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Structural and optical properties of azo dye doped polyether sulfone

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ABSTRACT

Casting technique was employed for preparation of polyether sulfone (PES) films with and without various mass fractions of azo dye. Dependence of certain physical properties was correlated with filling level (FL). It is observed that upon increasing the azo dye concentrations, FTIR spectrum shows obvious changes in intensity and position of some bands. These results manifested the conclusion about the specific interaction in polymer matrices and hence the occurrence of complexation. UV-Vis. data indicate the presence of a well defined $\pi-\pi^*$ transition associated with the formation of conjugated electronic structure and the decrease in the optical energy gap was correlated to increase of the degree of disorder and overlap in the localized states. X-ray diffraction pattern (XRD) reveals the amorphous nature of the pristine and dye-doped polymer and shows the random distribution of the filler within the polymeric matrix. Scanning electron micrograph (SEM) suggests the dependence of morphological structure on dye concentration. © 2014 Trade Science Inc. - INDIA

KEYWORDS

Azo dye;
PES;
FTIR;
UV-visible;
X-ray diffraction;
SEM.

INTRODUCTION

Polymeric materials have attracted considerable attention due to their stunning growth of interesting potential applications. Among such materials Polyether Sulfone (PES) which is a preferred material because it is a relatively new high temperature amorphous engineering thermoplastic with excellent thermal stability and oxidative stability^[1]. Due to its optimum insulating properties, PES has applications in the electronics, automobiles industries, medical sector, food sector and aircraft cabins^[2]. Polyethersulfone (PES) is finding extensive use in electronics due to its excellent dielectric property. In

fact, PES is now rapidly becoming the material of choice for membrane preparation of hollow fibers^[3,4], stable substrate for the deposition and thermal processing of semiconductor thin films^[5], sensors applications^[6], etc. Moreover, PES is commercially available, relatively inexpensive and exhibits a high glass transition temperature ($T_g \sim 503$ K).

Azo dyes constitute a major part of all commercial dyes employed in a wide range in the textile, leather, pharmaceutical, plastics, paints and food industries. The color of the azo dyes is determined by the azo bond (-N=N-) which is the most active bond in the dye molecule and their associated chromospheres and

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auxochromes. Additionally, Azo dyes have potential applications in the fabrication of devices for optical switching^[7], holographic recording grating^[8], and integrating circuits^[9], etc. The working principle of these devices, either reversible or permanent, hinge on the polarization-dependent refractive index and absorption induced by the photoisomerization of azo dyes, which enables the encoding of different kinds of macroscopic anisotropies with all optical techniques.

In recent years dye doped polymers have attracted the attention of materials researches, because the optical and electrical properties can be modified to a specification requirement by doping process.

The aim of the present work is to prepare and characterize azo-dye doped thermoplastic polymer and to examine their structural and optical properties.

MATERIALS AND METHODS

Materials

Polyethersulfone (PES) granular was obtained from General Electric Company (Victrex 4100P), 5-(4-Antipyrinylazo)-4 phenyl-2-aminothiazol azo dye (Chemistry Dept., Mansoura Univ.) scheme 1, (DEMSO, 99.5%) was used as a solvent for film preparation. All the chemical products were used without any further purification. The studied doped-PES films with different ratio (x wt%) of azo-dye where, x = 0, 0.05, 0.1, 0.5, 1.0 and 2.0 were prepared by casting method. Dimethylsulfoxide (DEMSO) was used as a common solvent for both polymer and azo-dye to ensure homogeneity of the prepared samples. The solution of azo-dye was added to the dissolved polymer at a suitable viscosity. The mixture was cast to a glass dish and kept in a dry atmosphere at 323 K for complete evaporation and removal of any solvent traces. Different thickness of the same samples were prepared to be suitable for different measurements and the obtained films was in the range of \approx 30-120 μ m.

Characterization techniques

X-ray diffraction (XRD) scans were obtained using PANalytical X'Pert PRO XRD system using Cu K α radiation (where, λ = 1.540 Å, the tube operated at 30 kV, the Bragg's angle (2θ) in the range of (5-80°). Fourier Transform Infrared (FTIR) measurements

were carried out using single beam Fourier transform-infrared spectrometer (Nicolet iS10, USA) at room temperature in the spectral range from 4000-400 cm⁻¹. UV/vis. absorption spectra were measured in the wavelength region of 222-800 nm using spectrophotometer (V-570 UV/VIS/NIR, JASCO, Japan). Scanning electron micrograph of the studied samples was performed using SEM Model (Quanta 250 FEG, FEI Company, Netherlands), operating voltage at 30KV accelerating voltage. Surface of the samples were coated with 3.5 nm layer of gold to minimize sample charging effects due to the electron beam.

RESULTS AND DISCUSSION

Fourier transform infrared analysis

Fourier transform infrared (FTIR) spectroscopy was employed to identify the different structural groups in the polymer matrices and to retrace the structural changes result from introducing dopant material. Figure 1 shows FTIR absorption spectra of virgin PES and samples that doped with various levels of azo-dye at room temperature in the region 4000–400 cm⁻¹. The spectra of all samples are quite similar and a little obvious change was recognized. FTIR absorption bands positions and the assignments of all prepared samples

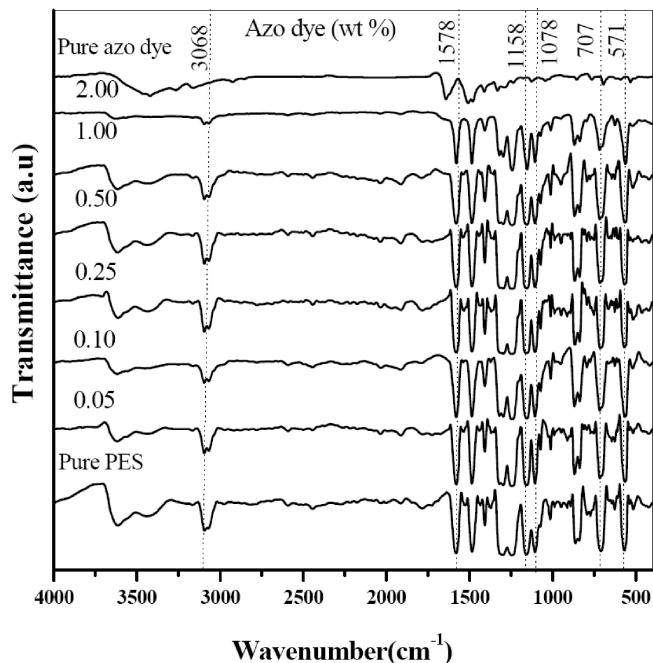


Figure 1 : Infrared spectra of PES films filled with various mass fractions of azo dye

TABLE 1 : FTIR band position and their assignments for the prepared samples

Band position (cm ⁻¹)	Band assignment	Ref.
3600-3309	Broad intermolecular hydrogen bonded,-OH stretching	10-14
3122-3033	Aromatic C-H) asymmetric stretching	14-16
2441	Moderately weak S-H stretching	15
1997-1690	Overtone-combination bands	15,16
1578,1484, 1400	The C=C ring stretching	15,16
1306	Sulfone group,O=S=O asymmetric stretching	19,20
1245	Aromatic ether (Ar-O-Ar) asymmetric stretching	19,20
1158	Sulfone group,O=S=O symmetric stretching	19,20
1109	S=O stretching	15
1078	C-O-C stretching	15,19,21
1015,989	In-plane C-H bending	15
913	Out- of- plane C-H bending	15
846	C-C stretching	15
707	C-S	10
571	SO ₂ Scissoring	15,16
516	Weak S-S stretching	15

are listed in TABLE 1.

From the spectra of all prepared samples, a broad band at 3309–3600 cm⁻¹ is associated with the stretching vibration peak of hydroxyl group (OH) of water molecules^[10] which were trapped in the prepared samples pores^[11] and may be due to the intermolecular or intramolecular type of hydrogen bonding^[12] of the samples. Since, the method of preparation may indeed result in the presence of entrapped water^[13]. This behavior was also observed by Belfer and colleagues for PES membrane where the water molecules were difficult to be removed completely^[14], despite the fact that all the prepared samples were kept in an oven for 4 days at 70 °C.

Aromatic C-H asymmetric stretching vibration occurs in the range 3122 - 3033 cm⁻¹ which its intensity decreases with increasing filler concentration. The band at about 2441 cm⁻¹ is assigned to weak S-H stretching^[15] which shifts to lower wavenumbers at high concentrations of azo dye. In the 1997 –1690 cm⁻¹ region,

a series of weak combination and overtone bands appears and the pattern of the overtone bands reflects the substitution pattern of the benzene ring^[15,16].

Skeletal vibrations, representing C=C stretching vibration of benzene ring occurs in the region 1578–1400 cm⁻¹ indicates the formation of small conjugated polyene sequences, which are presumably responsible for the color of the fillers-treated PES. The changes which appear in this band for different FLs point to the possibility of azo dye to be attached to a C=C group in the side chain of the PES molecule. The band at 1109 cm⁻¹ refers to S=O srtretching^[15]. It is remarkable that, the present double bonds segments are considered as suitable sites for polarons and/or bipolarons to be in the polymeric matrix^[17,18].

The asymmetric stretch of the aromatic ether group (Ar-O-Ar) appears at 1245 cm⁻¹. The band at 1306 cm⁻¹ refers to O=S=O asymmetric srtretching of the sulfone group^[19]. The band at 1158 cm⁻¹ refers to O=S=O symmetric srtretching of the sulfone group^[20]. The absorption band due to the symmetric stretching of C-O-C appears in the region 1078 cm⁻¹^[21].

C-H in the plane deformation vibration appears at 1015, 989cm⁻¹ while Out-of-plane aromatic C–H bending (monosubstituted benzene ring) appears at 913 cm⁻¹^[15]. Low-frequency bands are of little use in determining the nature of ring substitution since these absorption patterns result from the interaction of O=S=O and C-H out-of-plane frequencies. The intensity of the bands also decreases with increasing the FLs of filler. In the pristine PES, SO₂ scissoring is observed at 571 cm⁻¹^[19,20]. There is an obvious decrease in the intensity of this band at high dopant levels, namely (W=2.0 wt %).

From the infrared spectra, it can be noticed that the doping with azo dye causes some observable changes in the spectrum of PES in the range 1000-400 cm⁻¹. It induces slight changes in the intensities of some absorption bands which indicate that the dopant is complexed with the polymer matrix^[22].

The absorption band at about 846 cm⁻¹ is attributed to C-C stretching^[15] and that at about 913 cm⁻¹ is assigned to C-H bending^[23], exhibits changes in its intensity behavior with azo-dye content. The apparently changes in the intensity of this peak with azo dye means that this peak is sensitive to some type of defects, which may be head-to-head [(h-to-h)] and tail to- tail [(t-to-

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t)] polymer chain defects^[24]. The h-to-h defects are found to be suitable site for polarons and/or bipolarons in the PES matrix^[23]. C-S stretching vibration at 707 cm⁻¹ is also observed^[25] while, the absorption region 516 cm⁻¹ corresponds to weak S-S stretching^[15].

From the IR spectra, it is observed that upon increasing the filler azo dye concentrations, some of the peaks are shifted, some bands increased and some of them decreased with respect to the pure PES. These results manifested the conclusion about the specific interaction in polymer matrices and hence the occurrence of complexation.

Ultraviolet and visible analysis

Ultraviolet-visible (UV-Vis.) spectroscopy is an important tool for investigation as it gives an idea about the value of optical band gap energy (E_g). The absorption of light energy by polymeric materials in UV and visible regions involves promotion of electrons in σ , π , and η orbital from the ground state to higher energy states which are described by molecular orbitals^[26]. Doping the polymers leads to the formation of new defects and new charge states.

The results of absorption studies with UV/Vis. spectrophotometer in the wavelength range of 228-800 nm

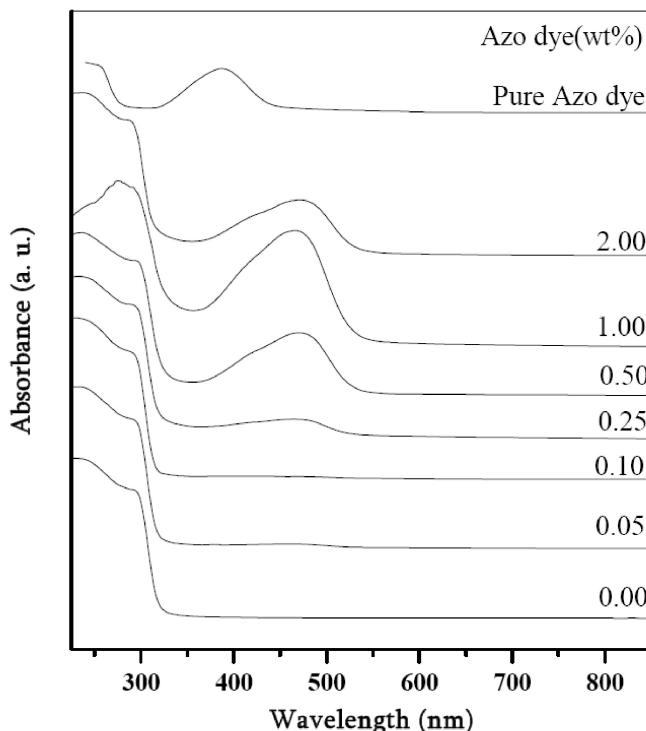


Figure 2 : UV/Vis. spectra of pure PES, pure azo dye and PES filled with different concentrations of azo

carried out on virgin and filled samples are illustrated in Figure 2 .

The optical absorption spectrum of the virgin sample shows an absorption edge which has a sharp decrease with increasing wavelength upto~313 nm, followed by plateau region. The observed spectra are characterized by the main absorption edge for all curves that shifted towards higher wavelength with increase the filler content. These shifts of the spectra indicate the complexation between the filler and the polymer, and may also be due to change in crystallinity, which reflects the variation in the optical energy gap, due to adding the filler^[27]. It is evident that optical absorption increases with increasing filler content and this absorption shifts from UV/Vis. towards the visible region for filled samples. The increase in absorption may be attributed to the formation of a conjugated system of bonds due to bond cleavage and reconstruction^[21].

Small band at about 235 nm in UV spectra is considered due to the transition $n \rightarrow \pi^*$ for polymer^[28]. The absorption band (shoulder) of pure PES at about 274 nm may be attributed to the $\pi \rightarrow \pi^*$ transition which comes from unsaturated bonds, mainly (C=C and/or S=O) in polymeric macromolecule^[29] which observed in FT-IR spectra. It is observed that the spectrum of pure azo dye dissolved in DMSO (Common solvents for the studied system) has absorption peak at 390 nm. For PES films filled with different amount of azo dye, the spectra contain absorption peak at about 462 nm. It is reasonable to assign the observed bands to the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition of chromophoric groups. The FL dependence of the intensity of the band at about 462 nm is shown in Figure 3. The intensity of this band increases as the azo dye content increases, where their positions are slightly changed.

Figure 3 provides an evidence for incorporation of azo dye into PES matrix. These results manifested the conclusion about the specific interaction in PES matrices and hence the occurrence of complexation.

Determination of optical energy gap (E_g)

The optical absorption method can be used for the investigation of the optically induced transitions and can provide information about the bond structure and energy gap in crystalline and non-crystalline materials^[30].

The study of optical absorption gives information

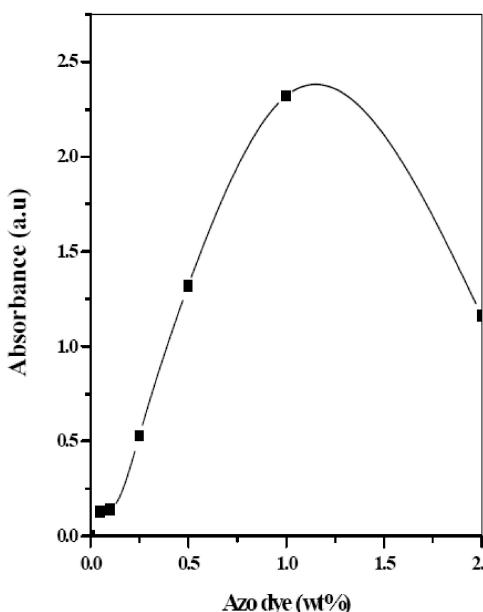


Figure 3 : FL dependence of absorption peaks at 462 nm.

about the band structure of organic compound. Semiconductors are generally classified into two types: (i) direct band gap and (ii) indirect band gap. In direct band gap, the top of the valence band and the bottom of conduction band both lie at the same zero crystal momentum (wave vector). If the bottom of conduction band does not correspond to zero crystal momentum, then it is called indirect band gap. In indirect band gap materials transition from valence to conduction band should always be associated with a phonon of the right magnitude of crystal momentum^[31]. Davis and Shalliday^[32] reported that near the fundamental band edge, both direct and indirect transitions occur and can be observed by plotting $(\alpha h\nu)^{1/2}$ and $(\alpha h\nu)^2$ as a function of photon energy ($h\nu$). The analysis of Thutupalli and Tomlin^[33] is based on the following relations:

$$(n\alpha h\nu)^2 = c_1 (h\nu - E_{gd}) \quad (1)$$

$$(n\alpha h\nu)^{1/2} = c_2 (h\nu - E_{gi}) \quad (2)$$

Where $h\nu$ is the photon energy, E_{gd} , the direct band gap, E_{gi} , the indirect band gap, n , integer, c_1 , c_2 , constants and α is the absorption coefficient.

The absorption coefficient (α) can be determined as a function of frequency using the formula^[34]:

$$\alpha = 2.303 \times (A/d) \quad (3)$$

Where A is the absorbance and d is the thickness of the sample under investigation.

By plotting $(\alpha h\nu)^{1/2}$ versus photon energy ($h\nu$), each linear portion indicates a band energy gap E_{gi} . It is will be noticed that the curves are characterized by the pres-

ence of an exponentially decay tail at low energy^[35].

From the linear parts of these curves as shown in Figure (4.a-g), the E_{gi} values of the films were calculated and are given in TABLE 2. The absorption edge shifts the higher wavelengths with increasing azo dye content. This suggests that the optical band gap decreases with azo dye content as shown in Figure 6. It is clear that E_{gi} decreases as W increases. This indicates that the azo dye filler significantly influence optical energy gap.

TABLE 2 : Absorption edge (λ_g), optical energy gap (E_g) and refractive index (n) for prepared samples.

Azo dye (wt%)	Absorption edge(λ_g) (nm)	E_g (eV)		n	
		E_{gd}	E_{gi}	n_{direct}	$n_{indirect}$
0.00	328.94	3.97	3.78	2.176	2.214
0.05	330.03	3.96	3.76	2.178	2.218
0.10	330.03	3.98	3.77	2.174	2.220
0.25	335.57	3.94	3.71	2.182	2.228
0.50	340.72	3.92	3.66	2.182	2.239
1.00	353.61	3.84	3.49	2.201	2.276
2.00	364.96	3.82	3.42	2.206	2.292

The experimental data have been obtained for equation (1). This behaviour indicated that the transitions are direct allowed transitions. So, we plot $(\alpha h\nu)^2$ versus photon energy ($h\nu$) in order to determine the optical band gap energies (E_{gd}) by drawing the extrapolation of the linear region of the curve to the $h\nu$ -axis as shown in Figure (5.a-g) The band gap energies (E_{gd}) obtained in the present work are given in TABLE (2).

The values of direct band gap and indirect band gap are showed in Figure 6.

It is clear from Figure 6 that the direct band gap and indirect band gap values showed a decrease with increase the filler concentrations. This decrease indicates that there are charge transfer complexes arose between the polymer and filler and may be attributed to the formation of defects in the polymeric matrix. These defects produce the localized states in the optical band gap. These overlaps are responsible for decreasing energy band gap when filler concentration is increased in the polymer matrix^[30]. In other words, the decrease in the optical gap results in an increase in the degree of disorder in the films. These results are supported by the data obtained from XRD studies in the present work.

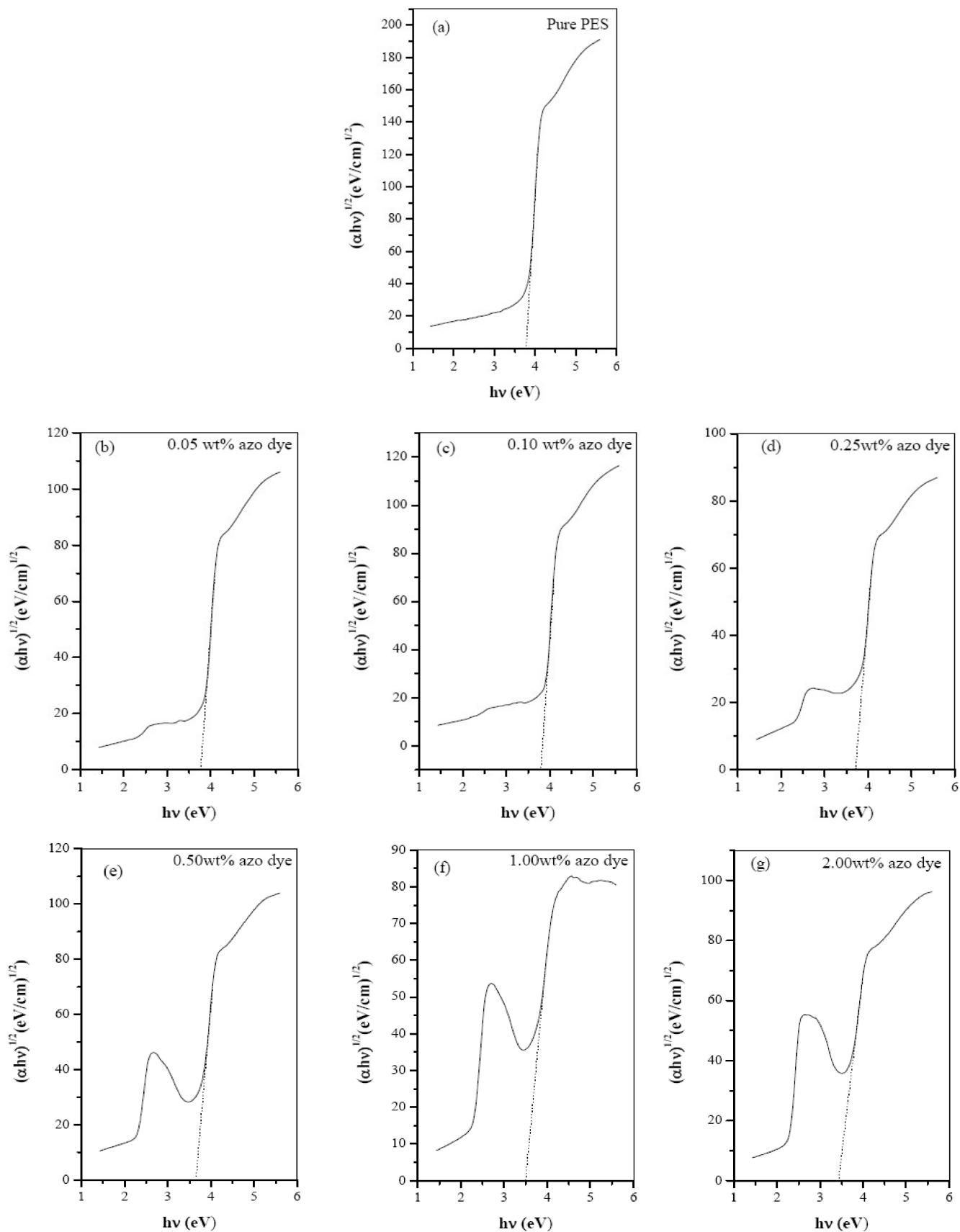
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Figure 4(a-g) : The relation between $(\alpha h\nu)^{1/2}$ versus photon energy ($h\nu$) for pure and filled polymer.

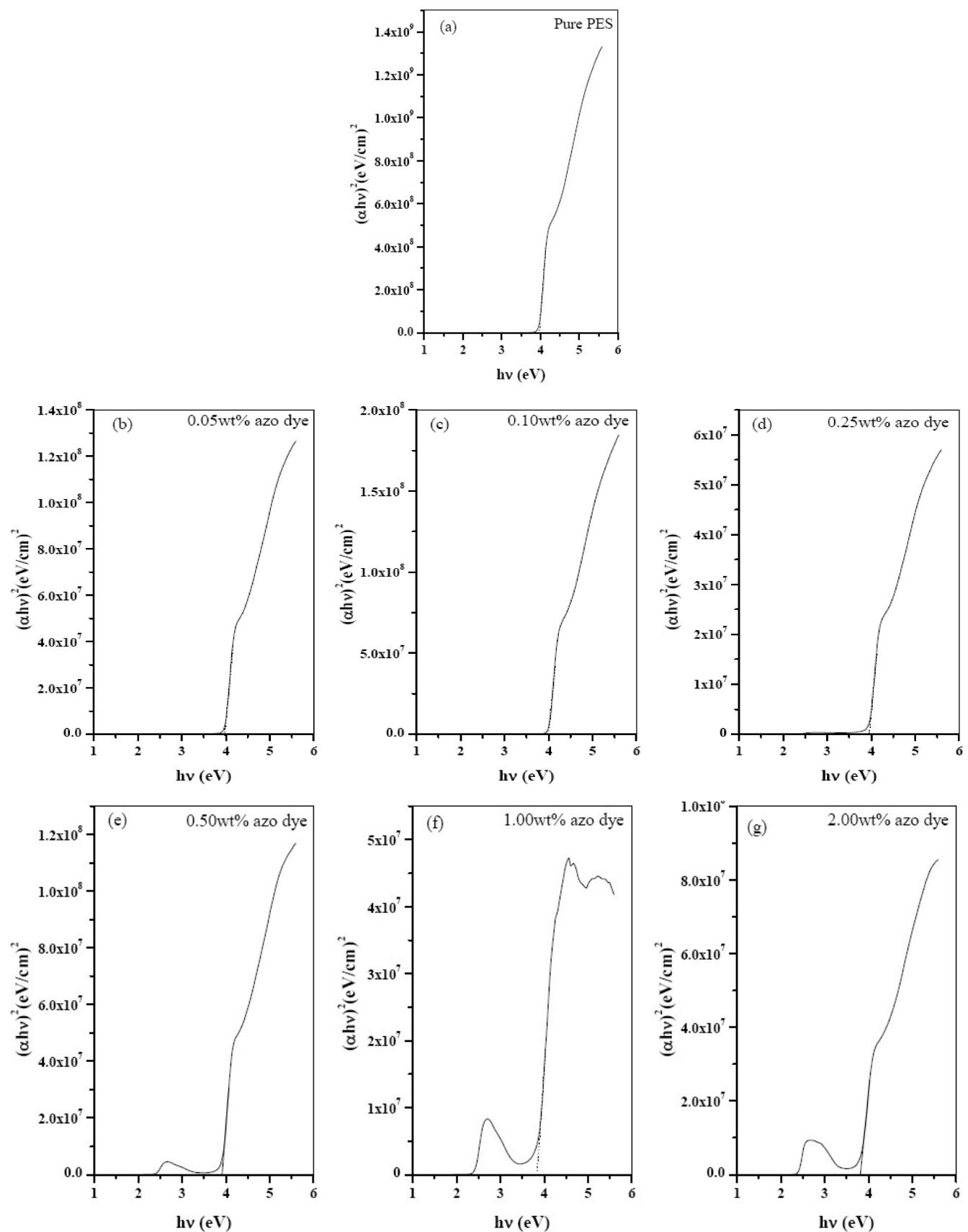


Figure 5(a-g) : The relation between $(\alpha h\nu)^2$ versus photon energy ($h\nu$) for pure and filled polymer.

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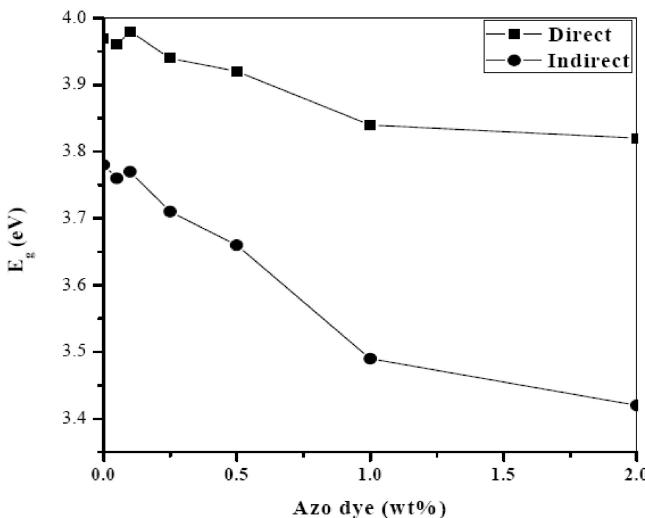


Figure 6 : The FL dependence of both the direct and indirect optical energy gap E_g for pure and filled

TABLE 3 : Peak position, peak intensity and area under the amorphous peak A_T for both pure PES and PES filled with azo dye

Azo dye (wt%)	Peak Position (2θ)	Peak Intensity	Area A_T (cm^2)
0.00	17.11	263	6.75
0.05	17.86	290	7.01
0.10	17.85	212	3.96
0.25	17.45	286	8.32
0.50	18.00	554	26.31
1.00	17.70	571	28.85
2.00	16.76	210	4.23

The shift in absorption edge was correlated with the optical band gap E_g , as TABLE.3, given by;

$$E_g = hc / \lambda_g \quad (4)$$

Where h is Planck constant and c is the velocity of light. The wavelength λ_g is determined by Tauc's expression^[36] from the intersection with the abscissa of the plot of (ε^2/λ) versus $(1/\lambda)$ where ε is the optical absorbance and λ is the wavelength. The absorption edge moves towards higher wavelengths.

Determination of the refractive index

In optics the refractive index n of a substance is a dimensionless number that describes how light, or any other radiation, propagates through that medium. Polymers with sulfone moieties exhibit n values above 1.72, depending on the degree of molecular packing^[37]. The refractive index (n) was examined by equation (5) derived by Dimitrov^[38].

$$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{\frac{E_g}{20}} \quad (5)$$

Where E_g is the direct and indirect band gap of the polymer sample, estimated by ultraviolet (UV) absorption edge analysis of samples. The values of the refractive index which are calculated from direct and indirect band gap are showed in TABLE (3) and Figure 7.

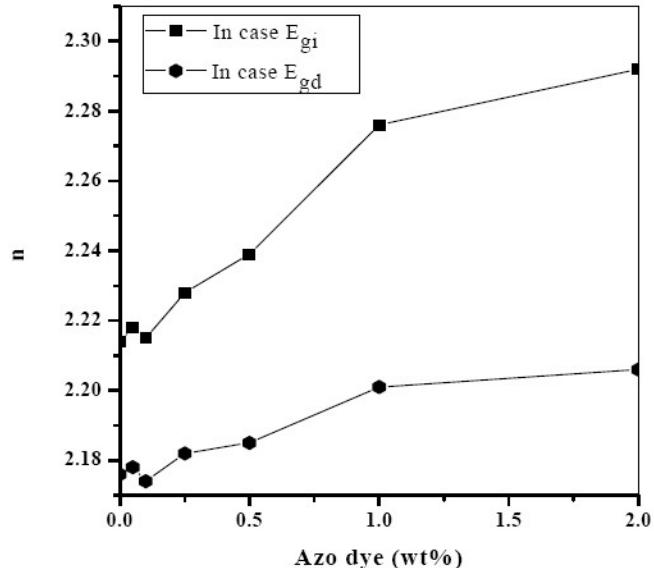


Figure 7 : The FL dependence of both the refractive index for pure and filled polymer.

It is clear that the refractive index of polymers increased with increase the filler concentrations. This increase indicates that there are decreases in the molar polarizability and to increasing the number of free electrons interacting with light^[39]. The refractive index changes suggest the applicability of PES films filled with azo dye for optical devices.

X-ray diffraction analysis

The polymer studied here is a complex polymer widely used in electronics and sensors applications falling in the category of polymeric materials that consist of crystalline and amorphous regions in different proportions. XRD measurements have been carried out for pristine sample and samples filled with various filling levels of azo dye to observe changes in structure and examine the crystallinity.

It is worth quoting that Blackadder and Ghavamikia^[40] reported on the problem of defining PES crystallinity. No evidence of X-ray crystallinity was found in any of the tested PES samples. They concluded

that PES is undoubtedly crystalline on the evidence of outward appearances. The main aim of this measurement is to check crystallization of PES films with and without filler. It must be pointed out that crystallinity of polymers is one of the factors determining their transport properties^[41].

Figure (8) shows the XRD pattern for virgin and azo dye filled PES films with various mass fractions W namely (W = 0.05, 0.1, 0.25, 0.5, 1.0, and 2.0 wt %). The diffraction pattern of virgin PES indicates that this polymer is mainly amorphous in nature and shows one prominent X-ray peak at $2\theta = 17.11^\circ$ ^[40,41]. A comparison in terms of peak position, peak intensity and the area under the amorphous peak A_T have been carried out and summarized in TABLE (3).

The present X-ray scans revealed a very significant change in the amorphous peak position due to the azo dye filling. These apparent changes in band position, especially at high concentrations of azo dye 2 (wt%), point out that azo dye used in this study seemed to alter

weakly the average intersegmental spacing of polymer chains (i.e. this implies that the lattice parameters changed significantly)^[41].

It is clear that A_T increases as W increases, until 1.0 wt % and then decreases indicating that the filler significantly influence the degree of crystallinity^[42]. At 1.0 wt % FL, the intensity of diffraction peak has a maximum value implying the more organized distribution of the azo dye filler in the polymeric matrix. With increasing the content of azo dye and at 2.0 wt % FL, there is a dramatic decrease in the intensity of the diffraction peak and the main amorphous peaks became broad and a significant decrease in its intensity was observed indicating that the distribution of the azo dye filler in the polymeric matrix became random. This behaviour demonstrates that complexation between the filler and the polymers took place in the amorphous regions^[43].

Scanning electron microscopy (sem)

Morphology of the studied samples was investigated with SEM to provide further information about the structural modifications of PES films due to filling with azo dye. Scanning electron micrograph (SEM) suggests the dependence of morphological structure on FL. Figure (9.a-g) shows the SEM micrograph of the surface of pure and filled films with different concentrations of azo dye at magnification 50,000 times. Figure (9.a) shows pure polymer morphology which is transparent and is shown to be in a uniform morphology revealing a rather smooth surface. Figure (9.b) shows some pits of different sizes between the PES and azo dye interface, which accompanied by pores of different size randomly distribution in a medium as shown in Figure (9.c). This can be attributed to the partial compatibility between the polymer and the filler.

By increasing the concentration of the additives to 0.5 wt %, granules of randomly distribution accompanied with a degree of roughness in a medium that appeared to be pure PES as shown in Figure (9.e). The grown ones were of different sizes and irregular shapes, uniformly distributed in the amorphous matrix, indicating the occurrence of a homogeneous growth mechanism, which is also observed in Figure (9.f).

On the other hand, the micrograph of W = 1.0 wt % as shown in Figure (9.f), the filler begin accumulate

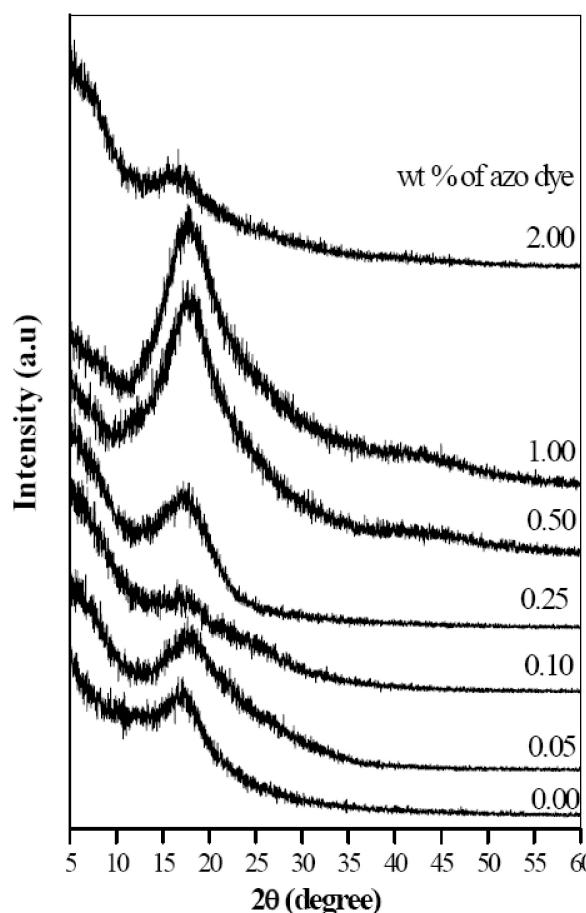


Figure 8 : The X-ray diffraction scans for variously filled and unfilled PES with azo dye.

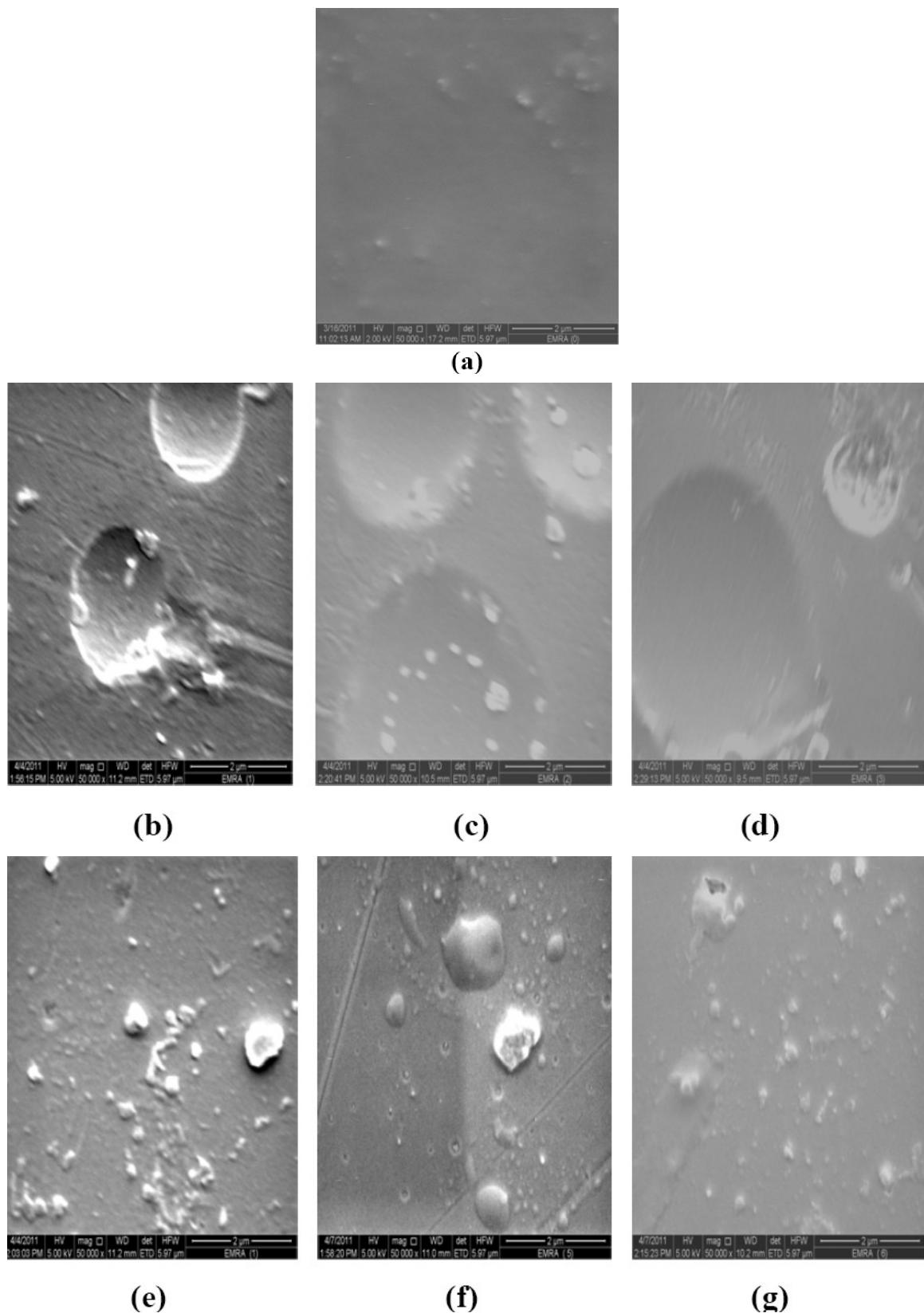
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Figure 9 : Scanning electron micrograph with and without filling by azo dye.

at the surface which increased and more apparent at W=2.0 wt %, as in Figure (9.g).

These results showed the formation of a crystalline phase, when PES was filled with azo dye, that increased

with an increasing W value of azo dye. These results confirmed the findings of XRD in this work. This indicates segregation of the filler in the host matrix and this may be confirmed by the interaction and complexation between filler and polymer.

CONCLUSION

Casting technique was employed for preparation of pristine and filled polyether sulfon (PES) films with various mass fractions of azo dye. Dependence of certain physical properties was correlated with filling level (FL). From the FTIR spectra, it is observed that upon increasing the filler azo dye concentrations, some of the peaks are shifted, some bands increased and some of them decreased with respect to the pure PES. It is remarkable that the present double bonds segments are considered as suitable sites for polarons and/or bipolarons. These results manifested the conclusion about the specific interaction in polymer matrices and hence the occurrence of complexation. UV/Vis. analysis showed that the spectra of the filled PES films exhibit a shift of sharp absorption edge towards longer wavelength. The shift of the absorption edge in the filled PES reflects the variation in the optical energy gap, which arises due to the variation in crystallinity within the polymer matrix. Results of the optical measurements indicated the presence of a well-defined $\pi \rightarrow \pi^*$ transition which comes from unsaturated bonds, mainly; C=C and/or S=O, which is responsible for electrical conduction in the samples. Also, it is observed that there is charge transfer complexes arose inside the prepared samples. The spectra of filled films showed a new band at about 462 nm assigned to the chromophoric group of azo dye. The intensity of this band increase with increasing the filling level. The appearance of this band confirms the occurrence of complexation between the filler and the host polymer.

The optical energy gap E_g was decreased by almost 10% at the highest concentration of azo dye 2.00 wt%. The refractive index (n) increased with increasing the filling level of azo dye. The change of n is a criterion of structure change. The refractive index changes suggest the applicability of PES films filled with azo dye for optical devices. X-ray scans revealed a very significant change in the halo position due to the azo dye filling. It

was observed that the fillers significantly influence the degree of crystallinity. SEM results shows the formation of a crystalline phase which increases with increasing filler content, indicates segregation of the filler in the host matrix, these results confirm the findings of XRD and optical measurements.

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