# NANOINDENTATION AND OPTICAL PROPERTIES OF HIGH DENSITY POLYETHYLENE/ZnO NANOPARTICLES COMPOSITES

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This article studies the effects of nano-zinc oxide (ZnO) on the structure, nano-indentation and optical properties of High Density Polyethylene (HDPE). The nanocomposite samples were prepared by melt-blending process. The nanoparticles were added with different ratio 0.5, 1 and 2 % wt/wt. The nanocomposites were characterized using X- ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). As well as the dispersion of the nanoparticles were examined by the field emission scanning electron microscope (FESEM). The effect of different content of nanoparticles on the nano-mechanical properties was investigated by a nanoindentation test method. Spectrophotometric measurements of the transmittance and reflectance at normal incidence in the wavelength range of 200 - 2200 nm were used to study the optical properties. As well as, we determine different optical parameters and the energy gap for the samples under investigation. FTIR and X-ray proved the presence of ZnO, while the FESEM results showed good dispersion of ZnO in HDPE matrix. Mechanical properties of the nanocomposites were improved; nanohardness and elastic modulus of nanocomposites samples improved than pure HDPE. The analysis of absorption coefficient for the samples indicated allowed direct transition and the energy gap increase with increasing of the ZnO wt%.

(Received July 23, 2018; Accepted September 25, 2018)

Keywords: HDPE, ZnO nanoparticles, FESEM, Nanoindentation and Optical properties

### 1. Introduction

Nanocomposites are considered as the next industrial revolution materials. Nanocomposites are new class of materials made by loading different nanoparticles organic, inorganic and natural materials as filler material in different polymer blends [1-5]. In the present era, the primary focus area is in identifying a nanocomposite material which is lighter in weight, eco-friendly, bio-degradable, cost-effective, as well as usage-oriented for a variety of applications.

Zinc oxide nanoparticles (nano-ZnO), is one of the important inorganic filler, which is a typical metal excess n-type ohmic semiconducting oxide with a wide and direct bandgap of 3.4 eV and a large exciton binding energy of 60 meV[6]. ZnO nanostructures with excellent electrical and optical properties are widely used as electronic and photonic materials for the field-effect transistor, UV light-emitters, transistors, gas sensors, acoustic wave devices, etc[7-9]. Recently, ZnO nanoparticles have attracted considerable attention as fillers for polymer nanocomposites, due to the reinforcement of ZnO NPs into the polymeric matrix enhances not only the optical properties but also mechanical[10]catalytic and antibactrial properties and so on [11].

One of the most important used polymer in the world polyethylene. Three kinds of PE can be produced commercially[12]. According to the Phillips and the Ziegler–Natta processes, the homo-polymerization of ethylene or the copolymerization of ethylene with a small amount of higher $\alpha$ -olefin can produce high-density PEt (HDPE)[13-15]. High-density polyethylene, HDPE has very high tensile strength due to its highly crystalline structure, compared to other kinds of PE. Its reasonable cost and low energy demand for processing make it ideal for many applications as packaging, bottles, films, pipes manufacture etc. In addition, HDPE has excellent mechanical

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properties, which make it ideal for industrial applications. As well as it has good barrier for humidity and resistance to abrasion and corrosion, and it presents inertness to the most of chemicals. Most of these mentioned properties can be improved when various inorganic compounds are incorporated into the polymer matrixes [15,16].

Although several studies have been performed in HDPE by addition different nanoparticles [16-18], studying the optical properties is very importance to determine band gap energy, optically active defects and band structure etc., which may be of permanent interest for several different applications for the nanocomposites [19-21]. For this reason in the present study nanocomposites of HDPE with nano-zinc oxide (ZnO) were prepared to evaluate the nanomechanical behavior and optical properties of HDPE by addition of ZnO nanoparticles.

### 2. Experimental

#### 2.1 Materials

High-density polyethylene pellet form, (density =  $0.931 \text{ g/cm}^{3}$ , melt flow index (MFI) ~ 3.0 g/min, and its melting point = 140 °C) was purchased from Exxon Mobil chemical (Riyadh, Saudi Arabia). Stearic acid, u.s.p 22, pharmaceutical chemicals, El Nasr pharmaceutical chemical co, Abu Zaabai, Egypt. Zinc oxide (ZnO), nanopowder<50 nm particle size (BET), product of USA, MW 81.29 g/mol , assay 97%, Sigma – Aldrich. Coupling agent is vinyl triethoxysilane(VTES), percent 97%, F.W = 190, B.P = 160-161 °C, Alfa AesarGmbh S Co KG. Zeppelin Strasse 76 Karl Sruche – Germany.

### **2.2 Preparation of samples**

Composites of HDPE are prepared by using method of melt blending, an accurate weight of high density polyethylene (HDPE) with the ingredients according to the formulations in Table 1 are melt mixed in a laboratory through a Haake Rheomex intermeshing twin-screw extruder (length=1100 mm, L/D=44). Firstly, HDPE pellets and stearic acid are melted at 140°C for 3 minutes at 40 rpm. Thereafter, different concentrations of nanoparticles ZnO are added gradually to the HDPE melt containing stearic acid till completely mixing are obtained for 7 min. Finally, the coupling agent is added gradually to the mixture (for 1 min at 40 rpm). The molten compositions containing of ZnO nanoparticles were immediately transferred from the mixer to an open roll – mill to form sheets. A sample of polymer sheet is then pressed in hydraulic press at 160 °C for 8 minutes (5 minutes preheating and 3 minutes at 20 MPa) to obtain a rectangular sheets of 2 mm thick. The molded sample is then cooled at the same pressure before opening the mould.

| Ingredients, phr <sup>a</sup> | Blank | Zn0.5 | Zn1 | Zn2 |
|-------------------------------|-------|-------|-----|-----|
| HDPE                          | 40    | 40    | 40  | 40  |
| VTES Coupling agent           | 0.4   | 0.4   | 0.4 | 0.4 |
| Stearic acid                  | 0.2   | 0.2   | 0.2 | 0.2 |
| Nano-ZnO                      | -     | 0.2   | 0.4 | 0.8 |

Table 1. Composition of HDPE/ZnO nanocomposites.

Note: <sup>a</sup> part per hundred parts of HDPE by weight

## **2.3 Characterizations and measurements for nanocomposites 2.3.1Fourier-Transform Infrared Analysis (FTIR)**

FTIR spectra for polymer nanocomposites was measured in the spectral range 4000–400 cm<sup>-1</sup>, this frequency region covers the absorption due to the fundamental vibrations of almost all the common functional groups of organic compounds. By using Bruker, Vector 22 infrared spectrophotometer. TheFTIR spectra were normalized and major vibration bands are associated with chemical group.

## 2.3.2 Field emission scanning electron microscope

The particle size distribution and surface morphology of nanoparticles embedded in polymer nanocomposites were examined using a high resolution scanning electron microscope (FESEM, Quanta FEG250 INSPECTS Company, Philips, Holland). The samples were sprayed with a thin layer of gold before being scanned.

#### 2.3.3 X-Ray diffraction

The X-ray diffraction (XRD) of the samples were recorded using Philips X-ray diffractometer model "X' Pert" with radiation source Cu K<sub> $\alpha$ </sub> (1.5405 Å) which operator at voltage (40 kV) and current (30 mA) with a step size of 0.02deg/min.

#### 2.3.4 Nanoindentation testing

Low load nanoindentation tests in this study were performed by Nano Test Vantage apparatus, (Micro Materials Co, UK, 2012). A spherical diamond indenter tip manufactured by Micro Materials was used. The specimens were mounted onto the nano-indentation fixture using a suitable adhesive. All tests were conducted at an approximate temperature of 27 °C. Typical experimental indentation parameters used for all measurements were as follows: Minmum load: 10 mN, the maximum load for all indents: 50 mN, Dwell time or holding time at maximum load: 20 s and number of indentation: 25 indent.

#### 2.3.5 Optical properties

The transmittance T ( $\lambda$ ) and reflectance, R ( $\lambda$ ) were measured at a normal incidenceof light in the wavelength range 200–2500 nm double beam spectrophotometer model (JASCO, V-570, UV–VIS–NIR) attached with constant angle specular reflection attachment (5°) with Al-mirror as reference.

The measurements of the transmittance T ( $\lambda$ ) and reflectance, R ( $\lambda$ ) were transformed to absolute values by making a correction to eliminate the absorbance and reflectance of the substrate by using a computer program comprising a modified search technique[22,23]in order to determine the optical constants.

The refractive index (n) and absorption coefficient  $\alpha$  for the samples at every wavelength were calculated using the following equation[24]:

$$\alpha = \frac{1}{d} \ln \left[ \frac{(1-R)^2}{2T} + \sqrt{\frac{(1-R)^4}{4T^2} + R^2} \right]$$
(1)

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

The values of the absorption index k can be calculated from the absorption coefficient using the relation  $k = \alpha \lambda / 4\pi [24]$ .

The experimental error in measuring the film thickness was taken as  $\pm 2\%$ , in T and R as  $\pm 1\%$  and in the calculated values of n and k as  $\pm 3.5\%$  and  $\pm 4\%$ , respectively.

## 3. Results and discussion

#### **3.1 Fourier-Transform Infrared Analysis (FTIR)**

The Fourier-Transform Infrared Analysis (FTIR) analysis is used to identify the interaction of polymer and nano filler materials to characterize the present of specific chemical groups. It is sensitive to morphology and, chain conformation of molecular environment which detection of transition between energy levels in the macromolecules[25, 26].(Fig. 1a). show the

FTIR absorbance spectrum of pure High Density Polyethylene (HDPE) film, while (Fig. 1b-1d) show the FTIR for HDPE/ZnO nanocomposites with different ZnO nanoparticle wt% (0.5,1and 2).

It is observed for pure HDPE a peak around 2978 cm<sup>-1</sup> related to the CH<sub>3</sub>. The peak at 2917and 2838 cm<sup>-1</sup> is corresponding to asymmetric and symmetric stretching vibration of CH2. The peak at 1468 cm<sup>-1</sup> is assigned to CH<sub>2</sub> bending deformation. The peak of Wagging deformation is found at 1366cm<sup>-1</sup>. The CH<sub>3</sub> rocking vibration observed at 1082 cm<sup>-1</sup> while CH2 rocking deformation is observed at about 724cm<sup>-1</sup>. However, the absorption band for ZnO was found in the region 550-500 cm<sup>-1</sup>[27]. As well as, it is observed a little shift in the peak in(Fig. 1b-1d), which is due to the effect of addition the ZnO nanoparticles but not modified drastically. Also, the figures show that the intensity of ZnO peak increased with the increase of ZnO% in the nanocomposites from 0.5wt% up to 2wt%.



Fig. 1.Infrared sepectra of pure HDPE and HDPE/ZnO nanocomposites.

## 3.2 X-Ray diffraction

X-ray diffraction patterns (XRD) shown in (Fig.2) for pure HDPE film and HDPE/ZnO nanocomposites was taken in a 2 $\theta$  range from 10° to70°. The pattern analysis confirms the crystalline structure of the investigated samples. For HDPE and HDPE/ZnO nanocomposites two basic peaks are shown at 2 $\theta$  (21.4° and 23.8°) which is due to (110) and (200) reflections associated with the orthorhombic structure for HDPE[28]. The angles of the two peaks did not change by the addition of ZnO which indicates that the interaction between ZnO nanocomposites and HDPE may be done by physical process.



Fig. 2. X-ray diffraction patterns of pure ZnO, HDPE and HDPE/ZnO nanocomposites with different wt%.

Fig. 2 shows another diffraction peaks at  $2\theta$  (31.7<sup>•</sup> 34.3, 36.26, 47.6, 56.6,62.7 and 66.06) related to ZnO nanoparticles [29]. They are in accordance with the zincite phase of ZnO which are typical for the hexagonal ZnO wurtzite structure with Lattice Constant, a = 3.245 Å; c = 5.207 Å and Space Group P63mc. The relative intensity of these peaks of HDPE/ZnO nanocomposites increases with increasing ZnO nanoparticles wt% in the nanocomposites. These changes will effect on the physical and optical properties of HDPE/ZnO nanocomposites.

### 3.3 Field Emission Scanning Electron Microscope FESEM:

When good compatibility between the polymer and the filler is achieved, it will lead to a significant morphological change. FESEM was used to confirm the dispersion of ZnO nanoparticles in the HDPE matrix and to show these morphological changes. The cross-sectional morphologies of the pure HDPE film and HDPE/ZnO nanocomposites with different ZnO NPs contents are shown in (Fig. 3). It is easy to observe that all the nanocomposites films show changes in the surface morphology than pure HDPE. The image of neat HDPE is homogenous and smooth, has no roughness (Fig. 3a). However, the addition of nano-ZnO in HDPE matrix creates a rough and heterogeneous surface as well as the roughness increases with increasing the concentration of ZnO in the matrix. Additionally, ZnO nanoparticles were dispersed evenly with no agglomeration in ZnO.5, Zn1 and Zn2 samples(Fig. 3 b-d). These observations indicate the incorporation of ZnO nanoparticles had a strong influence on the morphology of polymer[30-32], which means that VTES coupling agent, interacts with the ZnO nanoparticles preventing their aggregation and agglomeration. The presence of lone pair electron on oxygen atom in VTES may be combined with (Zn2+) in ZnO NPs during mixing and compressing process.



Fig. 3. FESEM images for Pure HDPE and the HDPE/ZnO nanocomposites with different wt%, (a) Pure HDPE, (b) HDPE/ 0.5wt% ZnO, (c) HDPE/ 1wt%ZnO and (d)HDPE/ 2wt%ZnO.

#### **3.4 Nanoindentation**

A typical loading–unloading curve for pure HDPE and the nanocomposites samples with various concentrations is presented in (Fig.4). As observed from the figure that the first portion of the curve (up to about 3500 nm depth) is straight line that indicates the pure elastic behavior of polyethylene while the remaining part of the curves indicates the transition from pure elastic to elastoplastic deformation.



Fig.4. Typical loading hold unloading curves for pure HDPE and the HDPE/ZnO Nanocomposites with different wt%.

Some important parameters have been obtained from the load-depth relation and have been collected in Table [2]. These parameters are maximum depth at peak load, nano-hardness H, and elastic modulus $E_s$ . The hardness and elastic modulus have been calculated using the method developed by Oliver and Pharr[33].

$$H = \frac{P_{max}}{A}$$
(3)

Where P<sub>max</sub> is the peak indentation load and A is the projected contact area at maximum load, and

$$E_{\rm r} = \frac{\sqrt{\pi} \,\mathrm{dP}}{2} \frac{1}{\mathrm{dh}} \frac{1}{\sqrt{A}} \tag{4}$$

Where,  $E_r$  is the reduced elastic modulus which accounts for the fact that elastic displacement occurs in both indenter and sample, and dP/dh = S (the slope of the upper portion of the unloading curve known as the elastic contact stiffness).

Thus the elastic modulus E of the sample can be deduced by:

$$\frac{1}{E_{\rm r}} = \frac{(1 - \nu_{\rm s})^2}{E_{\rm s}} + \frac{(1 - \nu_{\rm i})^2}{E_{\rm i}}$$
(5)

where,  $v_s$  is the Poisson's ratio for the sample; (for polymer approximately 0.2), vi the Poisson's ratio for the indenter (for diamond 0.07), Es the elastic modulus for the sample and Ei the elastic modulus for the indenter (1141 GPa is often used for a diamond). By rearranging Eq. (5), the equation for elastic modulus of the sample Es is:

$$E_{s} = \frac{1 - (v_{s})^{2}}{\frac{1}{E_{r}} - \frac{1 - (v_{si})^{2}}{E_{i}}}$$
(6)

| ZnO<br>nanoparticles<br>wt% | Maximum depth,<br>nm | Hardness,<br>GPa | Reduced modulus,<br>GPa |
|-----------------------------|----------------------|------------------|-------------------------|
| 0                           | 9829                 | 0.882            | 0.0564                  |
| 0.5                         | 7668.8               | 1.233            | 0.0702                  |
| 1                           | 6868                 | 1.253            | 0.0753                  |
| 2                           | 6612                 | 1.314            | 0.0777                  |

Table2.Mechanical parameters of the pure HDPE and HDPE/ZnO nanocomposites.

Initially Table [2] displays the average maximum depth at peak load for unreinforced HDPE sample is approximately 9829 nm, while for the nanocomposites are 7668.8, 6868, and 6612 for Zn0.5, Zn1, andZn2 reinforced samples, respectively. These values show that the depth at peak load is approximately about 21,26and 28 % lower than that of the pure HDPE for Zn0.5, Zn1 and Zn2 nanocomposites. The extent of nano-ZnO dispersion may have contributed to higher resistance to plastic deformation leading to a lower indentation depth [34, 35].

Additionally, it is observed from the table that the nano-hardness of the nanocomposites improved with increasing the ZnO content in the nanocomposite samples. In comparison with pure HDPE sample, nano-hardness increased by 0.39 and 0.42 and 48% (from 0.882 GPa to 1.233, 1.253 and 1.314GPa) for theZn0.5, Zn1 and Zn2 nanocomposites samples respectively.

Young's elastic modulus implies measure of bond strength between atoms; it can be altered either by substitution or by insertion of atom at lattice points. The elastic modulus calculated from the reduced modulus values according to equation (6). It is clear from (Table2) that the elastic modulus increased by 27, 33 and 37 % for Zn0.5, Zn1 and Zn2 nanocomposites samples respectively than neat HDPE (from 0.0564 GPa to 0.0702 to 0.0753 0.0777 and GPa), The nano-hardness and the modulus results are associated with the dispersion of the ZnO nanoparticles in the HDPE matrix. Good dispersion achieved for Zn0.5, Zn1 and Zn2 samples as (Fig 3) displayed, resulted in improving in nanohardness and the modulus compared with pure HDPE. The addition of hydrophilic NPs to hydrophobic HDPE polymer is expected to decrease the tensile mechanical strength and Young's modulus, but addition of coupling agent (silane) well improve the

compatibility between the NPs and HDPE during the heat mixing process, through improving the interfacial interaction between HDPE and the NPs [36, 37].

### 3.5 Optical characterizations

The absorption coefficient spectra for HDPE/ZnO nanocomposites near the fundamental edge is the most important optical constant, it used to estimate the band gap and which is type of internal transitions of sample. Which calculated from the transmittance T ( $\lambda$ ) and reflectance R ( $\lambda$ ) for pure HDPE and HDPE/ZnO nanocomposites with different weight% (0.5, 1 and 2) in the wavelength ranged from 200-2200nm. The function of photon energy (hv)for the samples are illustrated in (Fig. 5), the figure shows that the absorption coefficient decreased with the increase of ZnO nanoparticles in the HDPE matrix and the absorption edge shifts towards the higher energy.

Analysis of the optical absorption at the fundamental edge in terms of band-to-band transitions theory [38, 40]gives information about the optical transitions and band gap which given by the following equation[25, 41]:

$$(\alpha h \upsilon) = G(h \upsilon - E_g \pm E_{ph})^r.$$
<sup>(7)</sup>

Where G is a constant,  $E_g$  and  $E_{ph}$  is the optical band gap and phonon energy.



Fig. 5. The optical absorption spectrum of HDPE/ZnO nanocomposites films of different weight %.

The dependence of  $(\alpha hv)^r$  vs. (hv) near the absorption edge was plotted for different values of r and it was found the best fit at r =2 (Fig. 6), which indicated the transitions are direct allowed. The obtained energy band gaps are listed in Table 3. It is clear from the table the increase of the energy gaps values with increasing the of ZnO wt%, this is due to the change in the electronic structure of the polymer matrix, resulting in increases the degree of disorder in the films due to appearance of defect levels and various polaronic[42].



Fig.6.Dependence of  $(\alpha h v)^2$  on (h v) of (a) Pure HDPE, (b) HDPE/ 0.5wt% ZnO, (c) HDPE/ 1wt%ZnO and (d)HDPE/ 2wt%ZnO

The refractive index  $n(\lambda)$  forpure HDPE and HDPE/ZnO nanocomposites with different weight % is depicted in (Figs. 7). It is observed from the figure the presence of different peaks in the ultraviolet and visible region of spectra at 260 and 320 nm for pure HDPE, at 230, 280 and 390 nm for Zn0.5, at 230 and 410 nm for Zn1 and at 210,250and 420nm for Zn2, which can be explained by adopting multi-oscillator model. There are variations in the intensity and slightly in the position of these peaks. At wavelength,  $\lambda$ >900 nm, the spectral behavior of n is normal dispersion at which single oscillator model is applicable in this region. The dispersion theory describes the variation of the refractive index, which analyzed using the concept of the single oscillator model expressed by the Wemple and DiDomenico [43].It is an important in the research for optical materials.



Fig.7.Spectral behavior of refractive index, n, for Pure HDPE and the HDPE/ZnO nanocomposites with different wt%.

In the transparency region, the dispersion of the refractive index (n) in the region of very small values of the absorption constant k, under negligible damping can be expressed in the form [23].

$$(n^{2} - 1) = \frac{E_{d} E_{o}}{E_{o}^{2} - (h\nu)^{2}}$$
(10)

Where:  $E_d$  is the dispersion energy, which is measures the strength of interband optical transitions and  $E_o$  is the effective oscillator energy.

Linear least square fitting of  $(n^2-1)^{-1}$  against  $(hv)^2$  was plotted for pure HDPE and the HDPE/ZnO nanocomposites with different wt% (Fig. 8).  $E_o$  and  $E_d$  can be determined from the slope,  $(E_d E_o)^{-1}$ , and the intercept,  $E_o/E_d$ , on the vertical axis. The value of static refractive index  $\varepsilon_{\infty}$  can be obtained by extrapolating the relation in low energy limit  $(hv \rightarrow 0)$ . The calculated values of  $E_o$ ,  $E_d$  and  $\varepsilon_{\infty}$  are also illustrated in Table 3.

| ZnOwt% | $\mathbf{E}_{\mathbf{g}}$ | ε <sub>∞</sub> | ε <sub>L</sub> | $(N/m^*) \times 10^{45} g^{-1} cm^{-3}$ | (E <sub>o</sub> eV) | (E <sub>d</sub> eV) |
|--------|---------------------------|----------------|----------------|---|---------------------|---------------------|
| 0      | 2.89                      | 1.23           | 1.26           | 10                                      | 3.4                 | 0.79                |
| 0.5    | 2.9                       | 1.33           | 1.34           | 0.95                                    | 9.5                 | 3.2                 |
| 1      | 2.58                      | 1.196          | 1.25           | 12.2                                    | 2.41                | 0.47                |
| 2      | 2.12                      | 1.16           | 1.169          | 1.78                                    | 4.11                | 0.69                |

Table 3. Optical parameters of the pure HDPE and HDPE/ZnO nanocomposites.

Another dielectric constant,  $\varepsilon_L$  (n<sup>2</sup>), can be calculated from the following equation[44].

$$\varepsilon_1 = n^2 = \varepsilon_L - \frac{e^2 N}{4\pi^2 \varepsilon_a m^* c^2} \lambda^2 \tag{9}$$

where  $\varepsilon_L$  is the lattice dielectric constant,  $\varepsilon_o$  is the permittivity of free space, and N/m<sup>\*</sup> is the ratio of the carrier concentration to the electron effective.

It is observed in (Fig.9) the relation between  $n^2 \& \lambda^2$  is linearity at longer wavelengths. Extrapolation of these plots when  $\lambda^2 \rightarrow 0$  gives,  $\epsilon_L$  and from the slopes of the graphs N/m<sup>\*</sup> of the samples were calculated and listed in Table 3.



Fig.8.The relation between  $(n^2 - 1)^{-1}$  and the squared photon energy  $(hy)^2$  for HDPE/ZnO nanocomposites films of different weight %.



Fig.9.Plot of  $(n^2)vs.(\lambda)^2$  for Pure HDPE and the HDPE/ZnO nanocomposites with different wt%.

## 4. Conclusions

The objective of this study was to evaluate the nanomechanical behavior and optical properties of HDPE by addition of ZnO nanoparticles. HDPE/ZnO nanocomposites were prepared by using melt-blending method. The nanoparticles were added with different ratio (0.5, 1 and 2 wt%). FTIR and X-ray proved the presence of ZnO, while the FESEM results showed good dispersion of ZnO in HDPE matrix for samples contains 1 and 2 wt% ZnO. Mechanical properties of the nanocomposites were enhanced; nano-hardness for Zn1 and Zn2 nanocomposite samples are higher in comparison with the pure HDPE sample, however, Zn0.5 nanocomposite sample has lower value of nano-hardness than pure HDPE.

The analysis of absorption coefficient for the samples indicated allowed direct transition and the energy gap increase with increasing the of ZnOwt%. Fitting of the refractive index showedan normal dispersion at (transparent region) and anomalous dispersion at (absorption region). Other parameters of dispersion were calculated from the analysis of the normal dispersion curve using the single oscillator model.

### Acknowledgments

The author would like to acknowledge university of Tabuk for the financial support under research project number S-1438- 0136.

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