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Synthesis, Characterization and Thermal Degradation Oligomer and Monomer/Oligomer Metal Complex Compounds of 2-Methylquinolin-8-ol

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Synthesis, Characterization and Thermal Degradation Oligomer and Monomer/Oligomer Metal Complex Compounds of 2-Methylquinolin-8-ol

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The oxidative polycondensation of 2-methylquinolin-8-ol (2-MQ) has been accomplished by using H_2O_2 oxidant in aqueous alkaline medium. Optimum reaction conditions of the oxidative polycondensation and the main parameters of the process were established. At optimum reaction conditions, yield of the products was found to be 32.0%. The structures of the obtained monomer and oligomer were confirmed by FT-IR, UV-vis, ¹H-NMR, ¹³C-NMR and elemental analysis. The characterization was made by TG-DTA, using size exclusion chromatography (SEC) and solubility tests. The FT-IR, ¹H-NMR and ¹³C-NMR data shows that the polymerization proceeded by the C-C coupling of *ortho* and *para* positions according to -OH group of 2-MQ. The molecular weight distribution values of the product were determined by SEC. The number-average molecular weight (M_n), weight-average molecular weight (M_w) and polydispersity index (PDI) values of O-2-MQ were found to be 1980, 3312 and 1.681 for H_2O_2 oxidants, respectively. Thermal analysis results of O-2-MQ demonstrated to be stability against thermal decomposition. Thermal analyses of 2-MQ-Cd, 2-MQ-Co, 2-MQ-Cu, 2-MQ-Zn, O-2-MQ-Cd, O-2-MQ-Co, O-2-MQ-Cu, and O-2-MQ-Zn monomer/oligomer-metal complex compounds were investigated in a N₂ atmosphere between 15–1000°C.

Keywords: oxidative polycondensation; oligo-2-methylquinolin-8-ol; oligomer metal complexes; thermal degradation

1 Introduction

Polymers of phenols and aromatic amine compounds have found wide application in a number of areas such as coatings, laminates and photoresists (1, 2). Besides possessing good thermal properties, these polymers or oligomers can be doped to make them electrically conductive (3). The molecular weight distributions of polyphenols significantly influence functional properties (4). A large number of polymeric Schiff base complexes with a variety of central metal ions and ligand systems have been synthesized (5). These compounds have emerged as a new generation of material with tremendous potential in fields like superconducting materials, heat stability materials from Schiff bases of salicylaldehyde derivatives (6–10). The structures of some polymer–metal ion complexes have been reported in the literature (11-13). They also have useful properties such as paramagnetizm, electrochemical cell

and resistance to high energy (14-18). New oxovanadium (IV) Schiff base complexes derived from amino acids and aromatic o-hydroxyaldehydes were studied by Pessoa et al.(19). Conductivity properties of Cu (II), Co (II) and Ni (II) complex compounds of oligo-2-[(4-chlorophenyl) imino methylene] phenol were studied by Kaya et al. (20). Antimicrobial properties of oligophenols with a Schiff base substitute and their oligomer metal complex compounds were investigated by Kaya et al. (21). Copper (II), zinc (II) and cobalt (II) complexes of oligosalicylaldehyde had been synthesized and their thermal properties had been investigated by Mart et al. (22). Additional functions for oligophenols may be the cleaning of poisonous heavy metals in the industrial waste waters. In addition, Schiff base compounds had been used for the determination of transition metals in some natural food samples (23). Therefore, the synthesis of oligomer-metal complexes and oligomers are very important in analytic, environmental and food chemistry.

In this article, we have investigated the effects of different parameters such as temperature, times and initial concentration of H₂O₂ for the oligo-2-methylquinolin-8-ol in the medium alkaline. 2-MQ and O-2-MQ were characterized by using FT-IR, UV-vis, ¹H-NMR, ¹³C-NMR and elemental analysis, TG-DTA and SEC techniques. Also, 2-MQ-Cd,

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2 Experimental

2.1 Materials

2-Methylquinolin-8-ol, methanol, ethanol, dioxane, benzene, toluene, ethyl acetate, CCl₄, THF, DMF, DMSO, H_2SO_4 (98%), AgNO₃, KOH, hydrochloric acid (HCl, 37%), H_2O_2 (35% aqueous solution), Co (AcO)₂ 4H₂O, Cu (AcO)₂ 4H₂O, Zn (AcO)₂ 2H₂O, and Cd (AcO)₂ 2H₂O were supplied from Merck Chem. Co. (Germany) and were used as received.

UV-vis (λ_{max}): 204, 244, 306 and 335 nm. ¹H-NMR (DMSO): δ ppm, 9.41 (s, 1H, phenyl-OH), 7.06 (s, 1H, Ar-Ha), 7.37 (t, 1H, Ar-Hb), 7.35 (d, 1H, Ar-Hc), 8.18 (d, 1H, Ar-Hd), 7.42 (d, 1H, Ar-He) and 2.66 (s, 3H, -CH₃). ¹³C-NMR (DMSO): ppm, 152.98 (C1-ipso), 111.53 (C2-H), 127.39 (C3-H), 118.01 (C4-H), 126.84 (C5-ipso), 136.55 (C6-H), 123.02 (C7-H), 157.11 (C8-ipso), 138.28 (C9-ipso), 25.11(C10-H₃).

2.2 Synthesis of Oligo-2-methylquinolin-8-ol with H_2O_2 Oxidant

O-2-MQ was synthesized through oxidative polycondensation of 2-methylquinolin-8-ol with aqueous solutions of H_2O_2 (35%, in water) (3). The 2-MQ (0.795 g 0.005 mol) was dissolved in a 20 mL aqueous solution of KOH (10%, 0.005 mol) and placed into a 50-ml three-necked roundbottom flask (Scheme 1). It was fitted with a condenser, thermometer, stirrer, and an addition funnel containing H_2O_2 . After heating to 40°C, H_2O_2 was added drop wise over about 20 min. The reaction mixtures were stirred at the various temperatures and durations. The reaction mixtures were cooled to room temperature, and then, 0.005 mol HCl (37%) was

KOH, H₂O₂

 CH_3

HO

Óн

HO

ĊH

CH₃

Sch. 1. Synthesis of oligo-2-methylquinolin-8-ol.

HC

ĊH;

n



Sch. 2. Synthesis of 2-methylquinolin-8-ol metal complexes.

added. The unreacted monomer was separated from the reaction products by washing with ethyl acetate. The mixture was filtered and washed with hot water $(3 \times 25 \text{ ml})$ for separating from mineral salts and then dried in the oven at 110° C.

UV-vis (λ_{max}): 208, 248, 308 and 340 nm. ¹H-NMR (DMSO): δ ppm, 9.80 (s, 1H, phenyl-OH), 7.37 (t, 1H, Ar-Hb), 8.27 (d, 1H, Ar-Hd), 7.64 (d, 1H, Ar-He) and 2.75 (s, 3H, -CH₃). ¹³C-NMR (DMSO): ppm, 152.97 (C1-ipso), 111.54, 114.48 (C2-ipso), 127.40 (C3-H), 118.00, 123.99 (C4-ipso), 126.84 (C5-ipso), 136.57 (C6-H), 123.05 (C7-H), 157.12 (C8-ipso), 138.26 (C9-ipso), 25.15(C10-H₃).

2.3 Synthesis of 2-Methylquinolin-8-ol Metal Complexes

Solutions of Co $(AcO)_2 4H_2O$, Cu $(AcO)_2 4H_2O$, Zn $(AcO)_2 2H_2O$ and Cd $(AcO)_2 2H_2O$ (1.25 mmol) in methanol (15 ml) were added to a solution of 2-MQ and O-2-MQ (2.5 mmol) in methanol (10 ml). The mixture was stirred and heated at 80°C for 4 h (Scheme 2). The precipitated complex was filtered, washed with cold MeOH and then dried in a vacuum oven.

2.4 Synthesis of Metal Complex Compounds of Oligo-2methylquinolin-8-ol

Solutions of Co $(AcO)_2 4H_2O$, Cu $(AcO)_2 4H_2O$, ZnCl₂ and Cd $(AcO)_2 2H_2O$ (2.5 mmol) in methanol (30 ml) were added to a solution of 2-MQ and O-2-MQ (2.5 mmol/unit) in THF (20 ml). The mixture was stirred and heated at 80°C for 4 h (Scheme 3). The precipitated complex was filtered, washed with cold MeOH/THF (1:1) and then dried in a vacuum oven.

2.5 Characterizations

O-2-MQ was brown in powder form, and completely soluble in organic solvents such as DMF, THF, DMSO and alkaline solution. O-2-MQ was insoluble in hexane, benzene, toluene, ethyl acetate, CH_2Cl_2 , $CHCl_3$, CCl_4 and ethanol.



Sch. 3. Synthesis of metal complex compounds of oligo-2-methylquinolin-8-ol.

	Calculated (%, Found)						
Compounds	С	Н	Ν	Metal	$\mu_{\mathrm{eff}},\mathrm{BM}$	Yield, %	
2-MQ	75.47 (75.40)	5.66 (5.60)	8.81 (8.78)				
O-2-MQ	76.43 (75.79)	4.46 (3.88)	8.92 (8.54)	_		95.00	
2-MQ-Cu	63.23 (63.00)	4.22 (3.96)	7.38 (7.15)	16.74 (15.56)	1.77	87.50	
2-MQ-Zn	62.93 (62.52)	4.20 (3.89)	7.34 (7.10)	17.15 (26.23)	Diamagnetic	96.00	
2-MQ-Co	64.01 (63.78)	4.27 (4.05)	7.47 (7.28)	15.72 (13.28)	5.01	91.43	
2-MQ-Cd	56.02 (55.50)	3.74 (3.52)	5.54 (6.28)	26.49 (25.64)	Diamagnetic	93.06	
O-2-MQ-Cu	63.91 (62.55)	3.20 (3.00)	7.46 (7.22)	16.92 (14.05)	1.61	75.81	
O-2-MQ-Zn	63.60 (63.28)	3.18 (2.84)	7.42 (7.05)	17.33 (15.18)	Diamagnetic	76.31	
O-2-MQ-Co	64.70 (64.34)	3.24 (3.02)	7.55 (7.30)	15.89 (12.93)	4.85	86.42	
O-2-MQ-Cd	56.55 (56.33)	2.83 (2.45)	6.60 (5.28)	26.49 (23.39)	Diamagnetic	69.73	

Table 1. Elemental analyses data and yields of 2-MQ, O-2-MQ and metal complexes compounds

Monomer and oligomer metal complex compounds were insoluble in hexane, benzene, toluene, ethyl acetate, CH_2Cl_2 , $CHCl_3$, ethanol, methanol, CCl_4 and acetone. These monomer and oligomer metal complex compounds were soluble in DMSO and DMF.

The infrared and ultraviolet-visible spectra were measured by Matttson 1000 FT-IR (U.K) and UV-1700 Shimadzu (Japan), respectively. Elemental analysis was carried out with a Carlo Erba 1108 (Italy). The FT-IR spectra were recorded using a KBr disc (4000-400 cm⁻¹). UV-vis spectra of 2-MQ and O-2-MQ and metal complex compounds were determined by using methanol and THF. 2-MQ and O-2-MQ were characterized by using ¹H-NMR and ¹³C-NMR spectra (Bruker Avance DPX-400 and 100.6 MHz, respectively) recorded at 25°C by using deuterated DMSO as a solvent. Tetramethylsilane was used as internal standard. Thermal data were obtained by using a Shimadzu (Japan) Diamond Thermal Analysis system. The TG-DTA measurements were made between 15–850°C (in N₂, rate 10°C/min). SEC analyses were performed at 30°C using DMF/MeOH (v/v, 4/1) as eluent at a flow rate of 0.4 ml/min. A refractive index detector was used as detector. The instrument (Shimadzu 10AVp series HPLC-SEC system, Japan) was calibrated with a mixture of polystyrene standards (Polymer Laboratories; the peak molecular weights, Mp, between 162 and 19880) using GPC software for the determination of the number-average molecular weight M_n), weight-average molecular weight (M_w) and polydispersity index (PDI) of the polymer samples. For SEC investigations, a Macherey-Nagel GmbH & Co. was used (100 Å and 7.7 nm diameter loading material) 3.3 mm i.d. \times 300 mm columns. Magnetic measurements were carried out by the Gouy method using $Hg[Co(SCN)_4]$ as calibrant. Molar conductances of the oligomer-metal complexes were determined in DMSO (ca. 10^{-3} M) at room temperature using a Sherwood Scientific MKI Model conductivity meter. Metal analyses were determined by complexometric titration. The elemental analysis data of compounds are given in Table 1. The metal quantities of O-2-MQ-Cu, O-2-MQ-Zn, O-2-MQ-Co and O-2-MQ-Cd compounds were found to be 14.05%,



Fig. 1. Effect of H_2O_2 (mol L^{-1}) concentration on the yield of oligomer.



Fig. 2. Effect of 2-MQ (mol L^{-1}) concentration on the yield of oligomer.



Fig. 3. Effect of polycondensation reaction times and temperatures on the yield of oligomer.

15.18%, 12.93% and 23.39%, respectively, by gravimetric technique.

Results and Discussion 3.

Synthesis of O-2-MO 3.1.

2-methylquinolin-8-ol was oxidized with H₂O₂ oxidant in an aqueous alkaline medium at 80°C. The yield of O-2-MQ was 32.0% at optimum reaction conditions such as $[2-MQ]_0 =$ $[KOH]_0 = [H_2O_2]_0 = 0.1 \text{ mol/L}$, at 80°C for 3 h. The conversion of 2-MQ was 59.0% at the reaction conditions such as $[2-MQ]_0 = [KOH]_0 = [H_2O_2]_0 = 0.1 \text{ mol/L}$ at 50°C for 3 h. According to these values, under the same conditions, the yield of O-2-MQ decreased with the increasing of reaction temperatures. This reason may be the conversion from oligomer to monomer at the high reaction temperatures. The effects to the yield of the oxidative polycondensation reaction conditions were examined at the various conditions such as initial concentrations of monomer and oxidant and reaction times and temperatures and are given Figures 1-3.

3.2. Structure of O-2-MQ

SEC analyses of O-2-MQ were performed at 30°C using DMF/Methanol (v/v, 4/1) as eluent at a flow rate of 0.4 ml/min. The M_n, M_w and PDI values of O-2-MQ were found to be 1980, 3312 g mol^{-1} and 1.681, respectively.

The UV-vis spectroscopic studies were carried out with methanol and DMSO solutions of the 2-MQ and O-2-MQ, respectively. In the 2-MQ spectra, K bands of phenol and C_6H_5 -N = were observed, 246 nm and 335 nm, respectively. UV-vis spectra of O-2-MQ, K and R bands were observed, 248 nm and 340 nm, respectively. In the monomer and oligomer-metal complex compounds, the low intensity bands in the 645-610 nm and 655-640 nm range are consistent with $d \rightarrow d$ transitions of the Cu (II) and Co (II) metal ions, respectively. Because of diamagnetic properties of Cd (II) and Zn (II) complex compounds were not observed $d \rightarrow d$ transitions.

The FT-IR spectral data of 2-MQ, O-2-MQ and monomer and oligomer metal complexes are given Table 2. The FT-IR spectra of 2-MQ and O-2-MQ, bands of OH, (-C=N)_{cvclic} and -CH₃ groups were observed 3298, 1600 and 2920; 3320, 1602 and 2925 cm^{-1} , respectively. Metal-O and Metal-N vibrations of metal complex compounds were observed in $506-515 \text{ cm}^{-1}$ and $590-615 \text{ cm}^{-1}$, respectively.

The magnetic susceptibility measurements of 1.77 and 1.61 BM are indicative of the appearance of the Cu (II) complex compounds of monomer and oligomer, respectively, in a square planar geometry. The magnetic moment of Co (II) coplex compounds of monomer and oligomer have been found to be 5.01 and 4.85 BM, respectively. These values supports tetrahedral configuration (24). Zinc and cadmium coplex compounds of monomer and oligomer are diamagnetic and are likely to be of tetrahedral structures.

In order to identify the structures of monomer and oligomer, the ¹H-NMR spectra were recorded in DMSO-d₆. ¹H-NMR and ¹³C-NMR spectra of the monomer and oligomer are given in Figures 4-7, respectively. In the

Table 2. FT-IR spectral data of 2-MQ, O-2-MQ and metal complex compounds

	Wave number (cm^{-1})							
Compounds	-OH(str)	-OH (bend)	-C=N _{cyclic}	-C=C	-C-O	-CH ₃	Metal-O	Metal-N
2-MQ	3298	1325	1600	1572, 1508	1260	2920		
O-2-MQ	3320	1327	1602	1570, 1472	1246	2925		
2-MQ-Cu	3183	1330	1604	1570, 1508	1287	2918	514	604
2-MQ-Zn	3439	1341	1606	1568, 1508	1283	2927	510	600
2-MQ-Co	3387	1335	1605	1568, 1506	1279	2900	514	602
2-MQ-Cd	3443	1335	1593	1560, 1506	1280	2923	506	593
O-2-MQ-Cu	3360	1337	1604	1570, 1508	1287	2925	515	605
O-2-MQ-Zn	3424	1341	1592	1568, 1505	1283	2932	509	601
O-2-MQ-Co	3420	1329	1597	1568, 1506	1279	2923	514	615
O-2-MQ-Cd	3441	1333	1593	1564, 1510	1271	2923	510	590



¹H-NMR spectra of 2-MQ and O-2-MQ, the signals of phenyl -OH, and -CH₃ groups were observed 9.76, 16.11 and 9.61 ppm and 9.70, 15.82 and 9.58 ppm, respectively. The ¹H-¹³C-NMR spectra results of the O-2-MQ confirm the formation of oligomer units. Other phenol derivatives were also polymerized, and the results are reported in the literature (4). Crosslinking in the polymer structure is expected in those cases where the *ortho*, *para* positions in the corresponding monomer structure are unsubstituted. ¹³C-NMR studies on O-2-MQ indicate that the linkage between any two adjacent phenyl rings is largely at *ortho* and *para* positions (Figs. 6 and 7). The peak values for C2 and C4 observed in 111.53 and 118.01 ppm in the monomer and 114.48 and 123.99 ppm in the oligomer, respectively. In addition,



Fig. 5. ¹³C-NMR spectrum of 2-methylquinolin-8-ol.



Fig. 6. ¹H-NMR spectrum of oligo-2-methylquinolin-8-ol.

according to FT-IR spectra, no ether (C-O-C) linkage in the oligomer structure, has been supported to C-C coupling system. The FT-IR, ¹H- and ¹³C-NMR data shows that the polymerization proceeded by and the C-C coupling of *ortho* and *para* positions according to -OH group of 2-MQ.

The thermal degradation of monomer, oligomer and 2-MQ-Cu, 2-MQ-Zn, 2-MQ-Co, 2-MQ-Cd, O-2-MQ-Cu, O-2-MQ-Zn, O-2-MQ-Co and O-2-MQ-Cd monomer-oligomer-metal complex compounds were studied by TG-DTA analyses at the N₂ medium and some data derived from TG curves are summarized in Table 3, where the thermal stability in terms of onset temperatures, 20 wt% losses and residues are given. The initial degradation temperature and 0.0 wt% loss of 2-MQ and O-2-MQ was found to be 138 and 149°C and at 850°C, respectively. According to TG analysis, the initial degradation temperature of 2-MQ was lower than the oligomer. According to DTA curves, endothermic processes of the monomer and oligomer were observed in 155, 440 and 72, 186, 549°C, respectively.

A modification of the thermal stability by polycondensation or depending on the metal cation electro negativity is expected (15). Metal complex compounds of both monomer and oligomer show onset temperature values between 155–450 and 230–370°C range, respectively. This can also explain the differences, which appeared between the found and calculated elemental analysis values. Both 2-MQ-Cu and O-2-MQ-Cu demonstrated lower thermal stability against thermal degradation according to other oligomer-metal complex compounds.



Fig. 7. ¹³C-NMR spectrum of oligo-2-methylquinolin-8-ol.

		Т			DTA	
Compounds	^a T _{on}	^b W _{max.} T	20% wt loss	% Car.res. at 850°C	Exo	Endo
2-MQ	138	255	285	0.00		155, 440
O-2-MQ	149	380	370	0.00		72,186, 549
2-MQ-Cu	155	195	175	3.20		205
2-MQ-Zn	450	490	475	10.50		498
2-MQ-Co	250	300	277	28.45		103, 232
2-MQ-Cd	375	400, 500	405	13.45		403, 506
O-2-MQ-Cu	350	375	365	0.00		296, 394
O-2-MQ-Zn	370	415	432	44.05	756	410, 500
O-2-MQ-Co	230	300	270	22.45		585
O-2-MQ-Cd	345	380	370	15.55		396

Table 3. Thermal decomposition values of 2-MQ, O-2-MQ and metal complex compounds

^{*a*}The onset temperature.

^bMaximum weight temperature.

4 Conclusions

2-methylquinolin-8-ol was converted to oligomer by H_2O_2 oxidant in an aqueous alkaline medium. The yield of O-2-MQ was found to be 99% for the H₂O₂ oxidant. The M_n, M_w and PDI values of O-2-MQ were found to be 1980, 3332 g mol^{-1} and 1.681, respectively, using the H_2O_2 oxidant. The spectral analyses such as FT-IR, ¹H- and ¹³C-NMR have demonstrated the ability to unite from ortho, para carbons ring each other of formation oligomers from oxidative polycondensation of 2-MQ. The results of TG-DTA analyses were shown to have enough resistance against thermal decomposition of oligo-2methylquinolin-8-ol. According to TG analyses, weight losses of oligomer and monomer-metal complex compounds changed at 850°C as follows: Cu> Cd> Co> Zn and Cu> Zn> Cd> Co, respectively. As result, 2-MQ-Co and O-2-MQ-Zn demonstrated higher thermal stability against thermal degradation than monomer, oligomer and other oligomermetal complex compounds.

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