

Impact of ZnO Nanoparticle on the Structural and Optical Properties of Poly (vinyl alcohol) Film

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ABSTRACT

Using the cast technique thin films of poly (vinyl alcohol) (PVA) containing varying amounts of Zinc Oxide ZnO nanoparticles (0.001, 0.002, 0.003, 0.004 and 0.005) g were formed. Physical properties excluded structural; (X-Ray Diffraction) (XRD), Fourier Transform Infrared (FT-IR) spectroscopy and UV-Visible spectroscopy. XRD results showed the amorphous structure of PVA film and Hexagonal crystalline structure of ZnONPs, PVA/ZnO nanocomposite appears as pattern of PVA films so the peaks of ZnO don't appear in it. FTIR spectra revealed ZnONPs doesn't have any influence on polymer structure. The effects of the ZnONPs on optical characteristics of PVA like the absorption spectrum, transmission spectrum, energy band gap, absorption coefficient, extinction coefficient, refractive index, real and imaginary parts of dielectric constant has been studied using UV-Vis spectroscopy. This study demonstrates that increasing the number of ZnONPs has an effect on all of these parameters.

KEYWORDS: Nanocomposite, Optical Properties, PVA/ZnO Nanoparticle, XRD, FTIR, UV-Visible

الخلاصة

تم استخدام تقنية الصب لتحضير اغشية رقيقة من بولي فينيل الكحول (PVA) و PVA التي تحتوي على كميات مختلفة من الزنك اوكساييد (0.001، 0.002، 0.003، 0.004 و 0.005) غرام. تم اجراء فحوصات التي تتضمن حيود الاشعة السينية XRD ومطياف الاشعة تحت الحمراء FTIR ومطياف الاشعة فوق البنفسجية UV-Visible. حيث بين فحص XRD ان PVA يمتلك ترتيب عشوائي و ZnO ذات هيكل سداسي وعند دمج PVA مع ZnONPs لا يؤثر ZnONPs على بنية PVA. وجد ان زيادة ZnONPs تؤثر على الامتصاصية والنفاذية ومعامل الامتصاص ومعامل الانكسار ومعامل الاخمد والجزء الحقيقي والخيالي من ثابت العزل الكهربائي.

INTRODUCTION

Polymeric materials have piqued the interest of technological as well as scientific experts due to their broad range of uses. It is effective in creating new medical, industrial, and electrical applications because of its superior mechanical strength, lightweight, and optical characteristics [1, 2]. Metallic NPs have distinctive physical and chemical characteristics than bulk metals (high specific surface area, low melting points, mechanical strength, specific optical qualities, and specific magnetization) that could be useful in a variety of industrial applications [3]. ZnO can be defined as one of the semiconductors with a wide band gap (3.37eV) and a substantial excitation binding energy of 60meV at room temperature, which is significantly larger than other materials. It also has high transmittance and good electrical

conductivity. Because of its distinctive mix of exceptional chemical, physical, electrical, biological, optical, long-term environmental stability biocompatibility, cheap cost, and non-toxic qualities, ZnO has piqued attention as multifunctional inorganic NPs [4]. M. C. Golchha and V. S. Sangawar [5], used cast process to create Polystyrene (PS) thin films with varied ZnONPs concentrations as a filler (0, 0.5, 1, 3, and 5 wt. %). By analyzing optical absorption spectra with a UV-Vis spectroscopy, the impact of ZnONPs on optical characteristics of the Polystyrene like transmittance, absorbance, refractive index, extinction coefficient, absorption coefficient, and imaginary and real parts of the dielectric constant were examined. S. G. Dhole *et al.* [6], used Chemical synthesis to create a PANI-based nano-composite filled with ZnONPs. As an

oxidant, aniline hydrochloride with the ammonium persulphate utilized. The XRD approach utilized for investigating the structure of produced nanocomposite. The Wurtzite structure of the ZnONPs confirmed by the XRD pattern. The optical behavior regarding PANI-ZnO nanocomposite after doping ZnONPs in the polymer matrix investigated using UV-Vis's spectra. N. Bouropoulos *et al.* [7], used casting approach to create nano-composites of PVA and ZnO. Thermal de-composition of zinc acetate dihydrate had yielded the ZnONPs having a hexagonal Wurtzite structure with average diameters of 59nm, 82nm, and 150nm. UV-Visible absorption spectroscopy to determine the optical characteristics of nanocomposites. Results have revealed that all of the samples had near-band edge absorption values that were close to bulk value. At room temperature, the broadband dielectric spectroscopy used in order to evaluate the impact of average particle size on interfacial relaxation events. Interfacial polarization that is present in all of the 3 nanocomposite films, shifts to higher frequency values and its intensity decreases as average nanoparticle diameter decreases, according to dielectric measurements. In this work, the caste method used to prepare five samples of PAV with ZnO at different amounts. These samples are examined with XRD, FT-IR, and UV-Vis spectroscopy to check pure samples and the nanocomposite samples in terms of structure and optical properties change.

MATERIALS AND METHODS

Absorbance characterized as ratio of the intensity of the absorbed light (I_A) by material to light's incident intensity (I_0) [8]:

$$A = \frac{I_A}{I_0} \quad (1)$$

The coefficient of the absorption (α) calculated from spectrum of optical absorption based on the equation below [9].

$$2.303 \frac{A}{d} \quad (2)$$

where, d represents the thickness of the film in cm and A represents the absorbance. From the equation below, the extinction coefficient can be calculated (k) [10]:

$$k = \frac{\lambda \alpha}{4\pi} \quad (3)$$

where λ represents wave-length to the incident light. From the equation below the energy band gap can be calculated [11]:

$$\alpha h\nu = B(h\nu - E_g)^x \quad (4)$$

where, α represents the coefficient of absorption, h represents Planck's constant, ν represents frequency, E_g represents optical energy band gap between valence band and the conduction band, x represents power characterizing transition process and B represents a constant value. In particular, x could take values 3/2, 1/2, 3 or 2 for the transitions that designated as direct forbidden, direct allowed, indirect forbidden and indirect allowed respectively. The determination of optical energy band-gap value E_g includes plotting of $(\alpha h\nu)^{1/x}$ against $h\nu$. Reflectance (R) found from Absorbance (A) and transmission (T) values, using the following equation [12].

$$A + R + T = 1 \quad (5)$$

From the next equation, refractive index(n) can be calculated [13]:

$$n = \frac{1 + R^{1/2}}{1 - R^{1/2}} \quad (6)$$

where R : is the reflectance

From next equations, real (ϵ_r) and imaginary (ϵ_i) dielectric constant parts can be calculated [14]:

$$\epsilon_r = n^2 - k^2 \quad (7)$$

$$\epsilon_i = 2nk \quad (8)$$

Experimental work

PVA powder was obtained from (THOMAS BAKER) company, with an average molecular weight of 14000g/mole. And a nano-powder of Zinc Oxide with a molecular size of 80nm (supplied from China). By dissolving 0.5 g of PVA with (0.001, 0.002, 0.003, 0.004, 0.005) g of Zinc Oxide nanoparticle in 15ml of water that's been distilled at room temperature, stirred for 3–4 hours with a magnetic stirrer (hot plate) in a temperature range of 25-35°C to ensure that the PVA/ZnO films were dissolved completely. To get homogenous films, pour the solution into a glass plate with a diameter of 3 cm, allow gently for 3 to 4 days for the liquid to evaporate at room temperature. Using a digital micrometer type (Tasha) made in Japan with measurement accuracy (0.001) mm and measurement range (0-150) mm, the thickness of

the PVA film was obtained and was 0.1386mm and the thickness of the PVA/ZnO was 0.16562 mm. UV-Visible spectrophotometer used to measure the absorption and transmission spectra (T 70 / T 80 series UV/Visible spectrometer) in a wavelength range (200nm-900nm). Infrared spectroscopy (FTIR) was conducted for all films using FTIR spectrometer (type: Bruker-Tensor 27 with ATR unit).

RESULTS AND DISCUSSION

XRD Analysis

To look into sample's phase purity and crystalline structure. ZnONPs, pure PVA polymer, and PVA/ZnONPs analyzed using XRD [15]. Figure 1a exhibits the XRD pattern regarding ZnONPs. The peaks at 31.83°, 34.48°, 36.3°, 47.59°, 56.63°, 66.43°, 68° indexed to Hexagonal crystal structure of the ZnO with crystal planes (100), (002), (101), (102), (103), (200), (201). Those values are

consistent with International Center of Diffraction Data (ICDD) card No. (36-1451) [16]. XRD pattern of PVA shown in Figure 1b. A diffused and broad peak can be seen in Figure 1b at roughly $2\theta=19.4046$. This demonstrates that PVA is amorphous in nature [17]. XRD patterns of the PVA/ZnO nanocomposites in amounts of 0.003g and 0.005g shown in Figure 1c and 1d. Amorphous nature discovered after the addition of ZnONPs to PVA, with crystallinity distributed in PVA/ZnO nanocomposites. A detailed analysis of 2θ position regarding the XRD peak suggests that there is a shift in peak position toward lower 2θ value due to the addition of ZnONPs in PVA matrix. In PVA/ZnO nanocomposites, the crystallinity of ZnONPs observed to be disturbed. The fact that the peaks are changing shows that ZnONPs are incorporating into the PVA polymer matrix [18].

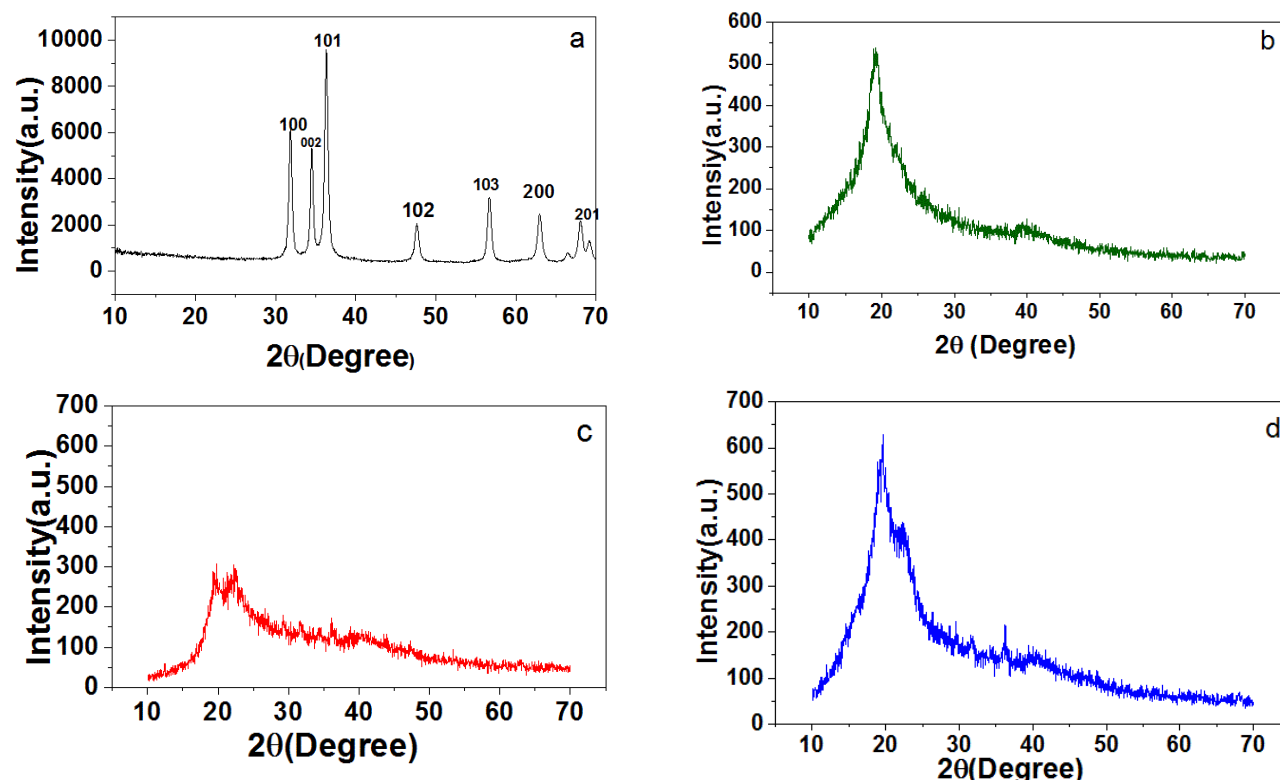


Figure 1. The XRD patterns of a) ZnONPs, b) pure PVA, c) PVA/ZnO Nano-composites for 0.003g and d) PVA/ZnO Nanocomposites for 0.005g.

FTIR Spectra

The FT-IR spectrum of pure PVA polymer depicted in Figure 2a. A large number of bands also featured in Table 1. Peaks exist between (625-970) cm^{-1} , indicating C-H bending out of the plane. Peaks between (1015-1300) cm^{-1} indicate C-O stretching vibration, whereas peaks between (1300-

1380) cm^{-1} show CH_2 bending vibration. Carbonyl stretching vibration ($\text{C}=\text{O}$) is shown by the peaks that developed between (1550-1780) cm^{-1} . The broad band at (3281.88) cm^{-1} refers to O-H hydrogen-bonded alcohols, while the two peaks at (2912.47) cm^{-1} and (2938.30) cm^{-1} suggest C-H stretch aliphatic vibrations, respectively. The FTIR

spectrum of pure ZnO-NPs powder shown in Figure 2b, infrared tests conducted to determine the nature and purity of metal NPs. Inter-atomic vibrations caused absorption bands in metals in the fingerprint region, i.e., below 1000cm^{-1} . The peak at 3750.27 cm^{-1} could be attributable to O-H stretching and deformation, which are both related to water adsorption on the metal surface. Furthermore, Zn-O stretching and deformation vibration are represented by the peaks at (669.14 and 1541.54) cm^{-1} , respectively. The metal-oxygen frequencies observed nm for the respective metal oxides are in accordance with literature vales [19].

For prove probable intermolecular interaction between the components of nanocomposites, FTIR spectra were studied for (PVA/ZnO) nanocomposites and for different amount (0.001, 0.002, 0.003, 0.004, 0.005) g as shown in Figure 2c. and to clarify the positions of the peaks that appeared with types of bonds may be seen in detail in Table 1. It observed that are no interactions between the nanoparticle and the polymer. The transmittance decreases marginally with increase in amount ZnONPs because of the increase in the density of the nanocomposites with increase in the number of nanoparticles.

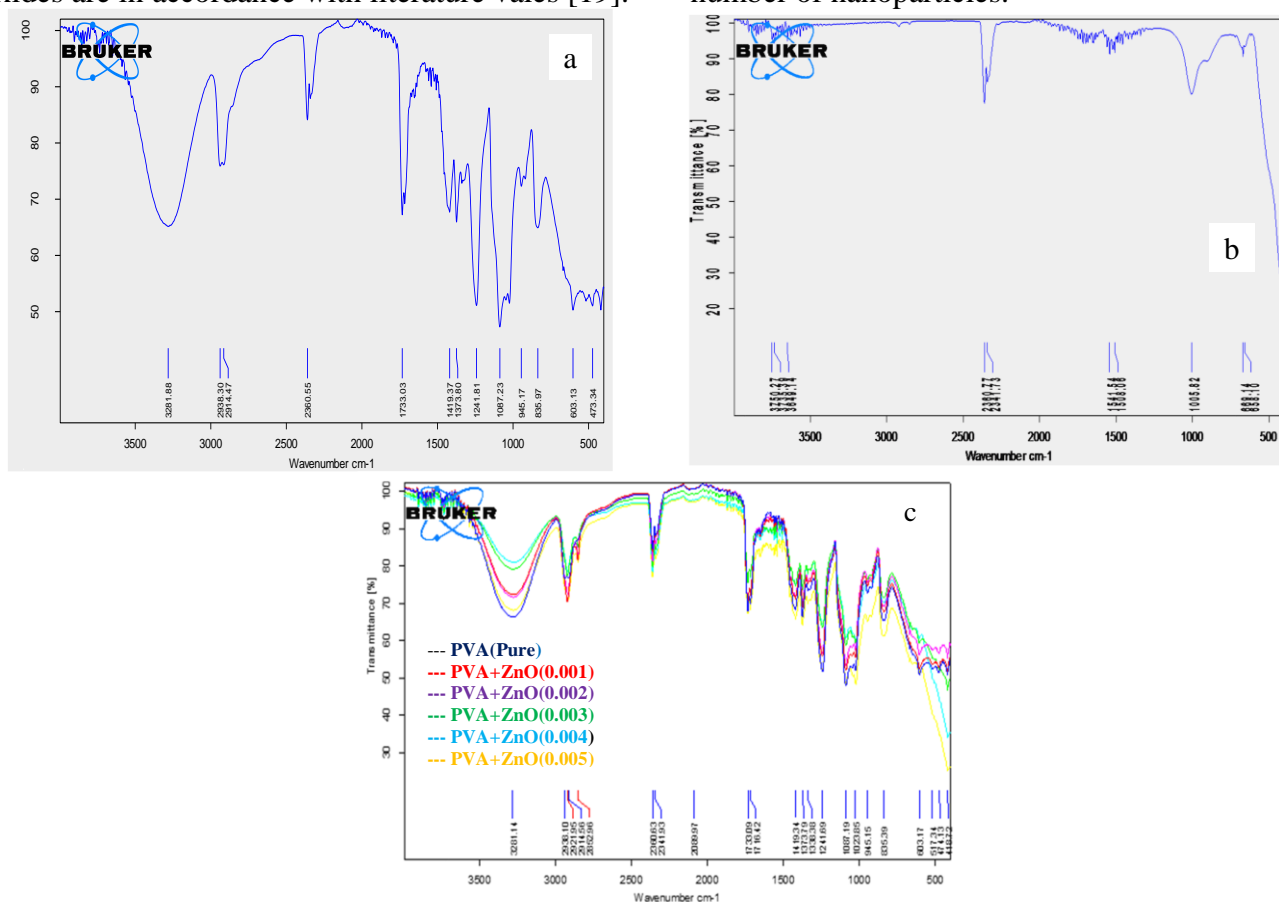


Figure 2. FTIR spectrum for a) pure PVA b) ZnO nanoparticles c) PVA/ZnO Nanocomposites films.

Table 1. FTIR-characteristic of pure PVA film and PVA/ZnO nanocomposites.

Bands	pure PVA	PVA/ZnONPs (0.001 g)	PVA/ZnONPs (0.002 g)	PVA/ZnONPs (0.003 g)	PVA/ZnONPs (0.004 g)	PVA/ZnONPs (0.005 g)
C-H Out phase bend ($625-970$) cm^{-1}	835.97	834.99	836.93	836.36	835.72	836.50
	945.17	945.53	945.04
C-O Stretch ($1015-1300$) cm^{-1}	1023.91	1022.18	1023.37	1021.67	1021.75
	1087.23	1087.30	1087.36	1087.55	1086.81	1087.27
	1242.81	1242.40	1242.07	1241.64	1241.40	1242.12
CH ₂ bending ($1300-1380$) cm^{-1}	1373.80	1374.05	1374.04	1373.95	1373.98	1373.94
C=O stretch ($1550-1780$) cm^{-1}	1716.57	1716.61
	1733.03	1733.77	1733.44	1733.60	1733.48	1733.17
C-Hstretch aliphatic ($2,800-3,000$) cm^{-1}	2914.47	2853.07	2852.70	2851.66	2852.30	2852.07
	2938.30	2922.12	2921.63	2919.54	2920.94	2920.16

Optical Properties

The UV-Visible absorption spectra regarding PVA/ZnO nano-composite films shown in Figure 3. As the amount of increase ZnO, the absorption spectrum increases. Adding varying amounts of filler material to polymer does not alter the polymer's chemical composition, yet it does give the combination new physical qualities. The absorption of pure polymers and nanocomposites in UV field increases more than in visible region as shown in Table 2. The impact of appended ZnONPs is clear; they have extremely high absorption in UV region, which increases with the increase in ZnONPs content [20]. Because of a unique weight of ZnONPs and formation of the inter-molecular hydrogen bonds between Zinc ions and surrounding OH groups of main chain PVA, absorption band changes. Because of the greater distance between from conduction to valence bands, the polymer exhibits a low absorbance [21].

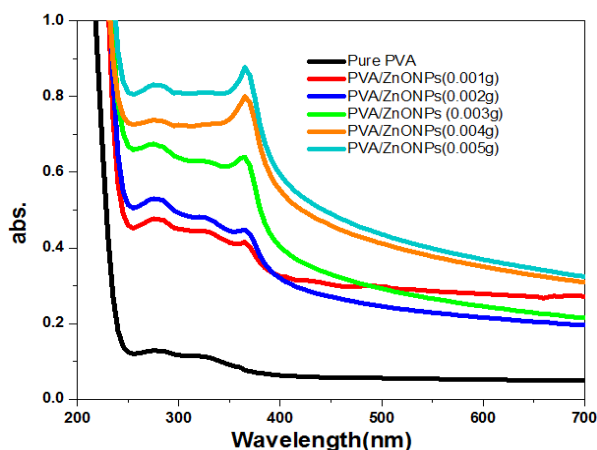


Figure 3. Absorption spectrum of pure PVA and PVA/ZnO nanocomposite films.

Table 2. The highest peak of the absorption spectrum of PVA/ZnO nanocomposite films.

Samples	Wavelength (nm)	Absorbance
pure PVA	280	0.128
PVA/ZnONPs (0.001 g)	280	0.475
	370	0.437
PVA/ZnONPs (0.002 g)	280	0.528
	370	0.437
PVA/ZnONPs (0.003 g)	280	0.67
	370	0.617
PVA/ZnONPs (0.004 g)	280	0.736
	370	0.784
PVA/ZnONPs (0.005 g)	280	0.829
	370	0.855

Figure 4 illustrates pure PVA polymer and PVA/ZnO nano-composite transmittance. Transmittance of pure PVA polymer is higher compared to that of the PVA/ZnO nano-composites. Pure polymers and nanocomposites have a lower transmittance in UV field than in visible range. Effects of the appended ZnONPs are clear; they have quite a low transmittance in UV range, which has dropped as the ZnONPs content has increased [22].

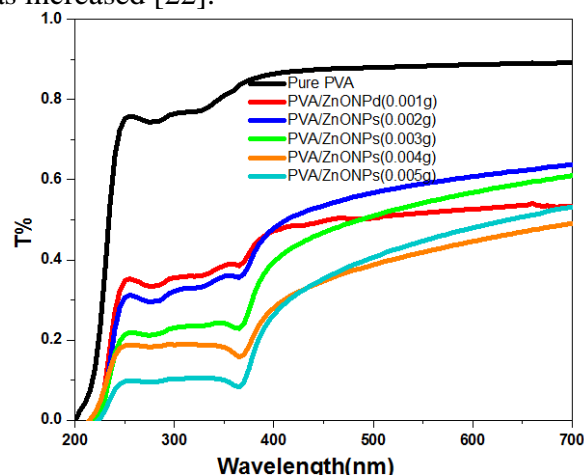


Figure 4. Transmission spectrum for PVA/ZnO nanocomposite films

Absorption coefficient (α) can be defined as a property regarding each absorber molecule or ion. It is specified as the material's capability for absorbing light with limited wave-length per unit length. The absorption coefficient could be calculated using Equation 2. The absorption coefficient values indicate the electronic transition nature. In a case where absorption coefficient is large ($\alpha > 10^4 \text{ cm}^{-1}$) at higher energy values, direct electronic transitions are expected, and electron and photon's energy momentum are preserved. At the low values of energy, indirect electronic transitions are expected since the coefficient of the absorption is low ($\alpha < 10^4 \text{ cm}^{-1}$). Figure 5 illustrates absorption coefficient regarding PVA/ZnO nanocomposite films as function of wavelength. The absorption coefficient value for all samples smaller than (10^4 cm^{-1}), as shown in Figure 5, has been derived, indicating indirect electronic transition.

Refractive index is a significant optical property that might be calculated with equation 6, which is dependent on reflectance. The nanocomposites'

refractive index varies as a wave-length function, as seen in Figure 6. Refractive index lowers as the amount of ZnO increases [22].

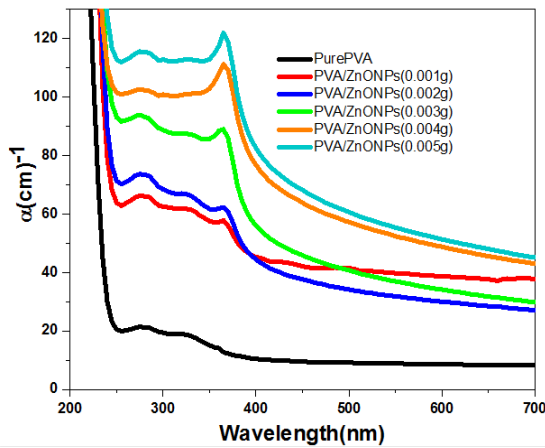


Figure 5. Absorption coefficient for PVA/ZnO nanocomposite films.

For PVA/ZnO nano-composite films, Figure 7 shows relationship between photon energy and absorption edge $(\alpha h\nu)^{1/2}$. Table 3 summarizes the energy gap values for all PVA/ZnO nano-composite film samples. It is seen that when the amount of ZnO grows, the energy gap values decrease. This drop is because of the addition of a new band gap level. This result is consistent with encouraging electron transport from valence to conduction bands [22].

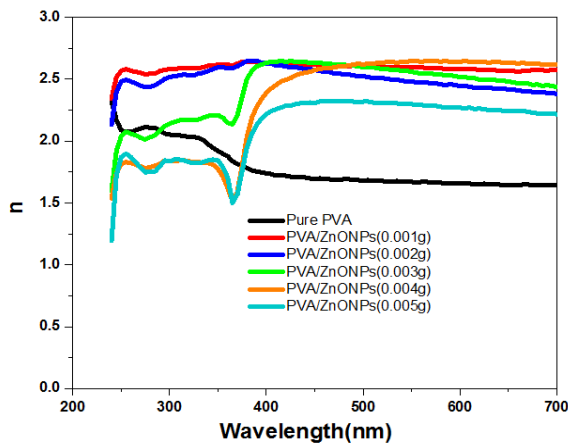


Figure 6. Refractive index for PVA/ZnO nanocomposite film for PVA/ZnO nano-composite film.

Equation 3 used to compute the extinction coefficient. The absorbance affects the extinction coefficient. Figure 8 illustrates variations of extinction coefficient(k) with wavelength for (PVA/ZnO) nano-composites. In addition, the coefficient of extinction rises with the increase for ZnONPs. Such behavior of the extinction coefficient could be a result of the high coefficient of absorption. At the longest wavelengths, there is

a lot of extinction. Higher absorption coefficient can explain this extinction coefficient pattern. Extinction is highest and highest at the longest wavelengths.

Equation 5 utilized in order to calculate the reflectance of PVA/ZnO nano-composite films, which illustrated in Figure 9. The variation in reflection spectrum of a nanocomposite as a function of wavelength. The reflection spectrum broadens due to the filler

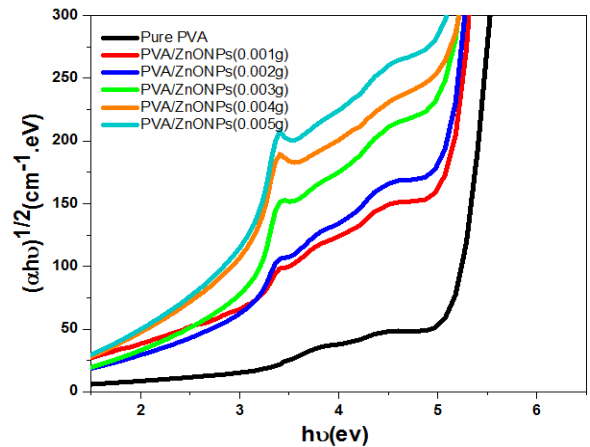


Figure 7. Optical energy gap for PVA/ZnO nanocomposite films.

Table 3. Energy gap (E_g) for PVA/ZnO nanocomposite films

Samples	Energy gap (eV)
Pure PVA	4.92
PVA/ZnONPs (0.001 g)	4.83
PVA/ZnONPs (0.002 g)	4.74
PVA/ZnONPs (0.003 g)	4.65
PVA/ZnONPs (0.004 g)	4.56
PVA/ZnONPs (0.005 g)	4.47

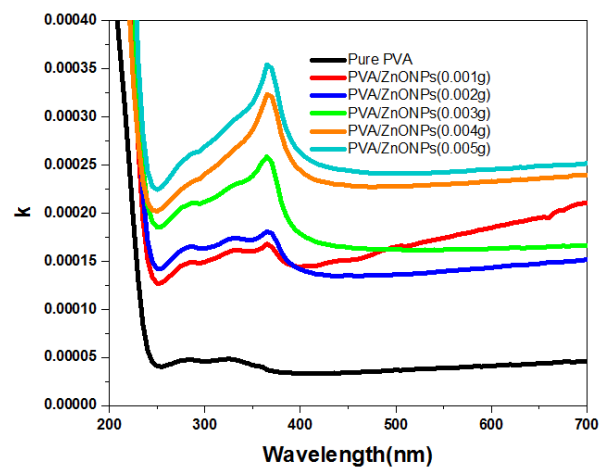


Figure 8. Extinction Coefficient for PVA/ZnO nano-composite films.

Optical constants are especially effective for defining the electronic band structure regarding the solids quantitatively utilizing optical reflectivity transmission and refraction data. In addition, they provide a method for determining solids' dielectric constants, which linked to the band structure.

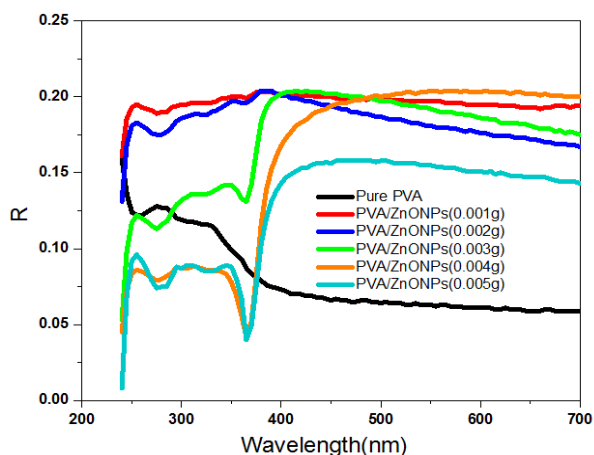


Figure 9. Reflection spectrum for PVA/ZnO nanocomposite films.

From Equation 7 and 8, the imaginary and real dielectric constants (ϵ_i & ϵ_r) for PVA/ZnO nano-composites were derived. Figures 10 and 11 show how the imaginary and real parts of dielectric constants of (PVA/ZnO) nanocomposites, as well as the imaginary parts regarding the dielectric constant of (PVA/ZnO) nanocomposites, change with wavelengths. It is inferred that the variation of ϵ_r is primarily influenced by (n^2) due to small values of (k^2), whereas the variation of ϵ_i is primarily influenced by (k) values, associated with variation of coefficients of absorption. In comparison to the imaginary dielectric constant, real dielectric constant has high values [22].

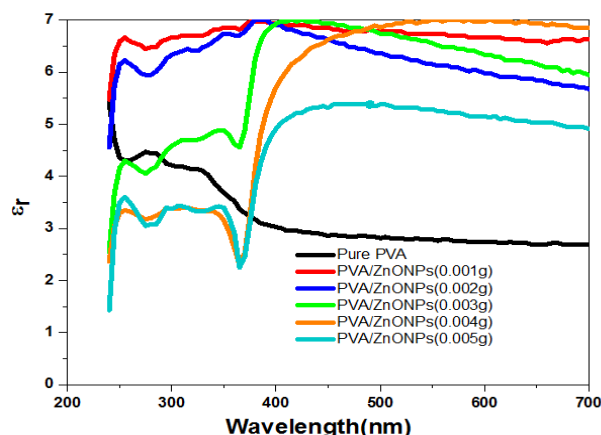


Figure 10. Real part of dielectric constant for the PVA/ZnO nanocomposite films.

CONCLUSIONS

The solution cast approach used to create nanocomposite thin films of PVA with varying amounts of ZnONPs. According to the XRD study, increasing the amount of ZnONPs in the

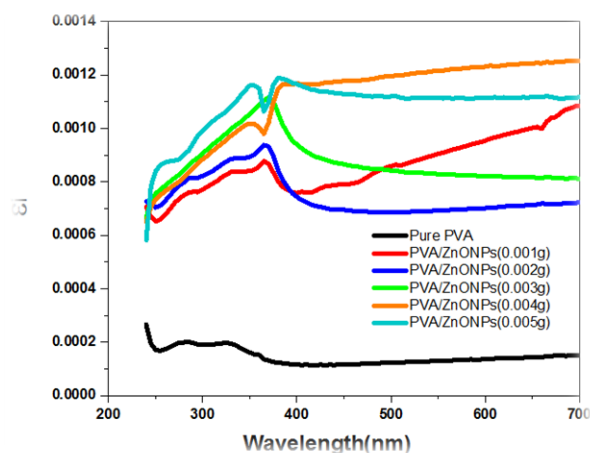


Figure 11. Imaginary part of dielectric constant for PVA/ZnO nanocomposite films.

system causes the ZnONPs peaks to completely disappear and/or nanocomposite. In addition, the differences in XRD spectrum suggest that NP doping caused a microstructure disparity in the polymer. Because no covalent bonds created between ZnONPs and PVA, FTIR demonstrated that ZnONPs have no detrimental effect on the polymer structure. In the case when the ZnO ratio in nanocomposites is increased, the extinction coefficient, absorbance, refractive index, absorption coefficient, real and imaginary dielectric constants of the PVA polymer

enhance. The energy gap and the transmittance to the PVA polymer decrease as the amount of ZnO nanocomposites increases.

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