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G. J. Kharadi<sup>a</sup>; S. C. Panchani<sup>a</sup>; K. D. Patel<sup>a</sup>

<sup>a</sup> V. P. & R. P. T. P. Science College, Sardar Patel University, Vallabh Vidyanagar, Gujarat, India

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# Studies on Some Coordination Polymeric Chains of Metal Ions with QM1N

G. J. Kharadi, S. C. Panchani, and K. D. Patel

V. P. & R. P. T. P. Science College, Sardar Patel University, Vallabh Vidyanagar, Gujarat, India

Coordination chain polymers of the type  $\{[M(QM1N)(H_2O)_2] \cdot H_2O\}_n$  (where  $M = Mn(II), Co(II), Ni(II), Cu(II), Zn(II)$ ,  $QM1N = 1,5$ -bis(8-quinolinolyl-5-methylenoxy)naphthalene) have been investigated. The synthesized coordination polymers have been characterized on the basis of elemental analyses, infrared spectra, electronic spectra, magnetic measurements and thermogravimetric analyses. All the coordination polymers are insoluble in acetone, ethanol, chloroform, methanol, benzene, DMF and DMSO. The thermal decomposition of the coordination polymers is studied and indicates that not only the coordinated water is lost but also that the decomposition of the ligand from the coordination polymers is necessary to interpret the successive mass loss.

**Keywords** coordination polymers, Freeman–Carroll method, transition metal, uninegative bidentate ligand

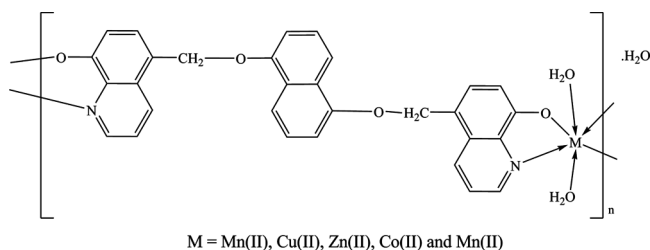
## INTRODUCTION

The interest in the construction of coordination polymers by linking transition metal ions with ligands has been constantly growing over the past years [1–7]. Coordination polymers (chelate polymers) are defined as materials in which metal ions are linked together with di- or polyfunctional ligands. Coordination polymers are usually known for their thermal stability [8,9]. However, other

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Address correspondence to K. D. Patel, V. P. & R. P. T. P. Science College, Sardar Patel University, Vallabh Vidyanagar 388 120, Gujarat, India. E-mail: drkdpatel64@yahoo.co.in; gaurangkharadi@yahoo.com



**Figure 1:** Suggested structure of the coordination polymers.

equally important applications have been reported such as their use as solar energy converters [10] and their ability to remove SO<sub>x</sub> and NO<sub>x</sub> from the environment [11]. The choice of the metal ions, the ligand design, the counter ions and the solvents can have a considerable effect on the final architecture of the coordination polymers and particularly its dimensionality [12].

The design of new coordination supramolecules and polymers based on transition metal compounds and multidentate organic ligands has attracted much interest in recent years [13–17]. Previously, Patel et al. have synthesized a series of transition heterochelates [18–20]. We prepared 1,5-bis[(8-quinolinolyl-5-methylenoxy)naphthalene], which reacts with transition metal ions like Mn(II), Co(II), Ni(II), Cu(II), and Zn(II). The coordination polymers have been characterized on the basis of elemental analysis, infrared spectra, reflectance spectra, magnetic measurements and thermal studies. The thermal analysis techniques were extensively used in studying the thermal behavior of coordination polymers [21–25]. The suggested structure of the coordination polymers is shown in Figure 1.

## EXPERIMENTAL

### Materials

All the chemicals used were of analytical grade. The compound 8-hydroxyquinoline, formaldehyde, 1,5-dihydroxy naphthalene and metal salt were purchased from E. Merck Ltd. (India). The organic solvents were purified by standard methods [26].

### Instruments

Carbon, hydrogen and nitrogen were analyzed with the PerkinElmer, USA 2400-II CHN analyzer. The metal content of the coordination polymers was analyzed by the EDTA titration technique [27]. Infrared spectra (4000–400 cm<sup>-1</sup>) were recorded on a Nicolet-400D spectrophotometer using KBr

pellets. The reflectance spectra of the coordination polymers were recorded in the range of 1700–350 nm (as MgO discs) on a Beckman DK-2A spectrophotometer. The magnetic moments were obtained by Gouy's method using mercury tetrathiocyanatocobaltate(II) as a calibrant ( $\chi_g = 16.44 \times 10^{-6}$  c.g.s. units at 20°C). Diamagnetic corrections were made using Pascal's constant. A simultaneous TGA/DTG and DTA had been obtained by a model 5000/2960 SDT, TA Instruments, U.S.A. The experiments were performed in a N<sub>2</sub> atmosphere at a heating rate of 10°C/min<sup>-1</sup> in the temperature range of 50–800°C, using an Al<sub>2</sub>O<sub>3</sub> crucible. The sample sizes are ranged in mass from 4.5 to 10 mg. The DSC was recorded using a DSC 2920, TA Instruments, U.S.A. The DSC curves were obtained at a heating rate of 10°C/min<sup>-1</sup> in a N<sub>2</sub> atmosphere over the temperature range of 50–400°C, using an aluminium crucible.

### Preparation of Polymers of 1,5-Bis(8-quinolinolyl-5-methylenoxy)naphthalene (QM1N)

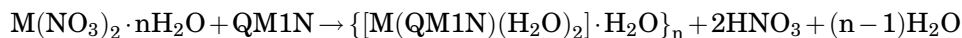
The literature procedure [28] with some modifications in the reaction time and work-up was followed. In a two-necked round-bottomed flask fitted with a magnetic stirrer, a reflux condenser and a dropping funnel, compound 5-chloromethyl-8-hydroxyquinoline hydrochloride (23 g, 0.01 mol) was dissolved in 1,5-dihydroxynaphthalene-A<sup>1</sup> (17.5 g, 0.1 mol) in an acetone-water mixture. The resulting mixture was refluxed for 3 h with occasional shaking. The resulting suspension, which contained a green precipitate, was made alkaline with dilute aqueous ammonia and then filtered. The solid product was collected and dried to give QM1N (71% yield). Analysis for C<sub>30</sub>H<sub>22</sub>N<sub>2</sub>O<sub>4</sub>: Calculated (%): C, 75.94; H, 4.64; N, 5.90; Found: C, 75.64; H, 4.34; N, 5.60.

### Synthesis of Coordination Polymers

The performed ligand QM1N (5 mmol, 3.13 g) was dissolved in 10 mL of DMF. To this solution, the methanolic solution of 25 mL metal nitrate (5 mmol) was added slowly with constant stirring over the period of 30 min in 1:1 molar ratio. The pH of the solution was adjusted 5–6 with the drop-wise addition of methanolic solution of sodium acetate. The resulting mixture was heated with stirring at a reflux temperature for 4–5 h. The obtained coordination polymers were filtered off, washed with hot water, hot methanol and diethyl ether and finally dried in a vacuum desiccator over anhydrous CaCl<sub>2</sub>. The coordination polymers are insoluble in all common organic solvents like methanol, ethanol, chloroform, acetone, benzene, dimethyl formamide and dimethyl sulfoxide.

## RESULTS AND DISCUSSION

The analytical and physical properties of the ligand QM1N and its coordination polymers are listed in Table 1. The following reaction describes the formation of the coordination polymers:



where M = Mn(II), Cu(II), Zn(II), Co(II) and Ni(II).

All the coordination polymers are insoluble in all common organic solvents. It was not possible to characterize them by conventional methods, like osmometry, viscometry, and conductometry, as they are insoluble. The nature of the ligand, high thermal stability, metal–ligand ratio (1:1) and insolubility in all common organic solvents suggest their polymeric nature.

### IR Spectra

The important infrared spectral band and their tentative assignments for the synthesized ligand and its coordination polymer were recorded as KBr disks and are discussed.

In the investigated coordination polymers, the bands observed in the region 3400–3450, 1295–1300, 860–870 and 715–717  $\text{cm}^{-1}$  are attributed to –OH stretching, bending, rocking and wagging vibrations, respectively due to the presence of water molecules [29]. The presence of a rocking band indicates the coordination nature of the water molecule [30]. The IR spectra of the QM1N show 1600  $\text{cm}^{-1}$  and 1380  $\text{cm}^{-1}$  corresponding to  $\nu(\text{C}=\text{N})$  and  $\nu(\text{C}-\text{O})$ , respectively; on complexation these peaks shifted to a lower frequency of 1588  $\text{cm}^{-1}$  and 1367  $\text{cm}^{-1}$  due to coordination polymer formation. The weak bands around 519  $\text{cm}^{-1}$  and 779  $\text{cm}^{-1}$  are attributed to the  $\nu(\text{M}-\text{O})$  and  $\nu(\text{M}-\text{N})$  stretching frequency [31].

### Magnetic Moments and Electronic Spectra

The information regarding geometry of the coordination polymers is obtained from their electronic spectral data and magnetic moments. For the Co(II) coordination polymer, the reflectance spectra exhibit the bands of medium intensity at  $\sim 9200$ ,  $\sim 18000$  and  $\sim 19000 \text{ cm}^{-1}$ , which are assigned to the transitions  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$  ( $\nu_1$ ),  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$  ( $\nu_2$ ) and  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})$  ( $\nu_3$ ), respectively, of an octahedral cobalt(II) coordination polymer [32] and the magnetic moment value is 4.19 B.M. The electronic spectra of the Ni(II) coordination polymer exhibit absorption bands at  $\sim 10500$ ,  $\sim 17400$  and  $\sim 24100 \text{ cm}^{-1}$  attributed to  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$  ( $\nu_1$ ),  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$  ( $\nu_2$ )

**Table 1:** Analytical and physical data of the ligand H<sub>2</sub>L and its coordination polymers.

Empirical formula of monomer unit	Color (yield %)	Found (Calcd.) (%)				$\mu_{\text{eff}}$ (B.M.)
		C	H	N	M	
QM1N	Yellowish brown (74)	75.64 (75.94)	4.34 (4.64)	5.60 (5.90)	–	–
{(Mn(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub> C <sub>30</sub> H <sub>26</sub> MnN <sub>2</sub> O <sub>7</sub>	Dark brown (65)	60.44 (60.23)	5.97 (5.70)	4.15 (4.07)	8.13 (8.06)	6.18
{(Co(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub> C <sub>30</sub> H <sub>26</sub> CoN <sub>2</sub> O <sub>7</sub>	Dark brown (61)	60.09 (60.00)	5.93 (5.83)	4.12 (4.03)	8.67 (8.58)	4.19
{(Ni(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub> C <sub>30</sub> H <sub>26</sub> N <sub>2</sub> NiO <sub>7</sub>	Dark green (71)	60.11 (60.01)	5.93 (5.77)	4.12 (4.09)	8.64 (8.52)	2.92
{(Cu(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub> C <sub>30</sub> H <sub>26</sub> CuN <sub>2</sub> O <sub>7</sub>	Dark brown (69)	59.68 (59.44)	5.89 (5.77)	4.09 (4.01)	9.29 (9.19)	1.78
{(Zn(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub> C <sub>30</sub> H <sub>26</sub> ZnN <sub>2</sub> O <sub>7</sub>	Brown (63)	59.52 (59.44)	5.88 (5.60)	4.08 (4.01)	9.53 (9.38)	Diamag.

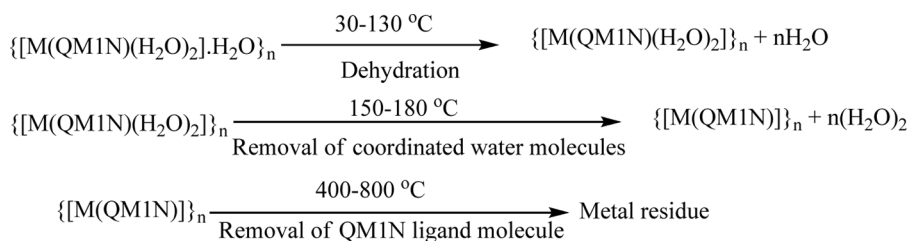
B.M. = Bohr Magneton.

and  ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$  ( $\nu_3$ ) transitions, respectively, in an octahedral geometry [33]. The value of the magnetic moment (2.92 B.M.) may be taken as additional evidence for their octahedral structure. The ground state of Mn(II) is  ${}^6A_{1g}$ . The Mn(II) coordination polymer in an octahedral field should have three transitions corresponding to  ${}^6A_{1g} \rightarrow {}^4T_{1g}$  ( $\nu_1 \sim 15000 \text{ cm}^{-1}$ ),  ${}^6A_{1g} \rightarrow {}^4T_{2g}$  ( $\nu_2 \sim 19600 \text{ cm}^{-1}$ ) and  ${}^6A_{1g} \rightarrow {}^4A_{1g}$ ,  ${}^4E_g$  ( $\nu_3 \sim 24500 \text{ cm}^{-1}$ ) in increasing order of energy. The magnetic moment value of the Mn(II) coordination polymer is 6.18 B.M., suggesting an octahedral geometry [34]. The Cu(II) coordination polymer displays a broad band at  $\sim 15500 \text{ cm}^{-1}$  due to the  ${}^2E_g \rightarrow {}^2T_{2g}$  transition, and the magnetic moment value is 1.78 B.M., which is close to the spin-only value (1.73 B.M.) expected for an unpaired electron, which offers the possibility of an octahedral geometry [35]. Magnetic moments of coordination polymers are summarized in Table 1. The Zn(II) coordination polymers are diamagnetic as expected for  $d^{10}$  systems.

## Thermal Behavior of the Prepared Coordination Polymers

The thermogravimetric analysis of the coordination polymers was carried out within a temperature range 30–800°C in a  $N_2$  atmosphere to establish their compositional differences as well as to ascertain the nature of associated water molecules. It has been observed that the loss of mass in the temperature range between 30–130°C for all coordination polymers corresponds to one lattice molecule. The coordination polymers show loss in mass corresponding to two water molecules in the range 150–180°C, indicating that this water molecule is coordinated to the central metal ion [36]. In the temperature range 400–800°C the ligand molecule is lost. In all of the cases the final products are metal oxides. These results are in good accordance with the composition of the coordination polymers. Thermoanalytical results (TGA) of the metal coordination polymers of QM1N are summarized in Table 2. The TGA curve of  $\{[Mn(QM1N)(H_2O)_2] \cdot H_2O\}_n$  is shown in Figure 2.

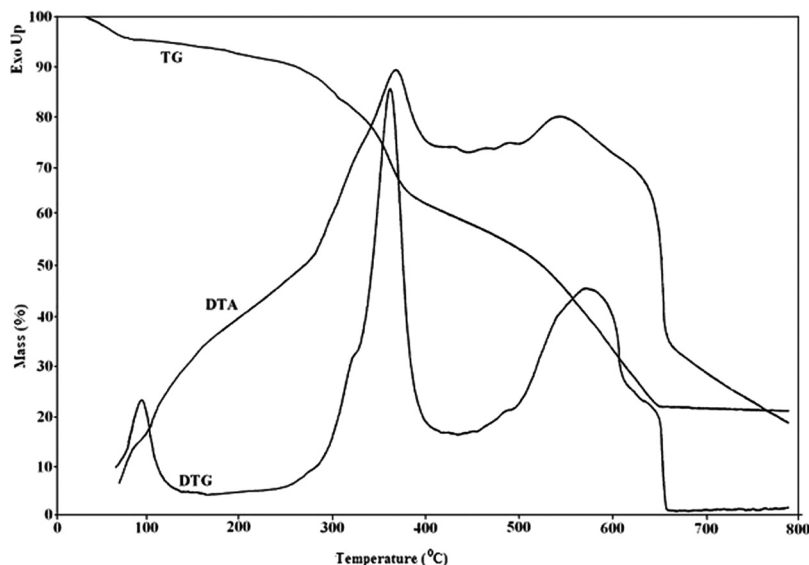
The stage of thermal decomposition of the coordination polymers can be written as under:



**Table 2:** Thermo-analytical data of the coordination polymers.

Coordination polymers	TGA range (°C)	Mass loss (% obs. (calc.))	Assignment	Activation energy Ea/kcal mol <sup>-1</sup>
{(Mn(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub>	50–120	3.42 (3.07)	loss of 1 lattice water molecule	3.33
	120–650	72.30 (72.17)	decomp. of ligand and formation of Mn <sub>2</sub> O <sub>3</sub>	
{(Co(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub>	50–130	3.28 (3.06)	loss of 1 lattice water molecule	3.38
	130–740	72.28 (72.02)	decomp. of ligand and formation of CoO	
{(Ni(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub>	50–130	2.96 (3.06)	loss of 1 lattice water molecule	3.63
	140–690	69.61 (69.37)	decomp. of ligand and formation of NiO	
{(Cu(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub>	50–130	2.86 (3.04)	loss of 1 lattice water molecule	3.63
	130–720	70.95 (71.52)	decomp. of ligand and formation of CuO	
{(Zn(QM1N)(H <sub>2</sub> O) <sub>2</sub> ) · H <sub>2</sub> O} <sub>n</sub>	50–130	2.57 (3.00)	loss of 1 lattice water molecule	3.61
	140–730	71.92 (71.31)	decomp. of ligand and formation of ZnO	





**Figure 2:** The TGA curve of  $\{(Mn(QM1N)(H_2O)_2)H_2O\}_n$ .

### Calculation of Thermodynamic Parameters of the Decomposed Coordination Polymers

The thermodynamic activation parameters of the decomposition process of the coordination polymers such as energy of activation ( $E_a$ ) and order of reaction ( $n$ ) were evaluated graphically by employing the Freeman–Carroll method [37] using the following relation:

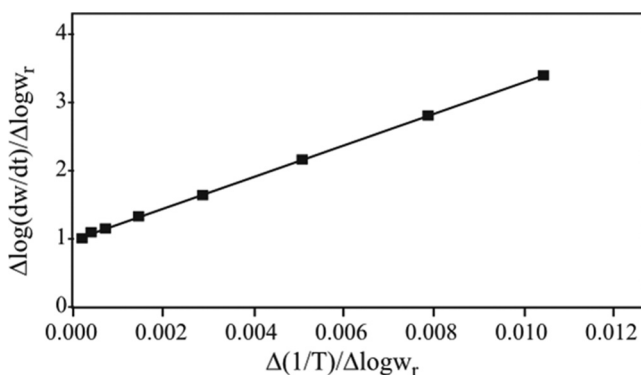
$$[(-E_a/2.303R)\Delta(1/T)]\Delta \log w_r = -n + \Delta \log(dw/dt)\Delta \log w_r \quad (1)$$

where  $T$  is the temperature in K,  $R$  is the gas constant,  $w_r = w_c - w$ ;  $w_c$  is the weight loss at the completion of the reaction and  $w$  is the total mass loss up to time  $t$ .  $E_a$  and  $n$  are the energy of activation and order of reaction, respectively. A typical curve of  $[\Delta \log(dw/dt)/\Delta \log w_r]$  vs.  $[\Delta(1/T)/\Delta \log w_r]$  for the Mn(II) coordination polymer is shown in Figure 3. The slope of the plot gave the value of  $E_a/2.303R$  and the order of reaction ( $n$ ) was determined from the intercept.

The thermodynamic activation parameters of the decomposition process of coordination polymers such as entropy ( $\Delta S^\ddagger$ ), pre-exponential factor ( $A$ ), enthalpy ( $\Delta H^\ddagger$ ) and free energy of the decomposition ( $\Delta G^\ddagger$ ), were calculated using the following relations [38]

$$E_a/RT_s^2 = A/\Phi_{\text{exo}}(-E_a/RT_s) \quad (2)$$

$$S^\ddagger = 2.303(\log Ah/KT)R \quad (3)$$



**Figure 3:** Freeman-Carroll plot for thermal dehydration of  $\{(Mn(QM1N)(H_2O)_2)H_2O\}_n$ .

$$H^* = E_a - RT \quad (4)$$

$$G^* = H^* - T_s S^* \quad (5)$$

Where,  $\Phi$  is the heating rate,  $K$  is the Boltzman constant,  $h$  is the Plank's constant and  $T_s$  is the peak temperature from the DTG curve. According to the kinetic data obtained from DTG curves, all the complexes have negative entropy, which indicates that the studied coordination polymers have more ordered systems than reactants [39].

## Thermal Analysis Data and Structural Interpretation

The TGA curves of Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) complexes consist of three stages of mass loss. The loss of one mole of lattice water molecules is a first order and the value of the energy of activation for the dehydration process is found to be 3.33–3.61 kJ mol<sup>-1</sup>. The second stage of mass loss shows that loss of metal coordinated water molecule [40]. The kinetic parameters, especially activation energy ( $E_a$ ), is helpful in assigning the strength of the bonding of ligand moieties with the metal ion. The calculated ( $E_a$ ) values of the investigated complexes, i.e., the stage of the formation of volatile gas products, are relatively high, and indicates that the ligand is strongly bonded to the metal ion [41]. From the above data, the suggested structure of the coordination polymers is shown in Figure 1.

## CONCLUSION

The design and synthesis of a new tetradentate ligand (QM1N) has been successfully demonstrated. New Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) coordination polymeric assemblies were prepared. All the coordination

polymers are insoluble in common organic solvents. It was not possible to characterize them by convectional methods, like osmometry, viscometry, or conductometry, as they are insoluble. The nature of the ligand, high thermal stability, metal–ligand ratio (1:1) and insolubility of these compounds suggest their polymeric nature. An octahedral geometry has been assigned to all the prepared polymers. Based on the activation energy values, the thermal stability of the coordination polymers in the decreasing order is: Ni(II), Cu(II) > Zn(II) > Co(II) > Mn(II) [42].

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