DOI: 10.17344/acsi.2021.6990 *Acta Chim. Slov.* **2022,** *69,* 73–80

Scientific paper

Synthesis of the New 1-(7-Methoxy-1-benzofuran-2-yl)-3-(4-methylphenyl)prop-2-en-1-one and Controlling of Spectroscopic, Optical and Conductivity Properties by Concentration

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Received: 06-04-2021

Abstract

Chalcone derivatives have an important place in science due to their different applications ranging from their semiconductor properties to biological properties. In this work 1-(7-methoxy-1-benzofuran-2-yl)-3-(4-methylphenyl)prop-2-en-1-one (**2**) has been prepared by condensation of 1-(7-methoxy-1-benzofuran-2-yl)ethanone with 4-methylbenzaldehyde in basic medium. The chemical structure of **2** was confirmed by elemental analysis, FT-IR, ^{1}H NMR and ^{13}C NMR. UV spectroscopic characteristics, absorption band edges, optical band gaps, refractive indices, environmental behaviors and conductivity properties of **2** in solutions at different concentrations were investigated in detail. With the concentration, we examined how the spectroscopic, optical and conductivity properties of **2** have changed and can be controlled.

Keywords: Benzofuran; chalcone; absorption band edge; optical band gap; refractive index; environmental behavior; conductivity properties.

1. Introduction

Benzofurans and their derivatives are used in the science of materials due to their significant fluorescence, absorption and quantum values,[1] electrochemical behavior, thermal stability,[2] blue-light emitting,[3] layered organic light-emitting diodes (OLEDs)[4] and organic electroluminescence (OEL)[5] properties. Studies in recent years have indicated that benzofuran and its derivatives are good for electron transport and as perforated injection materials.[6] Benzofurans exhibit significant optical properties due to the possibility of $\pi \rightarrow \pi^*$ transitions.[7] They also belong in the semiconductor class. This is due to delocalization and intermolecular charge transfer.[8–10] Conjugated organic molecules are very important in the second harmonic production due to their many delocalized π-electrons and provide easy charge transfer to the first order polarization (β) nonlinear optical (NLO) applications.[11,12] Chalcones are a class of flavonoids with significant biological activity.[13–15] Chalcones are also important because of their optical applications such as second harmonic generating materials in nonlinear optics and their photophysical properties,[16–19] holographic recording materials,[20] fluorescent probes for sensing of metal ions,[21] photo refractive polymers,[22] and micro environment in micelles.[23] The simplest chalcone structure is formed by two benzene rings that are connected by α,α-conjugated enol-keto groups.[24] Conjugated double bonds between aromatic rings are responsible for nonlinear optical properties.[25] The spectroscopic and photophysical properties of chalcone compounds can be easily changed by conjugation. Many researchers have revealed different types of transmitter and receiver groups to increase nonlinear optical coefficients. In recent years, chalcone compounds containing phenyl groups have been reported to be important materials to create a donor-accepting regulation for high SHG.[26] Therefore, it is important to study the NLO properties and molecular structures of new chalcone derivatives. Previously, benzofuran-substituted chalcones in which the electron releasing groups are attached to the phenyl ring showed improved electro-optical properties. For this reason, in this study, we have selected benzofuran-substituted chalcone derivative **2** comprising of methyl group. New chalcone derivative **2** was prepared using the reaction of 1-(7-methoxy-1-benzofuran-2-yl)ethanone with 4-methylbenzaldehyde in methanolic NaOH.

The aim of this study is to examine the effect of concentration on the spectroscopic, optical and conductivity properties and to investigate the important parameters for material science with device performance of compound **2**. These aims were accomplished with great success in this study.

2. Experimental

All chemicals for the synthesis of compound **2** were of analytical grade and were purchased from commercial sources. They were used without purification. Reactions were monitored by thin layer chromatography (TLC) using silica gel 60 plates. Büchi RE 11 rotary evaporator was used for solvent evaporation. A differential scanning calorimeter (Shimadzu DSC-50) was used for melting point measurement. A Leco CHNS-932 device was used for element analysis. NMR spectra were obtained by a Bruker AC 400 (400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR) in deuterated dimethylsulfoxide (DMSO-d_6) at 25 °C. Chemical shift values were recorded in parts per million (ppm). FT-Infrared (FT-IR) spectrum was recorded as KBr pellet on a Perkin-Elmer Spectrum One FTIR spectrometer. 1-(7-methoxy-1-benzofuran-2-yl)ethanone (**1**) was synthesized as described before.[27]

General Procedure for Synthesis of Chalcone 2

4-Methylbenzaldehyde (1 mmol) and 1-(7-methoxy-1-benzofuran-2-yl)ethanone (1 mmol) in MeOH (5 mL) were cooled to 0–5 °C, added and mixed. Then 3 mL of aqueous NaOH (1 mol/L) was added and stirred for 3 hours at room temperature. The solution was poured onto the water-ice mixture and precipitated solid was filtered, washed with dilute HCl and water, dried and crystallized from ethanol.

1-(7-Methoxy-1-benzofuran-2-yl)-3-(4-methylphenyl)prop-2-en-1-one (2)

Yield: 80%, m.p. 103–104 °C. IR (KBr) ν_{max} 3140–3001 (arom. C-H), 2966–2842 (aliph. C–H), 1653 (C=O), 1593 (C=C) cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.29 (s, 1H, 3-H), 7.81–7.79 (m, 4H, α,β-H, 12,14-H), 7.41 (d, *J* = 8.0 Hz, 1H, 4-H), 7.33–7.29 (m, 3H, 11,15-H, 5-H), 7.15 (d, *J* = 8.0 Hz, 1H, 6-H), 3.98 (s, 3H, OCH_3), 2.36 (s, 3H, R_2=CH_3). ^{13}C NMR (400 MHz, DMSO-d_6) δ 178.94, 153.77, 145.95, 145.27, 144.08, 141.66, 132.09, 131.07, 130.21, 129.52, 125.37, 121.19, 115.74, 115.58, 110.61, 56.36, 21.61. Anal. calcd. for $C_{19}H_{16}O_3$: C, 78.08; H, 5.48. Found: C, 78.10; H, 5.45.

Optical Measurements of Compound 2 Solutions

The solutions of the compound **2** in the chloroform solvent for 6.0, 4.0, 0.7 and 0.2 mM concentrations were precisely prepared. By using a digital vortex mixer (Four E's Scientific CO., Ltd.) the compound **2** was also better dissolved in chloroform. This process is very important in the solution technique for the most sensitive and accurate optical measurements. We recorded the optical measurements of the compound **2** solutions for selected concentrations with an UV-1800 Spectrophotometer (Shimadzu model) at room temperature.

3. Results and Discussion

3. 1. Synthesis and Characterization

First, 1-(7-methoxy-1-benzofuran-2-yl)ethanone was synthesized by the reaction of 1-chloroacetone and

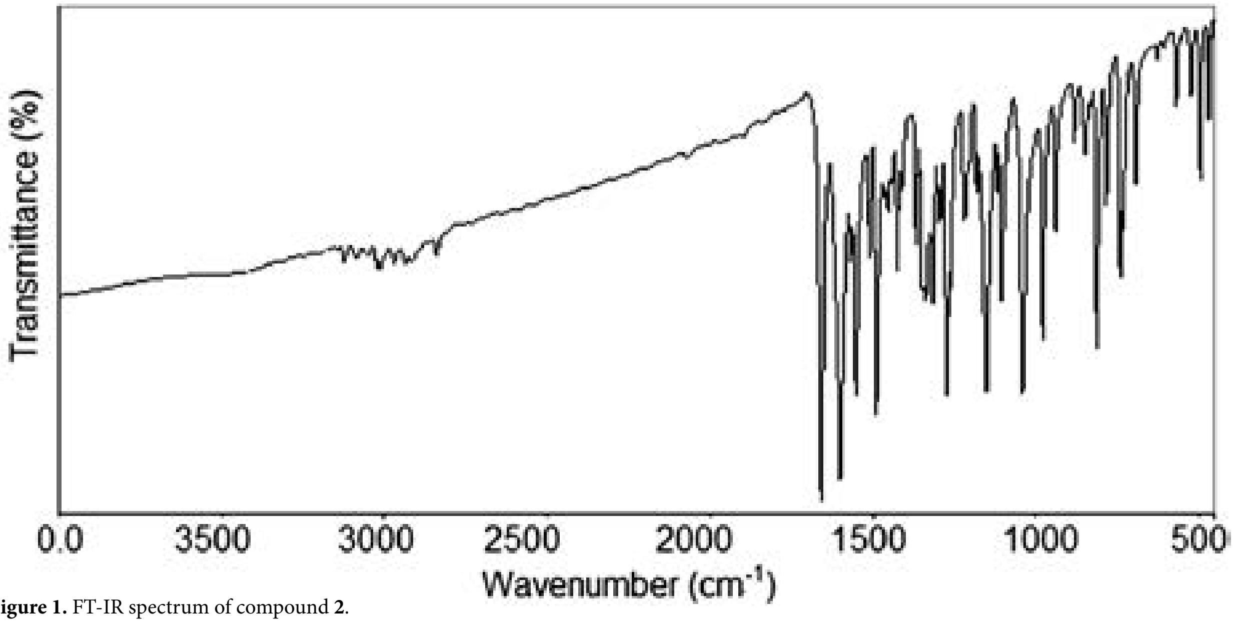

Figure 1. FT-IR spectrum of compound **2**.

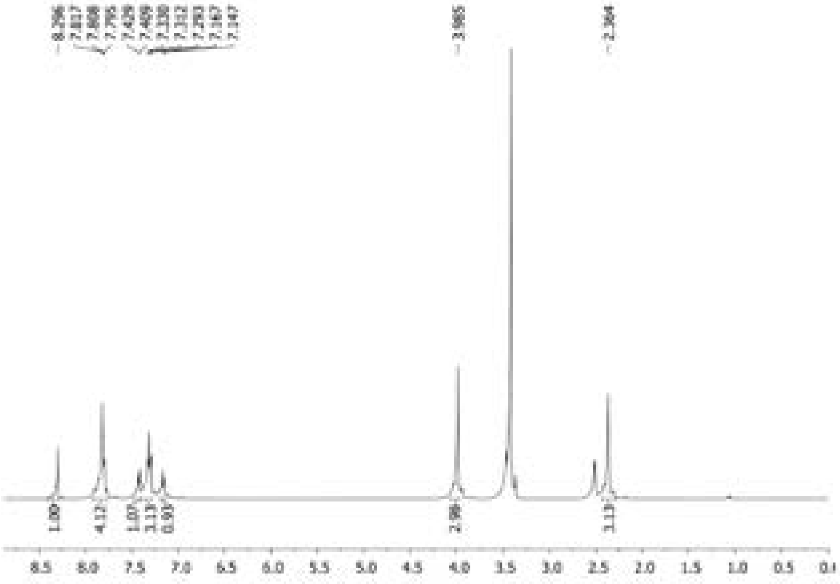

Figure 2. ^{1}H NMR (DMSO-d_6) spectrum of compound **2**.

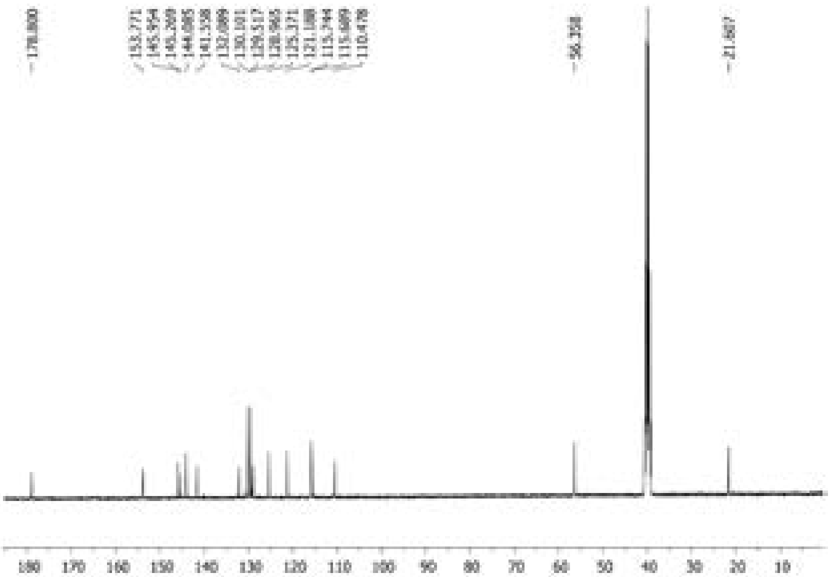

Figure 3. ^{13}C NMR (DMSO-d_6) spectrum of compound **2**.

3-methoxysalicylaldehyde.[27] Benzofuran-substituted chalcone derivative **2** was synthesized as shown in Figure 4. The most common method used for the synthesis of chalcones is the base-catalyzed Claisen–Schmidt reaction, which includes a condensation of a benzaldehyde derivative with an acetophenone derivative in ethanol or methanol with NaOH catalyst. For the characterization of **2**, FTIR, elemental analysis and NMR spectroscopy techniques were used.

In the FT-IR spectrum C=O stretching vibration of 1-(7-methoxy-1-benzofuran-2-yl)ethanol was observed at 1664 cm^{-1}. Also, C=O stretching vibration in chalcone structure was observed at 1653 cm^{-1}. The reason for the shifting of the C=O tensile vibration of chalcone to low wave numbers is the delocalization of π-electrons between the α and β carbons. This reduces the order of C=O bonds. After all, it increases the order of bonding between carbonyl carbon and a carbon atom. Another characteristic peak of the chalcone compound is the C=C stretching vibration at 1593 cm^{-1}. The most characteristic signals in ^{1}H NMR spectrum of the benzofuran-substituted chalcone was observed at 8.29 ppm (3-H belonging to the benzofuran ring) and at 7.81–7.79 ppm (α and β hydrogens of chalcone moiety) with a coupling constants about 15 Hz which characterizes the *trans* configuration. The β-H signal is in a lower area than α-H due to the resonance of π-electrons

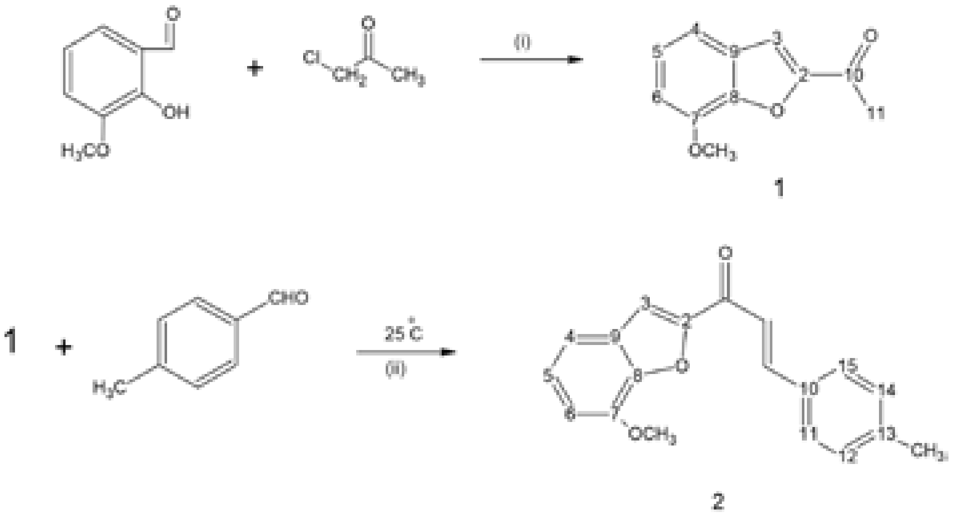

Figure 4. General synthesis of compounds **1** and **2**. Reagants and conditions: (i) K_2CO_3, acetone, reflux; (ii) NaOH, MeOH, r.t.

between α and β carbons with carbonyl group. In the ^{13}C NMR spectrum, carbonyl carbon was observed at about 178 ppm. The spectra FT-IR, ^{1}H NMR and ^{13}C NMR are given in Figures 1–3.

3. 2. UV Spectroscopic Characteristics and Absorption Band Edges of Compound 2 Solutions

Absorbance (*A*) is the amount of light absorbed by a solution (a sample). In other words, absorbance is a process of light that interacts with matter. Traditionally, absorbance measurements are carried out in a cuvette. We obtained UV spectra of the compound **2** solutions for 6.0, 4.0, 0.7 and 0.2 mM concentrations. Figure 5 shows the absorbance spectra of the compound **2** solutions at different concentrations. At higher molarities, it is normal that the absorbance curves do not appear completely. All absorbance curves of solutions below a certain concentration can be observed. As seen in Figure 5, all absorption curves are observed for 0.7 and 0.2 mM solutions. The maximum absorbance peak for 0.7 and 0.2 mM is observed at 347 and 345 nm, respectively. The compound **2** has a single peak and its absorbance decreases as the concentration is reduced.

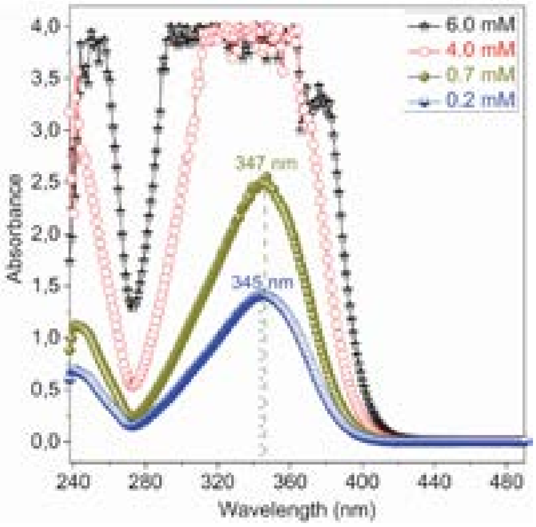

Figure 5. The absorbance spectra of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM concentrations.

Transmittance (*T*) is defined as light passing through an object that is not reflected or absorbed. It is relatively easy to determine the permeability levels of an object. Transmittance is the transmitted part of the incoming light. The transmittance spectra of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM are shown in Figure 6a. As can be seen in Figure 6a, all transmittance curves displays a peak at 273 nm and increase sharply in the range of about 350–420 nm. Transmittance increases with decreased concentration.

When examining the UV spectroscopic properties of a material, it is important to consider the absorbance band edge (E_{Abe}) of the material. Because this parameter is a specific parameter of any material, it gives very important information about the absorption and the behavior of the material in the environment. Therefore, we investigated the absorbance band edges of the compound **2** for different concentrations and we obtained the E_{Abe} values from the maximum peaks using the $dT/d\lambda$ curves vs. wavelength (λ) as seen in Figure 6b. While the concentration increased from 0.2 to 6.0 mM, the absorbance band edge of the compound **2** decreased from 3.002 to 2.877 eV. It is expected that the absorbance band edge decreases with increasing concentration as can be seen in the insert of Figure 6b. This result indicates that the absorbance band edge of the compound **2** can be controlled by concentration. Solutions with lower absorbance band edge are a desirable and preferred feature for metal-semiconductor based structures.

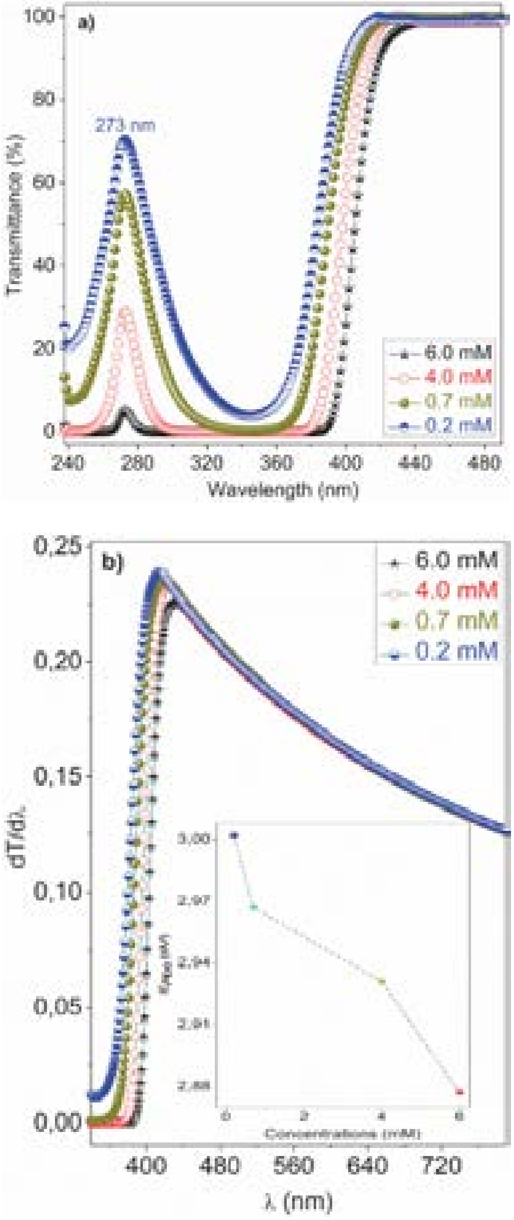

Figure 6. **(a)** Transmittance spectra and **(b)** dT/dλ curves vs. wavelength (λ) of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM.

3. 3. Controlling of Optical Band Gap of Compound 2 Solutions

When it comes to optical properties, one of the first parameters that comes to mind is the optical band gap (E_g) parameter. This parameter is very important for the determination of material and device performance. The E_g can be estimated from the Tauc model[28] given by the following equation:

$$\alpha(h\vartheta) = A(E - E_g)^m \tag{1}$$

where α is absorption coefficient, $h\vartheta$ and E are the photon energy, A is a constant and m determines the type of the optical transitions. We found m as ½, which corresponds to allowed direct optical band gap (E_{gd}). Therefore, the $(\alpha h\vartheta)^2$ curves vs. E of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM are plotted as seen in Figure 7a. By extrapolating the linear plot to $(\alpha h\vartheta)^2 = 0$, the E_{gd} values of the compound **2** at different concentrations were obtained. As seen in Figure 7b, the direct optical band gap of the compound **2** varies from 3.046 to 2.926 eV; that is, as the concentration increases, the optical band gap decreases. Materials with the lowest optical band gap are preferred for many electronic, optical and photonic devices, because the lowest band gaps weaken the mobility barrier of the carrier.

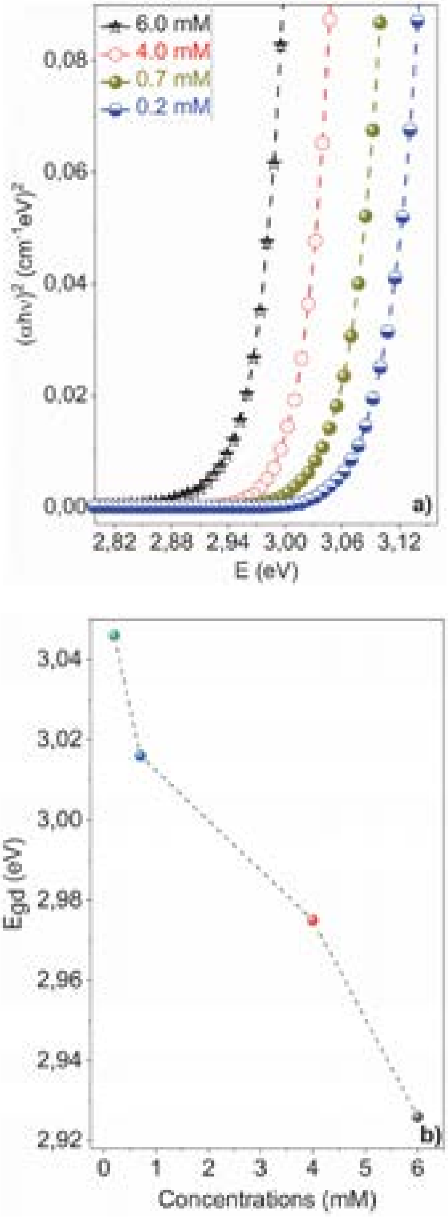

Figure 7. **(a)** $(\alpha h\vartheta)^2$ curves vs. E; **(b)** E_{gd} curves vs. concentrations of the compound **2** solutions.

3. 4. Controlling of Refractive Index of Compound 2 Solutions

Another parameter that comes to mind when talking about optical properties is the refractive index (n). The refractive index is a measure of the bending of a ray of light as it passes from one medium to another. The n can be explained with reflectance (R) and is calculated from following equation:[29,30]

$$n = \left\{ \sqrt{\frac{4R}{(R-1)^2} - k^2} - \frac{R+1}{R-1} \right\} \quad (2)$$

where $k = \alpha \lambda / 4\pi$. The refractive indices of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM were calculated from Equation 2. The n curves of the compound **2** solutions for different concentrations are shown in Figure 8a. As seen in Figure 8a, the refractive index increases with increasing angular frequency (ω). This behavior is known as normal dispersion behavior. In addition, it is observed that the index of refraction increases with the increasing concentration. This indicates that the refractive index can vary and can be controlled with different concentrations.

There are many relations such as Herve–Vandamme, Kumar–Singh, Moss, Ravindra and Reddy[31,32] describing how to obtain the refractive indices based on optical band gaps. The refractive indices of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM were obtained. The n curves vs. concentrations of the compound **2** for related relations are shown in Figure 8b. The refractive indices obtained from Reddy relation are the highest, while the refractive indices obtained from Ravindra relation are the lowest. Also, the index of refraction increases with increasing concentration for all correlations as seen in Figure 8b.

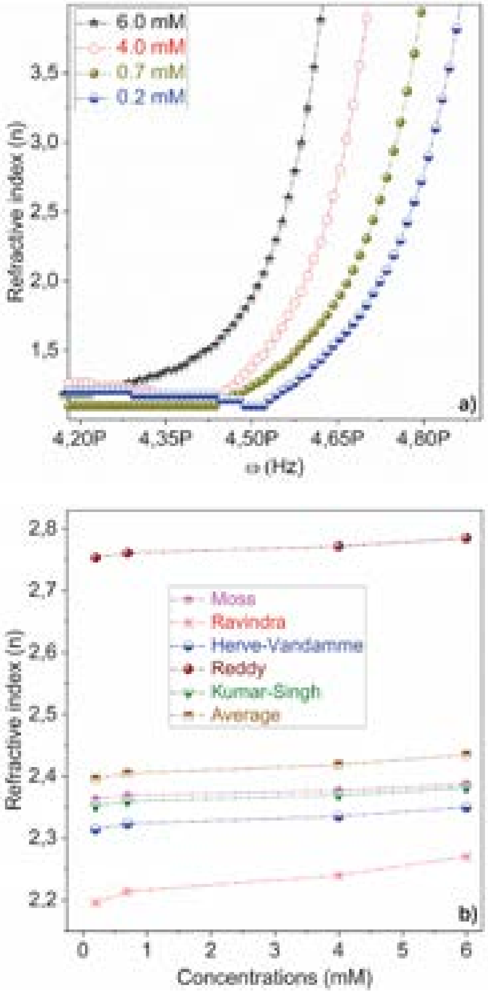

Figure 8. **(a)** n curves vs. angular frequency (ω); **(b)** n curves vs. concentrations of the compound **2** solutions.

3. 5. Environmental Behavior of Compound 2 Solutions

The behavior of a material in different environments, in other words, its interaction with light, is very important for the science of materials and devices. Incidence angle (Φ_1) and refraction angle (Φ_2) play an important role in investigating the behavior of the material in an environment. The Φ_1 is given by the following equation:[29,33]

$$\Phi_1 = \tan^{-1}\left(\frac{n_2}{n_1}\right) \quad (3)$$

We calculated the incidence angle of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM from Equation 3. The Φ_1 plots vs. E of the compound **2** for related concentrations are shown in Figure 9. These curves give the variation of the angle of incidence of the compound **2** according to the wavelength. In other words, it shows at which wavelengths the angles of incidence are decreasing and increasing. Also, as concentration increases, angles of incidence also increase. The Φ_2 is given by the equation:

$$\Phi_2 = \sin^{-1}\left(\frac{n_1}{n_2} \sin \Phi_1\right) \quad (4)$$

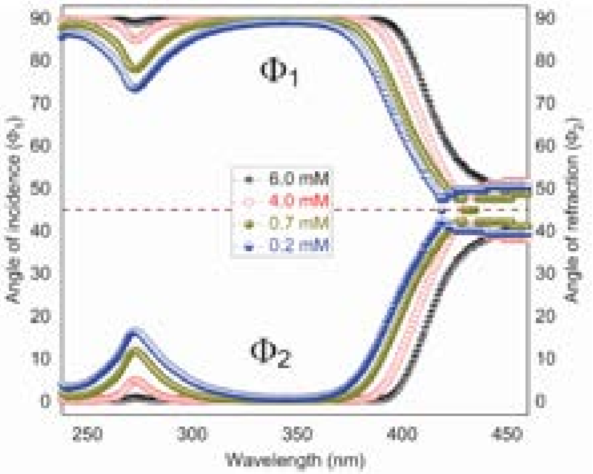

Figure 9. The incidence angle (Φ_1) and refraction angle (Φ_2) plots vs. E of the compound **2** at various concentrations.

The refraction angles of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM were calculated from Equation 4. Figure 9 indicates the Φ_2 plots vs. *E* of the compound **2** for these concentrations. As expected, the angles of refraction are smaller than the angles of incidence. The angle of refraction varies with varying wavelengths. On the other hand, as the concentration increases, the angle of refraction decreases.

3. 6. Conductivity Properties of Compound 2 Solutions

Conductivity plays an important role in materials science and device performance. Optical and electrical components of conductivity make a great contribution to electronic and optical applications. The optical conductance (σ_{opt}) and electrical conductance (σ_{elec}) properties can be explained by the following equations,[34] respectively:

$$\sigma_{opt} = \frac{\alpha n c}{4\pi} \tag{5}$$

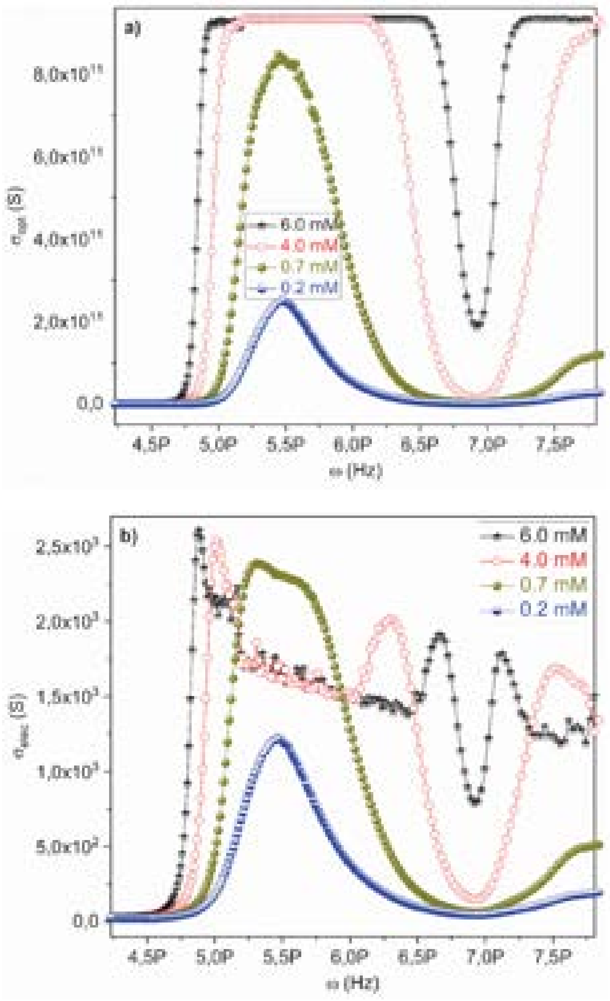

Figure 10. **(a)** σ_{opt} and **(b)** σ_{elec} curves vs. ω of the compound **2** solutions at 6.0, 4.0, 0.7 and 0.2 mM.

where *c* is the velocity of light. σ_{elec} is given as:

$$\sigma_{elec} = \frac{2\lambda \sigma_{opt}}{\alpha} \tag{6}$$

The σ_{opt} and σ_{elec} values of solutions of the compound **2** at 6.0, 4.0, 0.7 and 0.2 mM were calculated from Equations 5 and 6, respectively. The optical and electrical conductance curves vs. ω are shown in Figure 10a,b, respectively. As seen in Figure 10a, the compound **2** displays peaks at about 5.5 PHz at low concentrations, while the compound **2** displays pits at about 6.9 PHz at higher concentrations. The optical conductance values are of the order of 10^{11} S. On the other hand, the electrical conductance values are order of 10^3 S. As can be seen, both optical conductivity and electrical conductivity vary significantly with varying concentration and angular frequency.

4. Conclusions

We examined the effect of concentration on the spectroscopic, optical and conductivity properties and investigated the important parameters of the compound **2** for material science and device performance. The absorbance band edge of the compound **2** decreased from 3.002 to 2.877 eV, while the direct optical band gap of the compound **2** varies from 3.046 to 2.926 eV. The refractive index can vary and can be controlled by changing concentrations. Both, optical conductivity and electrical conductivity vary significantly with varying concentration and angular frequency. According to the data obtained, the compound **2** possesses promising properties for semiconductor and conjugated polymer technology and applications.

Acknowledgements

The authors thank Firat University for financial support of this work.

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Povzetek

Halkonski derivati so pomembni v znanosti, saj njihove uporabe segajo od polprevodniških lastnosti vse do bioloških učinkov. V tem delu smo s kondenzacijo 1-(7-metoksi-1-benzofuran-2-il)etanona s 4-metilbenzaldehidom v bazičnem mediju pripravili 1-(7-metoksi-1-benzofuran-2-il)-3-(4-metilfenil)prop-2-en-1-on (**2**). Kemijsko strukturo **2** smo potrdili z elementno analizo, FT-IR, ^{1}H NMR in ^{13}C NMR. Za različne koncentracije **2** v raztopinah smo podrobno določili UV spektroskopske lastnosti, robove absorpcijskih trakov, optične pasovne vrzeli, lomne količnike, obnašanje v odvisnosti od okolice in prevodnostne lastnosti. Preučili smo tudi, kako se spektroskopske, optične in prevodnostne lastnosti **2** spreminjajo v odvisnosti od koncentracije in kako lahko nanje vplivamo.