



**SYNTHESIS, CHARACTERIZATION AND ANTIFUNGAL ACTIVITY OF
COORDINATION POLYMERS BASED ON 5-CHLOROMETHYL-8-
HYDROXYQUINOLINE AND CIPROFLOXACIN DERIVATIVES**

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ABSTRACT:In the present study, 5-chloromethyl-8-hydroxyquinoline hydrochloride is reacted with ciprofloxacin to yield a novel ligand namely 1-cyclopropyl-6-fluoro-7-(4-((8-hydroxyquinolin-5-yl)methyl)piperazin-1-yl)-4-oxo-1,2,3,4-tetrahydroquinoline-3-carboxylic acid. The transition metal coordination polymers of the ligand were prepared using Cu²⁺, Ni²⁺, Co²⁺, Mn²⁺, Zn²⁺ and Cd²⁺ metal salts. The ligand and its all metal coordination polymers were duly characterized for elemental content, spectral features, M: L (metal: ligand) ratio, thermo gravimetric properties and magnetic moment. Antifungal activity of all the samples was tested against plant pathogens.

KEY WORDS: 8-hydroxyquinoline, ciprofloxacin, metal coordination polymers, thermal gravimetric analysis, antifungal activities.

INTRODUCTION:Amongst various quinoline based heterocyclic compounds, 8-hydroxyquinolines (8-HQs) have been found to display great metal coordinating ability and good metal recognition properties due to close proximity of the phenolic hydroxyl group to the heterocyclic N atomⁱ⁻ⁱⁱⁱ. The versatility of 8-HQs have attributed to their applications as OLEDs, fluorescent chemo sensors and corrosion inhibitors^{iv-vi}. Also, a wide range of medicinal properties such as antimicrobial, anticancer, anti-HIV, antineurodegenerative, anti-oxidant and anti-inflammatory have been reported for 8-hydroxyquinoline derivatives^{vii-xiii}. 5-chloromethyl-8-hydroxyquinoline (CMQ); one of the versatile derivatives of 8-HQ can easily be synthesized and is stable as its hydrochloride salt. CMQ has been found to possess anti-fungal as well as anti-cancer applications^{xiv, xv}. Ciprofloxacin (CF), a second generation broad spectrum fluoroquinolone, and many of its derivatives have shown promising activity against both Gram-positive and Gram-negative bacteria^{xvi}. Ciprofloxacin has a high oral bioavailability and a large volume of distribution. It is used for the treatment of a wide range of infections including urinary tract infections caused by susceptible bacteria^{xvii}. Also, the

metal complexes based on ciprofloxacin have been reported and some of them have displayed higher anti-bacterial activity than the parent drug ^{xviii}. Hence, it was thought of interest to accommodate CMQ and ciprofloxacin moieties in a single molecular framework and to synthesize a new ligand 1-cyclopropyl-6-fluoro-7-(4-((8-hydroxyquinolin-5-yl)methyl)piperazin-1-yl)-4-oxo-1,2,3,4-tetrahydroquinoline-3-carboxylic acid (CMQ-CF) which upon treatment with transition metal ions like Cu^{2+} , Ni^{2+} , Co^{2+} , Mn^{2+} , Zn^{2+} and Cd^{2+} yields metal coordination polymers. The novel ligand and its metal coordination polymers have been characterized on the basis of physicochemical analysis, spectroscopic techniques and thermal gravimetric analysis. Thermal analysis techniques are extensively used in studying the thermal behavior of metal chelates ^{xix, xx}. Thermogravimetry is a process in which a substance is decomposed in the presence of heat, which causes the breaking of bonds of the molecule ^{xxi}.

EXPERIMENTAL SECTION:

The elemental contents were determined by Thermo Finigen Flash1101 EA (Italy) and the metals of metal coordination polymers were determined volumetrically by Vogel's method ^{xxii}. To a 100 mg 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 CMQ-CF was recorded on 60 MHz NMR spectrophotometer. LC-MS of ligand was taken on LC-MSD-Trap-SL_01046. Magnetic susceptibility measurement of coordination polymers was carried out on Gouy Balance at room temperature. Mercury tetrathio cynato cobalate (II) $\text{Hg}[\text{Co}(\text{NCS})_4]$ was used as a calibrant. The electronic spectra of coordination polymers in solid were recorded at room temperature using MgO as reference. For determining the weight loss of the polymer samples at different temperatures, thermo gravimetric analysis (TGA) of CMQ-CF and its coordination polymers was carried out in a slow stream of air at $20^\circ\text{C}/\text{min}$. heating rate. Du pont thermo gravimetric analyzer (TC-10ATA-3000) was used for TGA. The melting points of the ligand and all the coordination polymers were measured using standard open capillary method. The in vitro antifungal screening of the ligand CMQ-CF and all the metal coordination polymers was carried out using a simple potato dextrose agar plate method against the fungi *Aspergillus Niger* (AN), *Botrydepladia Thiobromine* (BT), *Nigrospora* (NS), *Fusarium Oxyporium* (FO) as per the reported method ^{xxiii}.

GENERAL PROCEDURE:

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

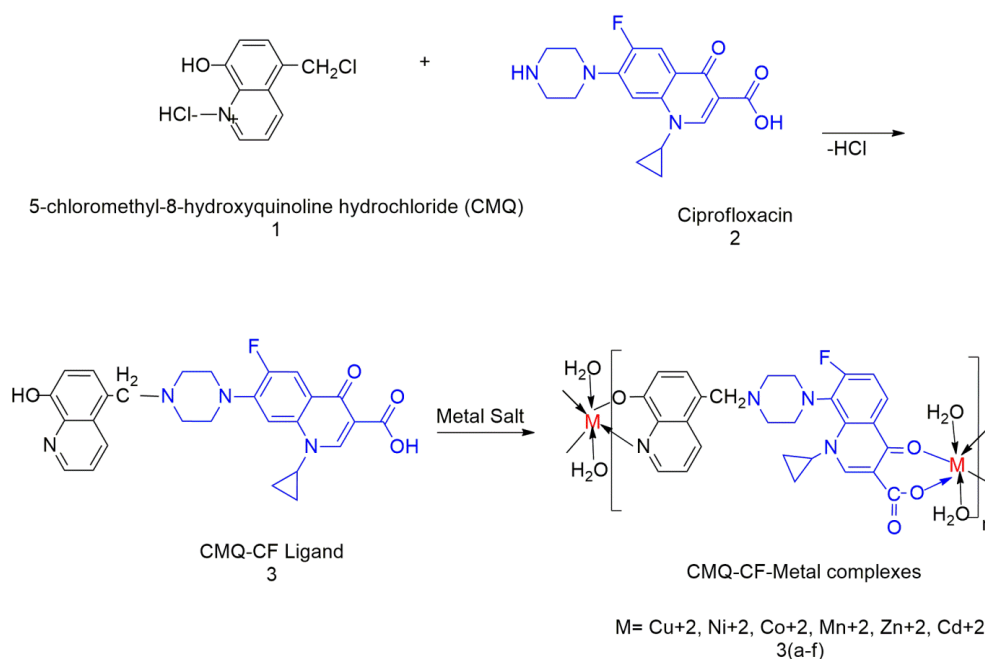
Method of preparation for synthesis of 1-cyclopropyl-6-fluoro-7-(4-((8-hydroxyquinolin-5-yl)methyl)piperazin-1-yl)-4-oxo-1,2,3,4-tetrahydroquinoline-3-carboxylic acid (CMQ-CF)[3]

5-chloromethyl-8-hydroxyquinoline (CMQ) was prepared by a reported method ^{xxiv, xxv}.

5-chloromethyl-8-hydroxyquinoline hydrochloride (0.25 mole) was added gradually to the mixture of ciprofloxacin (0.25 mole) and NaHCO_3 (0.2 mole) with continuous stirring. The mixture was then continuously stirred for 4 hours at room temperature followed by heating on water bath for about 3.5 hours at about 95°C . The resultant solid mass was filtered, washed with water and rectified spirit and then air dried. The sample did not melt up to 300°C .

Method of preparation for synthesis of coordination polymers of 1-cyclopropyl-6-fluoro-7-(4-((8-hydroxyquinolin-5-yl)methyl)piperazin-1-yl)-4-oxo-1,2,3,4-tetrahydroquinoline-3-carboxylic acid (CMQ-CF) [3(a-f)]

A solution of CMQ-CF (0.002 mol) in DMF (40 mL) was added drop wise to a solution of metal acetate (0.002 mol) in DMF (40mL) with stirring. Sodium acetate (0.60 g) was then added and appropriate pH was adjusted and then the reaction mixture was refluxed for 2.5 hours. The separated solid was filtered, washed with DMF (25 mL) and water and then air dried. The yields of the coordination polymers were almost quantitative.



Reaction Scheme

Compound 3: yield= 82%; Anal. Cald for C₂₇H₂₇FN₄O₄(490.53) Cald.: %C,66.11 ; %H, 5.55; %N, 11.42 ;%F, 3.87. Found: %C,66.1 ; %H,5.5 ; %N,11.4 ;%F,3.8. IR (KBR) (cm⁻¹): 3405-3345(-OHst.); 1390,962 (O-H); 3022, 2878 (C-Hst.);1590, 1507, 1472 (C-H bend.); 1690(COst.);1270-1245(C-Nst.); 1125(C-Ost.); 1628, 1580, 1465, 750(8-HQ characteristic bands). ¹H NMR (δ ppm):5.37 (s, 1H, -OH protons); 10.80 (s, 1H, -OH protons); 3.68 (s, 2H,-CH₂-); 3.45-2.64 (t, 8H,-CH₂-); 4.62 (t, 1H,-CH-); 3.92-3.68(d, 2H,-CH₂-); 1.40(m, 1H, -CH-); 0.85-0.60 (q, 4H, -CH₂-); 6.20-8.92 (m, 7H, Quinoline and Aromatic protons).LC-MS: 491.48 m/z.

Compound 3a:yield=78%; Anal. Cald for C₂₇H₂₅FN₄O₄Cu⁺²2H₂O (588.09) Cald.: %C,55.14; %H,4.29; %N,9.53;%F,3.23; %M,10.81. Found: %C,55.1; %H,4.3; %N,9.5;%F, 3.2; %M,10.8. IR (KBR) (cm⁻¹): 2965, 2830 (C-H st.); 1599, 1482(C-H bend.); 1725-1689 (CO st.); 1260 (C-N st.); 1120 (C-O st.); 1623, 1599, 1535,780 (8-HQ characteristic bands).

Compound 3b: yield=77%; Anal. Cald for C₂₇H₂₅FN₄O₄Ni⁺²2H₂O (583.23) Cald.: %C,55.60; %H,4.32; %N,9.61;%F,3.26; %M,10.06. Found: %C,55.5; %H,4.3; %N,9.6;%F 3.2; %M,10.0. IR (KBR) (cm⁻¹):2966, 2825 (C-H st.); 1610, 1485(C-H bend.); 1722-1695 (CO st.); 1265 (C-N st.); 1125 (C-O st.); 1625, 1608, 1534,782 (8-HQ characteristic bands).

Compound 3c: yield=70%; Anal. Cald for C₂₇H₂₅FN₄O₄Co⁺²2H₂O (583.47)Cald.: %C,55.58; %H,4.32; %N,9.60;%F,3.25; %M,10.09. Found: %C,55.5; %H,4.3; %N,9.5;%F,3.2;

%M,10.1. IR (KBR) (cm^{-1}): 2962, 2833 (C-H st.); 1620, 1480(C-H bend.); 1723-1695 (CO st.); 1263 (C-N st.); 1120 (C-O st.); 1625, 1608, 1530,780 (8-HQ characteristic bands).

Compound 3d: yield= 73%; Anal. Cald for $\text{C}_{27}\text{H}_{25}\text{FN}_4\text{O}_4\text{Mn}^{+2}2\text{H}_2\text{O}$ (579.48) Cald.: %C,55.96; %H,4.35; %N,9.67;%F,3.28; %M,9.48. Found: %C,55.9; %H,4.3; %N,9.6;%F, 3.2; %M,9.4. IR (KBR) (cm^{-1}): 2960, 2825 (C-H st.); 1599, 1480(C-H bend.); 1722-1699 (CO st.); 1260 (C-N st.); 1125 (C-O st.); 1625, 1603, 1537,784 (8-HQ characteristic bands).

Compound 3e: yield= 67%; Anal. Cald for $\text{C}_{27}\text{H}_{25}\text{FN}_4\text{O}_4\text{Zn}^{+2}2\text{H}_2\text{O}$ (589.92) Cald.: %C,54.97; %H,4.27; %N,9.50;%F,3.22; %M,11.08. Found: %C,54.9; %H,4.2; %N,9.4;%F,3.2; %M,11.0. IR (KBR) (cm^{-1}): 2960, 2825 (C-H st.); 1605, 1485(C-H bend.); 1715-1710 (CO st.); 1262 (C-N st.); 1125 (C-O st.); 1625, 1600, 1530,782 (8-HQ characteristic bands).

Compound 3f: yield= 76%; Anal. Cald for $\text{C}_{27}\text{H}_{25}\text{FN}_4\text{O}_4\text{Cd}^{+2}2\text{H}_2\text{O}$ (636.95) Cald.: %C,50.91; %H,3.96; %N,8.80;%F,2.99; %M,17.65. Found: %C,50.9; %H,4.0; %N,8.7;%F, 2.9; %M, 17.6. IR (KBR) (cm^{-1}): 2965, 2828 (C-H st.);1605, 1490(C-H bend.); 1725-1699 (CO st.); 1264 (C-N st.); 1120 (C-O st.); 1625, 1602, 1535,780 (8-HQ characteristic bands).

RESULTS AND DISCUSSION:

CMQ-CF ligand was an amorphous yellow powder. The C, H, N contents of CMQ-CF are consistent with the structure predicted (Reaction Scheme). The resultant ligand and all the metal coordination polymers do not melt upto 300°C . The analytical results show that the metal: ligand (M:L) ratio for all the coordination polymers is 1:1.

FT-IR spectra

The appearance of favorable bands in accordance with the reaction scheme suggest successful formation of the ligand and metal coordination polymers. Considerable differences were observed while comparing the IR spectra of the ligand with that of the metal coordination polymers which led to inference regarding the bonding of metal atoms to the functional groups. The broad band observed at $3405\text{-}3345\text{ cm}^{-1}$ in case of ligand attributed to the O-H stretching was absent in the spectra of coordination polymers indicating the involvement of oxygen of phenolic hydroxyl group and oxygen of -OH of carboxylic acid group in metal coordination. The shifting of C-N band from 1580 cm^{-1} to 1599 cm^{-1} indicates involvement of nitrogen of pyridine ring in metal chelation while forming coordination polymer^{xxvi}. The broad bands around $3000\text{-}3400$, 1240 , 815 and 700 cm^{-1} may be assigned to stretching, bending, rocking and wagging vibrations of the coordinated water molecules^{xxvii}. Appearance of new bands in spectra of coordination polymers at $\sim 1210\text{ cm}^{-1}$ is attributed to C-O-M stretching^{xxviii}. Sharp bands in the regions $\sim 560\text{ cm}^{-1}$ and $\sim 730\text{ cm}^{-1}$ can be assigned to M-N linkage, which indicates involvement of the nitrogen atom in coordination. Medium-intensity bands were observed at $\sim 675\text{ cm}^{-1}$ as a result of M-O coordination. The appearance of M-N and M-O vibrations supports the involvement of the N of pyridine ring and O of phenolic ring of the hydroxyquinoline moiety in metal coordination^{xxix}. Also, the C=O stretching frequency has shifted to higher frequency in spectra of metal coordination polymers which was observed at 1690 cm^{-1} in the spectra of ligand. This could be due to the involvement of the oxygen of the carbonyl group of ciprofloxacin moiety in metal coordination.

Electronic spectra and magnetic properties

The observed μ_{eff} values in the range 2.05-5.53 B.M are consistent with the above moieties. The value of magnetic moments and reflectance spectral data are represented in Table 1 for each coordination polymer which correlates with the structure assigned as the octahedral geometry^{xxx}.

Table 1 Diffuse electronic spectra and magnetic properties

Metal coordination polymers	Magnetic moment (BM) μ_{eff}	Electronic spectral data (cm ⁻¹)	Transition
CMQ-CF-Cu ⁺² .2H ₂ O	2.05	22796	Charge transfer
		15972	
CMQ-CF-Ni ⁺² .2H ₂ O	3.30	22980	³ A _{2g} → ³ T _{1g} (P)
		15605	³ A _{2g} → ³ T _{1g} (F)
CMQ-CF-Co ⁺² .2H ₂ O	3.87	22930	⁴ T _{1g} (F) → ⁴ T _{2g} (F)
		19616	⁴ T _{1g} (F) → ⁴ T _{2g}
		8945	⁴ T _{1g} (F) → ⁴ T _{2g} (P)
CMQ-CF-Mn ⁺² .2H ₂ O	5.53	23155	⁶ A _{1g} → ⁴ A _{1g} , ⁴ E _g
		17693	⁶ A _{1g} → ⁴ T _{2g} (4G)
		16485	⁶ A _{1g} → ⁴ T _{1g} (4G)
CMQ-CF-Zn ⁺² .2H ₂ O	Diamagnetic	-	-
		-	-
CMQ-CF-Cd ⁺² .2H ₂ O	Diamagnetic	-	-
		-	-

Thermal gravimetric analysis (TGA) of CMQ-CF ligand and its coordination polymers

The TGA data for ligand and all the coordination polymers are presented in Table 2. The weight loss of the polymer samples at different temperatures indicates that the degradation of the polymers is noticeable beyond 300°C. The rate of degradation becomes a maximum at a temperature lying between 400°C and 500°C. This may be due to accelerating by metal oxide which forms in situ. Each polymer lost approximately 55 percentages its weight when heated up to 700°C. Inspection of the thermo grams of all coordinated polymer samples revealed that all samples suffered appreciable weight loss in the range 150°C to 280°C. This may be due to the presence of coordinated water molecules. On the basis of the relative decomposition (% wt. loss) and the nature of thermo gram, the coordination polymers may be arranged in order of increasing stability as: Cu < Co < Cd < Ni < Mn < Zn. This trend also coincides with the stability order already reported for metal oxinates^{xxi} and for coordination polymers^{xxxii}.

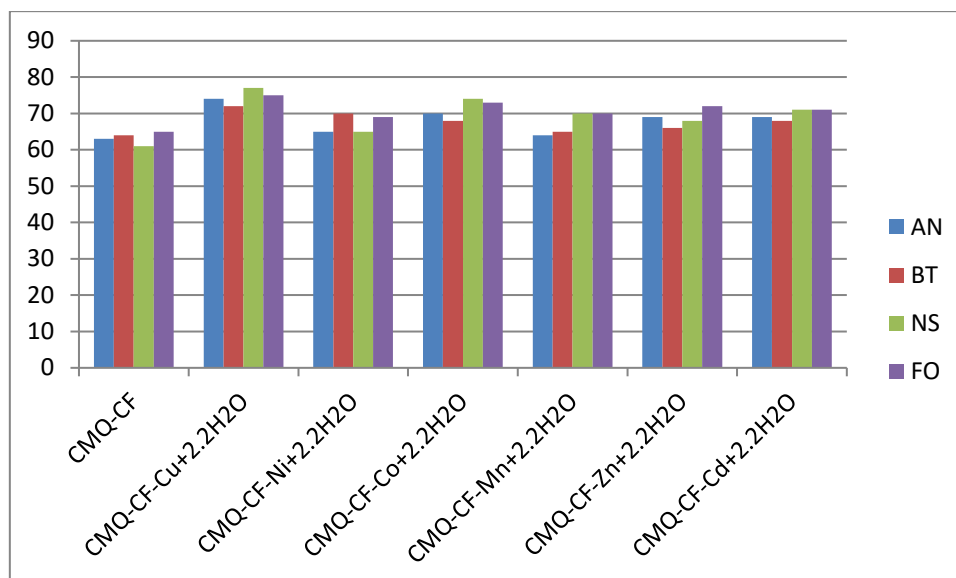
Table 2 Thermal gravimetric analysis (TGA) of CMQ-CF ligand and its coordination polymers

Ligand and its metal coordination polymers	% Weight loss at different temperatures (°C)						
	100	200	300	400	500	600	700
CMQ-CF	-	8.87	9.29	23.42	28.15	31.43	34.11
CMQ-CF-Cu ⁺² .2H ₂ O	1.12	8.75	11.70	26.33	30.43	33.36	37.03
CMQ-CF-Ni ⁺² .2H ₂ O	2.66	15.30	18.22	36.39	52.73	65.62	68.10
CMQ-CF-Co ⁺² .2H ₂ O	1.73	12.58	24.47	38.21	52.3	64.6	67.36
CMQ-CF-Mn ⁺² .2H ₂ O	2.32	9.94	13.61	34.23	53.20	66.06	68.49
CMQ-CF-Zn ⁺² .2H ₂ O	6.64	15.25	31.02	36.49	55.35	66.07	69.52
CMQ-CF-Cd ⁺² .2H ₂ O	1.75	12.58	24.44	38.51	52.60	64.82	67.44

Antifungal activity of CMQ-CF ligand and its coordination polymers

The screening of antifungal activity of CMQ-CF ligand and its coordination polymers (figure-1) reveals that the ligand is moderately toxic against fungi, while all the coordination polymers

are more toxic than ligand. Among all the coordination polymers, the Cu^{+2} coordination polymer has shown more toxicity against fungi.



CONCLUSION:

A novel ligand is successfully synthesized by condensation of CMQ and ciprofloxacin and its metal coordination polymers were prepared in good yield. In the metal coordination polymers, the ligand coordinates with two metal atoms at four coordination sites, with two water molecules. The elemental analysis and spectral data are found consistent with proposed structures of the ligand and metal coordination polymers. The octahedral geometry of the central metal atom for all the coordination polymers is confirmed by the study of electronic spectra and magnetic susceptibility measurements. The TGA reports support the coordination of two water molecules in all the metal coordination polymers and revealed the thermal stability order of the synthesized coordination polymers. A promising antifungal activity has been displayed by all the metal coordination polymers.

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CONFLICTS OF INTEREST:

None declared.

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