

Influence of Gamma Radiation on Optical and Morphology Properties for PS and MO/PS Composites

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ABSTRACT

In this work, the effect of gamma irradiation on optical characteristics of virgin polystyrene (PS) and its composite films was examined. The neat PS and its composites casted to be thin-films both before and after being doped with methyl orange (MO) at weight ratios of 1.0 % wt. /wt. Thickness of the obtained samples varied within the range of 0.2 ± 0.05 mm. The prepared samples were exposed to 1.0, 5.0, and 10 kGy of ⁶⁰Co (a gamma source) radiation. The optical properties were investigated using ultraviolet-visible spectra within a wavelength range of (280-1100 nm). The samples' absorption spectra were displayed both before and after irradiation. For PS and MO/PS nanocomposites, gamma radiation caused systematic changes in absorption with absorbed doses. The optical bandgap for PS decreased after adding methyl orange dye, and when the gamma ray dose increased from 1.0 to 10 kGy, the direct bandgap decreased from 4.24 to 3.90 eV for PS, and decreased from 4.13 to 3.4 eV for MO/PS. The abovementioned outcome demonstrated by the redshift in the transmittance spectra using Fourier Transform Infrared Spectroscopy (FTIR). The effects of γ -rays on the absorbance, absorption coefficient (α), and extinction coefficient (k) into all the samples were studied, and the findings showed an improvement in parallel with increasing radiation dose. The optical microscope images showed surface damage represented grooves, holes, bubbles, and cracks caused by samples photo-degradation. It was concluded that gamma rays could alter different characteristics of materials in terms of making them suitable for radiation detection, sensing, and dosimetry.

Keywords: FTIR, Gamma ray, Methyl Orange, Polystyrene, UV-VIS

1. INTRODUCTION

According to the research work related to polymer science, new elastomers, plastics, adhesives, coatings, and fibers are continuously developed. The new information is gradually consolidated, and standardized with the aid of important new theories about the combination between the technological structures of polymers, their physical properties, and their practical behavior. Thus, the concepts of kinetics, thermodynamics, and polymer chain structure are linked to each other in order to make polymer science stronger. [1, 2]. Ionizing radiation is a green way to create new materials with superior qualities. However, this could change the polymers features because the radiation can break bonds, and create free radicals that might be attached with the reactants to form new entities, giving conventional polymers exceptional properties [3, 4].

The materials and radiation dose have an impact on these properties. Radiation has a significant impact on polymeric materials because it can alter and enhance the physical characteristics of polymers. For instance, when polymeric materials expose to accelerated electrons, X-rays, ion beams, or gamma rays, reactive intermediates are created. These intermediates can subsequently trigger significant reactions

like degradation or cross-linking. The specified radiation dose has regulated these interactions [5, 6]. Different polymers respond to radiation doses in different ways, and these responses are intrinsically related to the polymers' chemical structures. Consequently, polymers' optical, electrical, mechanical, and chemical properties are changed [7, 8]. The radiation is limited to ⁶⁰Co and ¹³⁷Cs out of thousands of gamma emitters. In addition, gamma rays are electro-magnetic quantum waves with a wavelength that has a shorter and higher photon energy than light [9, 10]. Since gamma rays have become more significant in recent technological advancements, numerous researchers were examined their impact on various polymers comparable to what was previous studies have shown [11-15]. For photonic devices, polymers, particularly poly methyl methacrylate (PMMA) and PS are grown in a popular manner due to their low cost, high flexibility, ease of processing, and customizability [16, 17]. It is known that PS is a substantial component for both industry and academia. PS is a simple chemical compound with a saturated aliphatic chain, and an unsaturated aromatic ring [18-20]. PS is the most preferred polymer for numerous industrial applications due to its exceptional mechanical durability, electrical insulation, low density, strong thermal resistance, ease of molding and processing, and optical transparency

[21-23]. For healthcare sector, PS can play a pivotal role in manufacturing different types of lab apparatus such as Petri dishes and tissue culture, medicinal constituents, trays, containers, medicinal packages, constituents of heart pump, catheters, and epidurals [24-26]. On the other hand, methyl orange is an organic molecule with the molecular formula of ($C_{14} H_{14} N_3 Na O_3 S$), Methyl Orange is known by its International Nonproprietary Name (INN) as sodium 4-[(4-dimethylaminophenyl) diazenyl] benzenesulfonate. This orange powdery material is a poisonous dye that can be dissolved in water. It can be employed for calibrating a wide range of chemicals [27-29].

The aim of this work is to confirm the impact of gamma irradiation on the optical properties of neat PS and its composite films, which were doped with MO dye. The targeted application related to this work is manufacturing efficient dosimeters. Preparing samples of polymeric systems (pure and composite materials) by dissolution method, then studying of structural, optical properties on them before and after irradiation with γ -ray irradiation at

high radiation doses (1,5&10kGy) and studying the change in composition and properties of manufactured specimens.

2. MATERIALS AND INSTRUMENTATION

2.1 Sample Preparation

For the polymer film, Dichloromethane (CH_2Cl_2) was used to dissolve PS at varying quantities (7–12 wt. /vol), and vigorously stirred using a magnetic stirrer for an hour to create a homogenous solution. The highest concentration needed to produce a film was 10 % wt. /vol. The efficient casting required both bubble-free and easily alighted process from the petri dish. The sample thickness was 0.2 ± 0.05 mm. The SCRC Company supplied methyl orange ($C_{14}H_{14}N_3NaO_3S$), which was added as a dopant to the casted film. To create the MO/PS composites, methyl orange was firstly combined with dichloromethane at a concentration of (1.0 % wt. /wt.), and then dissolved in PS. Figure 1 shows the experimental procedure of casting for preparing the neat polymer and its composites.

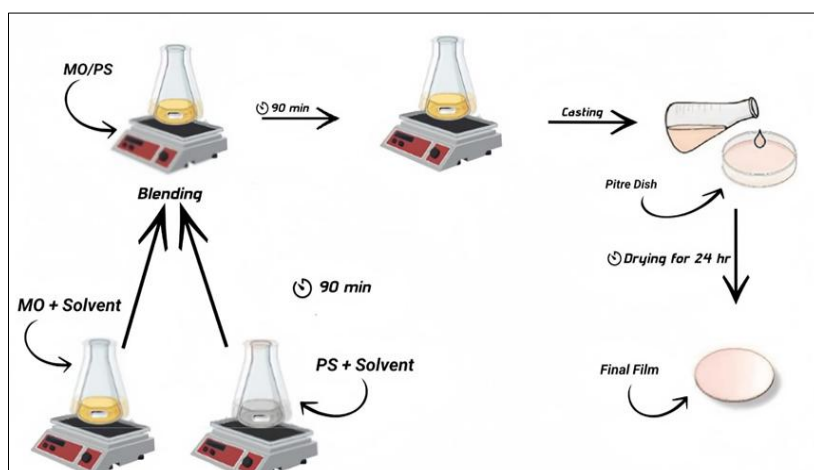


Figure 1. Casting method for preparing the neat polymer and its composites.

2.2 Instrumentation

To evaluate a precise particle size for Methyl orange, a particle size analyzer (90-plus) was utilized. Figure (2) illustrates the average particle size. The obtained effective diameter for the used nanoparticles was around 12094.1nm (about 12.0941 μm).

The samples were subjected to 14 Gy of gamma-irradiation per hour, with a half-life of 5.3 years, the commonly used Co^{60} Gamma-ray source emits mono-energetic 1.17 and 1.33 MeV γ -rays. Sealed radioactive source Co-60 made in India. And then used Ultraviolet UV/Visible device. Were carried

out using "Shimadzu -1900S" (Japan) in the range of (200-1200nm) to obtain the absorbance value as a function with wavelength. After that, used FTIR Spectroscopy were also conducted using "Shimadzu -1800S" (Japan) within a range of ($4000 - 400\text{ cm}^{-1}$) to determine the molecular structures of both PS as well as the irradiated and non-irradiated MO/PS composites.

The morphology for the prepared samples was analyzed using an Optical Microscope Analysis type MEIJI TECHNO CO.LTD (Japan). Each sample was examined using a magnification power of 100X.

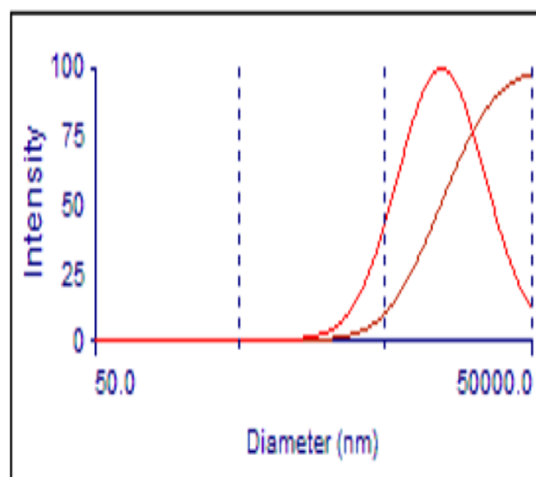


Figure 2. The lognormal size distribution of Methyl Orange.

3. RESULTS AND DISCUSSION

3.1 Effect of Adding MO with PS on Absorption Spectra

The maximum absorbance for MO/PS films was observed at 280 nm, then it rapidly decreased at wavelengths higher than 300 nm, according to Figure (3), which shows the UV-

Vis spectra of PS, MO/PS films, and corresponds to unirradiated samples. It is noticed that there was a shift towards longer wavelengths, which can be attributed to the occurrence of localized states within the energy gap. As a result, an increase in absorbance for MO/PS films was achieved. This outcome is in line to what was previously disseminated in [30-32].

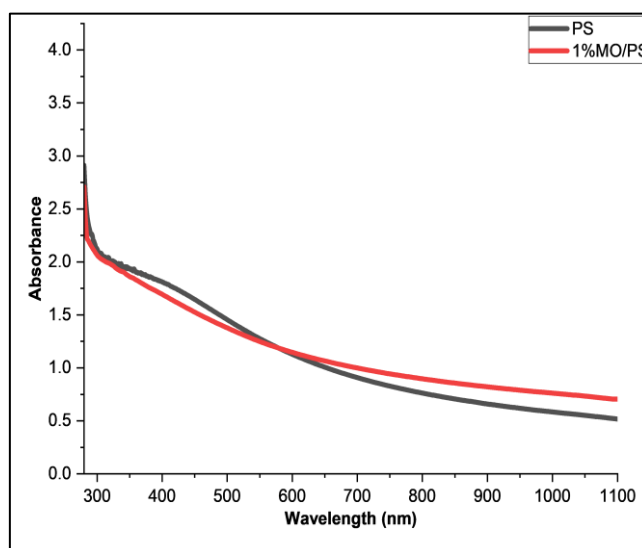


Figure 3. Comparison of absorption spectra for neat PS and MO/PS films before gamma irradiation.

3.2 Absorption Spectra of PS & MO/PS Before Gamma Irradiation

Figures (3&4) showed the optical absorption spectra of irradiated films for PS and MO/PS before and after irradiation. The absorption spectra for all films were obtained within a range of 280–1100 nm at three doses (1.0 kGy, 5.0 kGy and 10 kGy). The $\pi \rightarrow \pi^*$ electronic transition was responsible for the maximum absorbance peak that was located at 280 nm for all the films. Irradiation's primary effects include chain scission, cross-linking, as well as the creation of unsaturated products in the polymer chain. Irradiation causes the polymer to produce free radicals, which can then react with atmospheric oxygen to form

hydroxyl and carbonyl groups. The presence of carbonyl and hydroxyl groups in PS, and the creation of unsaturated groups was probably the main reason associated with the increment of absorbance upon gamma irradiation [15, 33, and 34]. The absorption decreased monotonically into the visible range after reaching its maximum value at 280 nm. Carbonium ion formation, which may be resulted from the interaction of ray-induced free radicals with conjugated structure, was responsible for the long tail in the visible region [15, 35]. It was evident that the absorption was moving towards the longer wavelengths, and gradually increased with absorbed doses.

This behavior can be attributed to the chemical reaction generated by ionizing radiation in polymers, which affected both the molecular structure and macroscopic characteristics. As a result, degradation products represented by electrons and holes were created in parallel with samples' exposure to radiation, which consequently

altered the optical absorbance [25]. The systematic promotion of the optical features with the absorbed doses of radiation inspires the optimism about the possibility of utilizing the prepared samples in sophisticated applications represented by dosimeters.

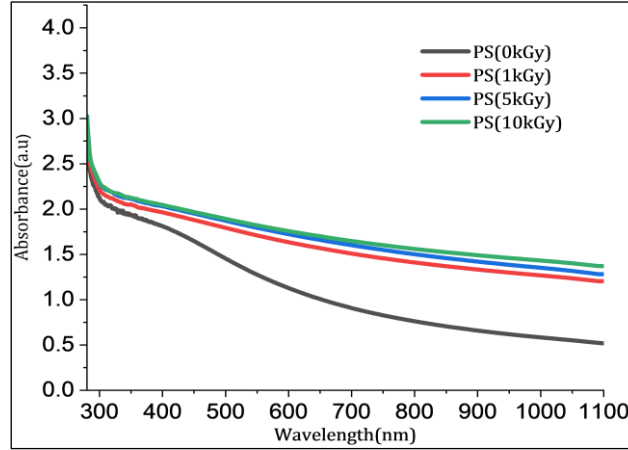


Figure 4. The absorption spectra for neat PS before and after exposure of gamma ray.

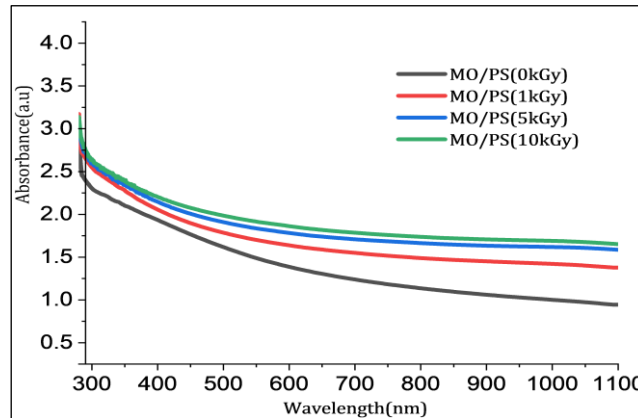


Figure 5. The absorption spectra for MO/ PS before and after exposure of gamma ray

3.3 Energy Gap of PS & MO/PS Before Gamma Irradiation

Band gaps, both direct and indirect Tauc's model is utilized to estimate E_g as follows: [36-39]

$$[(\alpha h\nu)]^m = A (h\nu - E_g) \quad (1)$$

The direct transition band gap was described at $m = 2.0$, and the indirect allowed transition was assumed to occur at $m = 1/2$, where A is an independent constant related to photon energy ($h\nu$). where is, h is Planck's constant is the frequency of light. The symbol α is the absorption coefficient and E_g is the optical band gap energy. A photon cannot be released in an indirect gap since the electron needs to transfer momentum to the crystal lattice, and go through an intermediate state. Figures 6 and 7 showed the data of $(\alpha h\nu)^{1/2}$ with the variation of the photon energy ($h\nu$) for MO/PS films and pure PS films. The linear portions were fitted to the x-axis at $(\alpha h\nu)^{1/2} = 0$ to obtain the band gaps.

The direct band gap transition and the evolution of the polymeric films' band gaps were nearly identical. It was noticed that the energy gap for PS was (4.25 eV) before irradiation, and then decreased to be (4.13 eV) after adding methyl orange dye. This outcome was due to the increase in local potentials within the energy gap, which is agree with previously reported studies [31, 40, and 41]. All band gaps energies decreased with further doses of gamma rays. The energy gap was decreased linearly with the exposure to the irradiation dose, which was explained by an increase in interface traps, and localized states with the irradiation dose. [33,42].

An increase in the structural disorder of the irradiated dyed PS may played a special role in the decrement of the band gap energy in parallel with increasing the radiation dose. The systematic decrement in energy gap for all samples with absorbed dose support the research orientation of using the prepared samples as a dosimeter within this range as regarded in previous sections. These samples contributed to

achieve the structural defects, which has a role in increasing the width of the localized states, and consequently reducing the value of the optical gap [1,37,43, and 44].

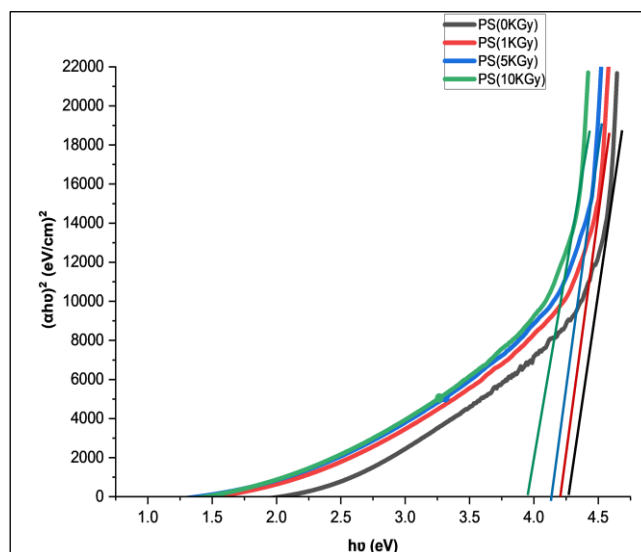


Figure 6. $(\alpha h\nu)^2$ against $(h\nu)$ for PS before and after gamma radiation at (1,5 & 10 KGy) doses.

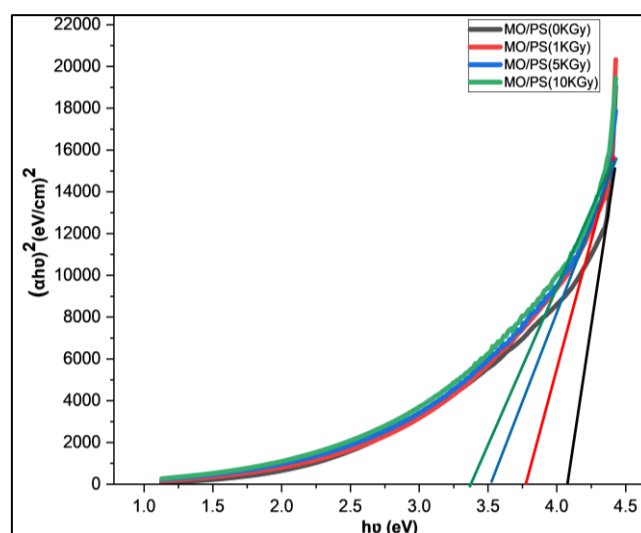


Figure 7. $(\alpha h\nu)^2$ against $(h\nu)$ for MO/PS before and after gamma radiation at (1,5 & 10 KGy) doses.

3.4 Absorption Coefficient of PS & MO/PS Before Gamma Irradiation

By using Lambert's law as a guide, the absorption coefficient (α) of the absorption edge was found [45-48]:

$$\alpha = \frac{2.303 \times A}{t} \quad (2)$$

Where t is the sample thickness, and A is the measured absorbance. Figures (8&9) shows the absorption coefficient of the PS and the other MO/PS composites before and after radiation with a dose of γ -rays, which was varied between (1.0 - 10 kGy). The figures showed that the absorption coefficient increased with the irradiation dose. This was ascribed to changes in absorption brought by photo-degradation, which was brought by radiation's increase in localized state [33, 49].

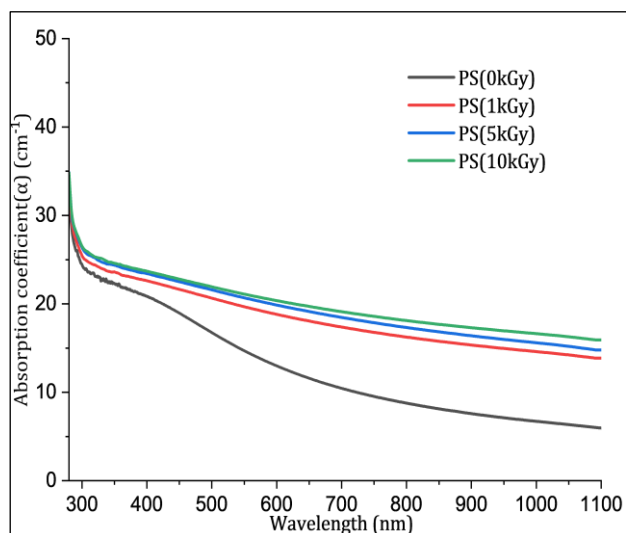


Figure 8. The relationship between PS's wavelength and absorption coefficient before and after three doses of gamma radiation.

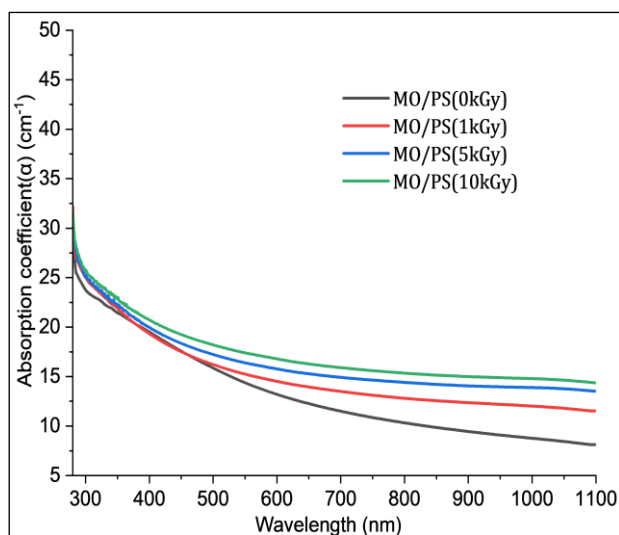


Figure 9. The relationship between MO/PS's wavelength and absorption coefficient before and after three doses of gamma radiation.

3.5 Extinction Coefficient of PS & MO/PS Before Gamma Irradiation

The extinction coefficient (K) could be obtained from the following equation [50-52]:

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

λ is the wavelength.

Figures (10 & 11) give an interpretation about the coefficient (K) behavior, which was almost identical to that one of the corresponding absorption coefficients (α) shown in figures (8 & 9). This outcome is in a good agreement with what was previously reported [1,25]. For the abovementioned reasons, the increment in absorption coefficient urged the extinction coefficient to rise with the irradiation dose; its peaks shifted toward long wavelengths, which was ascribed to a narrowing of the energy gap with the irradiation dose.

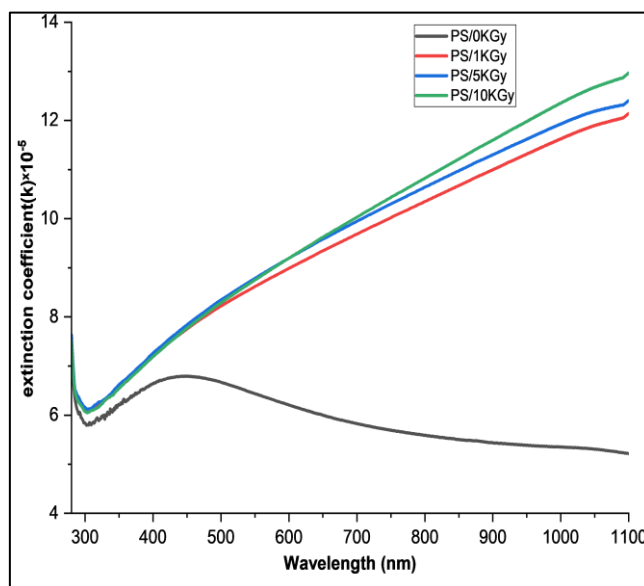


Figure 10. Relation between the extinction coefficient and wavelength of PS before and after gamma radiation for three doses.

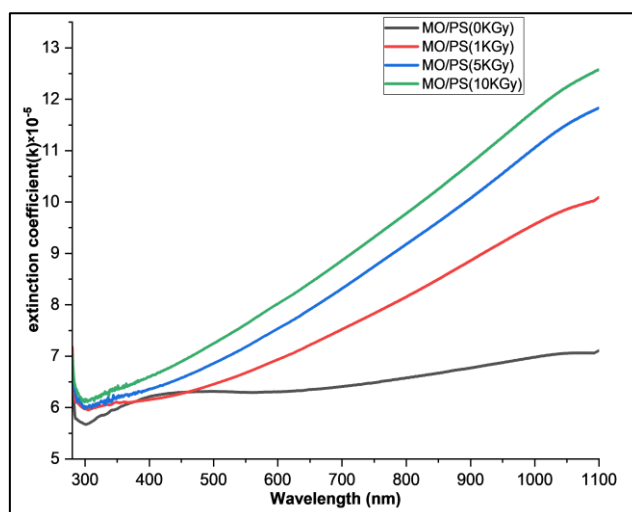


Figure 11. Relation between the extinction coefficient and wavelength of MO/PS before and after gamma radiation for three doses.

3.6 FTIR Spectra of PS and MO/PS Samples Before and After Gamma Irradiation

With gamma irradiation, FTIR was used to determine the molecular structures of both PS as well as the irradiated and non-irradiated MO/PS composites. The spectrum of the virgin PS and MO/PS films before irradiation is shown in Figure 12 and table 1. It was noted that three bands appeared at the range (2800-3060 cm^{-1}), which was ascribed to the C-H stretching of the aromatic and aliphatic groups; respectively. As shown in previous studies [53,54], the peak located at 3445.51 cm^{-1} was related to O-H stretching, and the peaks located at (1315.45 and 1338.60 cm^{-1}) was attributed to the symmetrical and asymmetrical stretching vibration of CH_2 bands. The peak located at 1598.99 cm^{-1} was related to stretching vibration of the C=C bond on the benzene ring, whereas bands at 908.47 and 941.26 cm^{-1} are attributed to C-O bond. Five bands varied from (630 – 908 cm^{-1}) are linked to the C-H out-of-plane bending vibration of the benzene ring [55, 56]. On the other hand, the spectra of (MO/PS) films exhibited similar behavior compared to pure

PS. The band appeared at 1600 cm^{-1} confirmed the presence of wagging C=C and the small shift occurred at 3564.64 cm^{-1} assigned to the stretching of O-H groups on films surfaces. It was noticed that the band of C-H beyond the plane bending was also related to the five bands. There was a shifting in almost bands, which was connected to the benzene ring hydrogen's C-H deformation vibration band. The C-O stretch appeared at (906.54 and 943.19 cm^{-1}) were vanished at C-H aliphatic, and at C-H aromatic; respectively. There was a physical interaction between the PS and MO ascribed to the variation into the band location (shifting) of the PS aromatic ring, which is in line with the literature [57, 58].

The powder of MO's spectra, which was exhibited at a range of 600–4000 cm^{-1} , included the typical bands of the azo band ($-\text{N}=\text{N}-$) at (1317–600 cm^{-1}), and the benzene ring, which was obvious at (1400–1602 cm^{-1}). The symmetric and antisymmetric stretching vibration at ($-\text{CH}_2$) appeared at (2820–2903 cm^{-1}). In addition, the asymmetrical stretching vibration of ($-\text{SO}_3\text{Na}$) set was attributed to the typical band at 1004 cm^{-1} . This outcome is in a good

agreement with the literature [45, 59]. After all films were exposed to gamma radiation at doses of 1, 5, and 10 kGy, FTIR spectra were obtained for each sample. It is noticed that FTIR spectra lightly change especially after irradiation with doses (5.0 and 10 kGy) for all (pure PS and MO/PS) as shown in table (1) as well as Figures (13 and 14).

Compared to non-irradiated PS, irradiated PS bands were influenced by radiation. They became broader and less intense, indicating structural changes brought by gamma irradiation. This outcome is in line with a previously reported study [14, 60]. It is emphasized that intensity of the band at 1591.27 cm^{-1} that corresponds to C=C stretching decreased as a result of gamma radiation, and disappeared at 630.72 cm^{-1} at doses of (5.0 and 10 kGy) for C-H out of phase bending. Moreover, bands at 964.41 cm^{-1} of C-O stretching were appeared at the dose of 1.0 kGy, and disappeared again at the doses of (5.0 and 10 kGy). With high dose of 10kGy, there was a disappearance of band 2835.36 cm^{-1} from the aromatic bond, and shifting towards

longer wavelengths from 3445.51 to 3566.38 cm^{-1} O-H hydroxyl stretching.

However, in MO/PS, the spectrum changed clearly, and it was evident that MO dye made an impact on it. The C-H band denoted the ring deformation vibration in the region of $(625-910)\text{ cm}^{-1}$. MO dye, which refers to (C-H) bending vibration and gamma effect, caused the reappearance of the peak of $(667.37)\text{ cm}^{-1}$ at 10 kGy after its disappearance at doses of 1.0 and 5.0 kGy due to the (C-H) deformation vibration band of benzene ring hydrogens. Furthermore, the benzene ring was specifically referenced by the peak at $(1598.99)\text{ cm}^{-1}$ for (C=C) stretching at dose of 10 kGy, which was related to MO dye, and gamma effect as well [61, 62]. At a range of $(1300-1380\text{ cm}^{-1})$ due to CH₂ bending, there was shifting at band (1336.67 cm^{-1}) . A new band appeared at doses of (1.0 and 5.0 kGy), and disappeared at the dose of 10 kGy. In addition, a new band appeared at 1327.03 cm^{-1} . At all doses, new bands appeared for C-H stretching aliphatic and aromatic [63, 64].

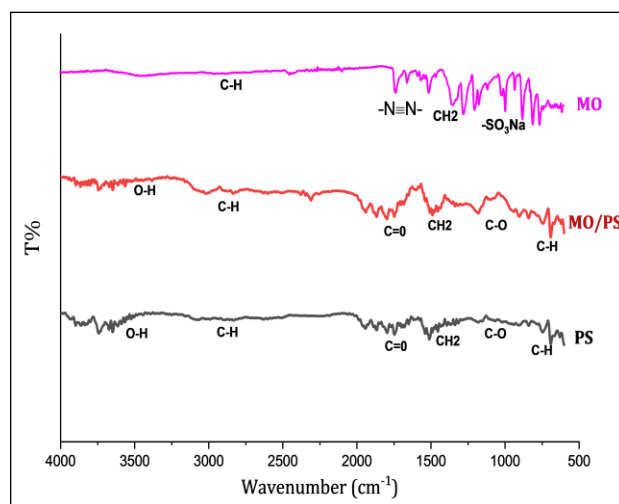


Figure12. FTIR Spectra of PS before and after doping with methylene orange(MO.)

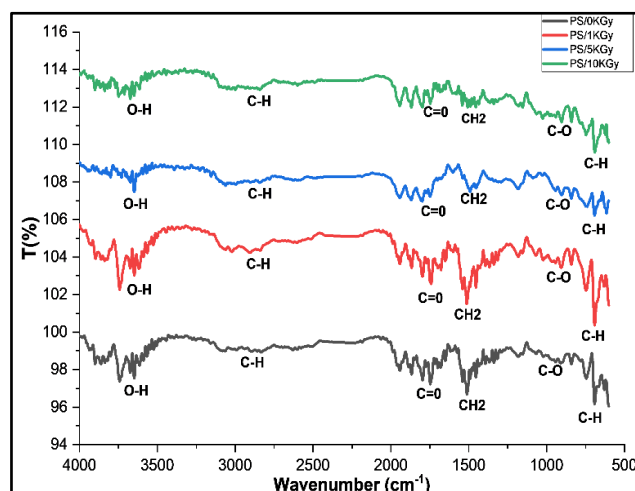


Figure (13): FTIR Spectra of PS before and after gamma ray exposure at three doses.

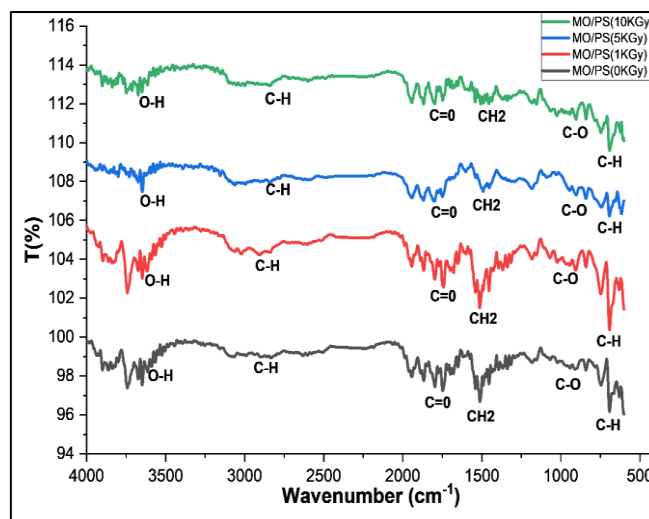


Figure (14): FTIR Spectra of MO/PS before and after gamma ray exposure at three doses.

3.7 Morphology

PS and MO/PS membranes must undergo morphological analysis in order to assess the material's surface features and structural properties before and after exposure γ -ray. Figure 15 showed the optical micrographs of pure PS films captured at doses of 1.0, 5.0, and 10 kGy before and after gamma irradiation. As shown in (a), PS was free of defects and bubbles prior to irradiation, indicating that no evidence of porous structure in the sample. The most notable effect of 1.0 kGy radiation was the release of gas. For the micrographs of (b-c), bubbles may form, and as dosage increased, more holes and grooves may become visible. Similar observations were previously reported [63, 64]. As observed in dose (10 kGy), PS photodegraded in the presence of oxygen, leading to deterioration and crack formation. When degradation occurred, the bubbles were burst as shown in micrograph (d), which is in line with the literature [65, 66]. While singlet oxygen undergoes a number of distinct reactions, the most

common one in the photo-oxidation of polymers was the creation of a hydro-peroxide through the oxidation of an olefin that contained an allylic hydrogen. This can then further break down, resulted in chain scission, and led to the formation of a carbonyl group terminal [65, 67].

Figure (16) shows that MO/PS was homogenous, and it had a bubble-free morphology with a few clusters of methyl orange as shown in micrograph (a), but no indication of a porous structure in the sample. The most notable result of high doses of radiation as shown in micrographs of (b-c) was the release of gas. As radiation dosages increased, more bubbles, rough surfaces, and lines (holes or grooves) were revealed. Similar morphological findings were reported [63, 68]. The majority of the bubbles burst at high dose irradiation (10 kGy) as shown in micrograph (d), whereas oxygen caused structural deterioration and crack formation. These cracks were increased, and clustering decreased with absorbed doses [65, 69].

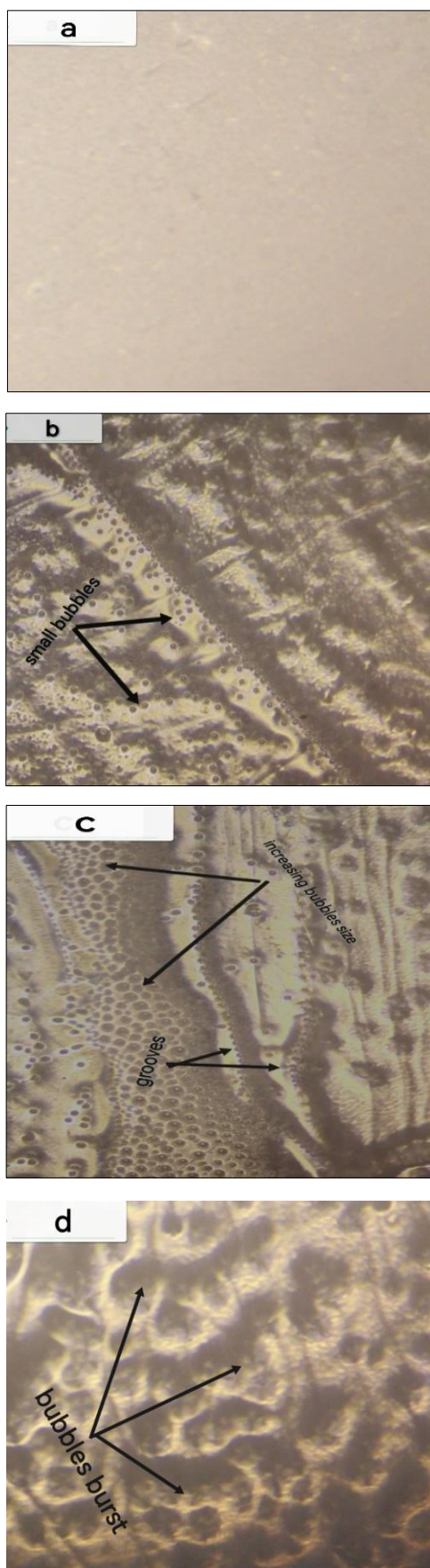


Figure 15. Optical micrographs of (a) pure PS film before gamma irradiation, and after irradiation with 1kGy (b), 5kGy (c), 10kGy(d).

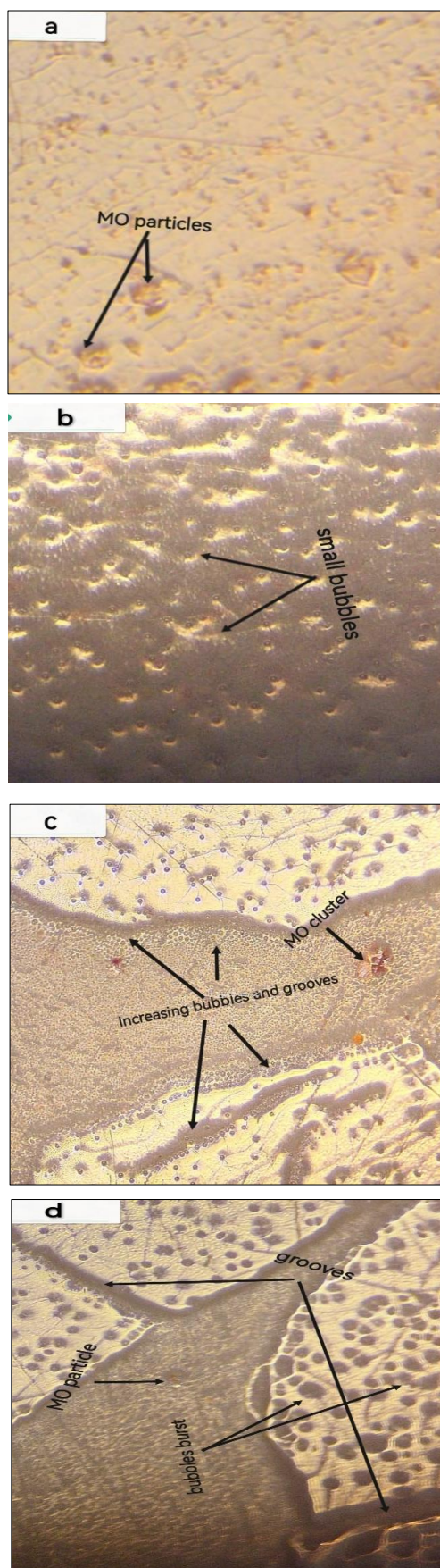


Figure 16. Optical micrographs of (a)MO/PS film before gamma irradiation, and after irradiation with 1kGy (b), 5kGy (c), 10kGy(d).

Table 1 FTIR-characteristic of PS and MO/PS before and after gamma ray exposure to three doses

Polymer System	(C-H) out of phase bending 625-910 cm ⁻¹	(C-O) Stretching 880-1010 cm ⁻¹	(CH ₂ -) Bending 1300-1380 cm ⁻¹	(CH ₂) Wagging C=C Stretching 1550-1610 cm ⁻¹	(C=O) Stretching 1550-1750 cm ⁻¹	(C-H) Stretching Elphatic 2800-3000 cm ⁻¹	(C-H) Stretching Aromatic 2800-3060 cm ⁻¹	(O-H) Hydroxyl Stretching 3340-3600 cm ⁻¹
PS (0kGy)	630.72 692.44 744.52 840.96 908.47	908.47 941.26	1315.45 1338.60	1598.99	1618.28 1651.07 1681.93 1747.51	2835.36 2889.37 2991.59	2835.36 2889.37 2991.59	3445.51
PS (1kGy)	632.65 690.52 746.45 840.96 906.54	906.54 943.19 964.41	1315.45 1338.60 1367.53	1598.99	1598.99 1651.07 1678.07 1741.72	2839.22 2904.80	3020.53 2839.22 2904.80 3020.53	3445.51
PS (5kGy)	692.44 740.67 840.96 902.69	902.69 943.19	1336.67	1600.92	1600.92 1678.07 1749.44	2833.43 2902.87 2993.52	2833.43 2902.87 2993.52	3560.86
PS (10 kGy)	690.52 746.45 840.96 902.69	902.69 941.26	1338.60	1591.27	1591.27 1651.07 1681.93 1747.51	2841.15 2999.31	2999.31 3101.54	3566.38
MO/PS (0kGy)	667.37 692.44 744.52 840.96 906.54	906.54 943.19	1315.45 1336.67	1600.92	1600.92 1651.07 1681.93 1747.51	2835.36	2835.36 3014.74	3564.43
MO/PS (1kGy)	690.52 742.59 840.96 906.54	906.54 943.19 966.34	1315.45 1338.60 1365.60	1600.92	1600.92 1678.07 1747.51	2858.51 2939.52	2858.51 2939.52 3072.60	3525.88
MO/PS (5kGy)	962.44 744.52 840.96 906.54	906.54 962.48	1315.45 1338.60 1367.53	1600.92	1600.92 1651.07 1681.93 1747.51	2858.51 2933.73	2858.51 2933.73	3523.95
MO/PS (10 kGy)	630.72 694.37 748.38 840.96 906.6	904.61 941.26	1327.03	1598.99	1598.99 1749.44	2843.07 2918.30	2843.07 2918.30 3022.45	3520.09

4. CONCLUSION

According to UV-vis spectroscopic studies of pure PS, and dyed unirradiated and irradiated films The highest absorbance appeared at 280 nm, and it rapidly decreased at wavelengths above 300 nm. Irradiation led to form free radicals in the polymer, and these radicals can react with oxygen in the air to compose carbonyl and hydroxyl groups. The absorption mechanism was identified as a direct allowed transition based on the optical absorption spectra, and the optical band gap. The decreased energy band gaps with the increased dose can be attributed to the increasing localized state, and interface traps that were induced by irradiation of the polymer film as the dose increased. The systematic decreasing in energy gap for both films with absorbed dose suggested the possibility of their

employment as efficient dosimeters within the range of (1.0, 5.0 and 10 kGy).

Both of absorption and extinction coefficient increased with increasing radiation dose for PS and MO/PS composites. The formation of a broad vibrational peak around 3445.51 cm⁻¹ for PS film, and around 3564.43 cm⁻¹ for MO/ PS film was emphasized by FTIR findings. This is because the -OH group absorbed moisture before and after irradiation by the γ-ray. The PS and MO interacted physically, which was explained by the PS aromatic ring's shifting or changing band location, and these interactions were increased after applying gamma radiation.

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