

Spectroscopic and band-gap characteristics of gamma irradiated ultra-high molecular weight polyethylene/gamma aminobutyric acid (UHMWPE/GABA) composites

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Abstract

The behavior of the absorption for pure and gamma ray irradiated (i.e. 30, 65 and 100 KGY) "polyethylene with ultra-high Mol. Wt. i.e (PE-UHMW)" within the visible range of electromagnetic spectral (i.e. 400-800 nm) has been investigated by utilizing the Muller-matrix spectro-polarimeter. The considerable changes have been observed in the tendency of absorption for treated samples as a result of physical and chemical changes, caused by radiation. In order to examine these changes that are caused by radiation in polymer structure, the Urbach edge method has been utilized to determine the optical activation energy, whereas the band energies (i.e. direct and indirect) and the "C" atom's numbers in the sample's cluster of C=C unsaturation have also been estimated by graphical method while employing the generalized form of the Urbach's relation and Tauc's relation respectively. According to the findings acquired in this work the Urbach energy is found to decrease with irradiation and has the lowest value for pure PE irradiated with 30 kGy (i.e. P-30) which is 98.56 MeV. Whereas for PE/GABA composites with 15% GABA by wt. of PE, irradiated with 100 kGy (i.e. PA-100) and for PE/GABA composites with 30% GABA by wt. of PE, irradiated with 100 kGy (i.e. PB-100) the values of Urbach energies are evaluated to be 122.45 MeV and 106.08 MeV respectively. The band energies (direct/indirect) have also followed the minimizing tendency with radiation treatment. Additionally the indirect energy band energies are discovered to possess lesser values in relation to direct energy band energies. The C atom's numbers in case of pure PE are 6 - 7 for 'direct' band energies and 7 - 9 for 'indirect' band energies whereas for PE/GABA composites with 15% GABA by wt. of PE (i.e. PA) the "C" atom's numbers for direct band energies are 7 while fluctuating from 7 - 8 for 'indirect' band gaps. In case of PE/GABA composites containing 30% GABA by wt. of PE (i.e. PB) the C atom's number is from 7 - 8 for 'direct' band energies and 7 - 9 for 'indirect' band energies.

Keywords: UHMWPE; gamma amino butyric acid; band-gap; Urbach energy; direct energy;

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I. Introduction

The significance of polymers possessing enriched surface and bulk properties is increasing day by day because of their benefits such as low weight, low cost, ease in processing and fabrication (Gul, McGarry et al. 2003). Ultra-high molecular weight polyethylene (UHMWPE) is a unique thermoplastic polymer, it belongs to polyethylene (PE) family and possessing an average molecular weight from 3,000,000 to 6,000,000 g/mol (Gul, McGarry et al. 2003, Sobieraj and Rimnac 2009). Because of its physical, chemical, mechanical, and biocompatible properties, it has been extensively used in the fields of orthopedic implants, electrical insulation, medicine, microelectronics engineering and food industry (Kumar and Pandya 1997, Buncick, Thomas et al. 2000, Sobieraj and Rimnac 2009, Bistolfi and Bellare 2011).

In order to utilize the UHMWPE for particular application of interest its physical and chemical properties need to be modified, for this purpose, the irradiation of polymers is significant and most reliable method at present (Kamal, Bashir et al. , Mishra, Tripathy et al. 2001, Brooks, Hole et al. 2002, Saad, Atwa et al. 2005). All the radiation induced physical and chemical processes result in modification of UHMWPE so as to utilize it for any particular application, e.g. to fabricate polymeric optical fibers, LEDs, optical sensors,

antireflective coatings etc. (Lee 1999, Brooks, Hole et al. 2002). It is vital to have knowledge about the optical properties and factors (e-beam radiations in this study) effecting these properties for abovementioned applications in the field of optics. This would legitimize the requirement of studying the effect of high energy radiation on optical properties of the UHMWPE in comprehensive detail(Kamal, Bashir et al.).

The research is going on to find/investigate the optical properties of pure and irradiated UHMWPE, in order to check the feasibility of using it for aforementioned applications. In this regard, Abdul-Kader (2009)(Abdul-Kader 2009) studied the modification in optical properties of ‘He’ ion bombarded UHMWPE by using photoluminescence and UV spectroscopy. In his work he made the correlation of ion fluence with optical band gaps and activation energies of irradiated samples of UHMWPE. It was discovered that energy band gaps and activation energies of samples decreased with an increase in the concentration of ions. Since this study is comprehensive as far as irradiating the samples with heavy ion like He ion but these results cannot be extrapolated/generalized for other type of high energy radiations like X-ray, gamma ray, e-beam and laser etc.

After that, Raghuvanshi et al. (2012) (Raghuvanshi, Ahmad et al. 2012) studied the effect of gamma rays irradiation on optical properties of UHMWPE. In this study the important properties of PE were focused that are: Urbach energy, direct and indirect band gap energies, and number of carbon atoms in a cluster of C=C unsaturation. As a consequence of the obtained results, it was proposed that UHMWPE could be used as material for radiation dosimeter. However, significantly higher values of gamma doses (i.e. 500–2000 kGy), thickness of PE sheets, and single data set of absorption experiment could make these results questionable for potential applications/readers.

In this study, effect of adding GABA on band gap properties of UHMWPE has been explored. The changes in spectroscopic and band gap properties of UHMPWE on irradiating it in the presence and absence of GABA are assessed and discussed

II. Materials and Methods

Material, Sample Preparation & Irradiation

Material, sample preparation & irradiation

Lab-grade UHMWPE in powder form (possessing an avg. Mol. Wt. of 3 to 6 million grams/mol) was acquired from Sigma Aldrich. The micron size films of UHMWPE powder were prepared, for this purpose the heating press machine which is accessible at PIEAS (Pakistan Institute of Engineering and Applied Sciences, 45650, Islamabad, Pakistan) having rise-up and calm-down rate of 10°C/min has been employed. The UHMWPE powder was pressed with the pressure of 200 bar, for 12 to 15 minutes at temperature of 140 °C, 160 °C, and 190 °C, respectively. Now in order to make the UHMWPE composites with GABA(Gamma-aminobutyric acid) specific amount of GABA was added into UHMWPE powder then this mixture was added into a beaker containing a small amount of distilled water. After stirring the mixture in the beaker, the beaker was kept in the oven at temperature of 80°C for some time in order to dry the mixture. Then the micron size films of the mixture/powder were prepared by using the above mentioned procedure. Then these films were cleansed with a chemical “acetone” to erase contaminants from their surface and then thickness of the films was measured. After this, these prepared films were classified into three main groups; i.e. pure samples, samples with 15% GABA concentration, and samples with 30% GABA concentration. Each sample was labeled with different code for its differentiation on the basis of radiation treatment and concentration of GABA.

Table 3.1: Samples codes with respect to pure UHMWPE and GABA concentration

Samples code*	GABA Concentration in %
P	0 % (Pure UHMWPE)
PA	15 % of UHMWPE
PB	30 % of UHMWPE

The prepared samples were then dispatched to PARAS, Lahore for irradiation with gamma dose at a rate of 1 kGy/pass. The samples have been irradiated at room temperature, about (~25°C) in open air with gamma dose of (30, 65, and 100 kGy) respectively. Then these samples have been characterized by employing the UV-VIS spectroscopy. The reason behind the irradiation of samples at or under 100 kGy was the polyethylene crosslinking saturation at or beyond 100 kGy.

Sample testing

The samples have been tested by employing an automatic Mueller matrix spectro-polarimeter controlled utilizing Axo scan TM (engineered by Axometrics, 2006) in this experiment. For each sample four to five readings have been obtained from different angles by putting them in different lateral-orientations

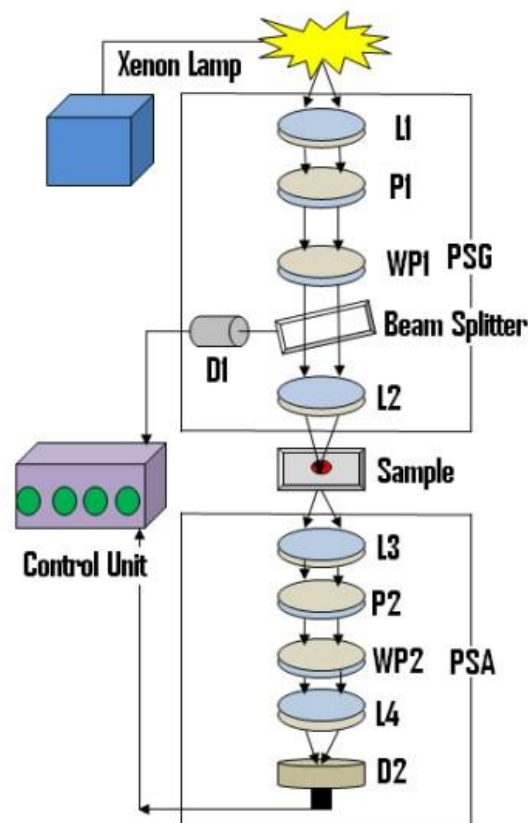


Figure-1: Schematic diagram of Mueller matrix spectro-polarimeter
(L = Collimating lens, P = Polarizer, WP = Waveplate, D = Detector)

The figure 1 shows the schematic diagram of the Mueller matrix spectropolarimeter which is used in this particular experiment (Ahmad, Ali et al. 2013). The Mueller matrix spectro-polarimeter (MMSP) setup comprised of a low-noise Xenon lamp having power of 150 W, which is working as an input light source. Spectro-polarimeter has an inherent diffraction grating monochromator, in order to select the desired wavelengths in the visible range of EM spectrum (i.e, 400 nm to 800 nm), with a precision of 6 ± 0.5 nm for our selected wavelength. The Mueller matrix spectro-polarimeter (MMSP) consists of two portions, one is named as the polarization state generator (PSG) and the other is named as the polarization state analyzer (PSA). These two portions are used for the generation and analysis of various polarization states, that are required for the Mueller matrix determination of sample. The polarization state generator (PSG) comprises of collimating lens which aligns the beam of light in a specific direction to travel through the cable of optical fiber. Then this collimated beam is passed through the set of polarizer that contains both the static polarizer and moving waveplate at high speed. Waveplate is such type of polarization element that is designed to generate a definite phase difference between the exiting beams for two orthogonal incident states of polarization and it is also called as retarder. Subsequently, the beam interacts with the beam splitter that diverts a small part of the beam to display the light output power, while the main beam moves through the lens positioned next to the beam splitter. This collimated beam of light then interacts with the sample and produces an output light which moves through the polarization state analyzer (PSA) of the instrument. The polarization state analyzer also comprises of collimating lens, set of polarizer and the detector like the polarization state generator. After interaction of light with the sample the beam of light passes through the lens of PSA and through the set of both stationary polarizer and moving waveplate. Eventually, the beam of light is focused by the lens on the detector which is connected to the computer. This kind of polarimeter is called as dual rotating retarder polarimeter (Azzam 1978, Goldstein 1992).

Data analysis and processing

This work basically focuses on determining the thermal annealing effects on the band-gap properties of pure and gamma irradiated UHMWPE. These properties are absorption-coefficient $\alpha(\nu)$, Urbach energy E_u , band-gap energies (i.e, direct/indirect) and C atom's number (N) in sample's C=C clusters. The absorption-coefficient $\alpha(\nu)$ has been determined from absorbance (A) while utilizing the following relation (Mujahid, Srivastava et al. 2005).

$$\alpha(\nu) = \frac{A}{l} \quad (3.1)$$

Where;

- l = Thickness or breadth of the sample in μm
- A = The absorbance it is defined as $A = \log (I_0/I)$

For amorphous materials, the absorption coefficient $\alpha(\nu)$ exponentially depends on the energy of the photon ($h\nu$) close to the band edge, and it has been described by the Urbach formula [90].

$$\alpha(\nu) = \alpha_0 e^{\frac{h\nu}{E_u}} \quad , \quad (3.2)$$

Where;

- α_0 is a constant,
- h is Planck's constant,
- ν is frequency of radiations used,
- E_u is the Urbach energy also called as optical activation energy. Fundamentally it is described as the thickness of extension of localized states (localized defect states) in forbidden energy band-gap (E_g) which is caused because of thermal vibrations of atoms or molecules in the crystal lattice/matrix (Buncick, Thomas et al. 2000, Raghuvanshi, Ahmad et al. 2012). These localized defects states in the band-gap region are responsible for the emergence of absorption tail in the absorption spectra. Which is termed as Urbach tail and energy associated with it is known as Urbach energy. In order to calculate the value of Urbach energy the inverse of the linear part of the slope in lower photon energy region is utilized for every single absorbance curve. Initially the Urbach relation was presented for crystals of alkali halide which turned out to work extremely effective for many amorphous materials as well. Although; Mott and Davies (Abdul-Kader 2009, Raghuvanshi, Ahmad et al. 2012) modified the Urbach formula, in order to have more generalized relation, which is given as:

$$\alpha(h\nu) = \frac{B(h\nu - E_g)^n}{h\nu} \quad (3.3)$$

Where;

- "h ν " is the incident photons energy,
- "E $_g$ " is the band-gap energy between valence band and conduction band,
- "B" is the constant it is depending upon transition probability inside the range of optical frequency, and
- "n" states the mode of electronic transition in K-space. Its value is taken as 2 for indirect allowed and 3 for indirect forbidden transitions whereas 1/2 for direct allowed and 3/2 for direct forbidden transitions.
- "N" represents the no. of carbon (C) atoms in each sample's cluster, is correlated with E_g and it has been calculated by utilizing the generalized form of Tauc's equation (Abdul-Kader 2009, Raghuvanshi, Ahmad et al. 2012).

$$N = \frac{2\beta\pi}{E_g} \quad (3.4)$$

Where;

- 2β is the band structure energy for a couple of neighboring π locations and the value of β is approximately taken as 2.9 eV for each $\pi \rightarrow \pi^*$ optical transitions in sample's -C=C-structure.

III. Results and Discussion

UV-Visible spectra obtained from MMSP for pristine and gamma irradiated samples of (PE-UHMW) are shown in figure. This spectrum describes the absorption of various doses of gamma ray with respect to particular wavelengths of incident and transmitted light from the sample. The samples have been irradiated with gamma ray of 30, 65, and 100kGy, consecutively, while wavelength range is from 400-800nm (i.e, optical window). The absorbance data of gamma irradiation doses from the sample have been explored.

Plotted results explained the behavior of the absorbance with increased doses of gamma ray in an optical window range. It can be seen in figure 2 that absorbance is decreasing with increasing dose of gamma ray on moving towards the longer wavelength. There is no considerable difference in the absorbance spectra/curve with the increased gamma dose. Moreover, there is not much difference in the peak of absorbance curve with increasing doses of gamma irradiation. The peak of absorbance curve for pristine sample is 2.3 at 408nm, whereas for 30 kGy it is 1.28 at 402nm, 1.3 at 402nm for 65 kGy and 1.35 at 405nm for 100 kGy samples.

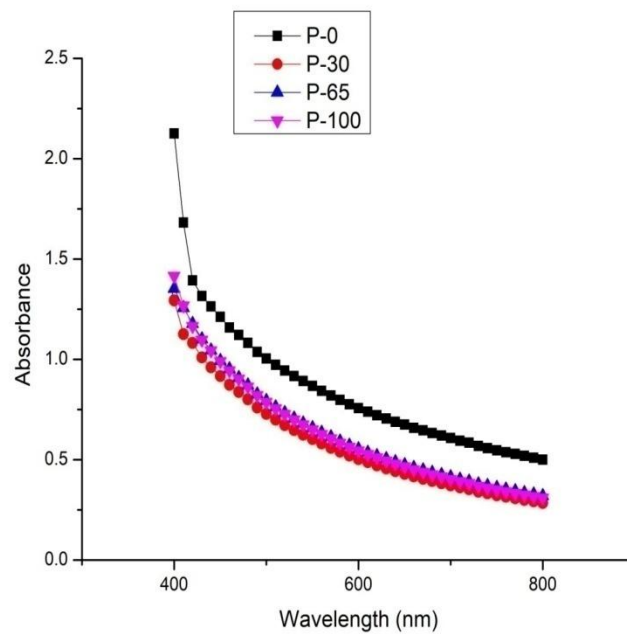


Figure 2: UV-VIS spectra of pure and gamma irradiated UHMWPE

Absorbance spectra for PE/GABA composites with 15% GABA by wt. (i.e, PA):

There is not much difference in the absorbance curve for pristine and 30k Gy irradiated sample and it can be seen from the graph that the absorbance curve obtained for 65 and 100 k Gy irradiated samples are overlapping. Moreover, the peak of absorbance curve have varying trend with respective doses of gamma irradiation. The peak of absorbance curve for pristine sample is 3.7 at 450nm, whereas for 30 kGy it is 3.0 at 472nm, 3.1 at 470nm for 65 kGy and 3.5 at 480nm for 100 kGy samples (please see figure 3).

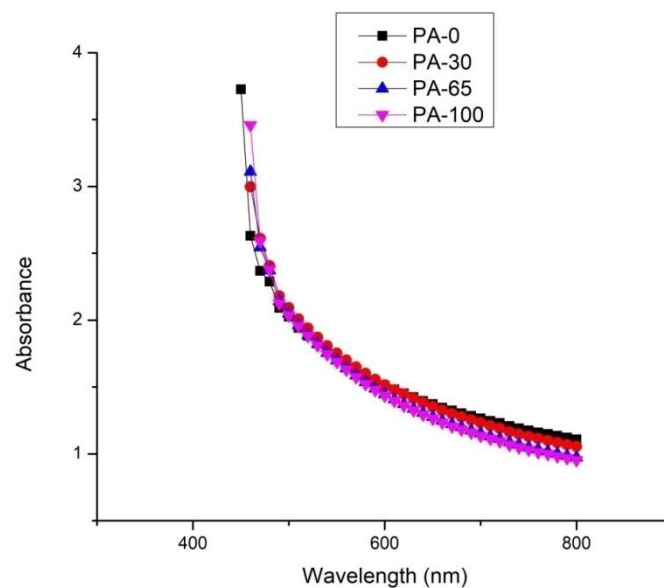


Figure 3: UV-VIS spectra of pure and gamma irradiated PE/GABA composites with 15% GABA by wt. (i.e, PA)

Absorbance spectra for PE/GABA composites with 30% GABA by wt. (i.e, PB):

The absorbance is decreasing with increasing dose of gamma irradiation. The decrease in absorbance is linear for 30 and 100 k Gy samples whereas it varies for 65 k Gy irradiated sample. The peak of absorbance curve for pristine sample is 2.6 at 470nm, whereas for 30 kGy it is 2.75 at 482nm, 2.9 at 460nm for 65 kGy and 3.5 at 475nm for 100 kGy samples (please see figure 4).

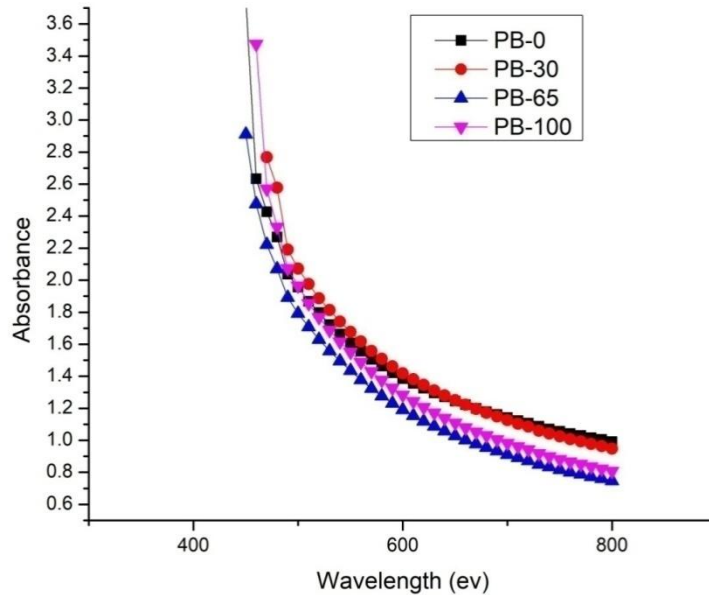


Figure 4: UV-VIS spectra of pure and gamma irradiated PE/GABA composites with 30% GABA by wt. (i.e, PB)

For calculation of the Urbach energy (E_u)

In order to evaluate the Urbach energy E_u for 0, 30, 65 and 100k Gy irradiated samples the natural logarithm of α has been plotted against photon energy ($h\nu$). In this plot the reciprocal/inverse of the slopes of the straight portions which associate with the lesser photon energy region is utilized to find the value of the optical activation energy (please see figure 5).

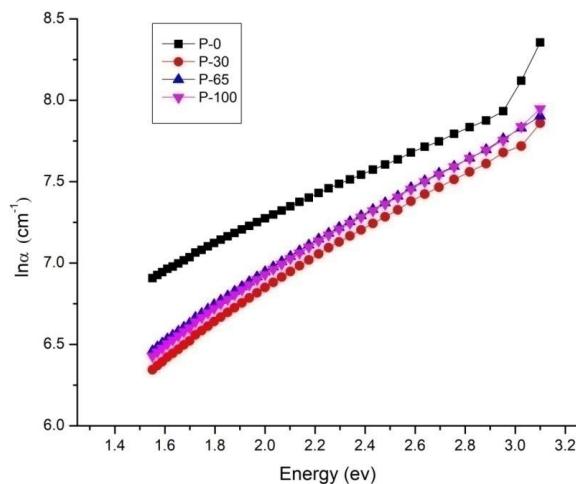


Figure 5: The natural logarithm of α plots as a function of photon energy ($h\nu$) for pure and gamma irradiated UHMWPE for Urbach energy (E_u) calculation

The determined values of E_u for pristine/pure and gamma ray irradiated samples of (PE-UHMW) indicates their behavior with the increasing dose of gamma ray. The E_u values were found to decrease for samples irradiated with 30kGy and 100kGy dose of gamma ray. But it increases for 65 kGy sample in comparison to 30 as well as 100 kGy samples though this increase is lower in relation to the pristine sample. It can be seen that the values of E_u for 30 kGy , 65 and 100 kGy irradiated samples are at lower side in comparison to pristine one one (see table-2).

Table 2: Urbach energy, energy band gap (direct and indirect) and carbon atoms in a cluster for pure and gamma irradiated polyethylene (i.e, P)

Dose Radiation (kGy)	Urbach's Energy (meV)	Band energy (eV)		'C' atom's numbers (N) in sample's cluster	
		Direct	Indirect	Direct	Indirect
P-0	133.17	2.88	2.56	~ 6	~ 7
P-30	98.56	2.76	2.38	~ 7	~ 9
P-65	101.83	2.48	2.05	~ 7	~ 8
P-100	99.17	2.69	2.22	~ 7	~ 7

Urbach energy (E_u) for PE/GABA composites with 15% GABA by wt. (i.e, PA) & composites with 30% GABA by wt. (i.e,PB) :

The determined values of E_u for pristine as well as gamma ray irradiated samples of (PE-UHMW) indicates their behavior with the increasing dose of gamma ray. The E_u values were found to decrease for 30, 65 and 100 kGy samples respectively. The values of E_u for PA & PB samples were found higher in number in comparison to pristine samples whereas the values of E_u for PA samples were found higher in relation to the PB samples (see figure 6 and table-3).

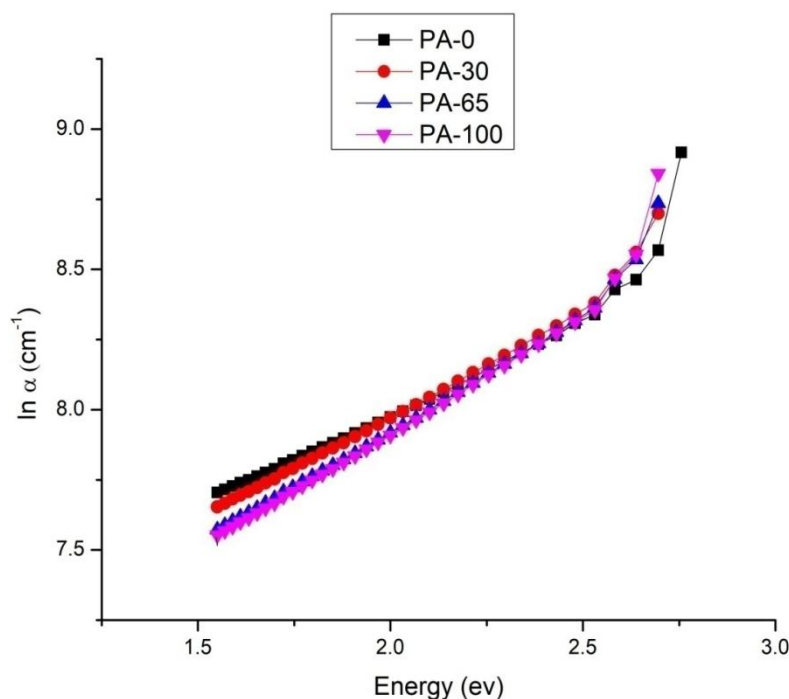


Figure 6: The natural logarithm of α plots as a function of photon energy ($h\nu$) for pure and gamma irradiated PE/GABA composites with 15% GABA by wt. (i.e, PA) for the Urbach energy (E_u) calculation

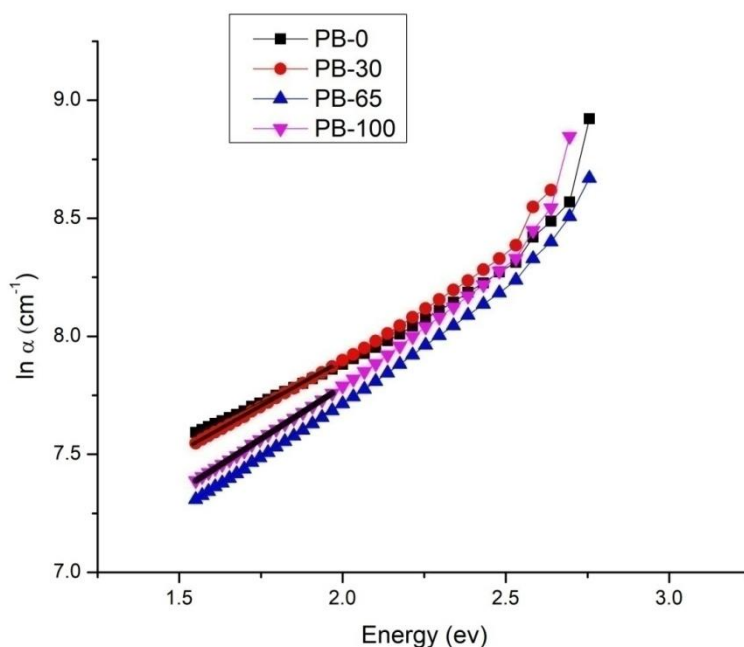
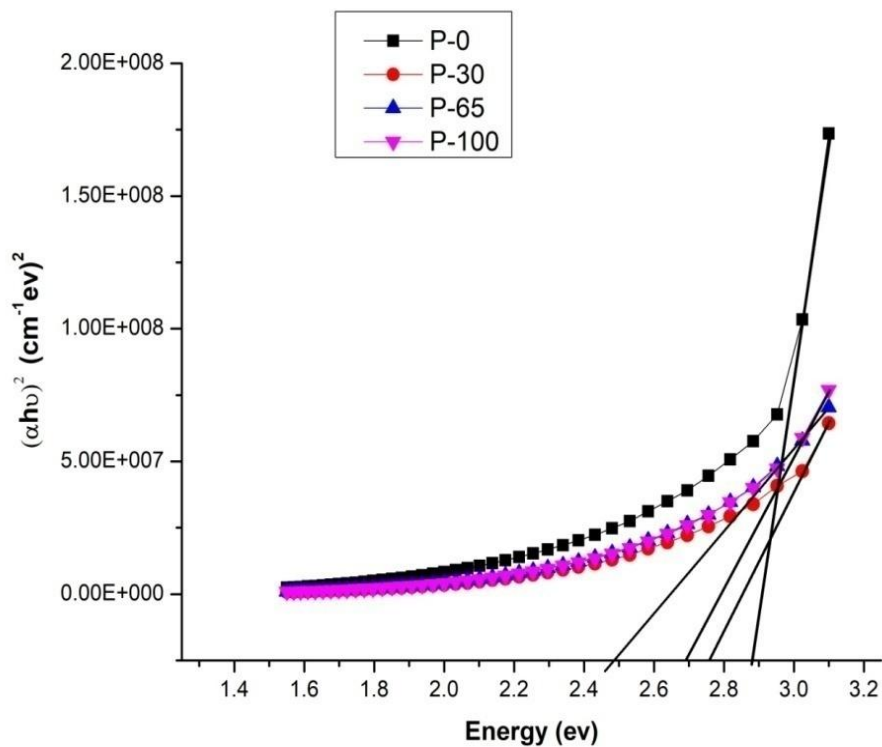


Figure 7: The natural logarithm of α plots as a function of photon energy ($h\nu$) for pure and gamma irradiated PE/GABA composites with 30% GABA by wt. (i.e, PB) for the Urbach energy (E_u) calculation

For the determination of direct energy band gap (DEBG) and indirect energy band gap (IDEBG)

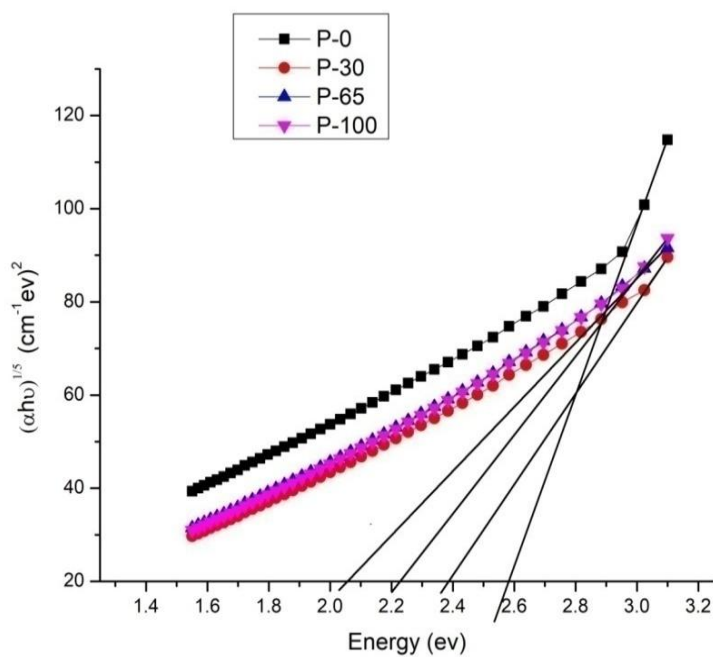
The values of $(\alpha h\nu)^2$ and $(\alpha h\nu)^{1/2}$ are plotted against photon energy ($h\nu$) for the calculation DEBG & IDEBG respectively. Different procedures can be employed for the calculation of band energies (direct/indirect) one of them is utilized in this work. Moreover, the C atom's numbers (N) in the sample's cluster of C=C unsaturation are also associated with the band gap energies.

The values of band energies (direct/indirect) from the graph have been acquired by intercepting the linear segment of the absorption edge at x-axis, representing photon energy axis (shown in figure). The impact of irradiation upon energy band may be described on account of C atom's numbers (N) in each sample's C=C structure. The no. of carbon atoms in the each sample's C=C structure have been calculated by utilizing the generalized form of Tauc's relation (Abdul-Kader 2009). It is pretty much clear that the relation between energy band gaps and "N" in sample's C=C structure is indirect i.e. greater is the "N" in sample's C=C structure lesser is the band energies and vice versa. It has also been published with specific facts previously by (Costa et al., 2008; Mehmood et al., 2014, 2013; Oral et al., 2008) that high energy irradiation of polyethylene results in increased C=C unsaturation inside its cluster (Costa, Carpentieri et al. 2008, Oral, Beckos et al. 2008)



a)

Figure 8 (a): Plots for determining direct energy band gap (DEBG) in (eV) for pure and gamma irradiated UHMWPE

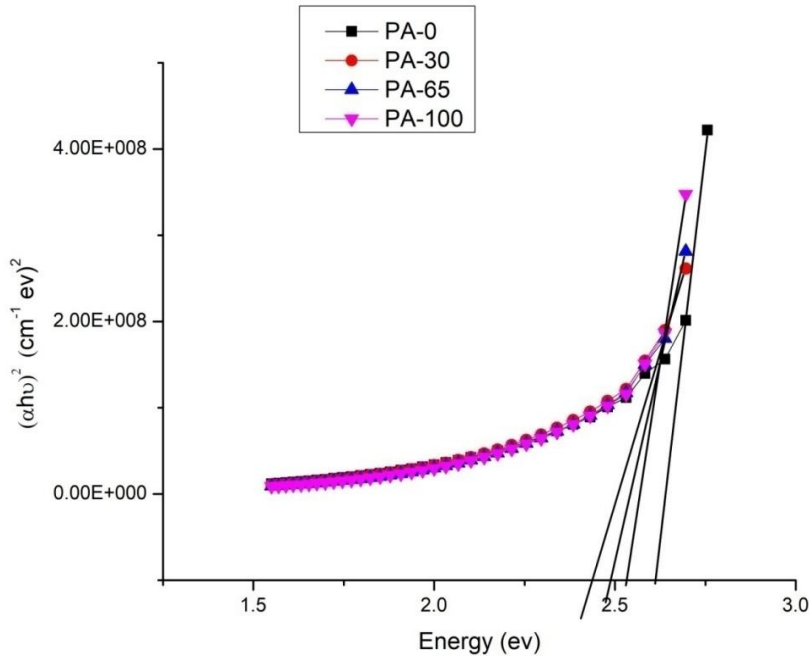


b)

Figure 8 (b): Plots for determining indirect energy band gap (IDEBG) in (eV) for pure and gamma irradiated UHMWPE

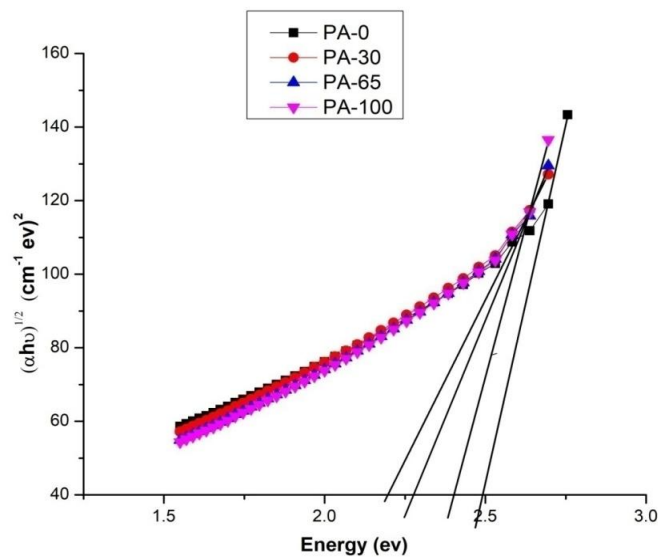
Direct energy band gap (DEBG) and indirect energy band gap (IDEBG) for PE/GABA composites with 15% GABA by wt. (i.e, PA) & composites with 30% GABA by wt. (i.e, PB) :

The DEBG & IDEBG and C atom's numbers (N) in C=C structure have generally shown the similar trend for PA & PB samples. As the band energies seem to be in indirect relation with broadening of absorption boundary i.e. more broader the absorption boundary is, more lesser the band energy is either the band energy is direct or indirect. Additionally in general the relation between band energies and "N" in sample's C=C structure is indirect i.e. greater is the "N" in sample's C=C structure lesser is the band energies and vice versa (indicated in the table below for PA and PB).



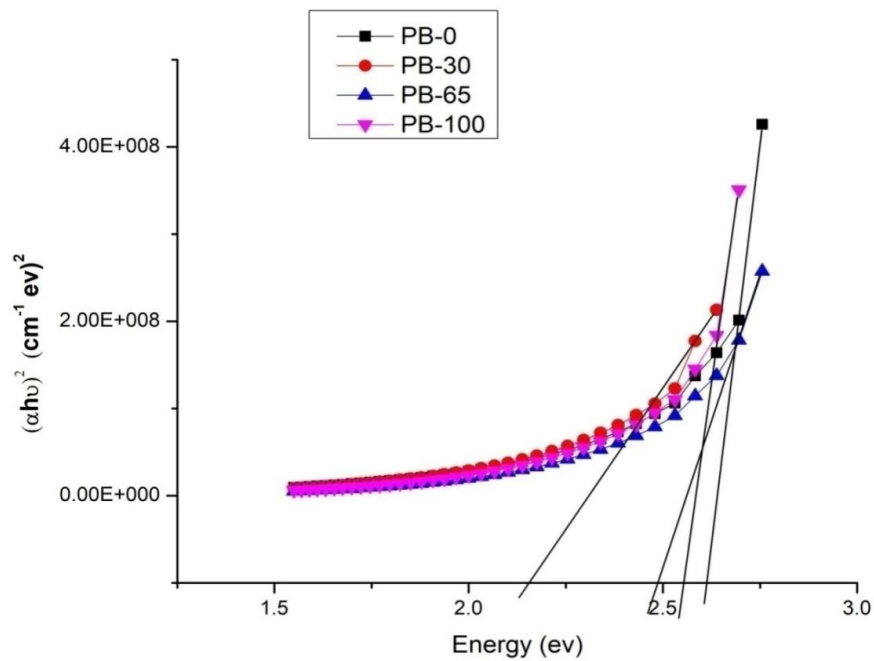
a)

Figure 9 (a): Plots for determining direct energy band gap (DEBG) in (eV) for PE/GABA composites with 15% GABA by wt. (i.e, PA)



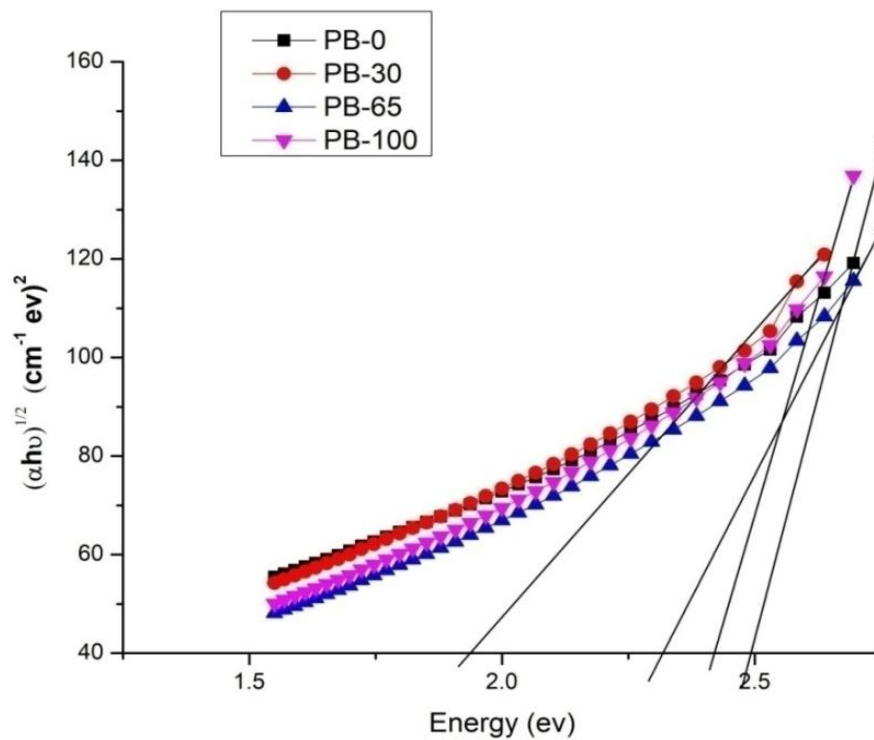
b)

Figure 9 (b): Plots for determining indirect energy band gap (IDEBG) in (eV) for PE/GABA composites with 15% GABA by wt. (i.e, PA)



a)

Figure 10 (a): Plots for determining direct energy band gap (DEBG) in (eV) for PE/GABA composites with 30% GABA by wt. (i.e, PB)



b)

Figure 10 (b): Plots for determining indirect energy band gap (IDEBG) in (eV) for PE/GABA composites with 30% GABA by wt. (i.e, PB)

Table 3: Urbach energy, energy band gap (direct and indirect) and carbon atoms in a cluster for pure and gamma irradiated PE/GABA composites with 15% GABA by wt. (i.e, PA)

Dose of Radiation (kGy)	Urbach's Energy (meV)	Band energies (eV)		'C' atom's numbers (N) in sample's cluster	
		Direct	Indirect	Direct	Indirect
PA-0	156.79	2.61	2.49	~ 7	~ 7
PA-30	136.54	2.43	2.19	~ 7	~ 8
PA-65	125.42	2.48	2.27	~ 7	~ 8
PA-100	122.45	2.53	2.40	~ 7	~ 7

Table 4: Urbach energy, energy band gap (direct and indirect) and carbon atoms in a cluster for pure and gamma irradiated PE/GABA composites with 30% GABA by wt. (i.e, PB)

Dose of Radiation (kGy)	Urbach's Energy (meV)	Band energy (eV)		'C' atom's numbers (N) in sample's cluster	
		Direct	Indirect	Direct	Indirect
PB-0	140.42	2.61	2.49	~ 7	~ 7
PB-30	120.82	2.15	1.94	~ 8	~ 9
PB-65	106.64	2.48	2.32	~ 7	~ 8
PB-100	106.08	2.55	2.42	~ 7	~ 7

IV. Conclusion

In this work, the band gap properties of pure (PE-UHMW) and its composites with GABA (Gamma-aminobutyric acid) have been examined by utilizing very sensitive Muller matrix spectro-polarimeter in order to assess the band gap properties of UHMWPE in the presence and absence of stabilizer i.e. GABA. The study is carried out for samples treated with three particular doses of gamma rays; (i.e. 30, 65 and 100kGy) specifically. A decrease in absorption with increasing dose of gamma towards the longer wavelength has been observed due to physical changes induced by radiations. In order to calculation of Urbach energy, band energies along with the "C" atoms numbers (N) in the sample's C=C structure generalized Urbach's relation and Tauc's relation are utilized. It has been observed that generally the Urbach energy and band energy followed the decreasing tendency for 30 as well as 100kGy irradiated samples whereas it increases for 65kGy treated samples. In case of GABA composites i.e. PA and PB samples, Urbach energy have shown decreasing tendency and band energies have shown increasing tendency for 30,65 and 100kGy irradiated samples consecutively but the values of direct as well as indirect band energies were lower in comparison to the pure samples.

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