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Structural and optical characterization of pure and SnZrO$_3$ doped PS based polymer nanocomposite

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Abstract

This report presents a straightforward, an efficient and a promising methodology for the preparation of nanocomposite polymer (NCP) systems composed of polystyrene (PS) as the host polymer with addition of SnZrO$_3$ filler. For structural study and optical properties determination of the prepared samples; x-ray diffraction (XRD), and ultraviolet-visible (UV–vis) spectroscopy was performed, respectively. From XRD pattern several new peaks of the composites appearance as an evidence of strong interaction between the polymer matrix and the filler. The crystalline size of nan–particles were estimated using Debye–Scherrer’s equation. The fundamental optical parameters, for instance refractive index ($n$), optical band gap energy ($E_g$), optical dielectric loss ($\varepsilon_i$), and optical dielectric constant ($\varepsilon_r$) have been determined in which the quantity of SnZrO$_3$ is effective. Tauc’s equation has been used in the specifying the direct and indirect band gap energy. The absorption edge ($E_a$) shifting from 4.42 to 3.74 eV is associated to the charge-transfer complexes creation within the composite films. There is an increasing in the $n$ from 1.2 to 2.54 for pure PS upon addition of 12 wt.% doped that indicates the existence of few interaction between photons and electrons. The optical band gap and transition type are studied in detail. The exponent value ($r$) has been identified from Tauc’s equation using the $\varepsilon_i$ against photon energy to measure optical band gap energy and specify the electronic transition types. This doping of filler has considerably increased the $\varepsilon_r$ from (1.5–6.4) for pure PS sample and doped SnZrO$_3$ nanoparticles.

1. Introduction

The unique properties of metal nanoparticles and nanocomposites make studies on the subject of one of the hot topics in terms of fundamental and technological aspects. The most common properties of these materials are desired optical, thermal, mechanical, electronic, and electrical properties [1, 2]. Then nanocomposites can be classified on the basis of chemical composition as aceramic nanocomposite, metal nanocomposites, and polymer nanocomposites [3]. From these materials one can improve the optical properties of polymer through addition of suitable nano filler with controlling the concentration [4]. The decisive factors in enhancing the polymer properties are the way of dispersion of the nano fillers and the nature of interaction with the polymer [5]. To deal with the band structure and energy gap in polymer matrices, it is proper to examine the optical absorption spectra of nanocomposite particles (NCPs) [6]. Therefore, such systems as optical materials are applicable in several different areas, for example, glass lens, camera lens, optical reflectors [7, 8]. Moreover, these modified materials are used in numerous areas for example light-emitting diodes and solar cell [9, 10], optoelectronics and photonics [11–13]. The study of the absorbance edge and index of refraction enable us to have a comprehensive understanding of the nature of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) of the linear and cyclic alkanes [14]. To be applicable in electronics, the non–polar polymers are needed that have lowest dielectric constant ($\varepsilon_r$) compared to those solid
polymers that known till now. Yang et al concluded that polymers that possess the lowest index of refraction are demanded as low-index claddings for waveguide applications [15].

The present work has focused on the study the optical properties of SnZrO₃ nanoparticles within the polystyrene (PS) host polymer using the UV–vis spectroscopy. It has also limited to tackle the effect of SnZrO₃ nanoparticles on the optical band gap decrement of PS based nanocomposite polymer (NCP) films. The occurrence of electronic transitions within energy level in the structure of the materials has been directly correlated to the photon energy. It has been revealed that the measurement and understanding of the optical properties of materials are key factors on deciding the suitability for various applications.

### 2. Experimental details

The raw materials in this study are pure polystyrene (PS) and SnZrO₃ nanoparticles that purchased from Sigma Aldrich. The solution casting methodology was used in the preparation of nanocomposite polymer (NCP) films based on PS. To prepare the solution of PS, one gram of PS powder was dissolved in 40 ml Toluene with stirring via a magnetic stirrer for 3 h until a homogeneous solution was obtained. To this solution, various amount of SnZrO₃ of 4, 8 and 12 wt. % nanoparticles were inserted under continuous stirring to prepare various composition of PS:SnZrO₃ (NCP) systems. Eventually, the casting process of the solutions of pure PS and PS:SnZrO₃ were performed in different glassy and dry Petri dishes and then permitted to evaporate at ambient temperature for two weeks until solid films without solvent was acquired. Subsequently, the PS polymer nanocomposite films were coded as NCP-0, NCP-1, NCP-2 and NCP-3 correspond to PS incorporated with 0, 4, 8, and 12 wt. % of SnZrO₃, respectively. Table 1 presents the composition of PS:SnZrO₃ nanocomposite polymer films.

The structural properties were examined using x-ray diffractometer (XPERT-PRO) equipped with a radiation source of Cu kα (λ = 0.154 nm) in the 2θ range (10°–70°) at a scan rate of 2° min⁻¹. The optical properties of pure PS and PS:SnZrO₃ films were conducted at room temperature using double beam UV–vis-NIR spectrometer (Lambda 25 model) from Perkin Elmer.

### 3. Results and discussion

#### 3.1. X-ray diffraction

It is well-known that structural study needs for acquiring x-ray diffraction spectrum. The XRD spectra of pure PS and PS:SnZrO₃ nanocomposites polymer films are shown in figure 1. It is seen that the relatively largest specific diffraction peak at 2θ locates between 15°–22° is due to the amorphous nature of polystyrene [16, 17]. Comparison of the crystal structure of the PS nano-composite films and pure PS it is seen that there is no alteration upon addition of nanoparticles from the XRD patterns. It has been documented that pure PS does not produce sharp peaks, instead possesses an amorphous halo [17]. The appearance of several diffraction peaks at scattering angles 2θ = 28.25°, 30.65°, 32.05°, 43.95°, and 44.95° of the nanocomposite samples is characteristic. It is also observed that the intensity of these peaks increases upon addition of the SnZrO₃. To determine the crystalline sizes, Debye–Scherrer’s equation is used [18, 19].

\[
D = \frac{k \lambda}{\beta \cos \theta}
\]

Where; D refers the particle size, λ refers the x-ray radiation wave length (Cu kα radiation λ = 0.154 nm), k is a constant and having a value of 0.9, full width at half maximum (FWHM) in radian is referred by β, and Bragg angle of peak is referred by θ. From the XRD analysis, the average sizes of SnZrO₃ nanoparticles have been estimated and found to be 30.82 nm. The required parameters shown in equation (1) for the calculation of the size of nanoparticles are exhibited in table 2.

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| Table 1. Composition of PS based Nanocomposite Polymer Films. |
|---------------------------------------------------------------|
| Designation | PS (g) | SnZrO₃ (wt%) | SnZrO₃ (g) |
|------------|--------|-------------|------------|
| NCP-0      | 1      | 0           | 0.0000     |
| NCP-1      | 1      | 4           | 0.0416     |
| NCP-2      | 1      | 8           | 0.0869     |
| NCP-3      | 1      | 12          | 0.1363     |
3.2. Optical properties

3.2.1. Absorption study

The optical response of the polymer has been changed significantly with addition of SnZrO3 nanoparticles. UV–vis spectroscopy provides insight into optical band gap energy by giving the value. Promotion of electron in $\pi$, $\sigma$, and n- orbits from the ground state to higher energy states is performed by the absorption of light in the ultraviolet and visible regions as described by molecular orbital theory [20]. The spectra of pure PS sample and the doped ones with SnZrO3 nanoparticles are shown in figure 2. It is noted that upon the addition of SnZrO3 nanoparticles, the absorption increases considerably. It is also seen that the absorption peaks in the spectra of the samples with wavelength are sharp and it is non-exponential. This feature of absorption peaks with wavelength for the whole samples indicates the dominancy of crystalline nature of the samples. Based on these results, the behavior of absorption coefficient ($\alpha$) versus photon energy (eV) should be nonexponential [21]. The variation in the absorbance value of the pure PS sample with increasing the quantity of SnZrO3 nanoparticles would be correlated to influence of dopant quantity. The increase in dopant results in decreasing the transparency of the sample that attributed to that there is a modification of the molecular configuration [22]. The photograph image shown in figure 3 shows a realized nanocomposite film.

![Figure 1](image1.png)

**Figure 1.** The XRD patterns for the (a) Pure PS (NCP-0), (b) NCP-1, (c) NCP-2, and (d) NCP-3 samples.

### Table 2. Nanoparticle size from Debye-Scherre’s equation.

| $2\theta$ degree | cos $\theta$ | D (nm) | $D_{\text{Average}}$ (nm) |
|-----------------|-------------|--------|--------------------------|
| 28.25°          | 0.969       | 27.5   | 30.82                    |
| 30.65°          | 0.964       | 27.63  |                          |
| 32.05°          | 0.961       | 41.43  |                          |
| 43.95°          | 0.927       | 28.74  |                          |
| 44.95°          | 0.924       | 28.84  |                          |

3.2.2. Refractive index study

It is known that the samples optical parameters can be determined due to the analysis of reflection and absorption spectra. The refractive index ($n$) is one of the optical properties represents the lowering in the rate of the light speed in a medium [23]. The optical refractive index ($n$) of the medium, is one of the crucial parameters that can be obtained by taking into consideration both reflectivity($R$) as well as extinction coefficient ($K$) of the film in the following relationship [24].

$$n = \left[ \frac{1 + R}{1 - R} \right] + \sqrt{\frac{4 \times R}{(1 - R)^2}} - K^2$$  \hspace{1cm} (2)

Where; $K$ is proportional to both wavelength ($\lambda$) and absorption coefficient ($\alpha$) directly and proportional to the film thickness ($d$) inversely through $K = \alpha \lambda / 4\pi d$. The $n$ alteration against wave length for all samples is presented in figure 4. It is clearly seen that the $n$ values increase with increasing SnZrO3 nanoparticles content.
Today, the demand for optical materials with having large $n$ in the field of filters, ophthalmic lenses, optical adhesive, relatively high reflective, antireflection coating and advanced optoelectrical fabrications increases [25]. This change in the $n$ value is an evidence of occurring interactions between photons and electrons in the films. For example, at the high wavelengths, the films show non-dispersive behavior and $n$ become constant. Here in, the $n$ at infinite wavelength ($n_\infty$) for the films of the pure PS and wt.%SnZrO$_3$ dopant was measured [26, 27]. A series of small values of $n$ tabulated in the current work reveals that PS based nanocomposite polymer are very qualified as low-index cladding for waveguide applications and presented in table3 [21]. The obtained $n$ values as illustrated in figure 4 indicates that the insertion of SnZrO$_3$ filler into PS polymer matrix can alter the refractive index of the composite films and as a result boost the $n$ value from 1.2 to about 2.54. The magnitude of refractive index, $n$ relies on the identity (chemical composition) of materials, for example, 1.33 and 1.0 are recorded for water and air, respectively [28]. Moreover, the refractive index, $n$ of most polymer materials and titanium oxide pigment are 1.5 and 2.5, respectively. The value of refractive index of 1.3 to 1.7 for conventional polymers has been documented [29] while higher value has been reported for most of inorganic materials [30, 31]. Jin et al [8], documented that the refractive index of PMMA can be improved from 1.49 to 1.839 upon the addition of 20 wt.% of TiO$_2$ which is lower than that obtained in this work for PS incorporated with SnZrO$_3$ nano-particles.

**Figure 2.** The absorption spectra of pure PS sample and doped with SnZrO$_3$ nanoparticles.

**Figure 3.** The photograph image of pure PS and nanocomposite films.
3.2.3. Absorption edge and band gap study

The optical absorption coefficient ($\alpha$) reflects the capacity for absorbing light by materials. To determine the vibrational bands as well as electron transitions in energy states, it is proper to perform optical analysis. It is also allowed to calculate the value of materials energy band gap via effectively analysis of UV–vis spectroscopy. For example, the band gap energy is measured from an electron jump in the absorption process defined as absorption edge ($E_a$). The $E_a$ is a region where an electron is induced to excite by photons from low energy states to higher energy states [22, 32]. The $\alpha$ represents the relative decrease in rate if light intensity, which can be measured from the absorbance ($A$) due to the Beer–Lambert’s equation [33, 34].

$$\alpha = 2.303 \frac{A}{d}$$  \hspace{1cm} (3)

Where; $d$ is the sample thickness.

The $\alpha$ for pure PS and its composites are shown in figure 5. It is observed that there is a shift in the $E_a$ to lower photon energies ($h\nu$) for the system of PS:SnZrO$_3$. Estimation of $E_a$ for the samples were conducted via extrapolation of the linear part of $\alpha$ as opposed to $h\nu$ to zero value of absorption. The estimated $E_a$ values are presented in table 3. These relatively high values are directly correlated to the charge transfer occurrence in the composite complexes films [35]. The optical band gap energy for the doped samples decreases as evidenced from this shifting in $E_a$ toward the lower photon energy [24].

Investigation of optical absorption provides insight into the band structure of solids. In terms of band gap, materials, such as semiconductors/insulators are normally categorized into two categories; direct and indirect band gaps. In the former one, the top of the valance band (VB) and the bottom of the conduction band (CB) both lie at same zero wave vector (crystal momentum). In contrast, the bottom of the CB is not corresponded to zero wave vector in indirect band gap semiconductor [36, 37]. To calculate optical energy gap ($E_g$), Tauc’s equation can be applied as shown below [23, 27].

![Figure 4. The refractive index spectra of pure PS sample and doped with SnZrO$_3$ nanoparticles.](image)

Table 3. Refractive index ($n$) and Absorption edge ($E_a$) values of PS based Nanocomposite polymer films.

| Films    | $n_{\infty}$ | Absorption Edge $E_a$(eV) |
|----------|--------------|----------------------------|
| NCP-0    | 1.2          | 4.42                       |
| NCP-1    | 2.08         | 4.35                       |
| NCP-2    | 2.32         | 3.84                       |
| NCP-3    | 2.54         | 3.74                       |

Figure 4. The refractive index spectra of pure PS sample and doped with SnZrO$_3$ nanoparticles.
Where; B is a constant, $u_h$ refers the energy of photon. The value of $r$ is transition dependent factor. For example, the value of $r$ takes $\frac{1}{2}$ and 2 for allowed direct and indirect transition, respectively. The variation of $\alpha u_h^2$ and $\alpha u_h^{1/2}$ against $u_h$ for pure PS and PS:SnZrO$_3$ nanocomposite samples are shown in figures 6 and 7.

From the plots of $\alpha u_h^2$ and $\alpha u_h^{1/2}$ against $u_h$, the value of direct and indirect band gap can be determined. Importantly, it is straightforward to calculate the optical band gap ($E_g$) from equation (4) and estimate from the linear portion extrapolation of the plot to the axis of photon energy. The values of optical band gap ($E_g$) are presented in table 4. It is confirmed that with addition of wt.% of SnZrO$_3$ nanoparticles, the value of optical band gap ($E_g$) decreases. This is caused by introducing new states into the optical band gap. These new levels said electron crossing t VB to these local levels to the CB. As a consequence, with increasing SnZrO$_3$ nanoparticles content, the band gap decreases [38]. The decision accurately on choosing of value of the exponent $r$ using Tauc’s equation is basically difficult in which the acquired optical band gap values vary significantly [32]. So, to identify accurately the electronic transition kind and band gap, the optics dielectric loss ($\varepsilon_i$) parameter has to be studied as discussed in next section.

\[
\alpha u_h = B \left( u_h - E_g \right)^r
\]
3.2.4. Optical dielectric study

The dielectric function relies strongly on the materials band structure. The optical spectroscopic techniques are powerful and straightforward in dealing with the materials band structure determination [6]. The availability of \( n \) and \( K \) values make determination of the complex dielectric constant (\( \varepsilon^{*} = \varepsilon_{r} + i\varepsilon_{i} \)) of the NCP be easily using the equations shown below [39, 40].

\[
\varepsilon_{r} = n^2 - K^2 = \varepsilon_{\infty} - \frac{\varepsilon^2}{4\pi^2\varepsilon_0 m^*\Lambda^2} \tag{5}
\]

\[
\varepsilon_{i} = 2nK \tag{6}
\]

Where; \( \varepsilon_{\infty} \) refers the dielectric constant in vacuum, \( \varepsilon_{\infty} \) is the residual dielectric constant, \( c, \Lambda, \varepsilon \) have usual meaning and \( \frac{N}{m^*} \) is the ratio between the density of the localized electronic levels and the effective mass.

Herein, as a replacement to Tauc’s equation, \( \varepsilon_{i} \) technique is used in the estimation of the band gap and also the electronic transitions nature. Figure 8 illustrates the variation of \( \varepsilon_{i} \) corresponds to photon energy for all the films. On the basis of the \( \varepsilon_{i} \), the optical band gap was obtained and tabulated in table 4. This is obtained from the linear part intersection on the \( x \)-axis in the figure 8. In a comparison view, determination of the electronic transition kinds from plots obtained from Tauc’s equation (see figures 6, 7) is less precise to that of \( \varepsilon_{i} \) (see figure 8). Interestingly, from the data analysis, it is clarified that the type of electronic transition of all samples are direct allowed type [24]. It is also achieved that estimation of optical band gap and specification of the electronic transition nature from both the \( \varepsilon_{i} \) spectra versus \( h\nu \) and Tauc’s model are easily performed. However, the \( \varepsilon_{i} \) is simpler and takes less time to carry out the data analysis [35]. Schematically the role of nano-filler on band gap reduction is shown in scheme 1. Based on optical dielectric loss results and Tauc’s model; direct allowed transition type is dominant. Schemes 2(A), (B) shows the direct and indirect transition types in insulating and semiconducting materials [41]. the results of the present work follow the direct transition as shown in scheme 2(A).

![Figure 7. Plot of \((\alpha h\nu)^{1/2}\) of pure PS sample and doped with SnZrO3 nanoparticles as a function of photon energy \( h\nu \).](image)

Table 4. Optical Band Gaps from Tauc’s Model and \((\varepsilon_i\text{ versus }h\nu)\) for all of the samples.

| Films Code | Direct optical band gap energy (eV) | Indirect optical band gap energy (eV) | Optical Band gap energy from \((\varepsilon_i\text{ versus }h\nu)\) (eV) |
|------------|----------------------------------|----------------------------------|----------------------------------|
| NCP-0      | 4.44                             | 4.35                             | 4.42                             |
| NCP-1      | 4.38                             | 4.28                             | 4.35                             |
| NCP-2      | 3.95                             | 3.70                             | 3.88                             |
| NCP-3      | 3.88                             | 3.66                             | 3.80                             |
Figure 8. Optical dielectric loss spectra of pure PS sample and doped with SnZrO₃ nanoparticles as opposed to photon energy $h\nu$.

Scheme 1. Effect of SnZrO₃ nano-filler on Band gap reduction.

Scheme 2. Direct and indirect transitions.
Figure 9 exhibits these optical dielectric constant ($\varepsilon_r$) values correspond to the wave length for pure PS sample and doped with SnZrO$_3$ nanoparticles. It is interesting to see that at the absorption edge, the $\varepsilon_r$ decreases for all samples. The relatively high value of the real part of the $\varepsilon_r$ can be related to its direct correlation with the square of the $n$ and with the minus of the $K$ as mathematically shown in equation (5) [42, 43].

It is important to notice that this increase in $\varepsilon_r$ value from 1.5 to 6.4 can be correlated to the increasing of density of states. There is a direct relation of ($\varepsilon_r$) parameter with the density of states inside the forbidden gap of the polymeric samples as established previously [44, 45]. the increase of density of states through the band gap region is responsible for the remarkable reduction in optical band gap.

4. Conclusions

In conclusion nano-composite polymer films of PS:SnZrO$_3$ have been prepared with tuned refractive index and reduced optical band gap using the casting methodology. The effect of SnZrO$_3$ on the nano-composite samples is confirmed from the appearance of additional diffraction peaks and increasing the intensity of these peaks win increasing nano-filler concentration. The average crystallite size of nanoparticles were found to be 30.82 using Debye–Scherrer’s formulae. It was found that the absorption increases remarkably upon addition of SnZrO$_3$ nano-particle. The absorption edge shifted to lower photon energy side with increasing nano-particle concentration due to the introduction of new stated into the band gap region. The increase of optical dielectric constant is an evidence for the increase of new stated and thus increasing of density of states. The refractive index was increased from 1.2 to 2.54. The optical band gap was studied in detail using two methods. The charge-transfer within the composite samples is responsible for decreasing the $E_a$ and consequently reducing the optical band gap. The plot of ($\alpha h\nu)^2$ and ($\alpha h\nu)^{1/2}$ against $h\nu$ are used accurately in the determination of the direct and indirect type of the optical band gap and also showing decreasing trend with increase of SnZrO$_3$ content. Ultimately, it is specified from $\varepsilon_r$ that the electron transition obeys the direct transition. In conclusion transition type which is a complicated subject in optical materials study can easily be specified through the optical dielectric loss plot.

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