

Spectral and Physiochemical Studying for Some 8-HQ Complexes

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Abstract

In this work, some organometallic complexes with featured characteristics were prepared by linking metallic ions such as Al^{3+} , Zn^{2+} and Ba^{2+} to the 8-hydroxyquinoline ligand. Two different solvents, dimethylformamide (DMF) and dimethylsulphoxide (DMSO), were used to form the solutions of the prepared complexes. The spectral characteristics in the UV, visible and IR regions, and physiochemical characteristics such as melting point, electrical conductivity, solubility and gas chromatography and mass spectrometry, of the prepared complexes were studied. Also, the effect of solvent type on the spectral characteristics of these complexes were determined. The prepared complexes and their spectral and physiochemical characteristics were assessed to be used as laser dyes.

1. Introduction

The 8-hydroxyquinoline is a necessary compound in many applications due to its capability to arrange with different metallic ions as bidentate via nitrogen atom of quinoline ring and oxygen atom after deprotonation of hydroxyl group [1-4]. The 8-hydroxyquinoline referred to as oxine, as shown in Fig. (1), is a well-known fluorogenic reagent in analytical chemistry. It is widely employed as a good fluorogenic ligand binding with several metal cations due to its low quantum yield in aqueous and some organic solutions. It has been widely studied from both experimental and theoretical viewpoints and it has analogous properties, such as intensive fluorescence in concentrated acids, lack of fluorescence in water or alkanes or making hydrogen-bonded clusters with water, ammonium or alcohol molecules [5-8]. Ye [9] has mixed hydrated lithium hydroxide and 8-hydroxyquinoline in anhydrous ether to prepare 8-hydroxyquinoline lithium and washed product repeatedly with anhydrous ether. Then he has dried the product using infrared radiation to obtain LiQ white powder with a yield of 90%.

The derivatives of 8-hydroxyquinoline were intensively used to synthesize fluorescent chemo-sensors of high sensitivity employed in many applications, especially sensing and imaging of metal ions of important biological and environmental importance [10-12]. The tris(8-hydroxyquinoline) aluminum (Alq_3) is commonly used in organic light emitting diodes (OLEDs) as an electron transport/light emitting layer [13]. The properties of the metal/organic interface affect the characteristics of such devices, the understanding of the interaction between metals and organic molecules is important [14,15]. The Alq_3 is known as a fluorescent complex showing electroluminescence with a high quantum yield [16]. This complex embedded in anodic porous alumina (APA) has attracted significant interest in connection with optical applications [17,18]. In 1985 notation metals from a mixture, the difference in fluorescence lifetimes was determined for the single determination of several (six) group II and group III [19]. The 8-hydroxyquinoline and its complexes occupy a uniquely important position in analytical chemistry [20,21]. Because of the intense luminescence and high stability of these complexes such as Alq_3 and Znq_2 , they are incorporated in OLED devices as emissive layers [22-24]. The fast achievements in nanotechnology have created new opportunities to use Znq_2 in bioanalysis and biosensors. Recently, research works on Znq_2 showed that the metal ions forms a supramolecular tetrameric structure composed of a Zn_4O_8 core via oxo-bridging of the 8-quinolinolato ligands [25,26]. The 8-Hq bivalent metal complex (MQ_2) is an attractive type of luminescent material, which has broad applications, MQ_2 for example, 8-hydroxyquinoline barium (Baq_2), 8-hydroxyquinoline magnesium (Mgq_2) [27-29].

In this work, binary and ternary organometallic complexes are prepared and the effects of solvent type on the spectroscopic and physiochemical characteristics of these complexes are determined and studied.

2. Chemicals And Materials

All chemicals and reagents were used in this project and were listed in Table (1).

2.1 Physical and spectroscopic measurements

In order to characterize precursors, ligands and their complexes, the following characterizations were considered:

Melting points

Melting points of compounds were obtained by an Electro-thermal Stuart SMP40.

FT-IR spectra

A Nicolet Impact 410 spectrophotometer was used to record the Fourier-transform infrared (FT-IR) spectra using KBr disk in the wavenumber range of 400-4000 cm^{-1} .

Electronic spectra

Electronic spectra were recorded in the spectral range of 190-1100 nm using by SPEKOL-2000 double-beam UV-visible spectrophotometer supplied by PG Instruments, Ltd. (UK).

Conductivity measurement

Electrical conductivities measurement of the complexes were recorded at room temperature for 1 mg/mL solutions of the samples in acetone solvent. The conductivity was measured in $\mu\text{S}/\text{cm}$ using Metrohm 712 conductivity meter calibrated with 0.01 M of KCl solution. Glass vial containing deep eutectic solvents (DESs) was used in order to prohibit water absorption and an electrode was sealed very well by parafilm. This vial was put into a water bath and joined to a thermostat to control the temperature. The heating rate of the DESs samples was 1 K/min.

GC-Mass spectra

The gas chromatography and mass spectroscopy were performed on the ligands and metal complexes using Agilent Technologies 7890A GC-Mass spectrometer.

2.2 Synthesis of 8-hydroxyquinoline complexes with some metal ions

The metal solution of $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (0.241g, 1 m.mole) in 10 ml distilled water was stirred for ten minutes. The ligand solution HQ (0.435 g, 3 m.mole) is mixed with KOH solution (0.56 g in 20 ml H_2O). Finally a solution of 8-hydroxyquinoline was also added to the above metal solution. The final mixture was filtered and the precipitate was washed with ethanol and dried at 25°C for one day. Two complexes barium and zinc were prepared using the same procedure, but the difference was in the weights.

3. Results And Discussion

The metal complexes obtained were colored solids. The complexes were insoluble in water and soluble in some of the common solvents such as dimethylformamide (DMF), dimethylsulphoxide (DMSO) and chloroform, as shown in table (2). Results of the molar conductivity indicated that the complexes have non-electrolyte behavior. The increase in melting points of the prepared complexes confirmed the new connections between the ligand and metallic ions as well as their thermal stability. Similar method of preparation was considered to prepare Znq_2 and Baq_2 (2m.mole) [30-33]. The measurement of molar conductivity was carried out in acetone solvent at 25 °C and 10^{-3} M for the prepared complexes. The molar conductivity of the two complexes were ranging in 0.835-3.001 $\mu\text{S}/\text{cm}$.

Figure (2) shows the absorption spectra of the free ligand dissolved in two different polar solvents (DMF and DMSO) while Figures (3-5) show the absorption spectra of the three complexes (Alq_3 , Znq_2 and Baq_2) prepared in this work in same solvents at 25°C (room temperature) in the spectral range 300-600 nm. Recalling that the polarity index of DMSO is slightly

higher (7.2) than that of DMF (6.4), the ligand in DMSO shows higher absorbance than that in DMF. This may also be attributed to the difference in absorption coefficient of the solvents (1.85 cm^{-1} for DMF and 2.17 cm^{-1} for DMSO).

Referring to figures (3) and (4), the light absorbed by Alq_3 or Znq_2 induces the valence electrons to transfer from the ground states to excited states. A slight red shift was observed in the absorption peaks because of the $n \rightarrow \pi^*$ transition and this results in an increase in the intensity. The compound can absorb the radiation in the spectral range of 350-400 nm and transmit bluish-green radiation for Alq_3 and green radiation for Znq_2 . The orbital energies included in the electronic transitions were constant and quantized, therefore, sharp peaks are expected on the absorption spectra in this spectral region. The band at 389 is attributed to the charge transfer (C.T) [34,35] because each electronic energy level contains several vibrational levels, and transitions can occur to and from various vibrational levels, resulting in peak broadening. With existing different rotational levels in absorbing materials, the absorption condition is additionally complicated in few cases only, such as the vapor phase or nonpolar solvents.

As shown in Fig. (3), the Alq_3 in DMSO exhibits higher absorbance than Alq_3 in DMF, which is attributed to the difference in the polarity indexes and absorption coefficients of DMF and DMSO. However, no reasonable shifts in the two distinct absorption peaks of Alq_3 (320 and 386 nm) are observed. Similarly, the result shown in Fig. (4) for Znq_2 in DMF and DMSO, can be interpreted. However, the Alq_3 solution exhibits higher absorbance than the Znq_2 solution regardless the type of solvent. In case of Baq_2 complex, a red shift was observed from 384 nm in DMF to 406 nm in DMSO, as shown in Fig. (5), which is due to the higher index of polarity and dielectric constant of DMSO than that of DMF. As well, the Baq_2 in DMSO shows about 400% higher absorbance than Baq_2 in DMF.

Accordingly, both solvent type and polarity have reasonable effect on the absorption characteristics of these complexes. In general, they exhibit red shift with increasing polarity of the solvent. As well, the effect of DMSO as a solvent on the absorption characteristics is larger than the use of DMF. The behavior of charge transfer complex is ascribed to varying the solvent. The featured property of reaction environment is the complex interaction between solvent and solute because the polarity of solvent can affect the stability of the complex. The measurements of electronic transitions of charge transfer metal complex in various solvents give important information about the mechanism of their formation. The interactions between the donor and acceptor species in the solvated complex are interfered by the solvent [36,37].

Figures (6-9) showed the FT-IR spectra of the free ligand (8-Hq) and complexes prepared in this work in the wavenumber range $400\text{-}4000 \text{ cm}^{-1}$. The spectrum of the 8-Hq in Fig. (6) showed different bands at 3161 cm^{-1} due to OH stretching group which it has been disappeared for complex that confirmed the coordination being through the oxygen atom of hydroxyl group, and another band at 3049 cm^{-1} to C-H_{st} group aromatic vibration. Another band at 1576 cm^{-1} was to the C=N group which it has been shifted to lower frequency at 1538 cm^{-1} for Znq_2 complex because it confirmed the coordination being through the nitrogen atom of C=N group. While the band at 1500 cm^{-1} was due to the C=C aromatic stretching group [38-40].

The vibrations of benzene rings at 1576 , 1500 , 1466 and 1402 cm^{-1} are observed. The vibrations of C=N is at 1375 cm^{-1} , the bend vibrations of O-H is at 1281 cm^{-1} , and the stretch vibration modes of C-O are at 1217 cm^{-1} and 1203 cm^{-1} [41]. New bands were observed around 545 and 647 cm^{-1} due to the formation of Alq_3 (Fig. 7), around 503 and 570 cm^{-1} due to the formation of Znq_2 (Fig. 8), and around 478 and 535 cm^{-1} due to the formation of Baq_2 (Fig. 9). These bands were ascribed to M-N (metal-nitrogen) and M-O (metal-oxygen) due to coordination with metal ions [42-44]. Bands related to C-H group aromatic is detected at 3059 cm^{-1} , but the C-H group stretching for the aliphatic group is detected at 2921 cm^{-1} to aluminum complex, 3045 cm^{-1} to Barium complex and 2923 cm^{-1} to zinc complex [45]. Bands were observed in 1538 , 1572 and 1579 cm^{-1} zinc, barium and aluminum complexes respectively due to C=N group vibrations. Other bands were recorded at 1281 cm^{-1} for Zn-C-N, 1175 cm^{-1} for C-C_{bend} , 1138 cm^{-1} for Zn-C-O, and vibrations at 2923 and 3058 cm^{-1} were recorded

to C-H_{as} stretching and =C-H stretching, respectively. Similar mode of linking in Baq₂ and Alq₃ were confirmed by bands at 1196, 1165, 1136 and 1114 cm⁻¹ ascribed to C-C bond vibrations [46].

The GC-MS is the typical analysis method to determine the composition of the volatile fraction as it offers high separation power and provides useful spectra for the identification and quantification of compounds. It is commonly used to confirm the purity of organic compounds. In theory, GC retention times should be useful for identifying components in mixtures. The applicability of such data is limited by the number of variables that must be controlled in order to obtain reproducible results. Nevertheless, GC provides reliable tool of confirm the presence or absence of impurities and contaminants, either as elements or compounds, in the sample, provided that an authentic sample of the substance is available [47].

Figure (10) shows the GC-MS spectra of the three complexes (Alq₃, Znq₂ and Baq₂) prepared in this work. The GC analysis of Alq₃ and Znq₂ samples show high abundance when compared to that of Baq₂. Also, the full-width at half maximum (FWHM) is smaller for Alq₃ and Znq₂ than Baq₂, which may be attributed to the formation of smaller particles of Ba linked to the 8-Hq ligand. This result may be supported by the red shift observed in absorption spectra of Baq₂ when compared to those of Alq₃ and Znq₂. Finally, all prepared complexes show high purity as no traces of other elements than the precursors were found in the mass spectra.

4. Conclusion

In concluding remarks, three different metallic ions were linked to the 8-hydroxyquinoline ligand to form organometallic complexes and study their spectral and physiochemical characteristics. The linkage of the metallic ions to the ligand was confirmed as the corresponding vibration bands were observed on the FTIR spectra. The complex Baq₂ in both solvents showed higher absorbance than the Alq₃ complex, which showed higher absorbance than the Znq₂ complex. Similarly, all complexes in DMSO showed higher absorbance than those in DMF, which reflects that the DMSO is better than DMF as a solvent for the prepared complexes. No reasonable shift was observed in the absorption peaks of Alq₃ and Znq₂ complexes in both types of solvents, whereas a slight shift (~12nm) was observed in case of Ba₂, which exhibits the stability of absorption characteristics of the prepared complexes.

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Tables

Table (1) Chemicals used in this work

No.	Chemicals	Formula
1	Acetone	C ₂ H ₆ O
2	Dimethyl sulfoxide (DMSO)	(CH ₃) ₂ SO
3	Ethanol	C ₂ H ₅ OH
4	Methanol	CH ₃ OH
5	Chloroform	CHCl ₃
6	N,N-Dimethylformamide(DMF)	HCON(CH ₃) ₂
7	Potassium hydroxide	KOH
8	8-Hydroxyquinoline (8-HQ)	C ₉ H ₇ NO
9	Barium(II) chloride dihydrate	BaCl ₂ .2H ₂ O
10	Aluminum chloride hexahydrate	AlCl ₃ .6H ₂ O
11	Zinc(II) chloride	ZnCl ₂

Table (2) Some physical properties of the 8-hydroxyquinoline and its complexes

No.	Empirical Formula	Color	M.P (°C)	Conductivity (μS/cm)	Molecular Weight	Solubility				
						H ₂ O	Acetone	DMF	DMSO	CHCl ₃
1	C ₉ H ₇ NO	White	76		145.1	-	+	+	+	+
2	C ₂₇ H ₁₈ AlN ₃ O ₃	Yellowish-green	>300	1.983	459.4	-	—	+	+	+
3	C ₁₈ H ₁₂ ZnN ₂ O ₂	Light-green	>300	3.001	353	-	—	+	+	+
4	C ₁₈ H ₁₂ BaN ₂ O ₂	Light-green	>300	0.835	425	-	—	+	+	+

- Insoluble, + Soluble, — Partially soluble

Figures

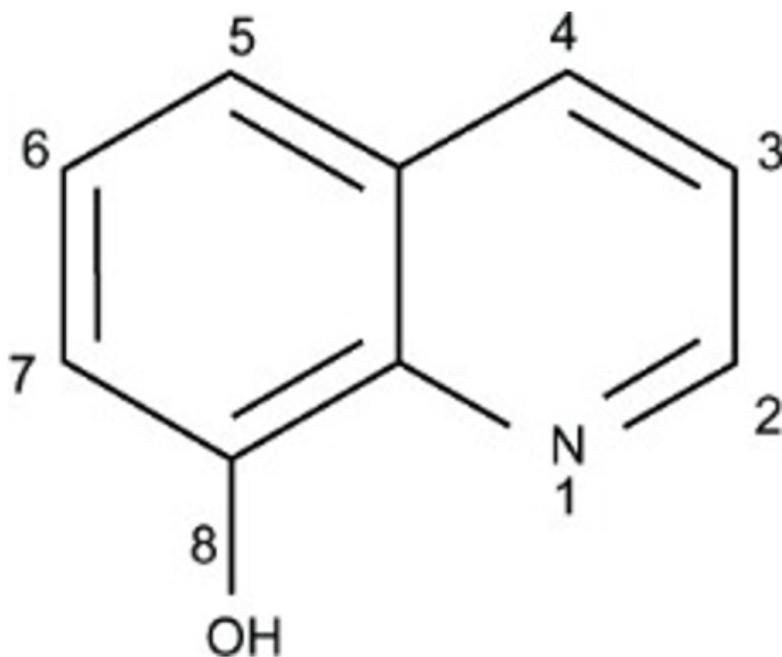


Figure 1

Structure of 8-hydroxyquinoline

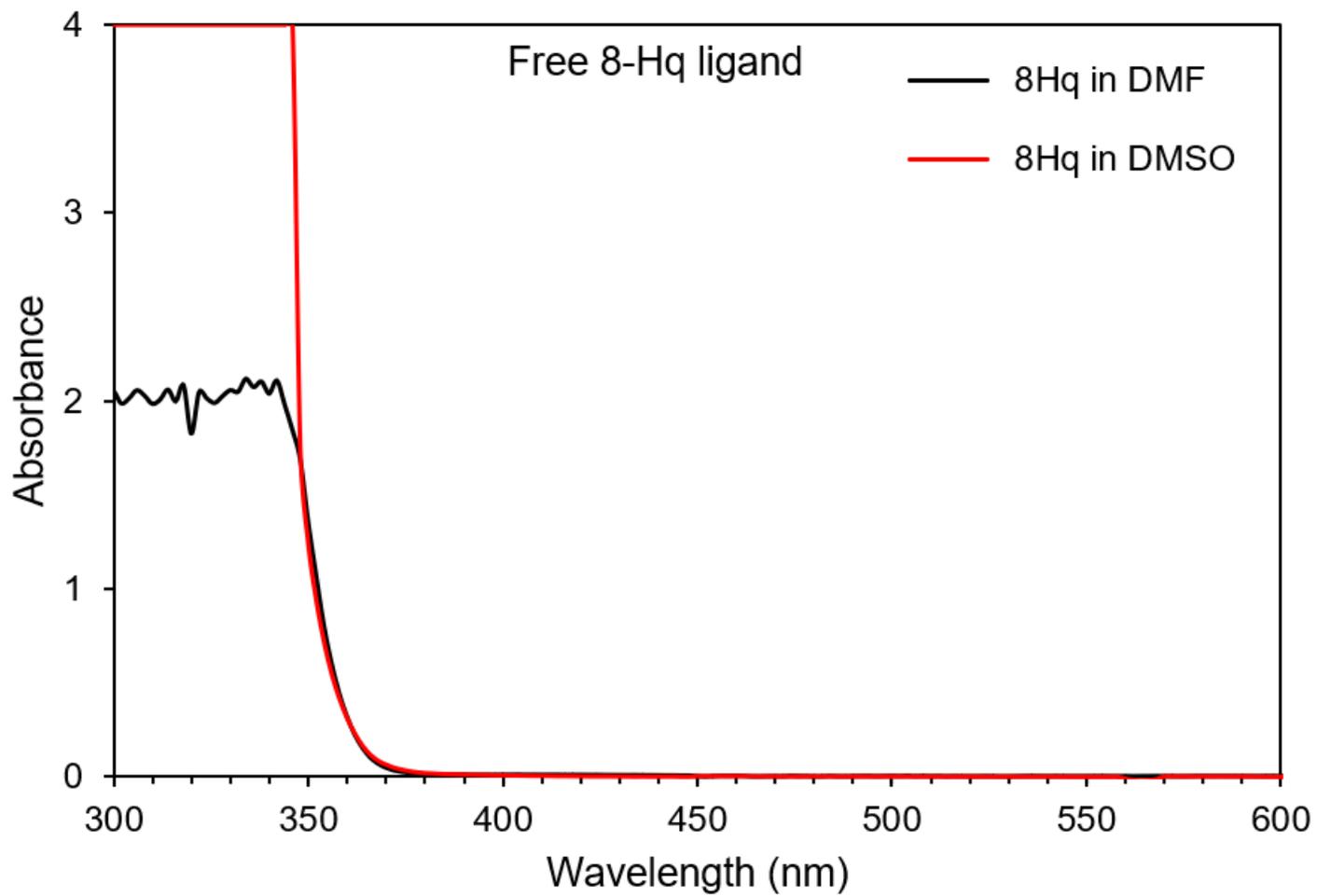


Figure 2

UV-Visible spectra of free 8-Hq ligand in DMF and DMSO solvents

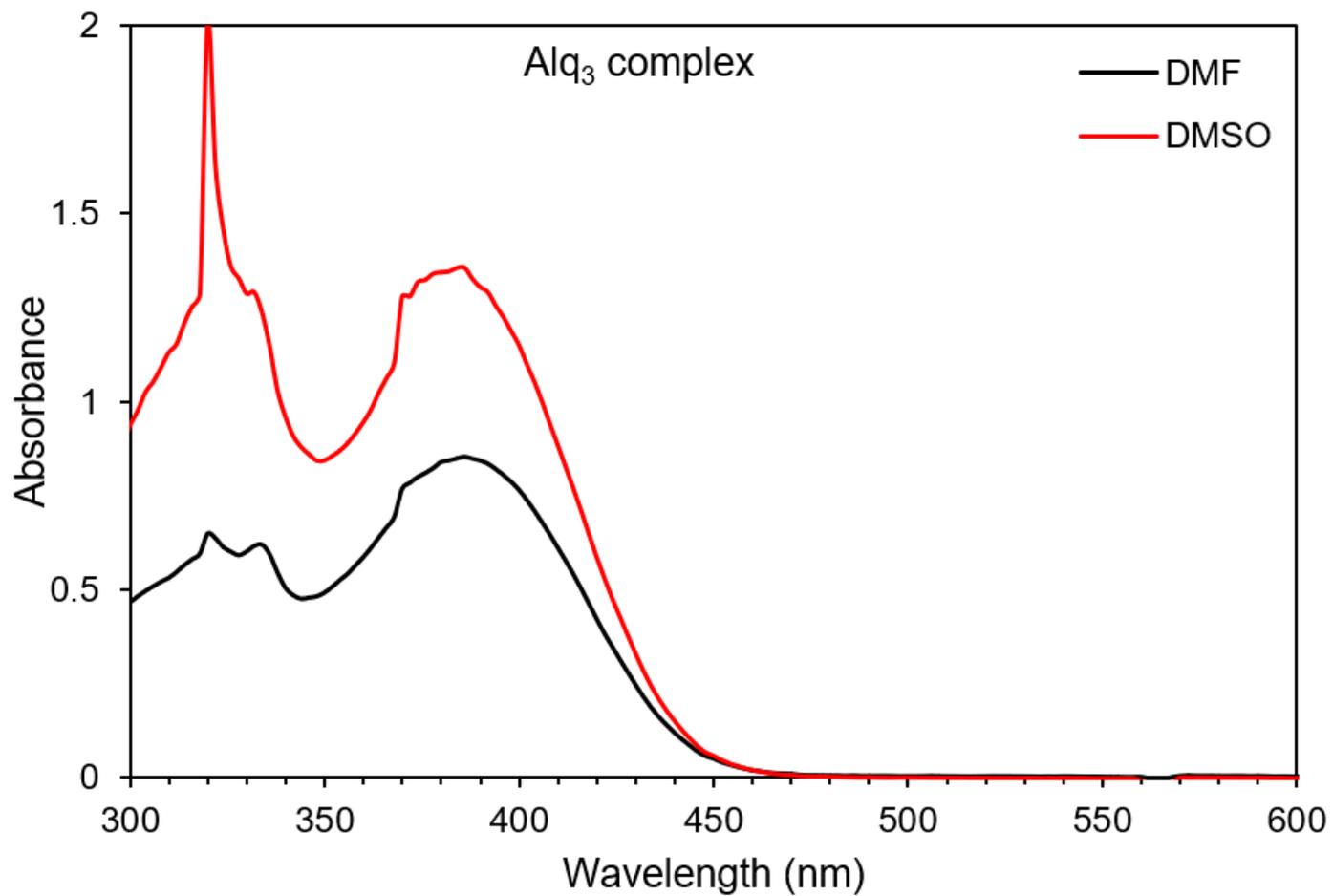


Figure 3

UV-Visible spectra of Alq₃ complex in DMF and DMSO solvents

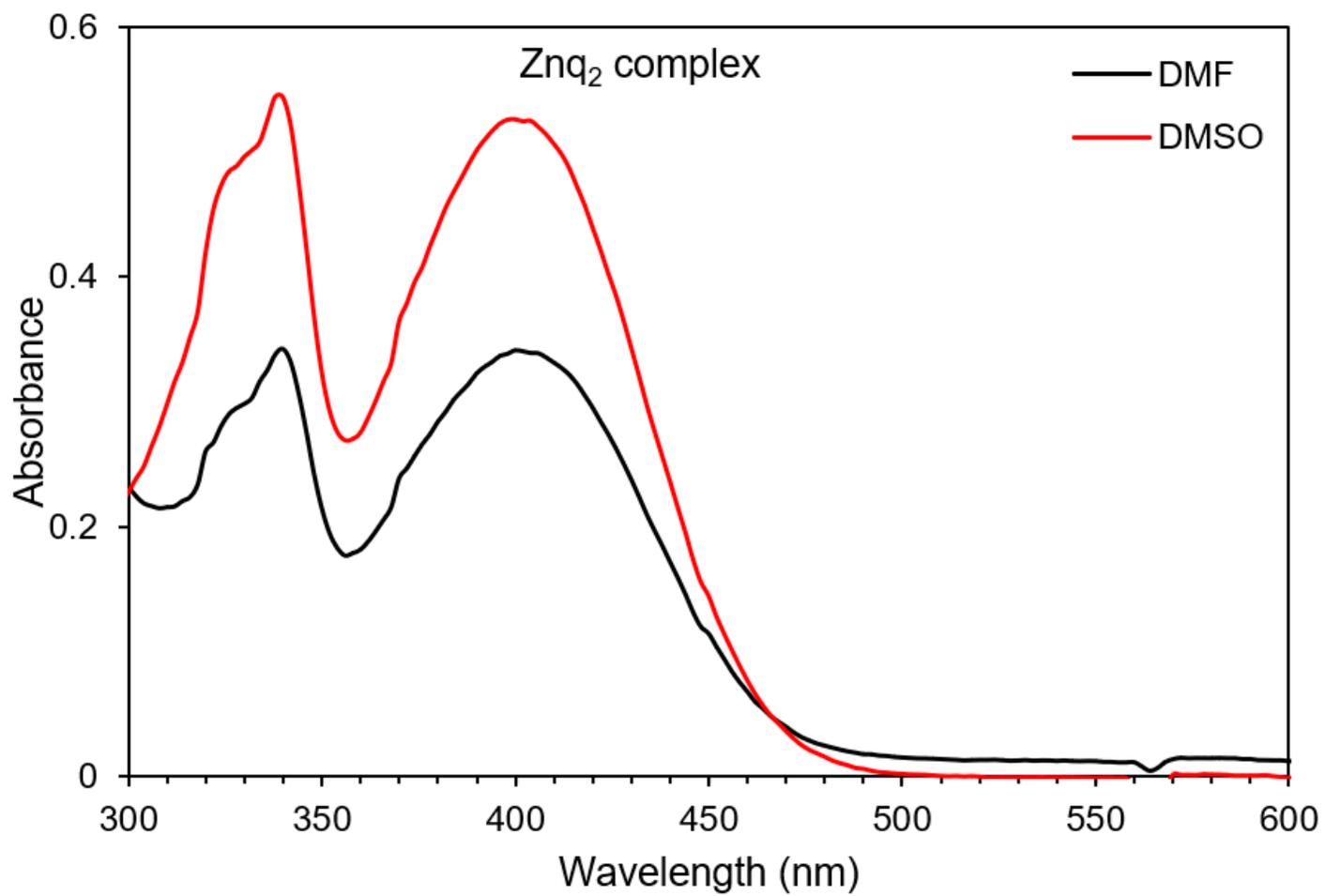


Figure 4

UV-Visible spectra of Znq₂ complex in DMF and DMSO solvents

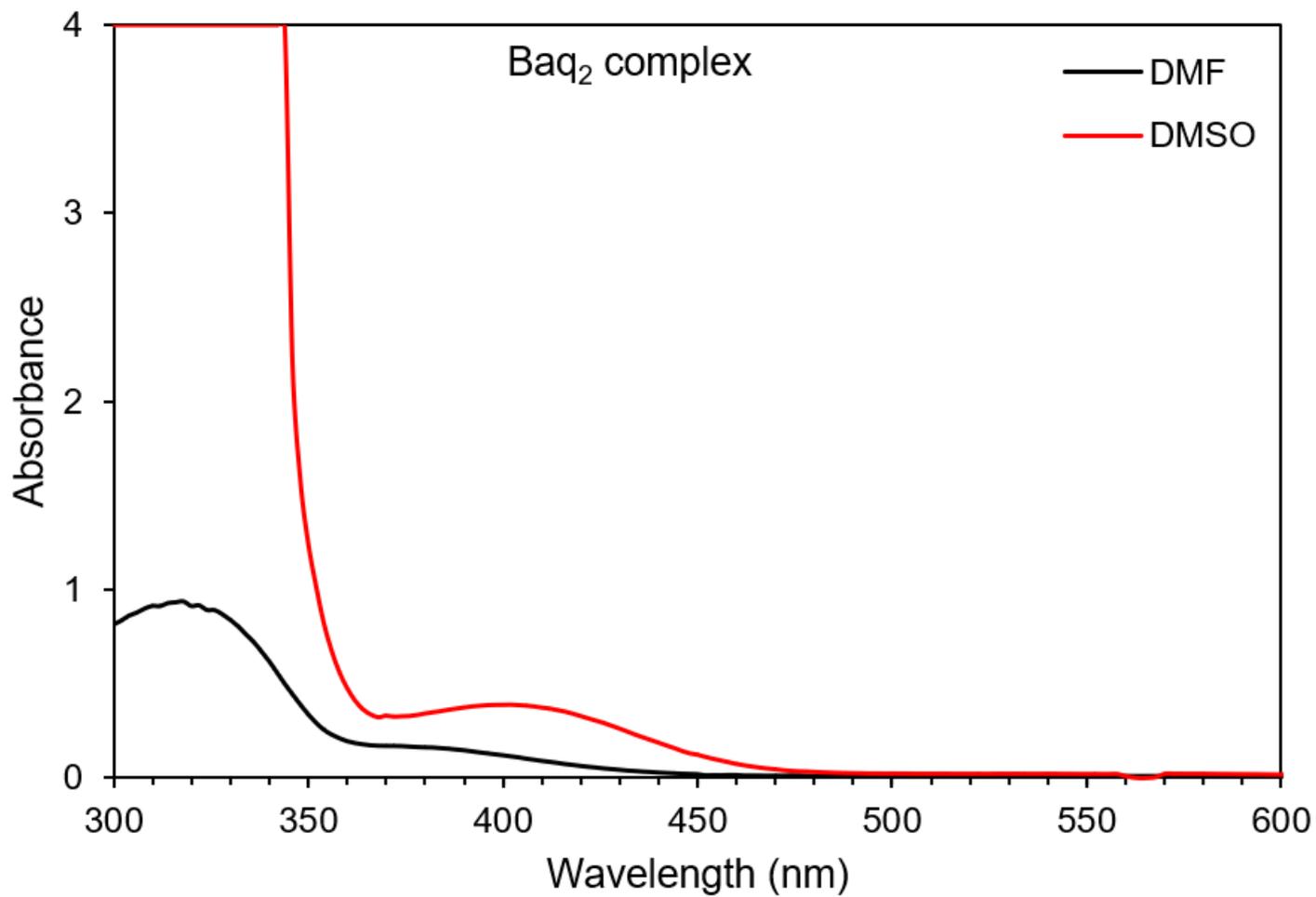


Figure 5

UV-Visible spectra of Baq₂ complex in DMF and DMSO solvents

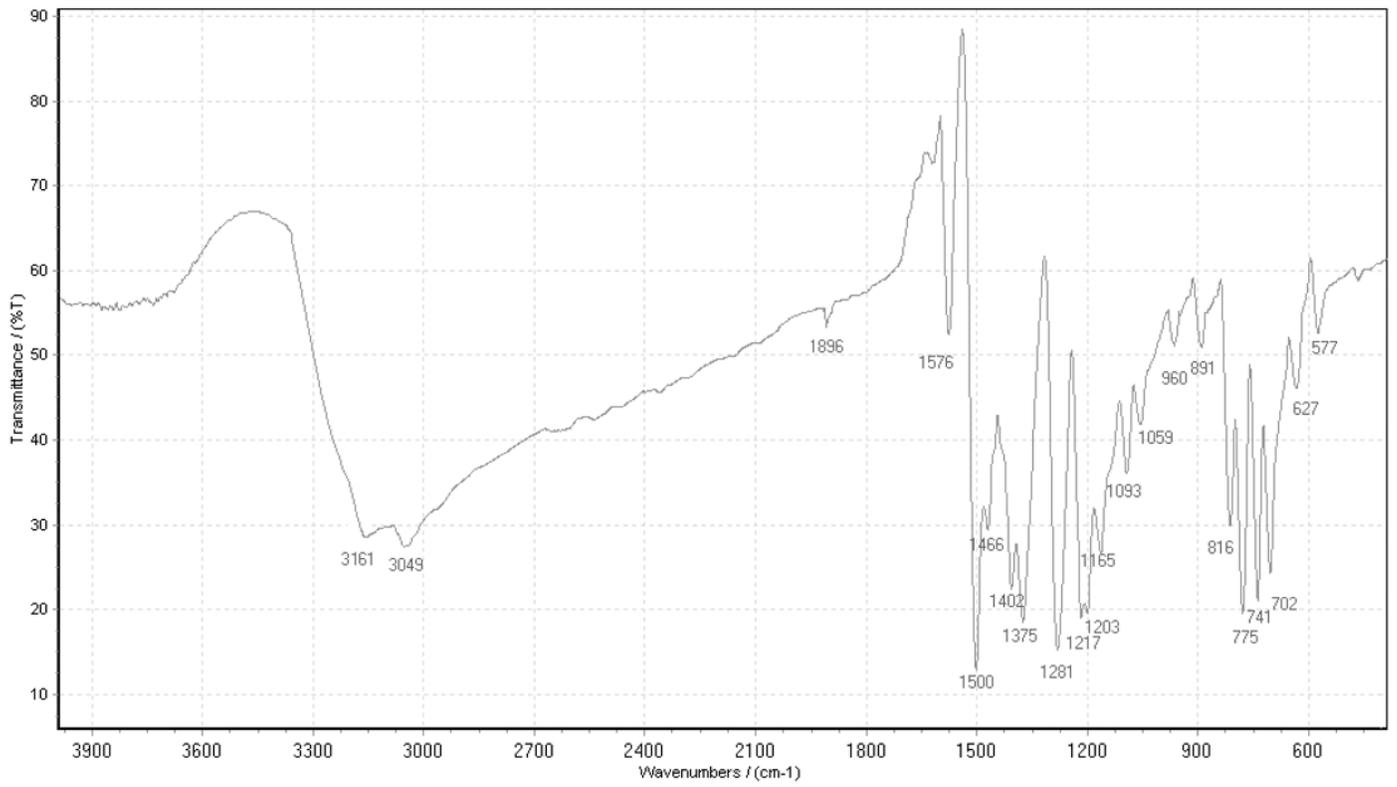


Figure 6

FT-IR spectrum of ligand 8-Hydroxyquinoline

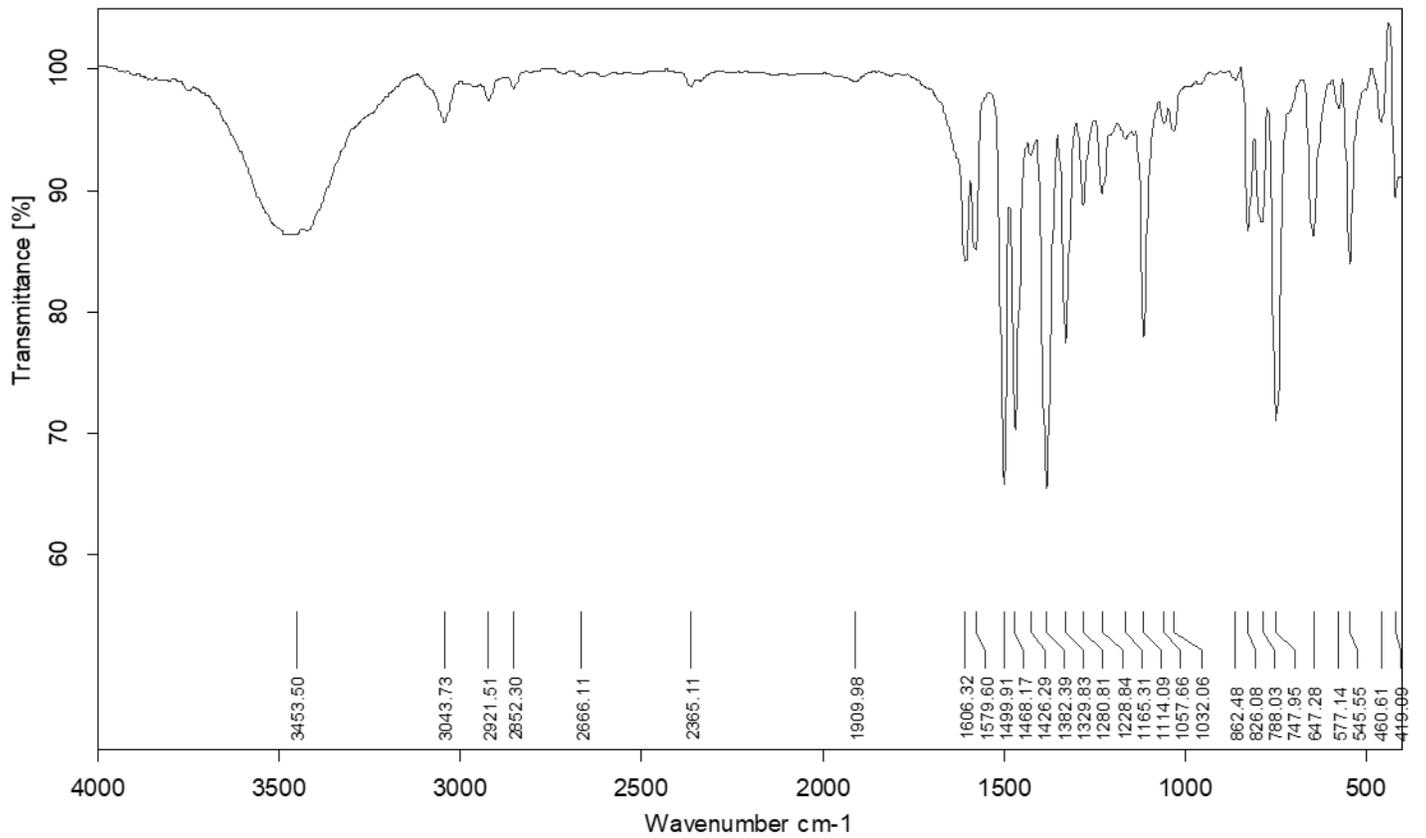


Figure 7

FT-IR spectrum of Alq3 complex

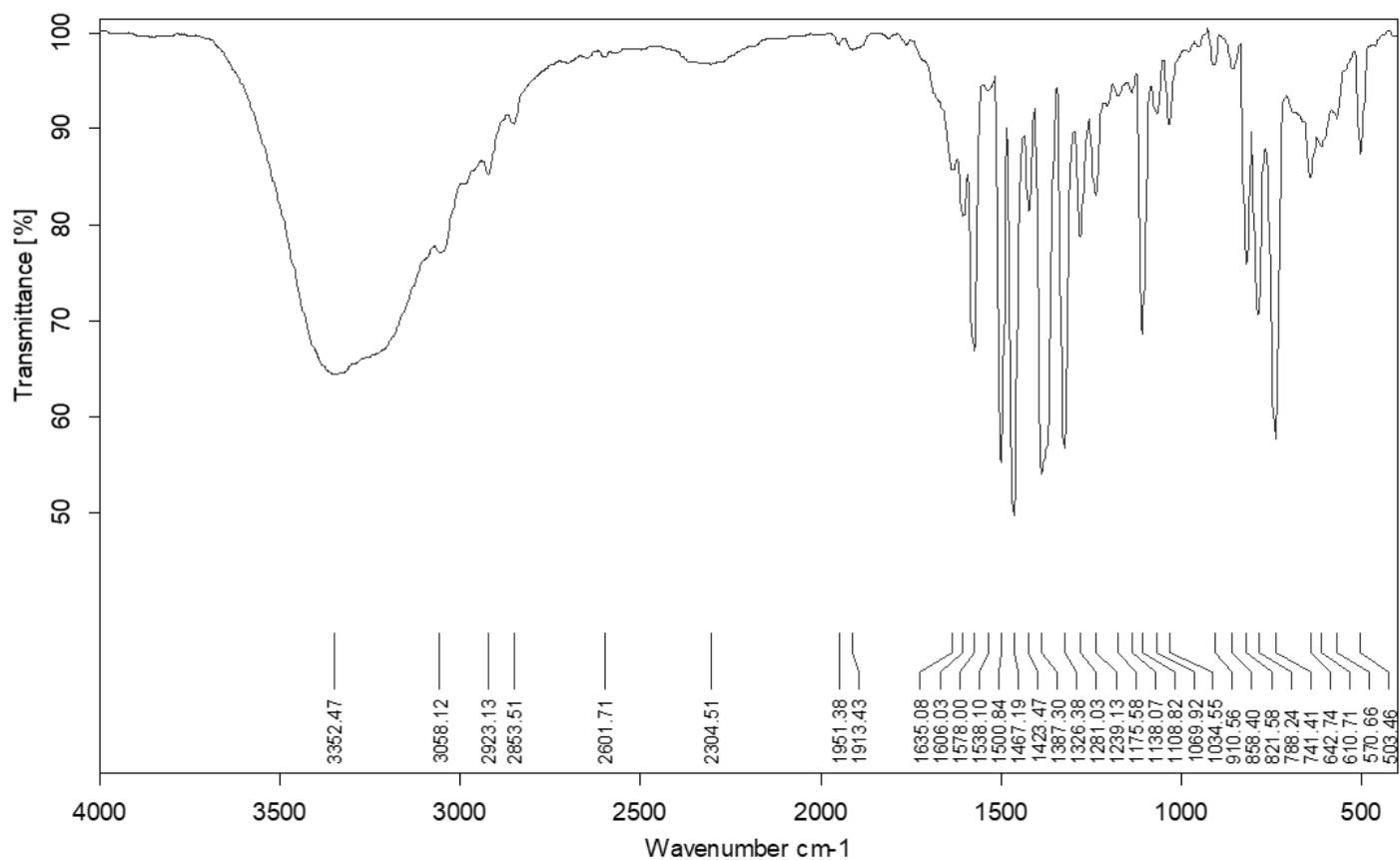


Figure 8

FT-IR spectrum of Znq2 complex

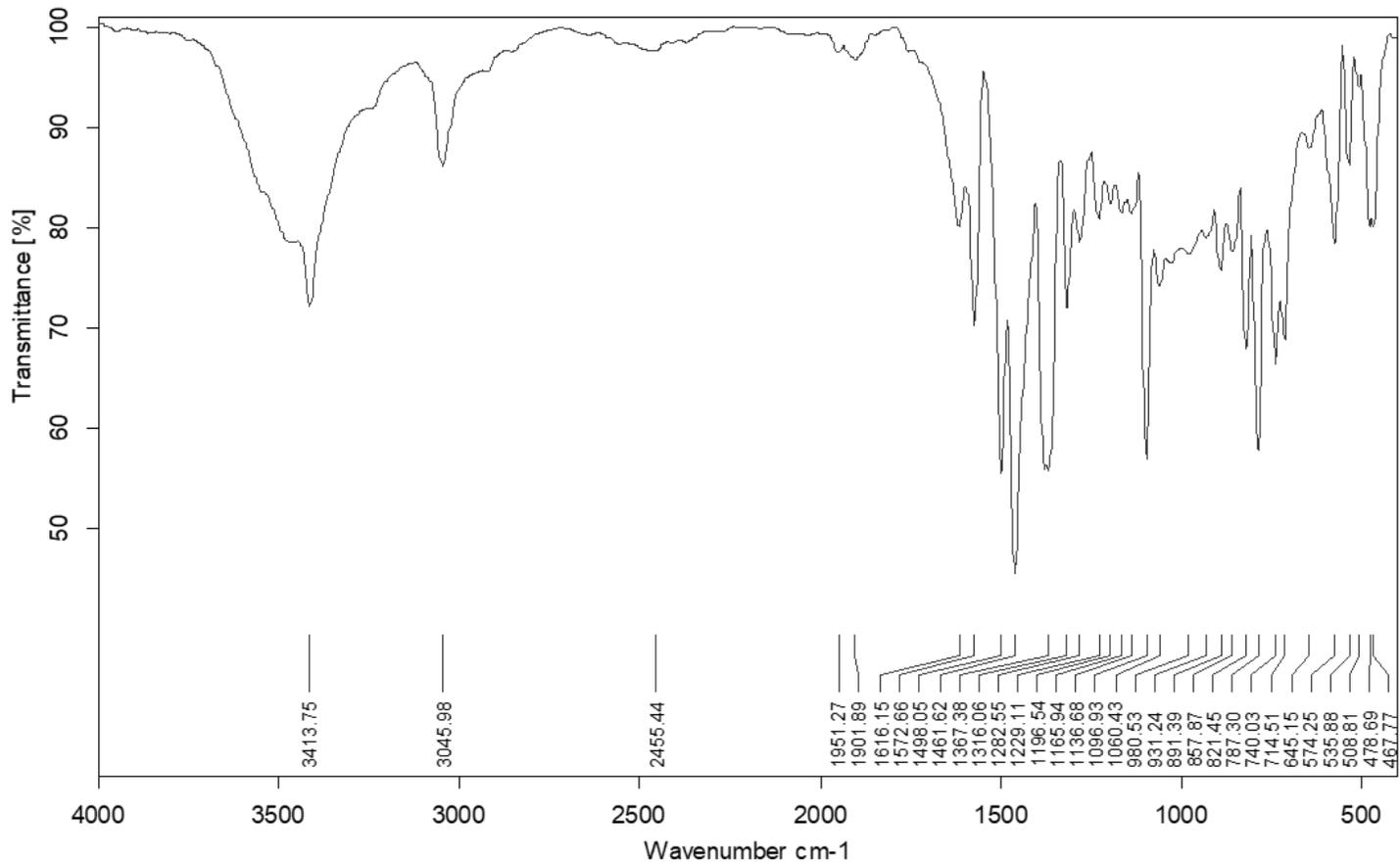
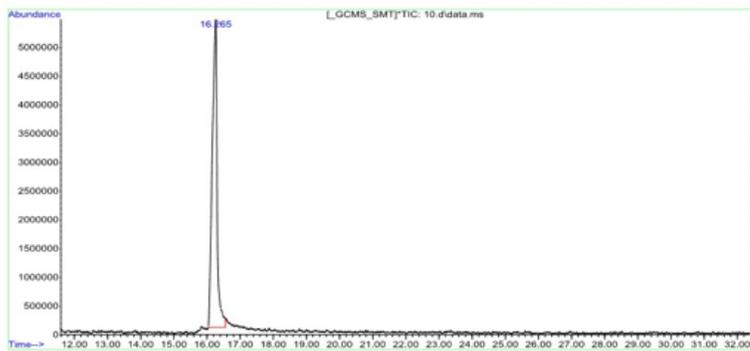
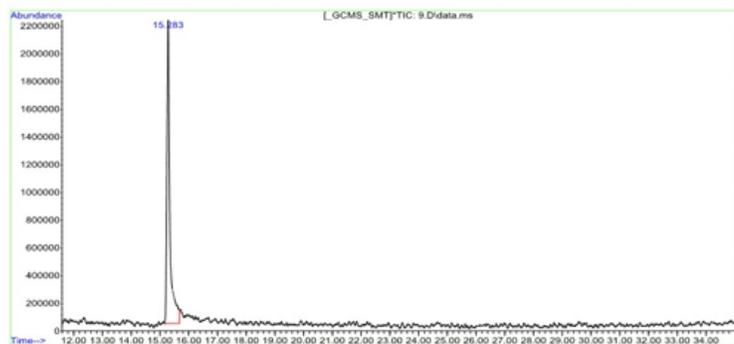


Figure 9

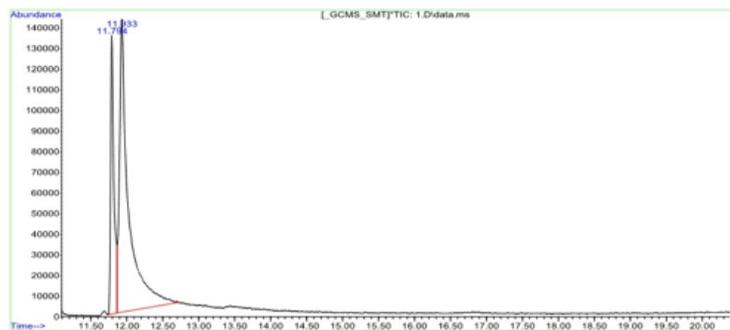
FT-IR spectrum of Baq2 complex



(a)



(b)



(c)

Figure 10

GC-MS spectrum of (a) Alq3 complex, (b) Znq2 complex, and (c) Baq2 complex