*IOSR Journal of Applied Physics (IOSR-JAP)*
*e-ISSN: 2278-4861.Volume 10, Issue 5 Ver. I (Sep. – Oct. 2018), 47-56*
*www.iosrjournals.org*

# Tuning the optical properties of Fe2O3 doped ZnO / Polystyrene composite filmsH.

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***Abstract:*** *The effect of addition of $Fe_2O_3$ doped ZnO nanoparticles on some optical properties of polystyrene has been studied.$Fe_2O_3$ doped ZnO nanoparticles was prepared by combustion method. Polymeric films based on polystyrene (PS) filled with different weight percentage (5, 7.5, 10, 12.5) were prepared by the casting method. The morphology of the prepared filler of $Fe_2O_3$ doped ZnO nanoparticles were verified by high-resolution transmission electron microscope (HRTEM). Structures of the prepared films were examined by x-ray diffraction (XRD), where the recorded pattern reveals the existence of wurtzite phase of hexagonal structure for ZnO nanoparticles doped with $Fe_2O_3$. The absorption, transmission and diffuse reflectance spectra were recorded in the visible range. The absorption coefficient, optical band gap, extinction coefficient, and refractive index of the casted films were calculated. The results showed a sharp decrease in the band gap from 4.54 to 2.82 eV with increasing the percentage ratio of $Fe_2O_3$ doped ZnO nanoparticles in PS matrix.*

***Keywords:*** *$Fe_2O_3$ doped ZnOnanoparticles, polystyrene, Inorganic/organic composite films, optical properties.*

Date of Submission: 03-09-2018 Date of acceptance: 18-09-2018

## I. Introduction

Inorganic/ organic composite material plays an important role in creating extensive usages in many areas. It is used in a wide search for high permittivity materials that have extensive variety ofof technologically important applications for example, microelectronic, embedded passive and electrostrictive devices. Polymer composites are used as electrically conductive adhesives and circuit elements in microelectronics and have been reported to possess anticorrosive behavior as metal components coatings [1].Polymer composites are utilized as electrically conductive glues and circuit components in microelectronics and have been accounted for to have anticorrosive conduct as metal parts coatingsOne of the most interesting polymers is the polystyrene; it has excellent physical and chemical properties. Polystyrene is an amorphous polymer with high transparency, ease of process and it is very important in many industrial applications [2, 3].It was reported that a conductive or semiconductive composite is formed by dispersion of sufficient quantity of metallic particle [4]. The dispersion of metals nanoparticles in the polymer matrix could commonly add unique physical properties to the associated matrixes such as responsiveness to mechanical, optical, thermal, magnetic, electric stimulation, etc to produce very useful nanocomposites [5, 6]. Also metals – polymer matrix is relatively not costly in the point of view of the processing techniques and manufacturing materials as compared to the expensive materials widely used in semiconductor processing industry [7]. Zinc oxide, ZnO is one of many inorganic materials. It has direct band gap (Eg = 3.27 eV) [8, 9] which is considered a promising candidate for optical and optoelectronic applications in nano-scale devices. [10,11]. In spite of these preferences of ZnO, its functional applications were constrained by some acquired disadvantages, for example the requirement of UV light activation, the low quantum efficiency, and the serious photo-corrosion [12]. To overcome these problems, ZnO matrix composite semiconductors containing some other reinforcing particles that have novel and advanced properties. Especially, because of the easy availability, nontoxic nature, biological and chemical stability, nanostructure semiconductor metal oxides such as TiO2, SnO2, CuO, WO3 and Fe2O3 have become promising photocatalysts in environmental improvement [13–21]. Among the metal oxides, hematite (α-Fe2O3) is the most thermodynamically-stable phase of iron oxide under ambient conditions with low cost, high resistance to photo-corrosion and environment-friendly features. It can be driven by visible light up to 600 nm due to the narrow band gap of 2.2 eV, and has been confirmed to be an important member of visible-light-responsive semiconductor photocatalysts [22-26]. Different methods have been used to synthesize various metal-polymer composites E.g. (i) Direct mixing of nanoparticles in the polymer [27] (ii) Sol-gel methods [28] (iii) in-situ techniques [29] and (iv) deposition method [30]. Many types of polymer composites have been studied by introduction of conductive filler such as, silver (Ag), nickel (Ni), Aluminum (Al), Ni/Co coated CNT in polystyrene composite and carbon nanotube (CNT) [31-35].

In this work,

- We successfully synthesized nanoparticles of $Fe_2O_3$ doped ZnO by combustionand prepared composite films of $Fe_2O_3$ doped ZnO/PS by casting method.
- The focus of this work is to investigate the structural and the optical properties ofthe casted films to get a material can be easily tuned for optical application by easier control of the composition.

## II. Experimental

$Fe_2O_3$ doped ZnOpowder was preparedby combustionmethodthrough mixing zinc nitrate, iron nitrate and urea as oxidizing agentwith a certain calculated ratios. The mixed powders were placed in porcelain crucible to be burned in the furnace at about 370 $^{\circ}$C, until the mixture homogenates, self- sustaining and a rather fast combustion with enormous swelling producing white foamy and voluminous $Fe_2O_3$ doped ZnO. Then, the temperature of the furnace was increased up to 500°C and the mixture was heated at this temperature for two hours before the furnace is switched off. Polystyrene (PS)was used as received without further purification with(MW= 35000 softening point (ASTM 28) 123-128$^{\circ}$C, density 1.06 g/mL at 25$^{\circ}$C from Sigma-Aldrich, Germany). The appropriate weight (5gm) of PS was dissolved in 100 ml of chloroform. The mixture was magnetically stirred continuously at room temperature for 2 hours until the mixture solution has a homogenous viscous appearance. The solution was left for 3 days before the addition metal oxides filler to it. Different weights of the prepared powder with (5, 7.5, 10 and 12.5 wt. %) were added to the chloroform and magnetically stirred vigorously to ensure a high dispersion of the added nanoparticles for 1 hour and then ultra-sonication for another 1 hour to prevent the nanoparticles agglomeration. The mixture then mixed with the PS solution and stirred again for1 hour then ultra-sonication for 1 h. The final product of the polymers PS reinforced with $Fe_2O_3$ doped ZnOnanoparticles was cast in glass Petri dishes and left 1 day for drying.

In order to characterize and identify prepared samples, high-resolution transmission electron microscope (HRTEM) JEM-2100 with an operating voltage is 200 keV and gun type is Lab 6 Emmittorwas used to analyze the morphology of the prepared filler. The structure of casted films was characterized by X-ray diffraction using analytical X'Pert PRO MRD diffract meter system having CuKα (λ =1.540598 Å) with 2θ = 10° - 90°. The optical measurements of the prepared films were investigated using UV- Vis spectrophotometer type JASCO 570.

The absorption coefficient of the prepared films was defined by the Beer-Lambert's law [44] as

$$\alpha(\upsilon) = \frac{2.303 \times Abs(\lambda)}{d}$$

where d and $Abs(\lambda)$ are the film thickness and the film absorbance, respectively.

The optical energy band gap of the presented films is related to the absorption coefficient by the following relation [47, 48]:

$$\alpha(\upsilon)h\upsilon = B(h\upsilon - Eg)^m$$

Where $E_g$, B, and $h\upsilon$ are the optical gap, constant, the incident photon energy, respectively and m is the index which can have different values of 1/2, 3/2, 2, and 3 depending on the nature of the electronic transition [49].

The extinction coefficient $(k)$ was calculated by the equation: $k = \frac{\alpha\lambda}{4\lambda}$

For normal reflectance, the refractive index can be determined from the relation:

$$n = \frac{1 + \sqrt{R}}{1 - \sqrt{R}}$$

## III. Results and discussion

The morphology of the prepared filler of ZnO doped with $Fe_2O_3$ was investigated by HRTEM. The photographs of HRTEM reveal a hexagonal crystal structure (wurtzite) of $Fe_2O_3$ doped ZnOnanoparticles as clearly seen from **Fig. (1a).** Theparticle size range is from 10-30 nm. Also the diffraction pattern of the selected area shown in the inset of **Fig.1b**reveals a polycrystalline structure of the prepared powder. The calculated d-spacing values from diffraction pattern shows all the characterized planes of ZnO; 2.76, 2.69,, 2.45, 1.62 and 1.45 Å are corresponding to the crystallographic planes (100), (101), (002), (110) and (103), respectively, while the plane (113) is related to the presence of $Fe_2O_3$.

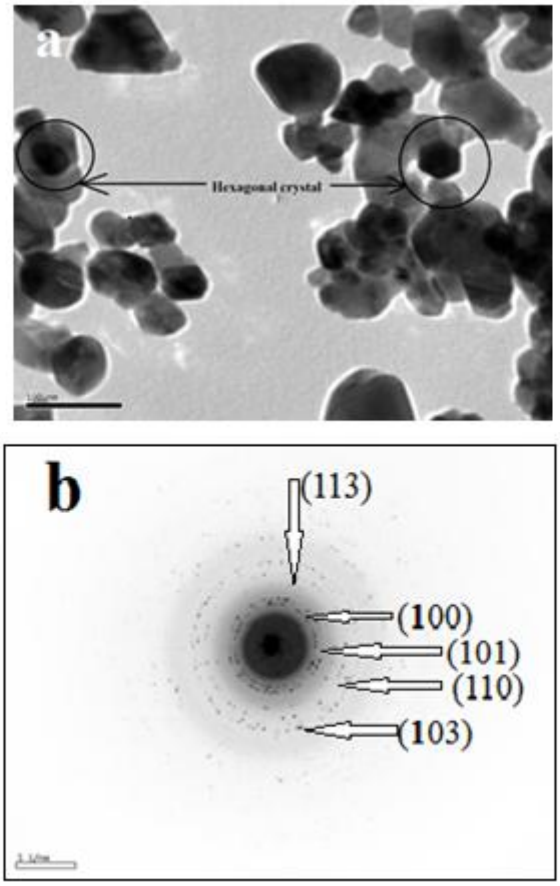


**Fig.(1a-c):** HRTEM photographs of $Fe_2O_3$ doped ZnOnanoparticles (a) image of prepared nanoparticles& (b) SAED diffraction pattern

XRD patterns of the casted films of PS filled with different weight percentages of $Fe_2O_3$ doped ZnO(5, 7.5, 10, and 12.5) are depicted in **Fig.(2)**. The XRD patterns, reveals that, all the formed peaks is indexed to the JCPDS card no. 36-1451, and belongs to the formation hexagonal wurtziteZnO phase with its characterized planes (100), (002), (101), (102), (110), (103) and (201), but there is no peaks related to the $Fe_2O_3$ as its percentage is less than 5 wt%. Also, the pattern indicates the presence of a hump at $2\theta = 10\text{-}20^{\circ}$ due to polystyrene. On the other hand, the XRD shows that the polystyrene has shown some dependence on the percentage of metal oxides of$Fe_2O_3$ doped ZnO filler ratio certainly, the PS peak is found to increase in broadness and decreased in intensity. By combining organic and inorganic material, the inorganic nano-sized particles of $Fe_2O_3$ doped ZnOable to assemble and can stabilize the monomers of polystyrene [40].As the content of $Fe_2O_3$ doped ZnOin PS matrix increases, the intensity of diffraction peak attributed to PS clearly decreases. This indicates that the $Fe_2O_3$doped ZnOcrystallites are well incorporated in PS matrix. From XRD pattern, the crystallite size (D) of $Fe_2O_3$ doped ZnOnanoparticles was calculated from the corrected FWHM of the most intense peak corresponding to (002) plane using Scherrer's formula and it listed in table (1). The reported data in table (1) explained that the crystallite size decreases with $Fe_2O_3$ doped ZnOcontent from 40 nm to 18.5 nm.

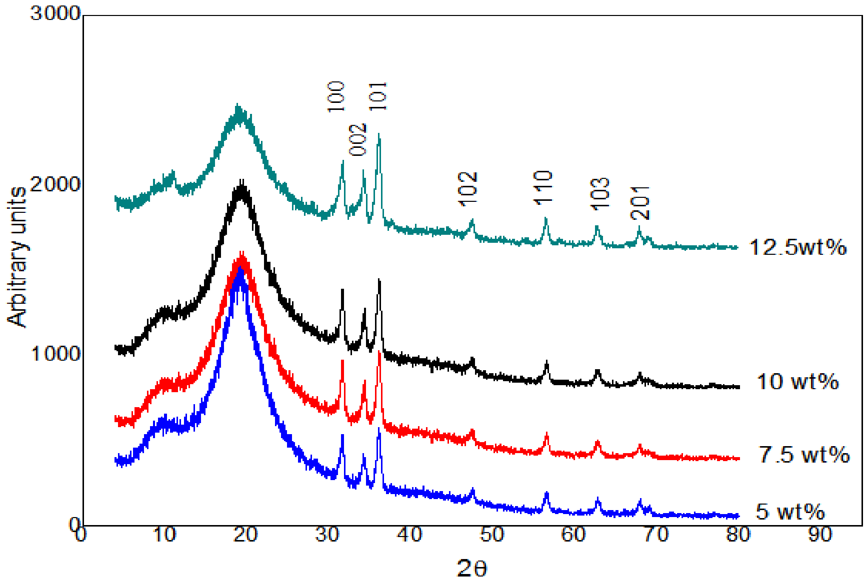
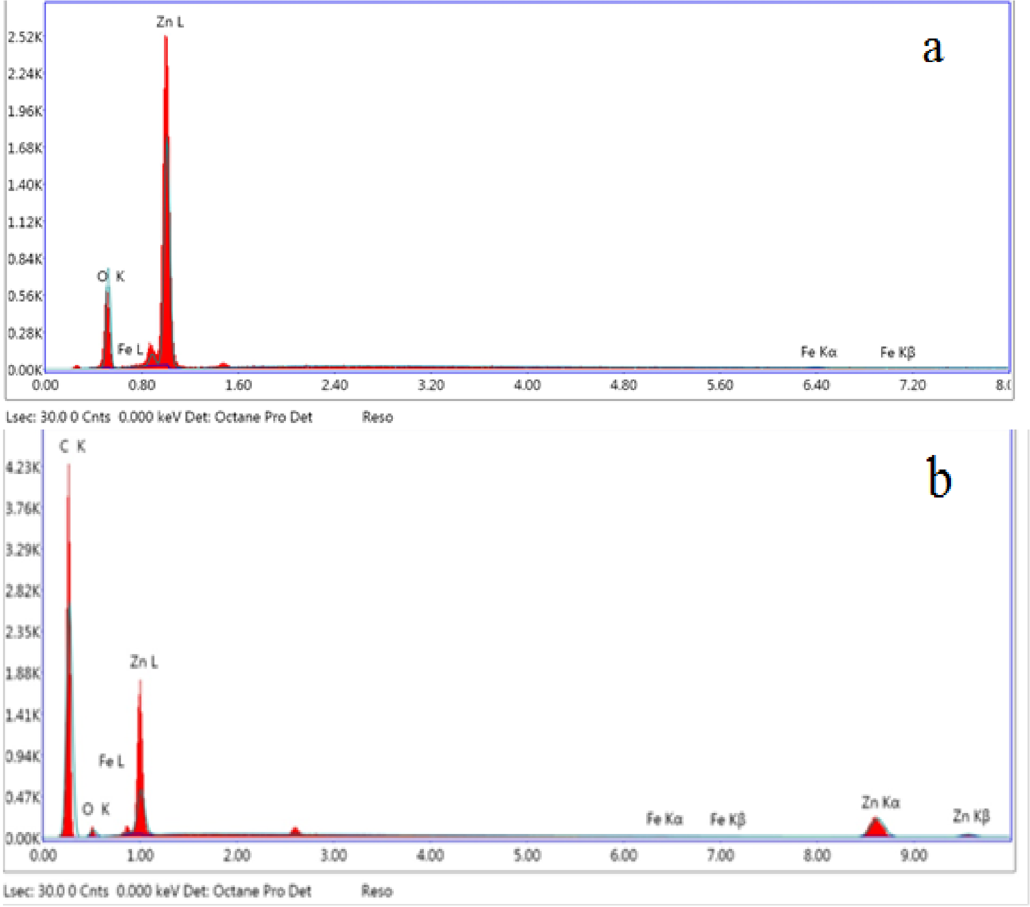


**Fig. (2):** X- ray diffraction patterns of $Fe_2O_3$ doped ZnO/ polystyrene films with different filler concentrations.

| $Fe_2O_3$ doped ZnO concentration | 5 wt. % $Fe_2O_3$ doped ZnO/PS | 7.5 wt. % $Fe_2O_3$ doped ZnO/PS | 10 wt. % $Fe_2O_3$ doped ZnO/PS | 12.5 wt. % $Fe_2O_3$ doped ZnO/PS |
|---|---|---|---|---|
| Crystallite size(nm) | 40 | 31.5 | 23 | 18.5 |

**Table (1): The crystallite size of $Fe_2O_3$ doped ZnO/PS nanocomposite films**

The EDX spectrum of both $Fe_2O_3$ doped ZnO powder and $Fe_2O_3$ doped ZnO/ PS films are shown in **Fig. (3a, b).**The characteristic peaks of Zn, O andFe are all found which indicate thepresence of $Fe_2O_3$ as doping in both powder and composite films, also there is no any impurities in the represented samples.



.**Fig. (3):** EDX images for (a) Fe2O3 doped ZnOpowder & (b) Fe2O3 doped ZnO/ PS films The optical absorption spectra are very important to understand the optical behavior of the samples. The optical band gap of the prepared powder of$Fe_2O_3$ doped ZnO can be determined from the relation between the diffuse reflectance (R) and the wave length (λ) as shown in **Fig. (4)**. The diffuse reflectance spectra illustrate absorption response in the range 400-600 nm due to the presence of $Fe_2O_3$ as previously reported [41]. The band energy gap value is3.4 eV which can be deduced from the onset of the linear increase in the diffuse reflectance [42, 43].

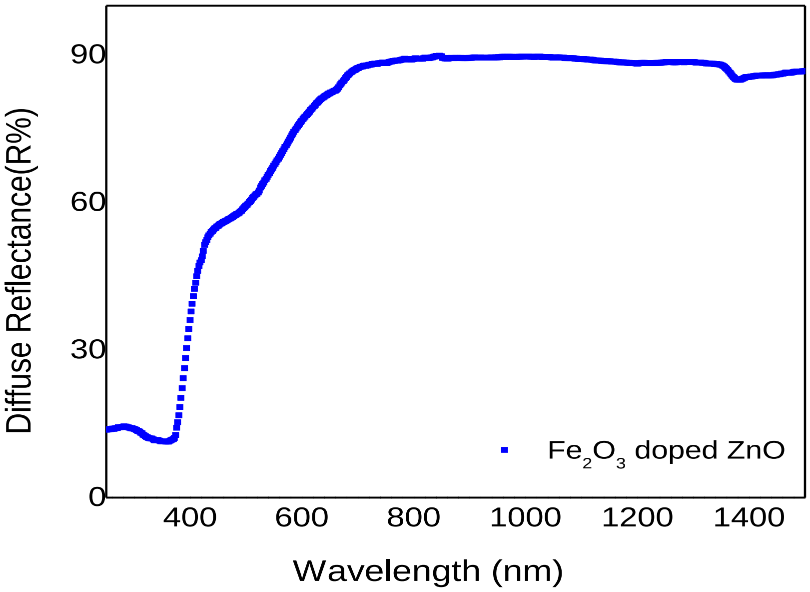


**Fig. (4)**: Diffuse reflectance for $Fe_2O_3$ doped ZnO.

The optical absorption spectrum of $Fe_2O_3$ doped ZnO/PS composite films with different concentration of $Fe_2O_3$ doped ZnOrange from (5 to 12.5 wt %) can be shown in **Fig (5)**. This figure shows an increase in the absorption with increasing the concentration of the filler in the PS also with decreasing the wavelength due to nanocrystalline [44]. The absorbance spectrum shows that $Fe_2O_3$ doped ZnO/PScomposite films have two-edge absorption. The absorptions at lower wavelengths can be attributed to ZnO. The absorptions at the higher wavelength (visible region) are ascribed to the $Fe_2O_3$ where the band gap energy of bulk $Fe_2O_3$ is 2.2 eV[22-26].The absorbance spectrum shows a splitting in the absorption band at 350 nm and 374 nm. The two absorption bands started to appear with the concentration of 7.5wt%. The splitting in absorption band can be attributed to the transition of the electrons from the valence to the conduction bands of ZnO [45, 46]. Where the intensity of these bands increases and become sharper with increasing the addition of $Fe_2O_3$ doped ZnOpowder in the polymer matrix and a slight shift towards longer wavelengths in the range from 374nm to 376.4 nm. This observed redshift is an indication of the incorporation of$Fe_2O_3$ into ZnO powder [42, 47].

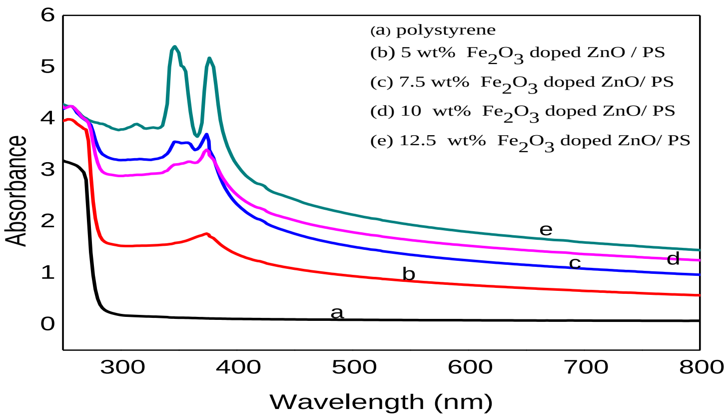


**Fig. (5)**:UV visible absorption spectra of polystyrene and composites films of $Fe_2O_3$ doped ZnO/ PS.

The optical transmission spectrum of PS and $Fe_2O_3$ doped ZnO/PS nanocomposite films with different concentration of $Fe_2O_3$ doped ZnOrange from (5 to 12.5 wt. %) can be shown in **Fig (6)**. This figure explained that the transmissiondecreased as the $Fe_2O_3$ doped ZnO nanoparticles increased due to the formation of the layer of covalent bonds formed between $Fe_2O_3$ doped ZnO filler and PS chains lead to decrease in the transmission of the incident light especially at the shortest wavelengths. The transmittance of pure PS reach to 80% while, $Fe_2O_3$ doped ZnO/PS films shows a decreasing in the transmission.

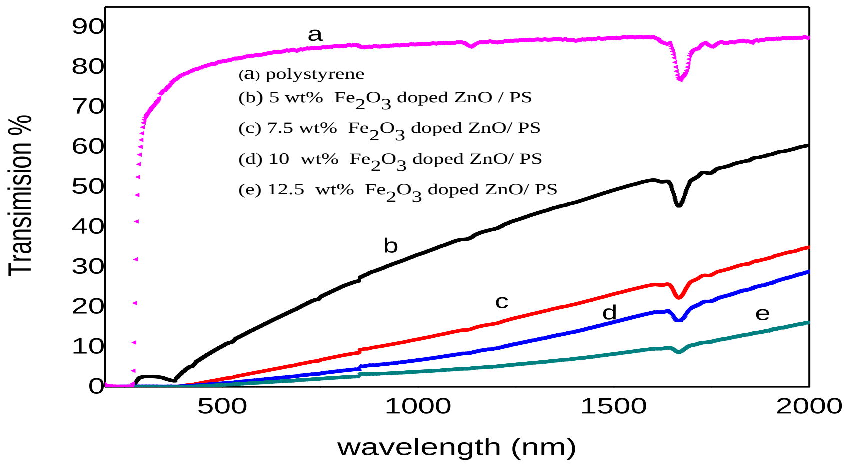


**Fig. (6)**:UV visible transmission spectra of polystyrene and composites films of $Fe_2O_3$ doped ZnO/ PS.

**Fig (7)** shows the relation between the absorption coefficient and the photon energy. The absorption coefficient α has a small and constant value at low photon energy because of the photon energy is not enough to transfer the electron from the valence band to the conduction band **(hυ<Eg)**, while at high energies, the absorption coefficient increases and hence a great possibility for electron transitions. The absorption coefficient increases as the concentration of $Fe_2O_3$ doped ZnO nanoparticles at higher energy which indicate the crystalline nature of the samples [48] and as previously discussed from x-ray investigation. So the absorption coefficient indicates the nature of electron transition, it is expected that indirect transition of electron can occur as the values of the absorption coefficient are low ($\alpha < 10^4$) $(cm)^{-1}$, as agree with [49]. Also, the absorption coefficient curves shifts to low photon energy values with increasing $Fe_2O_3$ doped ZnO concentration.

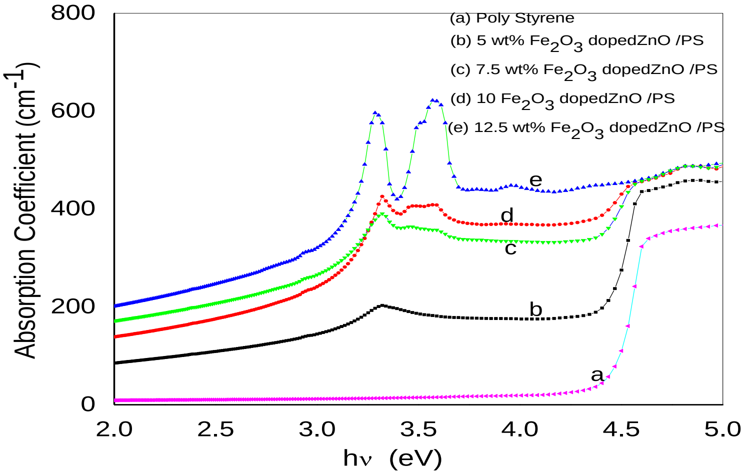


**Fig. (7)**: Absorption coefficient- photon energy relation for all investigated samples.

The allowedindirect energy band gaps of the $Fe_2O_3$ doped ZnO/PS composite films are illustrated by plotting $(\alpha h\upsilon)^{1/2}$ versus hυ as clearly shown from **Fig. (8a-e).**

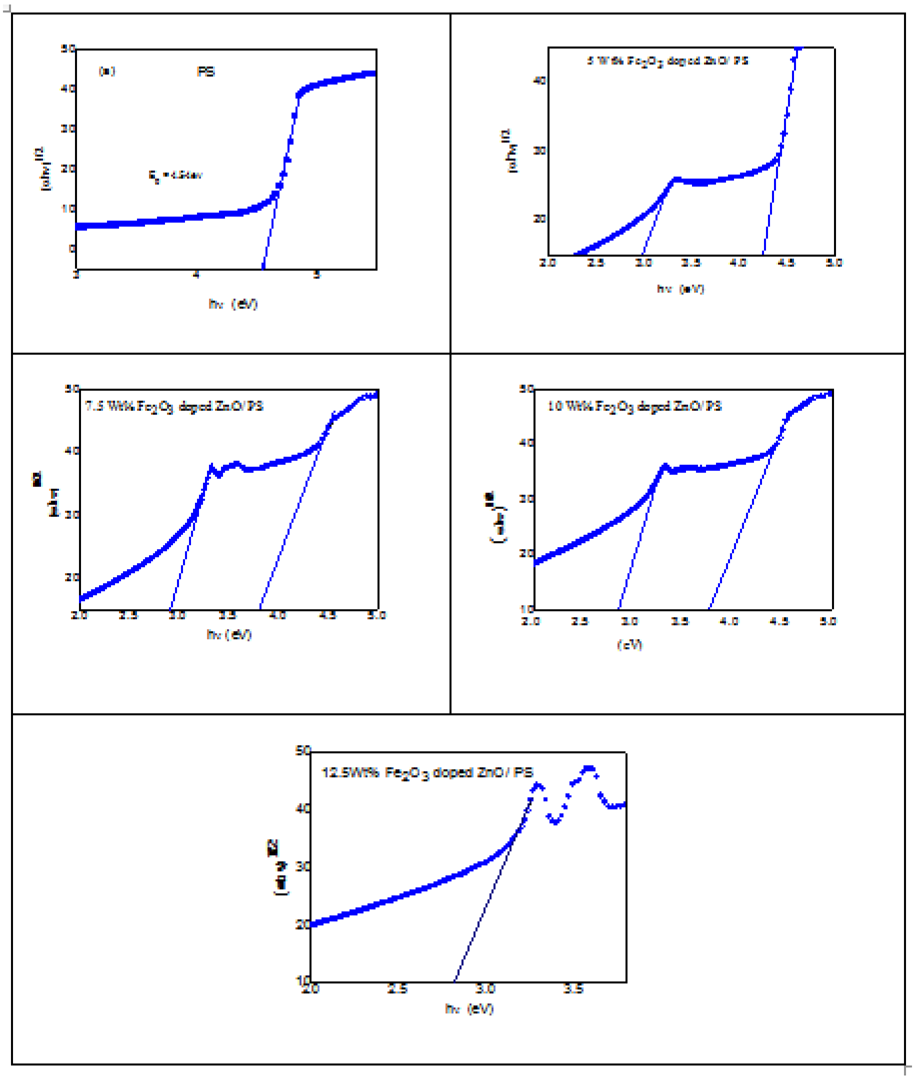


**Fig. (8)**:Optical band gap values of polystyrene and $Fe_2O_3$doped ZnO/PS composite films as a function of photon energy.

The energy gap can be obtained by the extrapolated line to the photon energy (hʋ) axis.The pure PS shows one indirect optical energy gap while $Fe_2O_3$ doped ZnO/PS samples show two indirect optical energy gaps except for sample with 12.5 wt. %$Fe_2O_3$ doped ZnO. The values of indirect optical energy gap were listed in **table (2).**

| Concentration | $E_{g1}$(eV) | $E_{g2}$ (eV) |
|---|---|---|
| PS | _ | 4.55 |
| 5 wt. % $Fe_2O_3$ doped ZnO/PS | 2.99 | 4.25 |
| 7.5 wt. % $Fe_2O_3$ doped ZnO/ PS | 2.94 | 3.81 |
| 10 wt. % $Fe_2O_3$ doped ZnO/PS | 2.86 | 3.76 |
| 12.5 wt. % $Fe_2O_3$ doped ZnO/PS | 2.82 | |

**Table (2): The values of allowed indirect transitionenergy gaps of $Fe_2O_3$ doped ZnO/PSnanocomposite films.**

The obtained energy gap values were found to decrease with increasing concentration of $Fe_2O_3$ doped ZnO in PS matrix. One possible interpretation of this observation is that the insertion of $Fe_2O_3$ doped ZnOnanoparticles in the host matrix introduces multiple states (additional energy levels) in the polystyrene structure and thus decreasing the band gap energy between the valence and conductions bands. Also,the decrease of band gap may be due to the strong electronic state exchange interaction. This interaction leads to merging the bands belonging to $Fe_2O_3$ and ZnO which in turn reduce the two band into one band as seen for

12.5 wt.%$Fe_2O_3$ doped ZnO / PS sample in **table (2).**The variation in the value of deduced energy gap was previously reported [42, 50].

The extinction coefficient as a function of wavelength can be shown from **Fig. (9).** Thevalue of extinction coefficientk increases with increasing $Fe_2O_3$ doped ZnOconcentration in the polymer matrix. The increment of $k$ values with wavelength reveals that some interaction takes place between photons and electrons. The small extinction coefficient ($\approx 10^{-4}$) indicates that the composite samples still are transparent [50, 51], while the increase in the value of extinction coefficient at higher concentrations indicate higher absorption coefficient. The variation in the value of extinction coefficient is important in the application of optical devices [52]

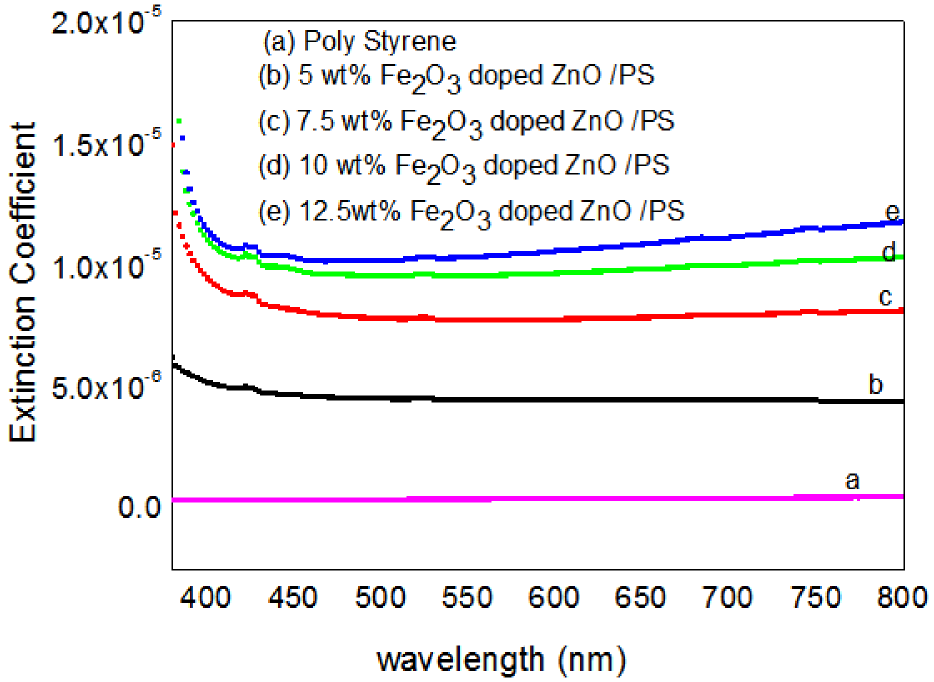
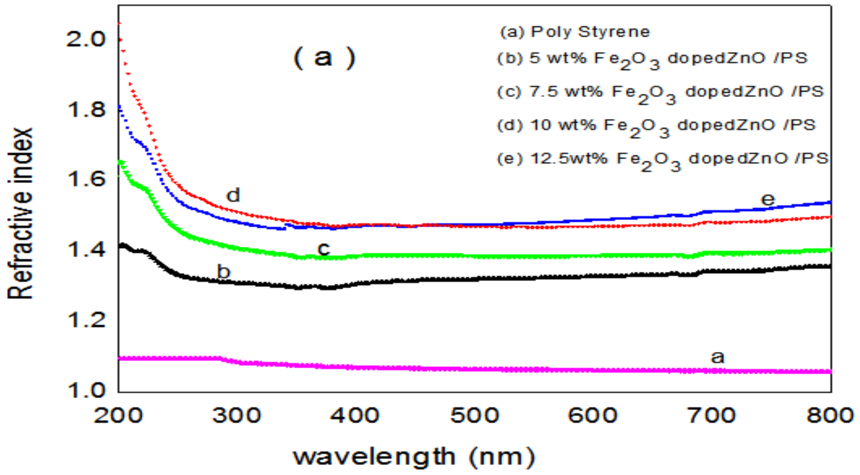


**Fig. (9):** The Extinction coefficient relation with wavelength for the prepared composites films with different filler concentrations.

Fig. (10a) shows the relation between the refractive index and the wave length. The refractive index for pure PS film is constant in the range from (200-800 nm), while it decreases rapidly only at lower wavelengths range (200-300nm) for Fe2O3 doped ZnO/PS films and is constant at higher wave length. The values of the refractive index were taken from the intersection with$Y$-axis as can be clearly shown from Fig (10b). It is clearly observed that the refractive index increases as the Fe2O3 doped ZnOwt % increases in PS, this may be attributed to the increasing the packing density of the investigated films. The small value observed for the refractive indexes from 1.1 to 1.6is an indication that these composites films are suitable as low-index claddings for waveguide applications [53]. The optical properties in this work confirm that the refractiveindex and energy gap are strongly correlated.



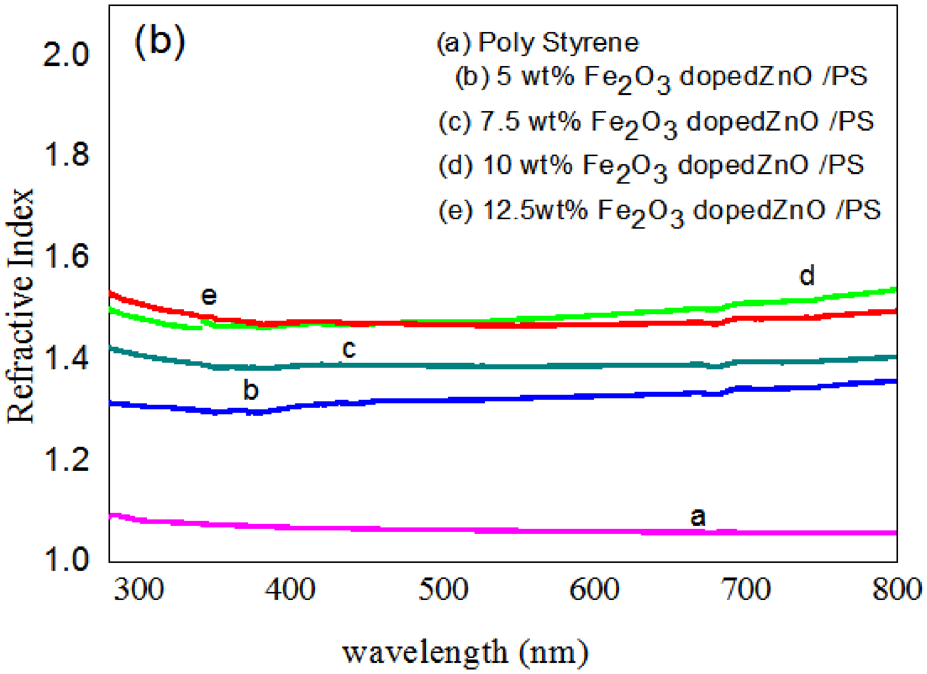


**Fig. (10):**The refractive index vs. wavelength for the prepared composites films with different filler concentrations.

## IV. Conclusion

- Polymeric films based on polystyrene (PS) filled with different concentration of ZnO doped with $Fe_2O_3$ nanoparticles were prepared by solution casting method.
- Fine scale nanoparticles of wurtzite structure of $Fe_2O_3$ doped ZnOwere investigated by HRTEM and XRD analysis respectively.
- The optical properties of prepared composite material were performed by means of UV-Vis technique. The results showed an increase of absorption coefficient, extinction coefficient and refractive index with increasing the percentage ratio of $Fe_2O_3$ doped ZnO nanoparticles in PS matrix.
- Moreover, the calculated optical band gap sharply decreased from 4.54 to 2.82 eV with increasing the filler percentage.
- Finally, we can conclude that $Fe_2O_3$ doped ZnO nanoparticles addition enhanced the optical properties of PS. So the composite material will be suitable as low-index claddings for wave guide applications. Also, it will be suitable for application of photo electronic devices.

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IOSR Journal of Applied Physics (IOSR-JAP) is UGC approved Journal with Sl. No. 5010, Journal no. 49054.

Abomostafa*a " Tuning the optical properties of Fe2O3 doped ZnO / Polystyrene composite filmsH.." IOSR Journal of Applied Physics (IOSR-JAP) , vol. 10, no. 5, 2018, pp. 47-56.