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Synthesis, Characterization and Biological Activity of Metal Chelates Based On 8-Hydroxyquinoline and 1, 2, 4-Triazol

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ABSTRACT

The present study aims to synthesize a novel ligand namely, 5-((3-(trifluoromethyl)-1H-1,2,4-triazol-1-yl)methyl)-8-Hydroxyquinoline (TFMTHQ) by reacting 5-chloromethyl-8-hydroxyquinoline (CMQ) hydrochloride, a versatile derivative of 8-hydroxyquinoline with 3-trifluoromethyl-1-H-1,2,4-triazole. The co-ordinate metal chelates of the ligand TFMTHQ were prepared using divalent metal ions viz., Cu^{2+}, Co^{2+}, Ni^{2+}, Mn^{2+}, Zn^{2+} and Cd^{2+} by a simple method. The ligand TFMTHQ and its all metal chelates were duly characterized for elemental content, spectral features, M: L (metal: ligand) ratio, and magnetic moment. The results of electronic spectral studies and magnetic properties indicated octahedral geometry for all the metal chelates. All the synthesized compounds were tested for antibacterial activity against gram positive and gram negative bacteria. Antifungal activity of all the samples was tested against plant pathogens such as Aspergillus Niger (AN), Botrydepladia Thiobromine (BT), Nigrospora Sp. (NS), and Fusarium Oxyporium (FO). The results showed good antimicrobial activity of all the metal chelates.

Keywords: 8-hydroxyquinoline, 1,2,4-triazole, metal chelates, spectral studies, magnetic properties, biological activity.

I. INTRODUCTION

Due to the vicinity of –OH group to the N atom of pyridine ring 8-hydroxyquinoline, a nitrogen containing heterocyclic compound, possess a broad spectrum of applications as metal chelating and metal recognising agents [1-3].The pharmacological properties of 8- HQs as anticancer, antiproliferative, antialzheimer and antineurodegenrative agents[4-6] have been explored extensively. Also, 8-hydroxyquinoline derivatives based metal chelates have been reported as potential corrosion inhibitors, fluoroscent chemo-sensors and OLEDs [7-9]. The therapeutic applications of 8-hydroxyquinolines extend as antibacterial, antifungal, anti-

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inflammatory, anti-diabetic antioxidants, anti-tubercular and anti-HIV agents [10-14].

5-chloromethyl-8-hydroxyquinoline (CMQ) is a versatile derivative of 8-HQ that is quite simple to synthesize and stable in its hydrochloride form.CMQ derivatives have been reported as effective antimicrobial, antioxidant and anticancer agents [15-17].

1,2,4-triazole, an azaheterocyclic compound, has been a prominent scaffold possessing wide ranging biological and medicinal applications such as antimicrobial, anticancer, anti-inflammatory, antitubercular, antiviral, antiparasaitic, analgesic, antihypertensive and so on[18,19].

A large variety of compounds contain trifluoromethyl group possessing enhanced pharmaceutical applications [20, 21]. Also, it has been recently reported by Ihsan Ullah et al that triazoles containing $-CF_3$ group display increased potency as therapeutic agents [22].

Hence, in continuation with our previous work [23] it was thought to combine these two pharmaceutically effective moieties, viz.,CMQ and 3-trifluoromethyl-1-H-1,2,4-triazole in a single molecular framework followed by synthesising various metal chelates by reaction of the ligand with various divalent metal ions such as Cu^{2+}, Co^{2+}, Ni^{2+}, Mn^{2+},Zn^{2+} and Cd^{2+} in order to investigate their biological efficacy.

In the present work, the synthesis of ligand5-((3-(trifluoromethyl)-1H-1,2,4-triazol-1-yl)methyl)-8-Hydroxyquinoline (TFMTHQ) is carried out by reaction between 5-chloromethyl-8-hydoxyquinoline hydrochloride and 3-trifluoromethyl-1-H-1,2,4-triazole which in turn reacted with Cu^{2+}, Co^{2+}, Ni^{2+}, Mn^{2+},Zn^{2+} and Cd^{2+} metal salts in order to synthesize co-ordinated metal chelates.. The characterization of the ligand was done by using various spectroscopic techniques such as FTIR, ^{1}H NMR and LC-MS. Electronic spectral studies and magnetic susceptibility measurements have been performed for determination of geometry of the metal chelates. Ligand and metal chelates were further incorporated for analysing their antibacterial and antifungal activities.

II. METHODS AND MATERIAL

All the chemicals were purchased from the local market and are used without further purification.

Preparation method for Synthesis of 5-((3-(trifluoromethyl)-1H-1,2,4-triazol-1-yl)methyl)-8-Hydroxyquinoline (TFMTHQ) :

5-chloromethyl-8-hydroxyquinoline (CMQ) was prepared by reported method [24, 25].

To 5-chloromethyl-8-hydroxy quinoline (CMQ) hydrochloride (0.15 mole) in ethyl alcohol, 3-Trifluoromethyl-1H-1, 2, 4-Triazole (0.15 mole) was added. To this, 0.15 mole $NaHCO_3$ was added. The mixture was refluxed for 4 hours with occasional stirring. The reaction mixture was cooled, filtered and washed with ethyl alcohol and recrystallised from petroleum ether (b.p.60-68°C). The product yield is 72%. The melting point of the ligand TFMTHQ is 133-134° C (uncorrected).

Preparation method for synthesis of metal chelates of 5-((3-(trifluoromethyl)-1H-1,2,4-triazol-1-yl)methyl)-8-Hydroxyquinoline (TFMTHQ) :

The metal chelates of TFMTHQ with Cu^{2+}, Co^{2+}, Ni^{2+}, Mn^{2+}, $Zn^{2+}$and Cd^{2+}salts were prepared in two steps.

Step-I Preparation of TFMTHQ solution:

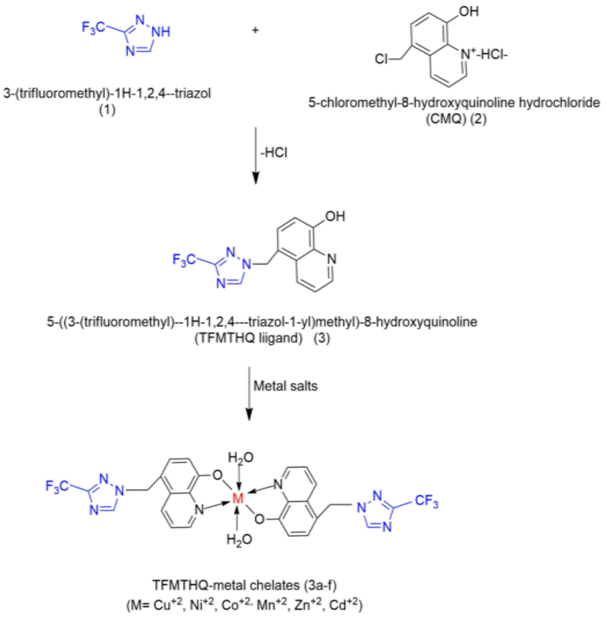
TFMTHQ (0.1 mol) was taken in 500 ml beaker and formic acid (85% v/v) was added up to slurry formation. To this slurry water was added till the complete dissolution of TFMTHQ. It was diluted to 100 ml.

Step-II Synthesis of TFMTHQ-metal-complexes:

In a solution of metal acetate (0.01 mol)in acetone: water (50:50 v/v) mixture (40 ml) the 20 ml of TFMTHQ solution (containing 0.02 M TFMTHQ) 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. The melting point of all the metal chelates was determined by open capillary method and all the chelates are found to be stable up to 250°C.

III.MEASUREMENTS

The elemental contents were determined by Thermo Finigen Flash1101 EA (Itally) and the metals of metal chelates were determined volumetrically by Vogel's method [26]. To a 100 mg chelate sample, each 1 ml of Conc. HCl, H_2SO_4 and $HClO_4$ were added and then 1 g of 0.1 N $NaClO_4$ 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 TFMTHQ was recorded on 60 MHz NMR spectrophotometer. LC-MS of ligand was taken on LC-MSD-Trap-SL_01046. Magnetic susceptibility measurement of the synthesized chelates was carried out on Gouy Balance at room temperature. Mercury tetrathio cynato cobalatate (II) $Hg[Co(NCS)_4]$ was used as a calibrant. The electronic spectra of chelates in solid were recorded at room temperature. MgO was used as reference. A fungal activities of the samples were investigated by reported methods [27,28].



(Reaction Scheme)

IV. RESULTS AND DISCUSSION

5-((3-(trifluoromethyl)-1H-1,2,4-triazol-1-yl)methyl)-8-Hydroxyquinoline (TFMTHQ)was prepared by condensation of 5-chloromethyl-8-hydroxyquinoline (CMQ) hydrochloride with 3-trifluoromethyl-1-H-1,2,4-triazole. The resulted TFMTHQ ligand was an amorphous yellow powder. The C, H, N contents of TFMTHQ (table-1) are consistent with the structure predicted (Reaction Scheme). The results show that the metal: ligand (M:L) ratio for all divalent metal chelates is 1:2.

Table 1. Physicochemical analysis of TFMTHQ ligand and metal chelates

Empirical formula	**% yield**	**Molecular weight (gm/mol)**	**Elemental analysis**				
			% C	**% H**	**% N**	**% F**	**% M**
			Calc. **Found**	**Calc.** **Found**	**Calc.** **Found**	**Calc.** **Found**	**Calc.** **Found**
$C_{13}H_9N_4OF_3$	72	294.24	53.06	3.08	19.04	19.36	-
			53.0	3.0	19.0	19.3	-
$C_{26}H_{16}N_8O_2F_6Cu^{+2}2H_2O$	74	688.03	45.52	2.64	16.33	16.61	9.26
			45.4	2.9	16.3	16.6	9.2
$C_{26}H_{16}N_8O_2F_6Ni^{+2}2H_2O$	72	681.19	45.84	2.66	16.45	16.73	8.62
			45.8	2.9	16.4	16.7	8.6
$C_{26}H_{16}N_8O_2F_6Co^{+2}2H_2O$	70	681.42	45.82	2.66	16.44	16.73	8.65
			45.8	2.9	16.4	16.7	8.6
$C_{26}H_{16}N_8O_2F_6Mn^{+2}2H_2O$	66	677.42	46.09	2.68	16.54	16.83	8.11
			46.0	2.9	16.5	16.8	8.1
$C_{26}H_{16}N_8O_2F_6Zn^{+2}2H_2O$	71	687.87	45.40	2.64	16.29	16.57	9.50
			45.3	2.9	16.2	16.5	9.5
$C_{26}H_{16}N_8O_2F_6Cd^{+2}2H_2O$	69	734.90	42.49	2.47	15.25	15.51	15.30
			42.4	2.7	15.2	15.5	15.3

A. FT-IR spectra

The FT-IR spectral frequencies for all the characteristic bonds of the ligand and all the metal chelates is represented in table-2 and are in good accordance with the structure proposed by the reaction scheme. Some important inferences could be drawn from the comparison of the IR spectrum of the ligand TFMTHQ with the spectra of the metal chelates.The involvement of phenolic oxygen in chelate formation could be inferred from the absence of -OH stretching frequency in IR spectra of chelates which is observed at 3450-2750 cm-1 for the ligand TFMTHQ. Also, a medium intensity bands present at ~675cm^{-1} suggest the M-O linkage and linkage of N of pyridine ring to the central metal ion could be inferred from the sharp bands at ~560 cm^{-1} and ~730 cm^{-1} in the IR spectra of metal chelates[29,30]. In the investigated metal chelates, the bands at around 3000-3600 cm^{-1} (very broad), 1240 ,815 and 700 cm^{-1} are assigned for stretching, bending, rocking and wagging vibration of water molecule and also the presence of rocking vibration

indicates the co-ordination of water molecule during chelate formation [31].

B. ^{1}HMR signal, δ ppm

The proton NMR signals of the ligand TFMTHQ are recorded as: 5.32 (s, 1H, -OH), 6.98–8.92 (m, 5H, Quinoline), 3.58 (s, 2H, N–CH_2-).

C. LC-MS(m/z)

The molecular ion peak for the ligand TFMTHQ is observed at 295.89(M^+).

D. Diffuse electronic spectra and magnetic properties

The data of electronic transitions and magnetic moments of metal chelates are shown in table-3. The observed μ_{eff} values in the range 2.53-5.51 B.M is of magnetic moments and reflectance spectral data of each complex co-relates with structure assigned as the octahedral geometry [32, 33].

E. Biological evaluation of TFMTHQ ligand and its metal chelates

The screening of antibacterial and antifungal activity of TFMTHQ ligand and its all complexes (table-4) reveals that the ligand is moderately toxic against bacteria and fungi, while all the chelates are more toxic than ligand. Among all the complexes the Cu^{+2} complex has shown more toxicity against bacteria and fungi.

Table 2 FT-IR spectral data of TFMTHQ ligand and metal chelates

Empirical Formula	**ν (-OH)**	**ν (C-H)**	**ν (C-F)**	**ν (C-N)**	**ν (C-O)**	**ν (8-HQ)**
$C_{13}H_9N_4OF_3$	3450-2750	3053, 2916, 2850 (st.) 1620,1448 (bend.)	750, 1296	1289	1176	1690, 1578, 1448 ,750.
$C_{26}H_{16}N_8O_2F_6Cu^{+2}2H_2O$	-	2938,2855(st.) 1594,1452(bend.)	741,1295	1231	1159	1641, 1574, 1513 , 741.
$C_{26}H_{16}N_8O_2F_6Ni^{+2}2H_2O$	-	2935,2852(st.) 1595,1454 (bend.)	750,1290	1232	1160	1638, 1570, 1510 , 750
$C_{26}H_{16}N_8O_2F_6Co^{+2}2H_2O$	-	2934,2857(st.) 1596,1450(bend.)	740,1292	1230	1156	1638, 1576, 1515, 745
$C_{26}H_{16}N_8O_2F_6Mn^{+2}2H_2O$	-	2936,2850(st.) 1590,1448(bend.)	745,1290	1235	1162	1645, 1578, 1518,740
$C_{26}H_{16}N_8O_2F_6Zn^{+2}2H_2O$	-	2936,2853(st.) 1592,1454(bend.)	752,1293	1235	1162	1641, 1570, 1512,740
$C_{26}H_{16}N_8O_2F_6Cd^{+2}2H_2O$	-	2936,2852(st.) 1596,1450(bend.)	745,1290	1235	1160	1643, 1575, 1516, 740

Table 3 Diffuse electronic spectra and magnetic properties

Metal chelates	**Magnetic moment μ_{eff} (BM)**	**Electronic spectral data (cm^{-1})**	**Transition**
TFMTHQ-$Cu^{+2}.2H_2O$	2.53	23446	Charge transfer
		15872	$^2B_{1g}\rightarrow{}^2A_{1g}$
TFMTHQ -$Ni^{+2}.2H_2O$	3.69	22584	$^3A_{1g}\rightarrow{}^3T_{1g}(P)$
		15372	$^3A_{1g}\rightarrow{}^3T_{1g}(F)$
TFMTHQ -$Co^{+2}.2H_2O$	4.64	22725	$^4T_{1g}(F)\rightarrow{}^4T_{2g}(F)$
		15263	$^4T_{1g}(F)\rightarrow{}^4T_{2g}$
		8940	$^4T_{1g}(F)\rightarrow{}^4T_{2g}(P)$
TFMTHQ -$Mn^{+2}.2H_2O$	5.51	23860	$^6A_{1g}\rightarrow{}^6A_{2g}{}^4E_g$
		18343	$^6A_{1g}\rightarrow{}^4T_{2g}$ (4G)
		16820	$^6A_{1g}\rightarrow{}^4T_{1g}$(PG)

TFMTHQ - $Zn^{+2}.2H_2O$	Diamagnetic	-	-
TFMTHQ - $Cd^{+2}.2H_2O$	Diamagnetic	-	-

Table-4 Biological evaluation of TFMTHQ ligand and its metal chelates

Ligand and its metal complexes	Zone of inhibition (in mm)				Zone of inhibition of fungus at 1000 ppm (%)			
	Gram + Ve		Gram -Ve					
	Bacillus Subtilis	Staphyloc occus aureus	Kllebsiella promioe	E.coil	AN	BT	NS	FO
TFMTHQ	13	6	9	13	63	62	60	68
TFMTHQ-Cu^{+2}	20	10	17	21	79	79	75	81
TFMTHQ-Ni^{+2}	16	7	10	13	77	74	70	77
TFMTHQ-Co^{+2}	12	8	15	15	76	76	73	79
TFMTHQ-Mn^{+2}	17	7	16	14	75	72	74	79
TFMTHQ-Zn^{+2}	14	9	19	14	73	73	75	80
TFMTHQ-Cd^{+2}	16	6	13	19	72	71	70	78

V. CONCLUSIONS

A novel ligand is synthesised by condensation of CMQ and 3-trifluoromethyl-1H-1,2,4-triazole and its metal chelates were prepared in good yield and were duly characterized. In the metal chelates, the ligand coordinates with one central metal atom at four coordination sites, with two water molecules. Structure proposed for the ligand and its metal chelates are consistent with results from elemental and spectral analysis. The geometry of the central metal ion was confirmed by electronic spectra and magnetic susceptibility measurements. All the data provide good evidence of complex formation. The complexes exhibited good antibacterial and antifungal activity.

Conflict of interest:

None declared

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