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*Full Length Research Paper*

# Spectrophotometric studies of charge transfer complex of 8-hydroxyquinoline with 1,4-benzoquinone

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**The interaction between donor 8-hydroxyquinoline (HQ) and π - acceptor 1, 4-benzoqinone (BQ) has been studied in the solution as well as in the solid state. Infrared spectroscopic data indicate that a charge transfer interaction between the donor and acceptor are due to π → π*/ n → π* transition by the formation of radical ion pairs. In solution state, the formation constant, the extinction coefficient, the free energy and transition energy of the resulting charge transfer complex (CTC) were determined. The optimum experimental conditions, such as time, temperature and solvents for the CTC formation were established.**

**Key words:** 8–Hydroxyquinoline, 1, 4–benzoqinone, the formation constant for the charge transfer complex and infrared spectroscopic.

## INTRODUCTION

Charge transfer complexes have been exclusively studied (Yoshida and Kobayashi, 1970; Ayad, 1997; El-Mossalamy and Ibrahim, 2004; Sharma et al., 2008; Aljaber and Nour, 2008; Al-Qaradawi et al., 2008; Bazzi, 2008; Refat et al., 2010; Khan et al., 2010; Frag et al., 2011) due to their wide application as ion sensors in the field of environmental science and in the determination of drugs based on the charge-transfer (CT) complexes formed with electron acceptors (Gutmann et al., 1968; Karuna et al., 2006). They also can be used as organic semiconductors (Fortner et al., 1992), photocatalysts (Wu et al., 2011), dendrimers (Thomas and Thayumanavan, 2004) and in the studying of redox processes (Krysiñk et al., 1999). More recently, attention has been given to the isolation and investigation of physical properties of some CT-complexes in the solid states. Some of these complexes show interesting electrical conductivity properties and have found applications in many form of electronics and solar cells (Gogulamurali et al., 1992; Parisi et al., 2002; Trotter and White, 1978). Charge transfer interactions formed between 8–hydroxyquinoline and acceptors in different solvents has been studied spectrophoto metrically (Khan et al., 2009; Khan and Ahmed, 2009).

The reactions of 8-hydroxyquinoline (HQ) and 1, 4-benzoqinone (BQ) with number of organic compounds in solid states also have been studied spectroscopyically (Singh et al., 2000; Barreto and Zaia, 1994; Singh et al., 1987; Singh and Singh, 1988). On the other hand, BQ is the parent molecule of a class of a heterocyclic aromatic compounds which play a relevant role in biological systems (Morton, 1965) and in the formation of charge-transfer salts (Zhou et al., 1994; Kuroda et al., 2008), most of the organic conductors based on charge-transfer complexes contain a BQ and its derivatives as the acceptor component (Mansour, 2005). HQ is used as analytical colorimetric agent (Windheuser and Chu, 1967), chelating agent (Chang et al., 2008) and in drugs (Ferrero and Torre, 1986). The present paper is concerned with the results obtained from electronic and absorption measurements on the chemical product formed in the reaction of HQ, as an electron donor and BQ as an electron acceptor. Physical parameters and structure of the reaction product is determined. The aim of this paper is to study the optimum experimental conditions (time, temperature and solvents) for the charge transfer complex formation.

## MATERIALS AND METHODS

HQ and solvents (Aldrich Chemicals Company) were of the highest purity and was used without further purification. HQ: Molecular

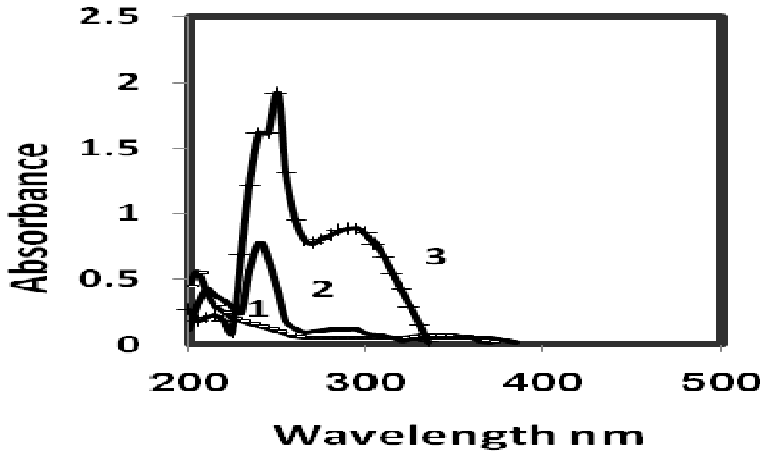



**Figure 1.** Electronic absorption spectra of 1- HQ, 2- BQ and 3- CT complex.

Formula: $C_9H_7NO$, Molecular weight: 145.16, Melting point: 76°C and Appearance: white crystalline needles. BQ: Molecular formula: 1,4-$C_6H_4O_2$, Molecular weight: 108.09, Melting point: 112.0 to 116.0°C and Appearance: yellow crystal. 1, 4–Benzoqinone was purified by sublimation.

**Preparation of standard solutions**

The concentration of HQ in the reaction mixtures was kept fixed at $0.4\times10^{-4}$ M in methylene chloride. Solutions of BQ were changed over a wide range from $0.1\times10^{-4}$ M to $1.2\times10^{-4}$ M in the same solvent.

**Synthesis of the solid CT complex from solution**

The solid CT complex [(HQ) (BQ)] was synthesized by mixing the saturated solution of HQ (1 mmol) in methylene chloride (10 ml) and a saturated solution of BQ (1 mmol) in methylene chloride (10 ml). The mixture was stirred at room temperature for 3 h, a brown solid was formed, and the precipitate was filtered off, washed several times with small amounts (5 ml) of hexane and dried over $CaCl_2$. The product was identified as [(HQ)(BQ)] ($C_{15}H_{11}NO_3$; MW = 253.26 and MP = 210°C), analysis: Calculated (Found), %C: 71.14 (70.94), %H: 4.38 (4.12), %N: 5.53 (5.78).

**Synthesis of the solid CT complex from solid**

HQ (solid) and BQ (solid) were grinding together in the stoichiometric ratio 1:1.The change of the mixture color (brown) has indicated the beginning of the reaction. For a week, it was occasionally mixed during this period. The reaction product washed several times with hexane to remove unreacted components. The product was dried and kept in desiccators.

**Micro structural studies**

A clean glass slide was kept at melting point of HQ in an oven and a small amount of HQ was placed on the slide and immediately melted. The temperature of the oven was lowered and the molten HQ allowed crystallizing in one direction with the help of a cover slip (a very thin layer crystallized). This was examined under microscope and photographed (Singh et al., 2001). The photograph showed a very thin layer of crystallized HQ which represented the microstructure of HQ. In a small beaker BQ was taken and heated so that the beaker was filled with its vapors. A slide of HQ was kept on top of the beaker filled with BQ vapors and covered with a bigger beaker and kept at 70°C for 2 min. HQ reacted with the vapors of BQ and the change in microstructure was photographed.

**Instruments**

Infrared (IR) measurements (KBr discs) were carried out on a Unicam-Mattson 1000 Fourier Transform Infrared (FT-IR) spectrometer. Elemental analyses of Carbon, Hydrogen and Nitrogen (CHN) were measured. All analysis was carried out at the Micro analytical unit in Cairo University. Electronic spectra were recorded at room temperature via both Shimadzu Ultraviolet (UV) Spectrophotometer model 1601 PC with quartz cell of 1 cm path length and Jenway 6405 Spectrophotometer each for certain reactions.

## RESULTS AND DISCUSSION

The reaction products obtained from the solution and the solid state reaction were brown colour. The elemental analysis, IR and the electronic spectra of the products are almost superimposable on each other. Charge-transfer interactions of HQ with BQ were investigated spectrophotometrically and found to form stable complex of [(HQ)(BQ)]. The donor site participated in CT complexation is the lone pair of electron on O atom of BQ. These complexes were easily synthesized from the reaction of HQ with BQ. IR, the elemental analyses, and Ultraviolet/Visible (UV-Vis) techniques characterize the [(HQ) (BQ)] charge transfer complex. The electronic spectra of the donor HQ, acceptor BQ and the resulting complex [(HQ)(BQ)] in methylene chloride were recorded in the visible range 200 to 500 nm (Figure 1). [(HQ)(BQ)] system has definite band at 295 nm due to the formation of CT complex. The nature of the binding of HQ and BQ

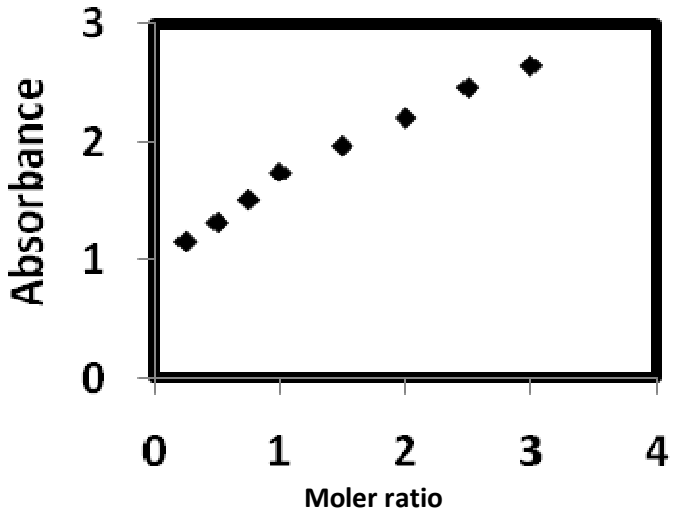



**Figure 2.** Molar ratio for the CT complex.

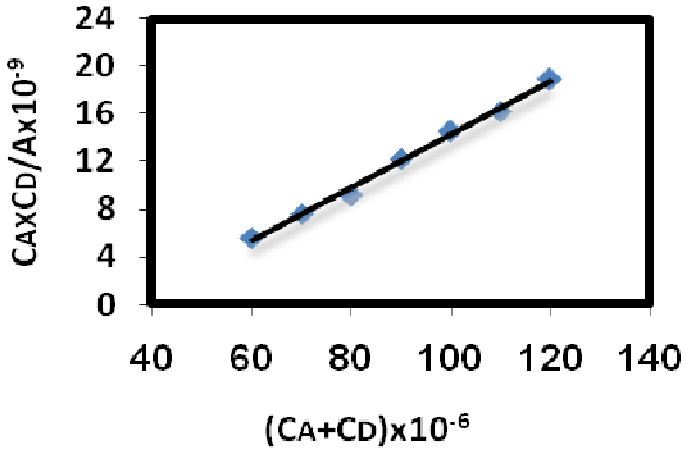



**Figure 3.** Relation between ($C_A$ x $C_D$ /A) and ($C_A$+ $C_D$) for the complex.

was determined by the molar ratio method (Yoe and Jones, 1944) indicated that a 1:1 complexation ratio has been occurred. The absorbance of the formed CT-complexes were measured and plotted as a function of the ratio $C_D$: $C_A$, according to the known method in ratios extending along the range from 1: 0.25 to 1: 3.00 (Figure 2).

The value of the formation constant, $K_{CT}$ ($mol^{-1}$) and the extinction coefficient, $\varepsilon_{CT}$ ($mol^{-1}cm^{-1}$) for the complex formed in solution was determine by using modified Benesi- Hildebrand equation (Benesi and Hildebrand, 1949). This equation can be written as follows:

$$(C_A \times C_D) / A = (C_A + C_D) / \varepsilon_{CT} + 1/ K_{CT}\, \varepsilon_{CT}$$

Where $C_A$ and $C_D$ are the initial concentrations of acceptor and donor, respectively, while A was the absorbance at the mentioned CT bands. On the plotting values of ($C_A$ × $C_D$/A) values against the ($C_A$ + $C_D$) values, straight lines were obtained with a slope of 1/ $\varepsilon_{CT}$ and intercept of 1/K$\varepsilon_{CT}$ (Figure 3). The data obtained throughout this calculation were given in Table 1. The standard free energy of complex in Table 1 can be determined from the formation constant by the following equation (Martin et al., 1969).

$$\Delta G^o = - RT \ln K_{CT}$$

Where $\Delta G^o$ was the free energy of the complex, R is gas constant, T is the temperature in Kelvin degree and $K_{CT}$ was the formation constant of complex. The complex were found to have small value of - $\Delta G^o$ value ($K_{CT}$ < 1,

$$D + A \leftrightarrow \underset{\text{CT complex}}{[D \rightarrow A]} \leftrightarrow \underset{\text{Radical ions}}{D^{+} + A^{-}}$$

**Scheme 1.** Donor-acceptor mechanism in charge-transfer process.

**Table 1.** Physical parameters data of the complex.

| Complex | $\lambda_{CT}$ ( nm) | $K_{CT}$ ($mol^{-1}$) | $\varepsilon_{CT}$ ($mol^{-1}$ $cm^{-1}$ ) | $E_{CT}$ (K cal $mol^{-1}$) | $\Delta G^*$ (K cal $mol^{-1}$) | $\Omega$ (s $cm^{-1}$ ) |
|---|---|---|---|---|---|---|
| [(HQ)(BQ)] | 295 | $27.723\times10^{3}$ | $4.484\times10^{3}$ | 97.14 | 4.99 | 0.72 |

**Table 2.** Effect of solvents on the position of the CT band of the complex in different organic solvents at 25°C.

| Solvent | Dielectric constant | $\lambda_{CT}$ (nm) | $E_{CT}$ (K cal $mol^{-1}$) |
|---|---|---|---|
| Chloroform | 4.73 | 290 | 98.61 |
| Methylene chloride | 9.10 | 295 | 96.94 |
| Acetone | 20.70 | 330 | 86.66 |
| Methanol | 24.00 | 310 | 92.25 |

$\Delta G^{o} < 1$), indicating a weak nature of the complex and spontaneous reaction between $D^{+}$ and $A^{-}$ (Scheme 1).

The values transition energy reported in Table 1 was calculated from CT band of the electronic applying the following equation (Briegleb, 1964).

$$E_{CT} = 1243.667 / \lambda_{CT}\ \text{nm}$$

## Conductivity

The resulting [(HQ)(BQ)] solution exhibit appreciable conductance (Table 1) which may be explained by the possible formation of charge transfer complex between the reaction partners in solution (Bhowmik and Bhattacharyya, 1988; Ramadan et al., 1991; Yakuphanoglu and Arslan, 2004; Al-Shuja'a et al., 2011).

## Effect of solvent

The wavelength of the CT band of the complex between HQ and BQ was measured in a number of solvents (Table 2). In the case of the oxygen containing solvents (methanol and acetone), no correlation appears between the polarity of the solvents and the transition energy. In free oxygen solvents (methylene chloride and chloroform), the transition energy of the molecular complex decreases as the polarity of the solvent increases. Accordingly, the transition energy of the molecular complex is inversely proportional to the polarity of the free oxygen solvents. The role of the polarity of the solvents arises from the stabilization of the excited state of the molecular complex through dipole - dipole or dipole - induced dipole interactions. Accordingly, the transition energy of the molecular complex is inversely proportional to the polarity of the no oxygenated solvent (Nour-El-Din and Mourad, 1983). The studies show that methylene chloride was the best solvent for the highest absorption intensity.

## Effect of reagent concentration, temperature and time

Under the optimum conditions, a correlation was obtained between absorbance (A) and the concentration (C) over the range 0.1 to $1.5\times10^{-4}$ M (Figure 4). It has been found that Beer's law was obeyed up to $0.6\times10^{-4}$ M. In order to select the suitable temperature for CT complex, it has been made at different temperatures ranged from 5 to 45°C. The absorbance of CT complexes is measured at the suitable wavelength. The effect of temperature on these CT complexes is shown in Figure 5. It has been observed from the given results that the absorbance attains a maximum absorbance at 25°C, thus the room temperature was selected as the optimum temperature. At temperature 25°C, the optimum reaction time is determined by following the color development spectrophotometrically. It is attained after 30, 35 and 45 min for HQ to BQ complex (Figure 6). The results obtained indicated that the developed colours remained stable at room temperature for at least 30 min.

## Micro structural studies

When a molten material is allowed to crystallize on a glass slide (only a very thin layer) in one direction, the structure observed under an optical microscopy is known

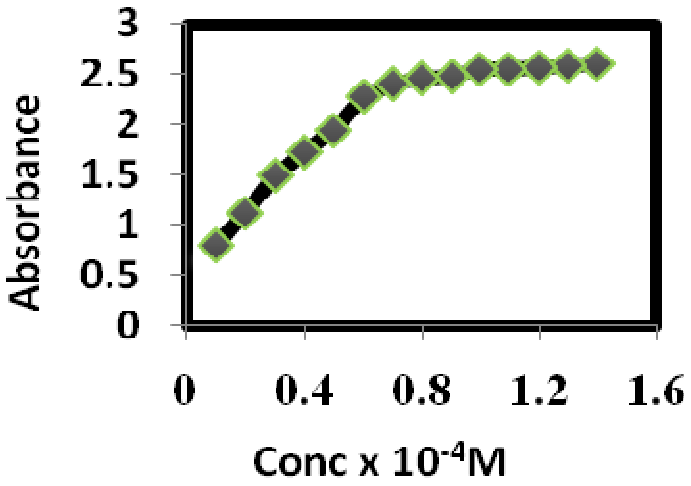



**Figure 4.** Effect of concentration.

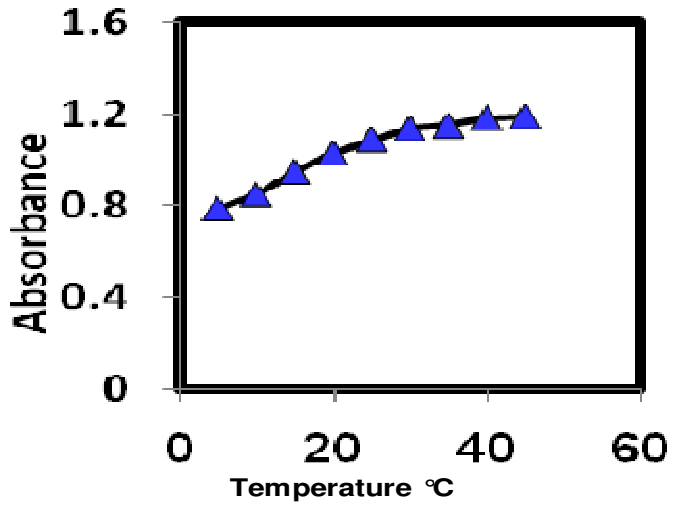



**Figure 5.** Effect of temperature.

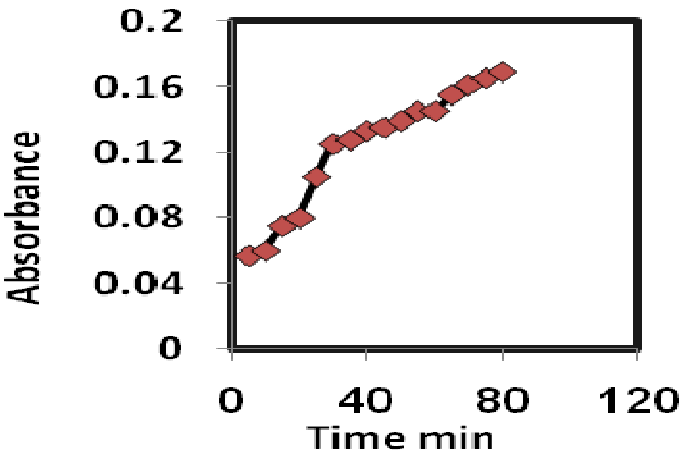



**Figure 6.** Effect of time on complex.

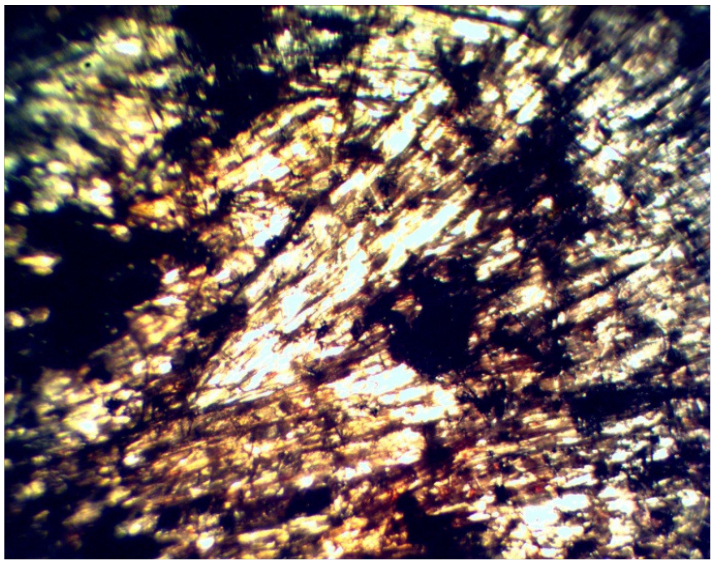


**Figure 7.** Microstructure of HQ exposed to vapors of BQ.

**Table 3.** Infrared frequencies and tentative assignments for HQ, BQ and the CT complex.

| Compound | υ (OH) | υ (C=O) + υ (C=C) | Hydrogen bond |
|---|---|---|---|
| BQ | | 1680 vs<br>1410 vs<br>1311 ms<br>1220 vs | |
| HQ | 3042 s, br | 1577 ms<br>1500 vs<br>1473 w<br>1376 ms<br>1274 vs | |
| HQ - BQ | 3410 s, br | 1645 ms<br>1499 ms<br>1455 w<br>1411 w<br>1375 w | 2363 v |

as the microstructure of the material. The slide of HQ was made and exposed to vapors of BQ. The structural changes are given in Figure 7, shows that HQ crystallizes from one point and spreads over in all the directions. When the slide covered with HQ was allowed to react with the vapors of BQ, reaction started at the central point from which crystallization started and spread in other directions (Rastogi et al., 1977; Singh et al., 1994).

## Infrared spectra

IR spectra of the characterized bands of HQ and BQ and [(HQ)(BQ)] were represented in Table 3. The formation of CT complex were associated by hydrogen bonding between one O atom of BQ and H atom of the OH group of HQ. However the majority of evidence suggested that the oxygen atom is preferred as a donation (n-π* interaction) (Gölcü et al., 2000; Singh et al., 2003; Refat et al., 2006). The shift of the bands of the acceptor to lower wavelength and those of the donor part to higher values reflects a donor to acceptor charge transfer of π-π*interaction, that is, $D_{HOMO} \rightarrow A_{LUMO}$ transition. On the basis of the results of complex obtained by elemental analysis, UV visible and IR analysis (Figure 8), the possible structure of the synthesized CT complex may

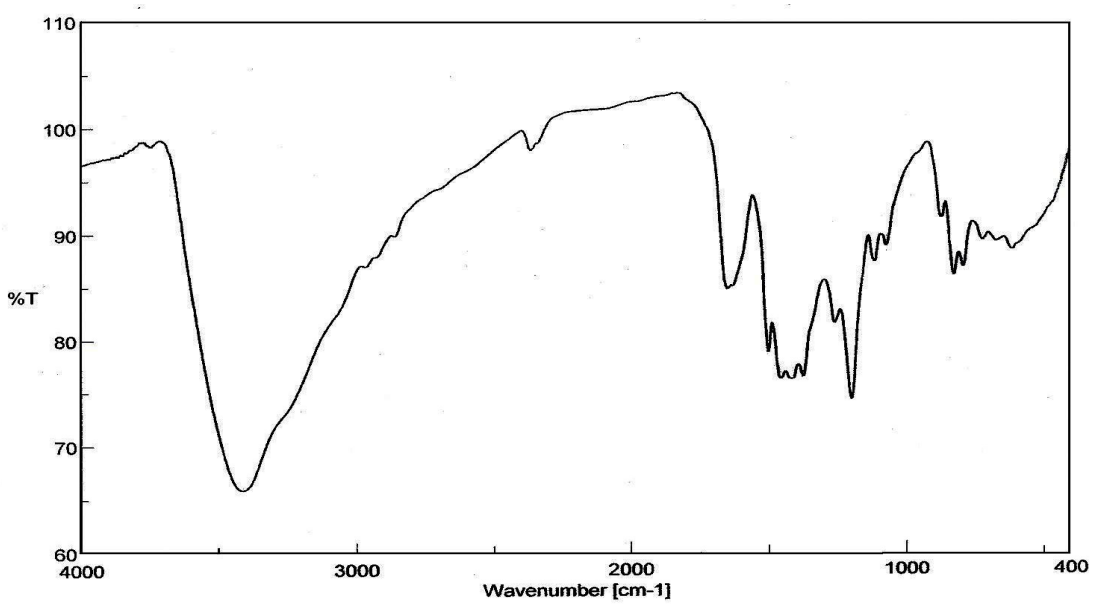
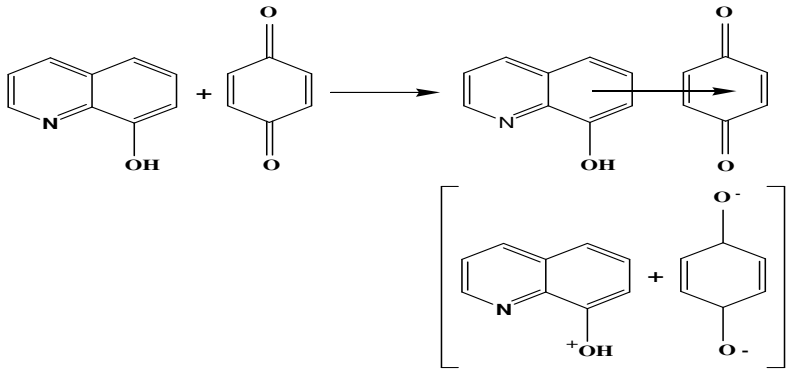



**Figure 8.** Infrared spectra of [(HQ)( BQ)] complex.




**Scheme 2.** Charge transfer transition between donor and acceptor.

be represented in Scheme 2

## Conclusion

The interactions between the electron donor HQ and the acceptor BQ was studied spectrophotometrically in methylene chloride. A new charge transfer complex was isolated and characterized through elemental analysis, infrared and electronic spectra. The stoichiometry of the product was found to be 1:1. Accordingly, the formed CT-complex have the formula [(HQ)(BQ)]. Benesi-Hildebrand and its modification methods were applied to the determination of formation constant $K_{CT}$, and molar extinction coefficient ($\varepsilon_{CT}$). The free energy and transition energy of the resulting CTC were determined. The best solvent, time and temperature for the complex formation were established. In solid state, reaction proceeds by the diffusion of BQ into the crystal lattice of HQ.

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