-en-1-one(ANFHQ) (3):

A mixture of 5-formyl-8-hydroxyquinoline(1) (0.025 mol) and 2-acetyl naphthalene (2) (0.025 mol) in ethanol(25 mL) were mix in a round bottom flask, 20 mL of 60% aqueous sodium hydroxide solution added drop wise. Resulting mixture was stirred for 3-4 hrs at below 10^{0}C, poured it into crushed ice and acidified with dilute HCl. The precipitate obtained was filtered and washed thrice with cold water. The resulting solid was allowed to air dry and recrystallized from ethanol. The yields was 79%, melting point was 213-214^{0}C.IR Spectral Features (cm^{-1}) shows at 3350(OH),1685(CHO), 1665(α,β-unsaturated ketones),1578(conjugated C=C), 2850, 1630,1470 (aromatic C-H), 1640, 1580, 1475 and 755(8-Hydroxy quinoline) and NMR Signals (δ ppm) at 7.32–8.20 (7H, m,Ar-H), 6.94-7.64 (2H, d, CH=CH),6.89-8.92 (m,5H, Quinoline), 5.56 (s,1H,OH).Elemental analysis of ANFHQ($C_{22}H_{15}NO_2$):Calc.%C 81.21,%N 4.30,%H 4.65; Calc.%C 81.2,%N 4.2,%H 4.6.

Synthesis of metal chelates of ANFHQ:

The metal chelates of ANFHQ with Cu^{2+}, Co^{2+}, Ni^{2+}, Mn^{2+} and Zn^{2+} metal ions were prepared in two steps. All the metal chelates were prepared in an identical procedure.

Preparation of ANFHQ solution:

ANFHQ (0.01 mol) was taken in 500 ml beaker and formic acid (85% v/v) was added up to slurry formation. To this slurry water was added till the complete dissolution of ANFHQ. It was diluted to 100 ml.

Table-1 : ANALYSIS OF ANFHQ LIGAND AND ITS METAL CHELATES

Empirical Formula	Mol. Wt. gm/mole	Yield (%)	Elemental Analysis							
			% C		% H		% N		% M	
			Cald	Found	Cald	Found	Cald	Found	Cald	Found
$C_{22}H_{15}NO_2$	325	79	81.21	81.2	4.30	4.2	4.65	4.6	-	-
$C_{44}H_{28}N_2O_4Cu^{+2}2H_2O$	715.54	75	73.79	73.7	4.47	4.4	3.91	3.9	8.88	8.8
$C_{44}H_{28}N_2O_4Co^{+2}2H_2O$	710.94	70	74.27	74.2	4.50	4.4	3.94	3.9	8.29	8.2
$C_{44}H_{28}N_2O_4Ni^{+2}2H_2O$	710.71	68	74.29	74.2	4.50	4.4	3.94	3.9	8.26	8.2
$C_{44}H_{28}N_2O_4Mn^{+2}2H_2O$	706.94	72	74.69	74.6	4.53	4.5	3.96	3.9	7.77	7.7
$C_{44}H_{28}N_2O_4Zn^{+2}2H_2O$	717.38	74	73.60	73.5	4.46	4.4	3.90	3.8	9.11	9.1

Synthesis of ANFHQ-metal-chelates:

In a solution of metal acetate (0.001 mol) in acetone: water (50:50 v/v) mixture (40 ml) the 20 ml of above mentioned ANFHQ solution (i.e. containing 0.01 M ANFHQ) was added with vigorous stirring at room temperature. The appropriate pH was adjusted by addition of sodium acetate for complete precipitation of metal chelate. The precipitates were digested on a boiling water bath. The precipitates of chelate were filtered off, washed by water and air-dried.

MEASUREMENTS

The elemental contents were determined by Thermo Finigen Flash1101 EA (Itally) the metals were determined volumetrically by Vogel's method [16]. To a 100 mg chelate sample, each 1 ml of HCl, H_2SO_4 and $HClO_4$ were added and then 1 g of $NaClO_4$ was added. The mixture was evaporated to dryness and the resulting salt was dissolved in double distilled water and diluted to the mark. From this solution the metal content was determined by titration with standard EDTA solution. Infrared spectra of the synthesized compounds were recorded on Nicolet 760 FT-IR spectrometer. NMR spectrum of ANFHQ was recorded on 60 MHz NMR spectrophotometer. Magnetic susceptibility measurement of the synthesized complexes was carried out on Gouy Balance at room temperature. Mercury tetrathiocynatocobalate (II) Hg [Co (NCS) $_4$] was used as a calibrant. The electronic spectra of complexes in solid were recorded on at room temperature. MgO was used as reference. Antifungal activity of all the samples was monitored against various fungi, following the method reported in literature[17].

RESULTS AND DISCUSSION:

The synthesis of 1-(2naphthalenyl)-3-(8-hydroxyquinolin-5-yl)prop-2-en-1-one (3) was prepared by aldol condensation of 5-formyl-8-hydroxyquinoline(1) with 2-acetyl naphthalene(2). The resulted ANFHQ ligand was an amorphous brown powder. The C,H,N contents of ANFHQ (Table-1) are consistent with the structure predicted (Scheme-1). The IR spectrum of ANFHQ comprises the important bands due to 8-quinolinol. The bands were observed at 1640, 1580, 1475, and 755 cm^{-1}.

TABLE-2 : SPECTRAL FEATRUES AND MAGNETIC MOMENT OF ANFHQ METAL CHELATES

Metal Chelates	μ_{eff} (BM)	Electronic spectral data (cm^{-1})	Transition
ANFHQ-Cu^{+2}	2.52	23453 13215	Charge transfer $^2B_{1g} \rightarrow {}^2A_{1g}$
ANFHQ-Ni^{+2}	3.72	22598 15372	$^3A_{1g} \rightarrow {}^3T_{1g}(P)$ $^3A_{1g} \rightarrow {}^3T_{1g}(F)$
ANFHQ-Co^{+2}	4.76	23735 19105 8926	$^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ $^4T_{1g}(F) \rightarrow {}^4T_{2g}$ $^4T_{1g}(F) \rightarrow {}^4T_{2g}(P)$
ANFHQ-Mn^{+2}	5.54	23237 19035 16843	$^6A_{1g} \rightarrow {}^6A_{2g}\ {}^4E_g$ $^6A_{1g} \rightarrow {}^4T_{2g}$ (4G) $^6A_{1g} \rightarrow {}^4T_{1g}$(PG)
ANFHQ-Zn^{+2}	Diamag.	-	-

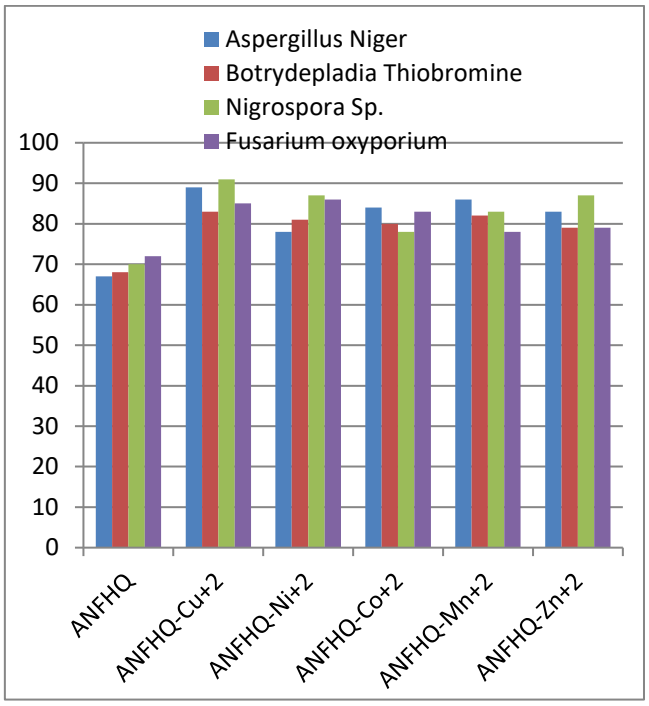
The broad band due to –OH group appeared at 3350 cm^{-1}. In this band the inflections are observed at 2970, 2980 and 2850cm^{-1}. The NMR spectrum of ANFHQ in DMSO indicates that the doublet of 2 H at 6.94-7.64

for CH=CH group. While the singlet at 5.56 δ ppm due to –OH group. The aromatic protons are appeared in multiplicity at 6.89–8.92 δ. Thus the structure of ANFHQ is confirmed as shown in Scheme-I.

The metal and C,H,N contents of metal chelates of ANFHQ (Table-I) are also consistent with the predicted structure. The results show that the metal: ligand (M:L) ratio for all divalent metal chelate is 1:2.

TABLE-3 : ANTIFUNGAL ACTIVITY OF ANFHQ LIGAND AND ITS METAL CHELATES

Sample	Zone of inhibition of fungus at 1000 ppm (%)			
	Aspergillus Niger	*Botrydepladia Thiobromine*	*Nigrospora Sp.*	*Fusarium oxyporium*
ANFHQ	67	68	70	72
ANFHQ-Cu^{+2}	89	83	91	85
ANFHQ-Zn^{+2}	78	81	87	86
ANFHQ-Ni^{+2}	84	80	78	83
ANFHQ-Co^{+2}	86	82	83	78
ANFHQ-Mn^{+2}	83	79	87	79



The infrared spectra of all the chelates are identical and suggest the formation of all the metalocyclic compound by the absence of band characteristic of free –OH group of parent ANFHQ. The other bands are almost at their respectable positions as appeared in the spectrum of parent-ANFHQ ligand. However, the band due to (M-O) band could not be detected as it may appeared below the range of instrument used. The important IR Spectral data are shown in Table-2.

Magnetic moments of metal chelates are given in Table-2. The diffuse electronic spectrum of Cu^{+2} chelates shows two broad bands around 13215 and 23453 cm^{-1}. The first band may be due to a $^2B_{1g} \rightarrow {}^1A_{1g}$ transition. While the second band may be due to charge transfer. The first band shows structures suggesting a distorted octahedral structure for the Cu^{+2} metal chelates. The higher value of the magnetic moment of the Cu^{+2} chelate supports the same. The Co^{+2} metal chelate gives rise to three absorption bands at 23735,19106 and 8926 cm^{-1}, which can be assigned $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$, $^4T_{1g}(F) \rightarrow {}^4T_{2g}$ and $^4T_{1g}(F) \rightarrow {}^4T_{2g}(P)$ transitions, respectively. These absorption bands and the μeff value indicate an octahedral configuration of the Co^{+2} metal chelate [21]. The spectrum of Mn^{+2}

polymeric chelate comprised two bands at 19035 cm^{-1} and 23237 cm^{-1}. The latter does not have a very long tail. These bands may be assigned to $^{6}A_{1g} \rightarrow {}^{4}T_{2g(G)}$ and $^{6}A_{1g} \rightarrow {}^{4}A_{2g(G)}$ transitions, respectively. The high intensity of the bands suggests that they may have some charge transfer character. The magnetic moment is found to be lower than normal range. In the absence of low temperature measuremet of magnetic moment it is difficult to attach any significance to this. As the spectrum of the metal chelate of Ni^{+2} show two distinct bands at 15372 and 22598 cm^{-1} are assigned as $^{3}A_{1g} \rightarrow {}^{3}T_{1g}(F)$ and $^{3}A_{1g} \rightarrow {}^{3}T_{1g}(P)$ transition, respectively suggested the octahedral environment for Ni^{+2} ion. The observed μeff values in the range 2.52-5.54 B.M are consistent with the above moiety [18].

The examination of antifungal activity of ANFHQ ligand and its all chelates (Table-3) reveals that the ligand is moderately toxic against fungi, while all the chelates are more toxic than ligand. Among all the chelates the Cu^{+2} chelate is more toxic against fungi.

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