Research Article

Investigation of Charge Transfer Complexes Formed between Mirtazapine and Some π -Acceptors

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Charge transfer complexes (CTC) of mirtazapine with tetracyanoethylene (TCNE), 2,3-dichloro-5,6-dicyano-*p*-benzoquinone (DDQ), and tetracyanoquinodimethane (TCNQ) have been studied spectrophotometrically in dichloromethane at room temperature. The stoichiometries of the complexes were found to be 1 : 1 ratio by the Job Method between mirtazapine and the acceptors. The equilibrium constants and thermodynamic parameters of the complexes were determined by the Benesi-Hildebrand and Van't Hoff equations. Mirtazapine in pure and dosage form was applied in this study. The results indicate that the formation constants for the complexes depend on the nature of electron acceptors and donor. And also the spectral studies of the complexes were determined by FT-IR and NMR spectroscopy.

1. Introduction

Mirtazapine(1,2,3,4,10,14b-hexahydro-2-methylpyrazino[2,1-a]pyrido[2,3-c][2]benzazepine) is the piperazino-azepine group of compound which has antidepressant therapeutic effects. It is a tetracyclic noradrenergic and specific serotonergic antidepressant which act as an antagonist of central presynaptic α 2-adrenergic autoreceptors and heteroreceptors as well as a potent antagonist of postsynaptic 5-HT2 and 5-HT3 receptors [1–3]. It also apparently causes a net activation of primarily 5-HT1A receptors [4]. Furthermore, mirtazapine is an antihistaminergic agent with a high affinity for histamine H1 receptors and manifests a very low affinity for dopaminergic receptors [5], and the chemical structure of mirtazapine is shown in Scheme 1.

Charge transfer complexation is an important phenomenon in biochemical and bioelectrochemical energy transfer process [6]. The electron donor-acceptor interactions have been widely studied spectrophotometrically in the determination of the drug based on the CT Complexes formation with some π -acceptors [7–9]. The interactions of the charge transfer complexes are well known in many chemical reactions such as addition, substitution, and condensation [10, 11]. The molecular interactions between electron donors and acceptors are generally associated with the formation of

intensely coloured charge transfer complexes, which absorb radiation in the visible region [12]. Electron donor-acceptor CT interactions are also important in the field of drug-receptor binding mechanism [13], in solar energy storage [14], and in surface chemistry [15] as well as in many biological fields [16]. On the other hand, the CT-reactions of π -acceptors have successfully been utilized in pharmaceutical analysis [17] and non-linear optical properties [18, 19].

In continuation of our studies of charge transfer complexes [20–22], this paper reports simple, direct, and sensitive spectrophotometric method for the determination of mirtazapine with some π -acceptors such as TCNE, DDQ, and TCNQ. Mirtazapine was used as drug both in dosage and pure form. Stoichiometries, equilibrium constants, and thermodynamic parameters of the complexes were determined. And also, the CTC of the mirtazapine- π -acceptors were determined by FT-IR and NMR spectroscopy.

2. Experimental

2.1. Materials and Spectral Measurements. The materials used in this study were obtained from local suppliers; TCNE (Merck), DDQ (Merck), TCNQ (Merck), mirtazapine tablets (Remeron Drage, Santa Farma Drug Company, Turkey).

SCHEME 1: Chemical structure of mirtazapine.

Dichloromethane (Merck) was redistilled before using. All laboratory reagents were freshly prepared.

The electronic absorption spectra were recorded in the region 900–200 nm using Shimadzu 2401 UV-Vis spectrophotometer with a quartz cell of 1.0 cm path length. The infrared spectra of the isolated complexes and the reactants were measured as a solid sample on Shimadzu IR Prestige 21 model FT-IR. ^1H NMR spectra were obtained by Varian 300 MHz Infinity Plus using CDCl_3 as the solvent.

2.2. Preparation of Standard Solutions

2.2.1. Acceptors. A stock solution of acceptors at a concentration of 1×10^{-2} M was prepared in different volumetric flasks by dissolving 12.8, 22.7, 20.4 mg of TCNE, DDQ, and TCNQ powder accurately weighed in dichloromethane and making up to 10 mL with the same solvent.

2.2.2. Mirtazapine. A standard solution of mirtazapine was prepared by dissolving 26.5 mg of pure mirtazapine in a 10 mL volumetric flask using dichloromethane.

2.2.3. Absorption Spectra. A 2 mL volume of mirtazapine and acceptors were scanned separately through a UV-Vis spectrophotometer to their wavelength of maximum absorption. When 2 mL of acceptor solution and 2 mL of donor solution were mixed, a color charge transfer complex was formed. The wavelength of maximum absorption of the resulting solution was determined by the spectrophotometer.

2.3. Stoichiometries of the Complexes. Job's method of continuous variations was used to determine the stoichiometries of the complexes [23, 24]. Master solutions of equimolar concentrations of the drug and acceptor in dichloromethane were used in this experiment. Aliquots of the solutions were varied alternately from 0.2 to 0.8 mL for donor and acceptor solutions to hold the total volume at 1 mL in the cuvette by using a 1 mL pipette. Average absorbances were obtained from three runs on the same sample and average values at 790–800 nm were subtracted from the average values at the maxima. The complex for the each reaction mixture was kept at 10 minutes at room temperature to form stable complexes before scanning.

2.4. Determination of Equilibrium Constants. Benesi-Hildebrand equation [25] was used for the determination of the equilibrium constants of the complexes. 0.53 mg of mirtazapine was weighed in the cuvette and added in 2 mL of 3×10^{-4} M acceptor solution. Then, each time 0.2 mL of 3×10^{-4} M acceptor solution was added in cuvette and absorption values were obtained at indicated wavelengths. After adding each time, waited for 10 min for getting stable complex. The UV-Vis spectrum was measured after each addition of 0.2 mL of solution. About 10 dilutions were run with each sample.

2.5. Thermodynamic Constants. The thermodynamic constants of the complexes between donor and acceptor were determined by Van't Hoff equation. 1.5 mL of 10^{-2} M mirtazapine and 1.5 mL of 10^{-2} M of acceptor from the stock solution were mixed and absorbances were obtained at the five different temperatures such as 7, 14, 21, 28, and 35°C. The thermodynamic parameters (ΔH° , ΔS° and ΔG°) were calculated by plotting $\ln(\text{Abs})/\epsilon - \ln(D_0 - \text{Abs}/\epsilon)/(A_0 - \text{Abs}/\epsilon)$ versus $1/T$ (°K).

2.6. Mirtazapine Tablets. Forty Remeron tablets were finely powdered and amount equivalent to 40 mg mirtazapine was accurately weighed. Transferred to a beaker containing 10 mL of dichloromethane and shaken for a while to dissolve the drug. Then, the solution was filtered to the 10 mL of volumetric flask and filled by dichloromethane to provide a theoretical 10^{-2} M solution of mirtazapine. 2 mL of acceptor solution was added to 2 mL of drug solution. The absorbance was determined 418, 708, and 850 nm with TCNE, DDQ, and TCNQ, respectively.

3. Results and Discussion

The absorption spectra of solutions containing donor and acceptors together exhibit new absorptions at longer wavelength than either the donors ($\lambda < 350$ nm) or the acceptors ($\lambda < 450$ nm) alone.

A solution of TCNE, DDQ, and TCNQ in dichloromethane had cream, orange, and yellowish-green color with the maximum wavelength lower than 450 nm, respectively. Yellow, brick red, and dark green colors were obtained on the interaction of TCNE, DDQ, and TCNQ acceptor solutions, respectively. The colorless solution of mirtazapine in dichloromethane was changed to colored solution, and it mentions a charge transfer complex formation. Scanning of the complex in the visible range between 400 and 900 nm showed the maximum peaks at 418, 708, and 850 nm, respectively, and the spectra are shown in Figure 1.

During the complexation, charge transfer transitions occur with the excitation of an electron from HOMO of the donor to LUMO of the acceptor. This is shown schematically in Scheme 2 in which $h\nu_{\text{CT}}$ depicts the energy of the CT transitions. The lowest energy CT transition will involve promotion of an electron residing in the high occupied molecular orbital (HOMO) of the donor to the acceptor

FIGURE 1: Mirtazapine (1) and charge transfer complexes of mirtazapine with TCNE (2), DDQ (3), and TCNQ (4) in dichloromethane at 21°C.

SCHEME 2: Charge transfer transitions for HOMOs of the donor and LUMOs of the acceptor.

as shown for $h\nu_{CT}$. Charge transfer transitions involving electrons in lower energy orbitals also are possible and would result in higher energy CT transitions as shown $h\nu_{CT}^1$.

The interactions between mirtazapine and π -acceptors give $n\text{-}\pi^*$ transitions and form radical ion pairs, such as radical cation and radical anions. Charge transfer transition reaction is shown in Scheme 3.

The stoichiometries of the complex formation were determined by Job's method of continuous variation and indicated as 1 : 1 ratio shown in Figure 2.

The formation constants (K_{CT}) and molar extinction coefficient (ϵ_{CT}) values of mirtazapine- π -acceptors (TCNE, DDQ, TCNQ) CT complexes were studied in dichloromethane at 21°C. The Benesi-Hildebrand equation was used for the calculations [25] and shown below:

$$\frac{[A_0]}{ABS} = \frac{1}{K \epsilon [D_0]} + \frac{1}{\epsilon}, \quad (1)$$

where $[D_0]$ is the concentration of the donor; $[A_0]$ is the concentration of the acceptor; ABS is the absorbance of the complex; ϵ is the molar absorptivity for the complex; K is the association constant of the complex.

Straight lines were obtained (Figure 3) by plotting the values $[A_0]/ABS$ versus $1/[D]$ and the results shown in Table 1 revealed that the K values of charge transfer complexes with

FIGURE 2: The plot of Job's method for mirtazapine with TCNE (+), DDQ (Δ), and TCNQ (O).

TCNQ are higher than the corresponding values with TCNE and DDQ. This is consistent with the decrease in electron affinity of TCNE relative to DDQ. On the other hand, the results indicate that the electron accepting ability of TCNQ is higher than that of DDQ and also electron accepting ability of DDQ is higher than that of TCNE. TCNQ has four strong electron withdrawing groups in conjugation with an aromatic ring which causes high delocalization leading to an increase in the lewis acidity of the acceptor. The results are compatible with the literature [26].

Thermodynamic parameters (ΔH° , ΔS°) of the CT complexes of mirtazapine with TCNE, TCNQ, and DDQ were determined from Van't Hoff and Beer-Lambert equations

$$\ln \frac{ABS}{\epsilon} = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \ln \left(\frac{[D_0 - ABS]}{\epsilon} \right) \ln \left(\frac{[A_0 - ABS]}{\epsilon} \right). \quad (2)$$

The slope of the plot was used to calculate enthalpies (ΔH°) and relative entropies (ΔS°) from the intercept of the plot and shown in Figure 4.

ΔG° values of the complexes were calculated from Gibbs free energy of formation according to the equation given below:

$$\Delta G^\circ = -RT \ln K_{CT}, \quad (3)$$

where ΔG° is the free energy of the charge transfer complexes; R , the gas constant ($1.987 \text{ cal mol}^{-1} \text{ } ^\circ\text{C}$); T , the temperature in Kelvin degrees; K_{CT} , the association constant of donor-acceptor complexes (Lmol^{-1}). The ΔH° , ΔS° , and ΔG° values of the complexes are given in Table 2.

The obtained results reveal that the CT complex formation process is exothermic and spontaneous. There is a good agreement with the literature values of the constants. When

TABLE 1: Formation constants of the complexes of mirtazapine with TCNE, TCNQ, DDQ in dichloromethane at 21°C.

Acceptors-drug	K_{CT} (L·mol ⁻¹)	Stoichiometry	r^2	λ max (nm)
TCNE	111	1 : 1	0.999	418
DDQ	199	1 : 1	0.998	708
TCNQ	220	1 : 1	0.996	850

SCHEME 3: The molecular structures of compounds and charge transfer transition between donor and acceptors.

FIGURE 3: Benesi-Hildebrand plots for mirtazapine with TCNE (+), DDQ (Δ), and TCNQ (O).

FIGURE 4: Van't Hoff plot for mirtazapine with TCNE (+), DDQ (Δ), and TCNQ (O) at 7, 14, 21, 28, and 35°C.

TABLE 2: Thermodynamic parameters of the complexes of mirtazapine with TCNE, DDQ, and TCNQ in dichloromethane at 7, 14, 21, 28, 35 °C for ΔS° , ΔH° and 21 °C for ΔG° .

Acceptors drug	ΔH° (cal mol ⁻¹)	ΔS° (Eu)	ΔG° (cal mol ⁻¹)	r^2
TCNE	-467	-164	-2751	0.996
DDQ	-3134	-2718	-3092	0.990
TCNQ	-3751	-3536	-3151	0.997

FIGURE 5: FTIR spectra of mirtazapine (a), mirtazapine-DDQ CT complex (b), mirtazapine-TCNE CT complex (c), mirtazapine-TCNQ CT complex (d) in the range 4000–600 cm⁻¹.

increasing electron affinity of acceptors, the values of the constants increases [27].

Infrared spectra of the electron donor (mirtazapine) and its CT complexes with using acceptors such as TCNE, DDQ, and TCNQ are shown in Figure 5. In the spectra of the CT complexes, each spectrum shows almost the main characteristic bands for both the donor and acceptor in each

case. This observation strongly supports the formation of the CT interactions between donor and acceptors. However, the bands of the donor and acceptors in these complexes reveal small shifts in both band intensities and wavenumber values from those of the free molecules. This is normal due to the expected changes of molecular symmetries and electronic structures of the reactants upon complexation.

FIGURE 6: ^1H NMR spectra of mirtazapine (a), mirtazapine-TCNE CT complex (b), mirtazapine-DDQ CT complex (c), mirtazapine-TCNQ CT complex (d).

For example, the $\nu(\text{CN})$ vibrations of TCNE alone are observed as a triplet at 2262, 2229, and 2214 cm^{-1} and the $\nu(\text{CN})$ vibrations of DDQ and TCNQ alone are observed at 2223 cm^{-1} and 2234 cm^{-1} , respectively. These vibrations occur at 2196, 2210, and 2193 cm^{-1} after complexation by mirtazapine-TCNE, mirtazapine-DDQ, and mirtazapine-TCNQ, respectively. Similar changes are also observed for the $\nu(\text{C}=\text{C})$ vibrations for each π -acceptors (TCNE, DDQ, and TCNQ) upon complexation. The $\nu(\text{C}=\text{C})$ of alone TCNE is 1502 cm^{-1} shifted to 1565 cm^{-1} upon complexation and the $\nu(\text{C}=\text{C})$ vibrations after complexation by DDQ and TCNQ were shifted from 1686 to 1565 cm^{-1} and from 1626 to 1541 cm^{-1} , respectively. The changes of the wavenumber values upon complexation is clearly associated with that the electron donation from mirtazapine is expected to go to the empty π^* orbitals of acceptors. Same kind of results such as shifting wavenumber values after complexation were observed in the literature [28, 29].

^1H NMR spectra of the electron donor (mirtazapine) and its CT complexes with using acceptors such as TCNE, DDQ, and TCNQ are shown in Figure 6. ^1H NMR of mirtazapine and the formed CT complexes were carried out in CDCl_3 . The

^1H NMR spectrum of mirtazapine shows the proton on C14b of piperazine ring of mirtazapine at δ 3.38 ppm as a dublet. In the ^1H NMR spectrum of mirtazapine-TCNE complex, the peak was shifted to δ 3.97 ppm as dublet. The ^1H NMR spectrum of mirtazapine-DDQ complex, the peak was found at δ 4.05 ppm as dublet. Similarly, the ^1H NMR spectrum of mirtazapine shows methylene protons on C10 of azepine ring in structure of mirtazapine at δ 4.54–4.49 and 4.36–4.32 ppm as dublet-dublet. In the ^1H NMR spectrum of mirtazapine-TCNE complex, these peaks were found between δ 4.56 and 4.46 ppm as one within the other. By studying the ^1H NMR spectrum of mirtazapine-DDQ complex, these peaks were found at δ 4.87–4.91 and 4.42–4.46 ppm as dublet-dublet. The ^1H NMR spectrum of mirtazapine-TCNQ complex shows similar results. Likewise, peaks of other methylene protons are shifted down field to higher ppm values and it confirms clearly that charge transfer complexes were formed.

4. Conclusions

In conclusion, the spectroscopic methods have advantage of being simple, sensitive, accurate, and suitable for routine analysis in laboratories. The methods used here are a single-step reactions and single solvent. Dichloromethane was used here as a solvent to avoid any interactions of solvent with donor and acceptors. The methods can be used as general methods for the spectrophotometric determination of drugs in bulk powder and in commercial formulations.

Conflict of Interests

The authors declare that they have no conflict of interests.

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