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Synthesis and characterization of a new organic semiconductor material

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HIGHLIGHTS

- Development of a new organic acetaminophen/Curcumin semiconductor material.
- The developed material has characteristics of an organic semiconductor.
- It has electrical conductivity comparable to available organic semiconductors.
- It has high optical transmittance and low permittivity/dielectric constant.

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ABSTRACT

The objective of this study is to create an ideal mixture of Acetaminophen/Curcumin leading to a new and improved semiconductor material, by a study of the electrical, thermal and optical properties. This new material will be compared with existing semiconductor technology to discuss its viability within the industry. The electrical properties were investigated using complex impedance spectroscopy and optical properties were studied by means of UV-Vis spectrophotometry. The electric conductivity σ, the dielectric constant ε_r, the activation energy E_a, the optical transmittance T and the gap energy E_g have been investigated in order to characterize our organic material. The electrical conductivity of the material is approximately 10^{-5} S/m at room temperature, increasing the temperature causes σ to increase exponentially to approximately 10^{-4} S/m. The activation energy obtained for the material is equal to 0.49 ± 0.02 ev. The optical absorption spectra show that the investigating material has absorbance in the visible range with a maximum wavelength (λ_{max}) 424 nm. From analysis, the absorption spectra it was found the optical band gap equal to 2.6 ± 0.02 eV and 2.46 ± 0.02 eV for the direct and indirect transition, respectively. In general, the study shows that the developed material has characteristics of organic semiconductor material that has a promising future in the field of organic electronics and their potential applications, e.g., photovoltaic cells.

1. Introduction

Conducting organics (CO) are an exciting new class of electronic materials which have garnered increasing interest since Shirakawa's report in 1977 [1] due to the fact that they combine both metal and organic properties. These conducting organics will establish themselves as key low-cost electronic materials. The structural and electrical properties of organic compound semiconductor (OCS) materials have been intensively investigated in the recent years due to their wide range of potential applications in light-emitting diodes, solar cells, organic transistors, battery materials [2], electrochromic devices [3,4], electromagnetic shielding [5], sensor technology [6], nonlinear optics [7], molecular electronics [8] and enzyme immobilization matrices [9,10]. The Organic semiconductor materials carry a potential for development in the

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search for relatively low cost photovoltaic modules for the production of domestic electricity. Unlike silicon cells, they can be easily manufactured on flexible substrates, allowing them to be easily integrated in to everyday objects. Several studies on the use of semiconducting polymers have been published in recent years [4]. In contrast to polymers, small molecules have the advantage that they can be deposited by evaporation or vacuum distillation without solvent and under high vacuum; this method allows obtaining a film of high purity. Many unsubstituted conducting organic systems have limited solubility as a result, they exhibit intractable and infusible properties. This is primarily due to the rigid rod nature of COs, arising from their extended-delocalization. Several approaches have been considered to improve the processability of conducting polymers [11]. The most effective method is the introduction of insulating organic matrices into them [12–18]. Organic semiconductors have certain advantages such as light weight and simplicity of design since the molecules are assembled together, and their biggest strength hallowing greater manipulation. In terms of manufacturing conditions and financial cost, unlike crystalline silicon whose production requires very high temperatures, their manufacture involves low energy costs and low environmental impact. They are evolving more and more into the world of electronics and information technology. They also are useful elements to replace silicon inorganic electronics.

Our attention was to limit the hepatotoxicity of Acetaminophen by adding Curcumin, and to increase the bioavailability of Curcumin. On this basis, it was thought to study certain physical properties, in order to find a relationship between physical and biological properties of the mixture. But coincidentally and after measuring the absorbance λ and the electrical conductivity σ of the blend it has been discovered that the blend may have semiconductor characteristics. Since Acetaminophen and Curcumin are widely used in the biological applications and of limited applications in electronics fields, the work has been directed and focused on such interesting research area of the organic semiconductors. In this paper, a new approach is introduced to produce a new organic semiconductor material from Acetaminophen (synthesized) mixed with powdered Curcumin (commercial) [50 wt%–50 wt%]. The new compounds were characterized by using complex impedance, UV–vis spectroscopy, thermogravimetric analysis (TGA) and X-ray diffraction.

2. Material and experimental

2.1. Material and sample preparation

The sample was prepared by mixing 01 g of powdered Curcumin (commercial) with 01 g of Acetaminophen (the commercial name is paracetamol) (synthesized) [50%–50%]. The chemical structures of the organic compounds are shown in Fig. 1. An amount of 7 ml of distilled water was added to each sample and was thoroughly mixed. This was placed into a microwave oven at 300 W for 10 min to get a homogenous semiconductor material. Finally, using a mortar, the mixture was crushed until a fine powder was obtained. The melting temperature was then measured using the powder and found to be 493 K. Discs samples of 13 mm diameter and 1 mm thickness have been prepared by compaction technique and used for the measurements of the electrical properties. Fig. 2 shows the disc samples that has been used.

2.2. Structural characterization

The structural characterization of Acetaminophen/Curcumin samples was performed by the XRD technique. The structural changes were analysed using Cu K_α radiation of wavelength $\lambda = 1.5406$ Å, produced by Bruker AXS D8 focus advance X-ray diffraction meter (Rigaku, Japan, Tokyo) with 'Ni-filtered'. The scans were taken in the 2θ range from 10° to 80° with a scanning speed and step size of 1°/mm and 0.01° respectively.

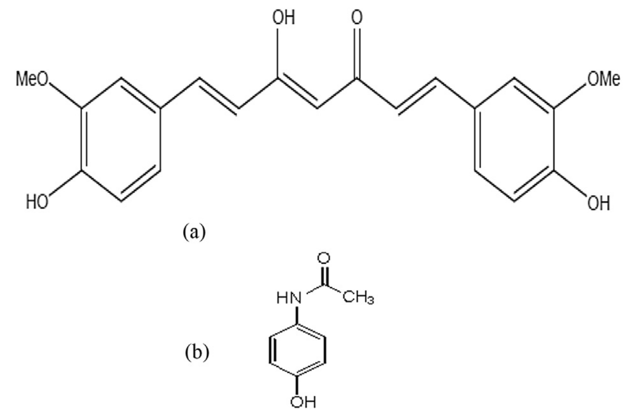

Fig. 1. The chemical structures of the organic compounds: (a) Curcumin and (b) Acetaminophen.

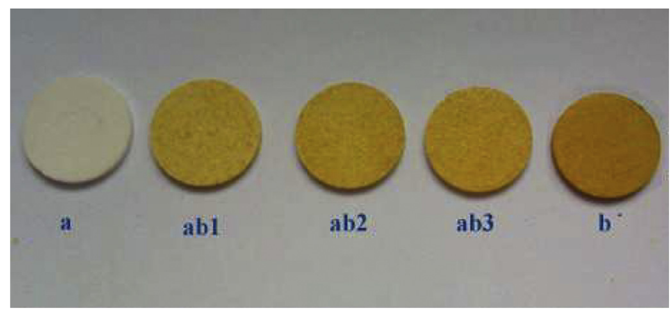

Fig. 2. Disc samples for electrical properties measurements. (a) Acetaminophen (100%); (ab1) Acetaminophen (75%)/Curcumin (25%); (ab2) Acetaminophen (50%)/Curcumin (50%); (ab3) Acetaminophen (25%)/Curcumin (75%); (b) Curcumin (100%).

2.3. Optical absorption characterization

The UV–visible spectrum of the developed organic semiconductor was obtained by using Shimazdu UV-1600 PC,UV–vis spectro-photometer of the range of 200–900 nm. The optical absorption of the prepared Acetaminophen/Curcumin samples was measured over the range of 300–800 nm and the energy band gap has been obtained.

2.4. Thermal performance characterization

Differential scanning calorimetry (DSC) experiment was performed by using TA instrument Q20-2487, for thermal characterization of the organic semiconductor. The sample for DSC analysis was cut into small pieces of 10 mg weight from the. Acetaminophen/Curcumin samples. All the experiments were carried out at 200 °C with heating rate of 10 °C min^{-1} in N_2 gas.

The thermogravimetric analysis (TGA, TA Instrument Q50-1555) of compounds were performed using an instrument equipped with a platinum pan. The sample was heated from 40 °C to 600 °C at a heating rate of 10 °C/min, while the chamber was purged continuously with N_2 gas at a rate of 100 ml/min. More details about the procedure and purpose of thermal tests are available in the work of Mourad and his group [19–26].

2.5. Electrical performance characterization

The electrical properties of Acetaminophen/Curcumin organic semiconductor were carried out by demonstrating the impedance spectrum. The complex impedance records the phase shift between the current through the sample and the alternating voltage imposed. This is a physical-chemical technique for studying the electrical properties. The conductivity and the permittivity (dielectric constant) are measured by varying the temperature in the range from 298 K (25 °C) to 487 K (214 °C), for each of the five frequencies (0.1 kHz, 0.5 Hz, 1 kHz, 10 kHz, and 100 kHz) in an inert environment, using an impedance analyser type Instek. The used apparatus comprises a test cell between two electrodes, where the sample was placed. The guard brass electrode was placed on the sample to have evenly distribution for the applied electrons and heat on the sample surface. The potential applied was 1 V.

3. Results and discussion

Differential scanning calorimetry thermograms (DSC) are utilised to determine the thermal behaviour of the pure and organic complex system. The DSC thermograms of pure Acetaminophen, pure Curcumin and Acetaminophen/Curcumin organic semiconductor are shown in Fig. 3. The endothermic peaks observed at 174 °C and 85 °C and shown in Fig. 3 are corresponding to the crystalline melting temperature (T_m) of pure Acetaminophen and pure Curcumin, respectively. The endothermic peak at 170 °C for the blend is obtained upon to the addition of Curcumin to Acetaminophen and shows the reduction in crystallinity of organic semiconductor samples. The melting enthalpy ΔH_m, estimated experimentally, demonstrates the heating point at which the substance changes its state from solid to liquid. The ΔH_m of pure Acetaminophen, pure Curcumin and Acetaminophen/Curcumin organic semiconductor is presented in Table 1.

The thermogravimetric curves presented in Fig. 4 show only 5% weight loss upon heating above 300 °C; 295 °C and 280 °C for Acetaminophen; Curcumin and Acetaminophen/Curcumin respectively (Table 1), demonstrating the thermal stability of the developed compounds in this study.

The crystallinity and morphology of organic semiconductors are often correlated with device performance. The crystallinity was characterized by X-ray diffraction, which showed fewer Bragg reflections of Acetaminophe, Curcumin and Acetaminophen/Curcumin blend. Fig. 5 shows the XRD patterns of the synthesized Acetaminophen/Curcumin powders together with pure Acetaminophen and Curcumin for comparison. The patterns were indexed according to the monoclinic (P2/n) crystalline structure of lattice parameter.

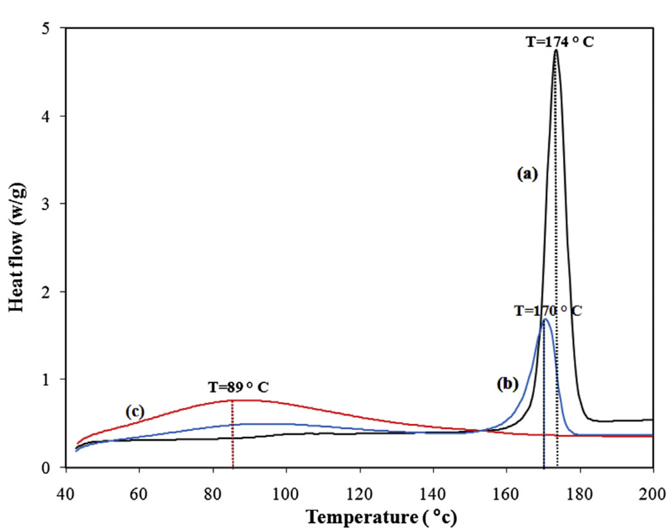

Fig. 3. DSC thermograms of (a) Acetaminophen, (b) Curcumin and (c) Acetaminophen/Curcumin.

The electrical conductivity for five different values of frequency (namely, 0.1 kHz, 0.5 kHz, 1 kHz, 10 kHz, and 100 kHz) was measured at room temperature and the device didn't detect any electrical value of the electrical conductivity that shows the materials are conductors. This illustrates that all the investigated samples are insulating materials as shown in Table 2.

To confirm if the investigated samples are a semiconducting materials or not, their behaviour with increasing temperature has been investigated. For the Acetaminophen and Curcumin, the device didn't detect any conductivity for the pure materials (Acetaminophen and Curcumin) over temperature range from 298 K up to 460 K, and from 298 up to 446 K, respectively. The measurements were conducted for the five different values of the frequency (0.1 kHz, 0.5 kHz, 1 kHz, 10 kHz, 0.1 kHz). This observation further confirms that the Acetaminophen and Curcumin are insulating materials. In a recent study of Dakhel et al. [27,28] has been used Curcumin as an insulator which again confirms that the Curcumin by itself is insulator material. On the other side, for a blend of Acetaminophen (50%)/Curcumin (50%), the device detects electrical conductivity at 100 kHz, however the device didn't show any evidence of the electrical conductivity for frequencies from 0.10 kHz up to 10 kHz. The measured electrical conductivity values are presented in Table 3 and Arrhenius curve in Fig. 6 in which the variation of the logarithm of the conductivity σ "log[σ]" versus the reciprocal of temperature (1000/T) at 100 kHz and power 1000 mV is shown. Two regions of electrical conductivity are observed in Fig. 6. The first region "Region I" is represented in the temperature range of 47 °C to 166 °C (which are corresponding to points 3.125 and 2.27 on the 1000 T/K axis) where the electrical conductivity oscillates around 10-5 S/m. This region "Region I" is not taken into consideration because the curve is nonlinear and it doesn't follow Arrhenius law. In the second region "Region II" which is represented in the range of 197 °C to 214 °C (that are corresponding to points 2.127 and 2.053 on the 1000 T/K axis), the curve is linear and follow Arrhenius law. In region II, a significant increase in the electrical conductivity is observed with the increase in the temperature. The electrical conductivity σ varies from 10-5 S/m to 10-4 S/m (Table 3). The value of the electrical conductivity σ is given by the Arrhenius equation [29,30]:

$$\sigma = \sigma_0 \exp\left[\frac{Ea}{K_B T}\right] \quad (1)$$

where σ_0 is a constant, E_a is the activation energy; kB is Boltzmann's constant and T the temperature in degrees Kelvin. There is an increase in electrical conductivity with increasing temperature, which implies that the more the temperature increases, the charge carriers exceed the activation energy barrier and participate in the electrical conductivity [31].

From the Arrhenius curve of the electrical conductivity (Fig. 6), the linearization of the Equation (1) determines that the value of the activation energy E_a which was found to be equal to 0.49 eV. In the study of Yakuphanoglu [32] on the electrical conductivity of the MEH-PPV:C70 (organic semiconductor material), the activation energy value for region II was determined from slope of log[σ] curve and found to be equal to 0.53 eV. The obtained activation energy values are typical values for the organic semiconductors. Thambidurai et al. [33] have investigated the electrical conductivity of CdS (inorganic semiconductor material) and 5.02% Co doped CdS

Table 1
Thermal properties of the pure and blend materials.

Material	Melting temperature T_m (°C)	Heat of fusion ΔH_m (j/g)	Initial degradation temperature T_i (°C)
Acetaminophen	174	175	300
Curcumin	85	93	280
Acetaminophen/Curcumin	170	109	295

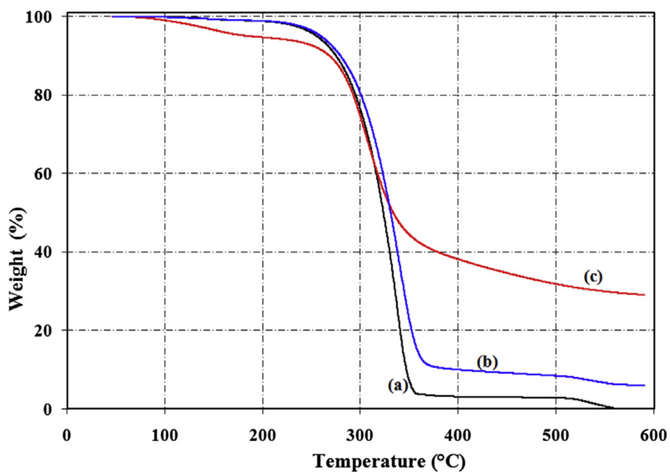

Fig. 4. TGA thermograms of (a) pure Acetaminophen, (b) pure Curcumin and (c) Acetaminophen/Curcumin semiconductor material.

Table 3
Variation of the electrical conductivity σ, of the sample Acetaminophen/Curcumin, with increasing temperature at 100 kHz.

Sample	Acetaminophen/Curcumin	
	T (K)	σ (S/m)
Region I	320	$1.7\cdot10^{-5}$
	439	$1.51\cdot10^{-5}$
Region II	470	$1.6\cdot10^{-5}$
	487	$2.18\cdot10^{-4}$

nanoparticles (inorganic semiconductor material). They have found that the activation energy values are 0.35 eV and 0.39 eV respectively. Comparison the activation energy values of the developed semiconductor organic material with that of organic [32] and inorganic semiconductor [33], it can be concluded that the developed material has activation energy between the organic and inorganic. These evidences reveal hat the investigated and newly developed organic material (Acetaminophen/Curcumin blend) has behaviour and characteristics of a semiconductors and worth further research.

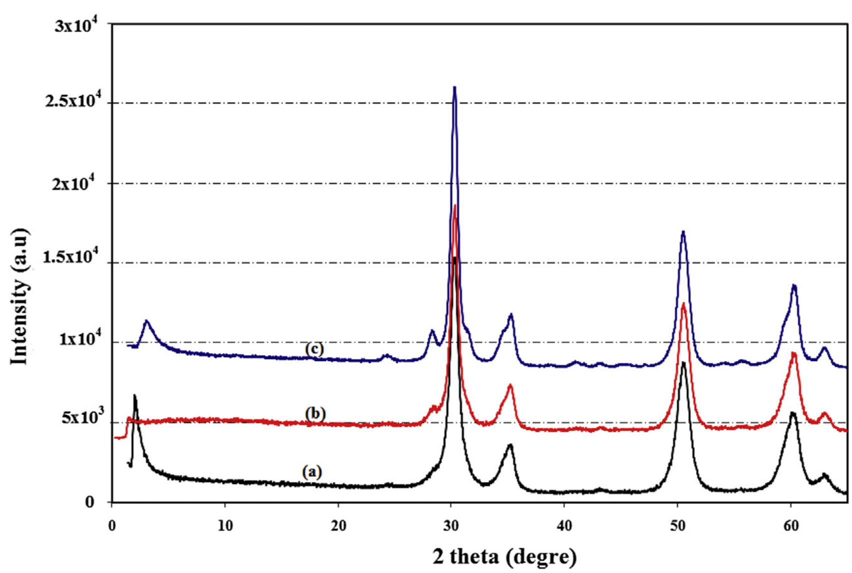

Fig. 5. XRD patterns of (a) pure Acetaminophen, (b) Acetaminophen/Curcumin semiconductor material and (c) pure Curcumin for comparison.

Table 2
The electrical conductivity of investigated samples at room temperature (300 K).

Sample	Temperature (K)	σ (S/m)
a	300	≈0.0
ab1	300	≈0.0
ab2	300	≈0.0
ab3	300	≈0.0
b	300	≈0.0

a: Acetaminophen (100%); ab1: Acetaminophen (75%)/Curcumin (25%); ab2: Acetaminophen (50%)/Curcumin (50%); ab3: Acetaminophen (25%)/Curcumin (75%); b: Curcumin (100%).

The value of the relative permittivity of Acetaminophen/Curcumin material was determined at room temperature and a frequency of 0.10 Hz, 0.50 Hz, 1 KHz, 10 KHz, and 100 kHz (Table 4). From this value, it can be seen that the investigated organic compound has a low permittivity or low dielectric constant. A comparison was performed between the relative permittivity ε_r of the investigated material (at a frequency of 100 kH and room temperature) and some organic and inorganic semiconductor [34] and presented in Table 5. It is observed that the relative permittivity ε_r of the investigated organic Acetaminophen/Curcumin

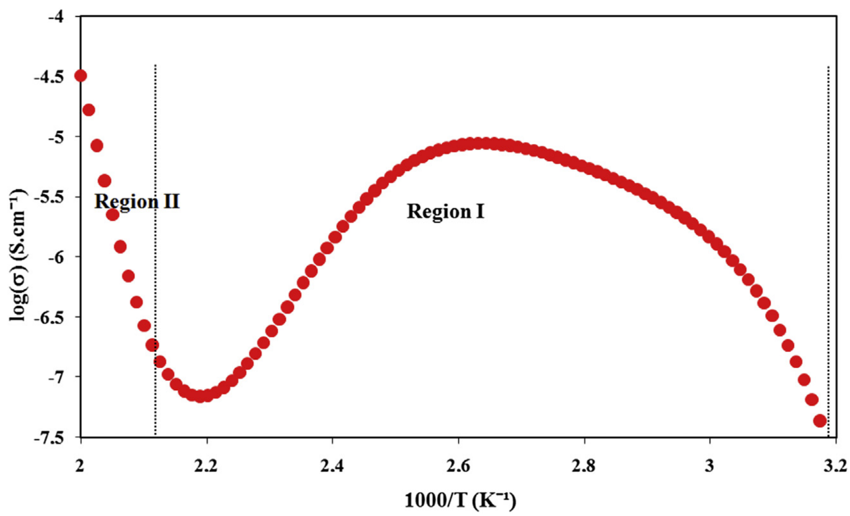

Fig. 6. The logarithm of electric conductivity as a function of the inverse of the temperature.

Table 4
Relative permittivity εr of acetaminophen/Curcumin.

Frequency	0.10 kHz	0.50 kHz	1.0 KHz	10 KHz	100 KHz
Permittivity ε_r	8.6256	7.1568	6.8256	6.9984	6.7717

Table 5
Comparison of relative permittivity between the investigated organic compound and some inorganic and organic semiconductors.

Semiconductors	ε_r	Reference
Ge	16	[24]
GaAs	13	[24]
Si	11.9	[24]
CdTe	10.2	[24]
Acetaminophen/Curcumin	**6.77**	
CdS	5.4	[24]
PTCDA	3.4	[24]
CuPc	2.5	[24]
PTCBI	2.0	[24]

semiconductor ($\varepsilon_r = 6.77$) is between the organic and inorganic semiconductors. The electrical properties of Acetaminophen/Curcumin were resumed in Table 6.

The UV-Visible measurements have been conduct to obtain the optical properties (absorbance, transmittance T and absorbance coefficient α). The results of these measurements are presented in Figs. 7–9.

The optical transmittance (T) presented in Fig. 8 was obtained to determine the absorption coefficient spectra (α) by using the following equation [10–12]:

$$\alpha = -\frac{1}{d}\ln\left(\frac{1}{T}\right) \tag{2}$$

where d is the width of the cuvette.

The optical transmittance spectra presented in Fig. 8 are normalized with regard to the basic line in the wavelength range 400–700 nm [35]. Two regions in these spectra can be clearly distinguish, these are:

Region I (High absorption region which is over a wavelength inferior to 500 nm): in this range the absorbance and transmittance spectra (Figs. 7 and 8) show a strong variation with increasing the level of absorbance and diminishing the level of transmittance. From this range the absorption coefficient spectra can be deduced [36] from the transmittance. Region ii (Transparency region): in this range of wavelength (>500 nm) the optical transmittance remains practically constant at 0.97 (97%) and the absorbance is typically equal to zero. In this region the refractive index n can be determined.

The absorption coefficient can also be obtained from the following empirical relation [37].

$$\alpha = 2.303\left(\frac{Abs}{d}\right) \tag{3}$$

Fig. 9 shows the variation of the absorption coefficient (α) determined from Equation (2) compared with that from empirical relation [37].

Equation (3) is valid for a weak reflection on the surface of the film. These conditions are better verified in this range of absorption where the material is absorbent [38]. Fig. 9 shows clearly a good agreement between the results obtained from the Equations (2) and (3) for the newly introduced semiconductor material.

The optical band gap (E_g) can be deduced from the absorption spectra by considering the Tauc formula [39]:

$$(\alpha \cdot h\upsilon) = B(h\upsilon - Eg)^2 \tag{4}$$

where h is Planck's constant, hυ is the photo energy, B is a material constant, of material, exponent number 2 depends on the type of transition.

A comparison was drawn between the spectrum transmittance of the investigated organic compound and those of other semiconductors available in the literature. It is observed form the semiconductor optical transmittance spectrum of Zinc Oxide (ZnO) that, its transmittance is over 90% [40]. and that of the investigated organic compound is more than 97%. This evidence reveals that the material has a good transmittance.

The optical band gap is an important factor in the

Table 6
Electrical properties of Acetaminophen/Curcumin material.

Frequency (kHz)	σ (S/m)	ε_r	Ea (eV)
100	10^{-5}	6.77	0.49

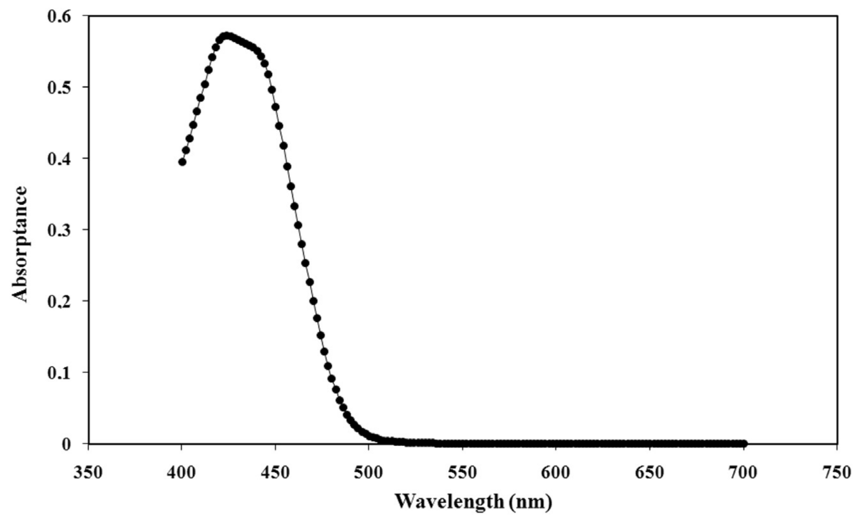

Fig. 7. Absorption spectra of Acetaminophen/Curcumin material.

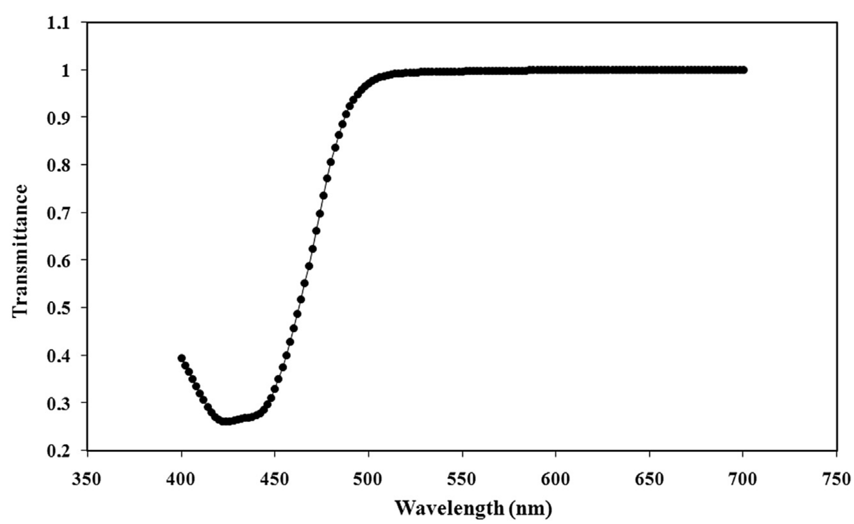

Fig. 8. Transmittance spectra of Acetaminophen/Curcumin material.

characterization of a semiconductor material A material is a semiconductor if the optical energy gap E_g at room temperature is between 1 eV and 4 eV.

A Tauc plot is a useful method of displaying the optical absorption spectrum of semiconductor material. It is used to determine the optical band gap or Tauc gap, in semiconductors which is used to characterize the practical optical properties.

According to Tauc, the dependence of the absorption coefficient α on the photon energy $h\nu$ for near-edge optical absorption in semiconductors takes the form $(\alpha h\nu)^{1/r} = k(h\nu - E_g)$ in which, Eg is the optical band gap, k is a constant and $r = 1/2$ for an allowed direct energy gap. The Tauc plot displays the energy of the light, quantity $h\nu$, on the abscissa and the quantity $(\alpha.h\nu)^{1/r}$ on the ordinate. α is the absorption coefficient of the material and the value of the exponent r represents the nature of the transition ($r = 2$ for indirect allowed transition). The plot has a distinct linear region that indicates the onset of absorption.

In order to determine the energy of the optical band gap, taking $r = 2$, $(\alpha h\nu)^{1/2}$ must be plotted versus $h\nu$ using the data obtained from the optical absorption spectra. The direct band gap of the semiconductor material is obtained by extrapolating the linear part to the abscissa (to the zero of the ordinate).

Tauc plot for the developed Acetaminophen/Curcumin semiconductor material is demonstrated in Fig. 10 and takes the form.

$$(\alpha \cdot h\nu)^{1/2} = c^{ste}(h\nu - E_{Tauc}) \quad (5)$$

From Figs. 10 and 11 the Tauc gap (E_{Tauc}) separating the extremities of the valence and the conduction bands is determined, assuming parabolic and non-perturbed variation by the extrapolation of the strong absorption part towards the weak energies according to the Equation (5).

The values obtained from Figs. 10 and 11 equal to 2.6 ± 0.2 eV and 2.46 ± 0.2 eV for direct and indirect band gap, respectively. Table 6 presents the band gap of some organic and inorganic semiconductors.

The direct gap band of CdS (inorganic semiconductor) was reported to be equal to 2.42 eV [41]. Khan et al. [42] have obtained values of 3.02 and 2.72 eV for the direct and indirect band gap of the inorganic semiconductor ZNNs. Yakuphanoglu [22] reported a value of 2.06 eV for the organic semiconductor MEH-PPV:C_{70} and a value of 3.32 for the organic semiconductor N-[5-methyl -1,3,4-

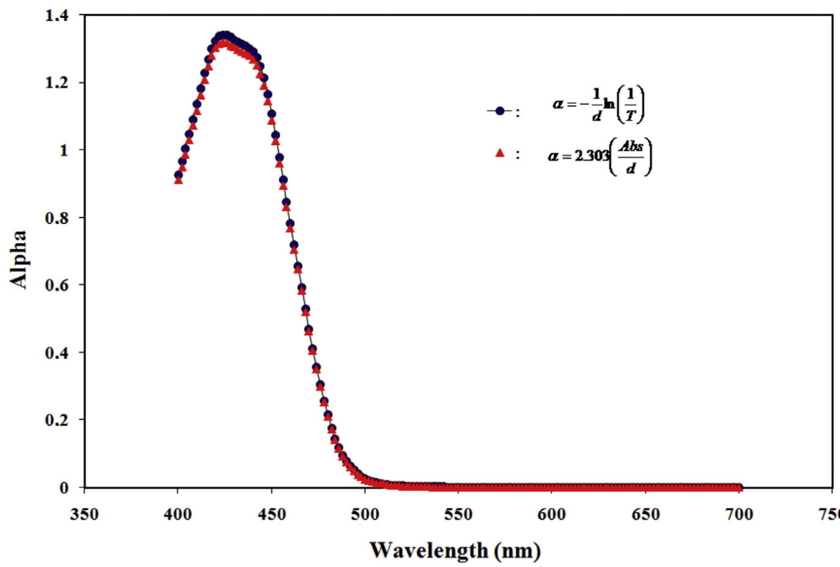

Fig. 9. Absorption coefficient, α, obtained from transmission spectra and empirical relation.

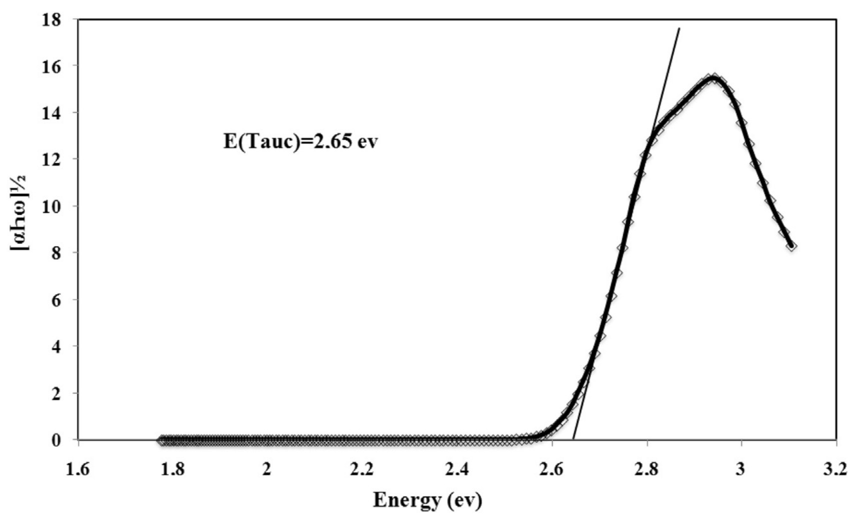

Fig. 10. Energy band gap (E_{Tauc}) for direct transition.

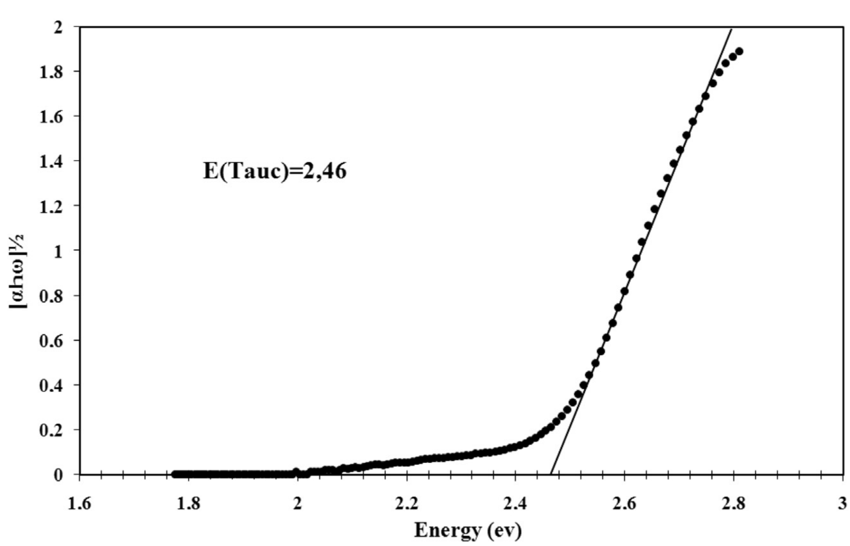

Fig. 11. Energy band gap (E_{Tauc}) for indirect transition.

tiyodiazole-2yl] dithiocarbamate [31]. Vazquez et al. [43] showed that the PTCDA organic semiconductor material has a gap band of 2.5 eV. In the light of the above the values of the developed semiconductor material are well comparable to the values reported in the literature and confirm that the it has the characteristics of semiconductors as it is well compared to the good semiconductors.

4. Conclusion

The structural, thermal, electrical and optical properties of the newly developed semiconductor material have been investigated using different characterization techniques. The results obtained in this study show that Acetaminophen/Curcumin organic semiconductor material possesses has a comparable conductivity to the available organic semiconductors. It has also a low activation energy ($E_a = 0.49$ eV) and permittivity (6.77) compared to the available organic semiconductors and silicon-based semiconductors, that has been studied by many research groups. The optical band gap energy of the material equals 2.6 eV and 2.46 eV for direct and indirect band gap, respectively. In addition, the material has high transmittance (97%). These properties are comparable to the available organic and inorganic semiconductors. Therefore the newly developed semiconductor material is of great importance and has a potential applications in the field of organic electronics and in the manufacturing of photovoltaic cells.

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