

Radiation Effects of Polycarbonate Films after Electron Beam Irradiation

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Abstract

Polycarbonate (PC) is a promising material for high-absorbed-dose detection. In this study, the effects of electron beam irradiation on the optical properties and structure of PC were investigated. Colourimeter, ultraviolet–visible absorption spectrum, X-ray diffraction, gel permeation chromatography, and thermal stability analysis were used to characterise the properties of PC after different doses (0–1000 kGy) of irradiation. The results showed that the colour of polycarbonate turned yellow after irradiation and that there was a good relationship between the chroma value and the absorbed dose. Furthermore, the visible light transmittance, crystalline region, crystal size, weight-average molecular weight, and thermal deformation resistance of PC decreased with the increase in the absorbed dose. The thermal stability of PC was improved by irradiation. This study provides an important basis for developing PC into radiochromic dosimeter.

Full Text

Radiation Effects of Polycarbonate Films After Electron Beam Irradiation

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Polycarbonate (PC) represents a promising material for high-absorbed-dose detection. This study investigates the effects of electron beam irradiation on the

optical properties and structure of PC. Colorimetry, ultraviolet–visible absorption spectroscopy, X-ray diffraction, gel permeation chromatography, and thermal stability analysis were employed to characterize PC properties after irradiation at various doses (0–1000 kGy). The results demonstrate that PC coloration shifts toward yellow after irradiation, with a strong correlation between chroma values and absorbed dose. Furthermore, visible light transmittance, crystalline region size, crystal dimensions, weight-average molecular weight, and thermal deformation resistance all decrease with increasing absorbed dose. Notably, irradiation enhances the thermal stability of PC. These findings provide a crucial foundation for developing PC-based radiochromic dosimeters.

Keywords: Polycarbonate, Electron beam irradiation, Radiochromic dosimeter, Radiation effects

Introduction

The radiation effects in polymers have been extensively investigated in recent years, with researchers seeking to enhance polymer material properties through ionizing radiation to broaden their application potential [1,2]. While chemical changes in polymers remain modest under moderate-dose irradiation, polymers typically consist of thousands or even tens of thousands of monomer units, meaning that even minor modifications can substantially alter their physical and chemical characteristics. Generally, polymer modification through ionizing radiation depends on both the polymer’s intrinsic structure and irradiation conditions, including radiation type, energy, absorbed dose, dose rate, atmospheric composition, humidity, and temperature.

Polycarbonate (PC) occupies an important position among polymers due to its low production cost, heat resistance, ease of processing, high impact toughness, high elastic modulus, creep resistance, and excellent optical transparency. These attributes have led to widespread applications in building materials, automotive manufacturing, medical instruments, aerospace, electronics, packaging, optical lenses, optical storage media, optical fiber production, and numerous other fields [3]. Consequently, studying the radiation effects in PC holds significant theoretical and practical value.

Numerous studies have elucidated how varying absorbed doses affect PC’s thermoluminescence behavior, degradation, optical density, and electrical, structural, and mechanical properties [4]. Abdul-Kader et al. (2018) examined gamma irradiation effects (150–950 kGy) on Makrofol LT 6-4 nuclear track detector films, revealing changes in PC crystal size and the occurrence of cross-linking and degradation at specific doses. They observed decreasing band gap energy, substantially increased dielectric constant, and diminished photoluminescent spectral peak intensity with increasing dose.

Zoul et al. (2020) demonstrated through spectrophotometric measurements of irradiated PC samples that optical density within the visible wavelength range could accurately determine doses up to 200 kGy. They also showed PC’s ability

to completely regenerate within months at doses up to 30 kGy and confirmed oxygen concentration's positive effect on fading rates [5]. Zoul et al. (2018) investigated fading and annealing characteristics of radiochromic PC dosimeters [6]. Jaleh et al. (2007) employed thermogravimetric analysis (TGA), Fourier-transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and electron paramagnetic resonance (EPR) to identify electron beam (EB) irradiation-induced changes in PC's physicochemical properties [7]. They observed increased cross-linking probability from 0 to 50 kGy, decreased decomposition temperature at high absorbed doses, and minimal crystallinity changes compared to unirradiated PC. EPR measurements revealed that higher doses produced more free radicals, which decayed faster due to increased recombination rates.

Chen et al. (2005) examined EB treatment parameters, including different doses, dose rates, and dose classifications, and their effects on PC structure and mechanical properties [8]. Their results showed that PC molecular weight degradation depended largely on dose, while mechanical properties such as tensile strength and ductility were temperature-dependent. Nouh et al. (2007) irradiated Makrofol detector samples with EB doses of 10–400 kGy [9], studying physicochemical properties through XRD, FTIR spectroscopy, color difference measurements, and EPR. Their findings indicated that the Makrofol detector exhibited limited degradation resistance, with a much lower tendency for cross-linking compared to several other solid-state nuclear track detectors.

Tang et al. (2014) investigated PC properties and structural changes under EB irradiation at glass transition temperature (T_g) using thermal analysis, XRD, and mechanical testing (tensile, hardness, wear, and impact properties) [10]. They found that irradiated PC sheet T_g increased at approximately 10 kGy then decreased at higher doses. Within 70 kGy, the ordered structure of the amorphous phase remained essentially unchanged. Wear resistance, hardness, and impact strength increased with dose, reaching maximum values around 10 kGy before decreasing. Tensile strength changes mirrored impact strength, while elongation at break dropped to half at 10 kGy. Under high-temperature irradiation, molecules rearranged through the synergistic effects of radiation and thermal migration, becoming densely packed in the matrix and thus improving material properties.

However, few studies have examined PC radiation effects across a wide absorbed dose range from the perspective of developing radiochromic film dosimeters. Radiochromic films undergo chemical reactions upon exposure to ionizing radiation, producing significant color changes without requiring additional processing through thermal, optical, chemical, or amplification methods. The radiation chemical yield of chromophores is proportional to absorbed dose, enabling dose determination through optical density or chroma measurements before and after irradiation.

Conventional dosimeters currently in use include calorimetric radiation dosimeters [11,12], ionization chamber dosimeters [13], chemical dosimeters

(Fricke [14–17], dichromate [18], cerium sulfate [19]), and solid dosimeters (radiochromic dosimeters [20–22], thermoluminescence dosimeters, radiophotoluminescence dosimeters [23]). Compared with other dosimeters, radiochromic film dosimeters offer advantages of simple operation, convenient testing, high resolution, good response consistency, and direct readability, making them ideal for measuring gamma and EB absorbed doses, electron accelerator field distributions, depth dose distributions, and energy. They hold wide application value in medical treatment, radiation processing, and other fields [24,25].

Among various radiochromic film dosimeters, the FWT-60 film dosimeter produced by Far West Technology Company (USA) demonstrates the best dosimetry performance. Using nylon as the base material and hexahydroxyethyl pararosaniline cyanide dye as the radiation-sensitive material, it offers a dose measurement range of 1–200 kGy. The American Institute of Standards and Technology has designated this dosimeter as the standard for radiation processing [26,27]. The FWT-60 series has become Far West Technology's exclusive worldwide product. In the 1980s and 1990s, Chinese research institutions including Guangdong Test Institute, Chemical Protection Research Institute, and Military Medical Science Research Institute developed radiochromic film dosimeters. However, these have not been widely applied or popularized due to small batch production and limited attention. Currently, domestic demand for radiochromic film dosimeters relies primarily on imports. To overcome this technical bottleneck, numerous research teams have pursued development of high-dose radiochromic film dosimeters, focusing on: dosimetry performance of FWT-60-00 radiochromic film dosimeters; formulation and mass production schemes; new high-sensitivity radiochromic film electronic and gamma dosimeters; proton irradiation effects and annealing characteristics; radiochromic film dosimeters for high-dose-level measurements; and PVB-based low-dose radiochromic film dosimeters. However, few studies have investigated radiation effects in radiochromic film materials potentially suitable for wide-range, high-dose measurements.

This study examines EB irradiation effects (0–1000 kGy) on the optical and structural properties of PC films, focusing on color, visible light transmittance, ultraviolet absorption, crystal structure, molecular weight, thermal deformation, and thermal stability after irradiation. Colorimetry, ultraviolet–visible absorption spectroscopy, XRD, gel permeation chromatography (GPC), and thermogravimetry (TG) were employed for these analyses. The results hold theoretical significance for polymer radiation effect studies and provide guidance for PC applications in high-absorbed-dose detection or as structural materials in high-dose irradiation fields.

II. Experimental Details

A. Sample Preparation

The PC films used in this study were manufactured by Jiaxing Diren Polycarbonate Co., Ltd., Zhejiang, China, with a thickness of 0.3 mm and density of 1.2 g/cm^3 . The film thickness is uniform, with molecular formula ($\text{C}_{16}\text{H}_{14}\text{O}_3$) [28]. Physical and chemical properties include: linear expansion rate of $3.8 \times 10^{-5} \text{ cm/}^\circ\text{C}$, thermal deformation temperature range above $135 \text{ }^\circ\text{C}$ and below $-45 \text{ }^\circ\text{C}$, and light transmittance of $90\% \pm 1\%$. The PC films were cut into $20 \text{ mm} \times 20 \text{ mm} \times 0.3 \text{ mm}$ squares and stored in sample boxes for subsequent use.

B. EB Irradiation

Irradiation was performed using the electron accelerator (IS1020) at Xianghua-Huada Biotechnology Co., Ltd., Hunan, China, with beam energy and power of 10 MeV and 20 kW, respectively. Sample boxes were placed on a conveyor belt and passed under a scanning horn multiple times to achieve the desired dose. PC films were irradiated at doses of 0, 50, 100, 200, 400, 600, 800, and 1000 kGy. Irradiation was conducted at room temperature in air atmosphere, with absorbed dose traced by silver dichromate dosimeter. The dose rate was 10 kGy/s, corresponding to 50 kGy per pass. All analyses were performed immediately after irradiation.

C. Chroma Analysis

The International Commission on Illumination (CIE) XYZ color space encompasses all color sensations perceivable by humans and serves as a standard reference for many other color space definitions [29]. A CS-5960GX colorimeter was used to analyze the chroma of irradiated PC films. The illumination source is an all-band balanced LED light source (CLEDs), with a dual optical array sensor. The measured wavelength ranges from 400 nm to 700 nm at 10 nm intervals.

D. Chroma Value Fading Characteristics

Studying fading characteristics is essential for developing PC as a radiochromic material. PC films irradiated at 200, 600, and 1000 kGy were stored in light-blocking boxes, and their chroma values were analyzed at days 0, 2, 5, 10, 20, 30, and 60 to determine temporal changes in chroma values.

E. UV-Visible Absorption Spectra Analysis

Absorption spectra of pristine and irradiated PC films were recorded using a UV-3600 spectrophotometer. The light source was set to automatic mode with conversion wavelength at 310 nm and slit width of 2 nm. The test wavelength range was 200–800 nm, with 0.1 nm resolution, 0.5 nm step size, and medium scanning rate. PC films were centered in the sample clip and positioned consistently to minimize optical path errors.

F. XRD Analysis

PC film crystal structure was characterized by XRD using a SmartLab SE diffractometer (Rigaku, Japan) with Cu-K α radiation (X-ray tube voltage: 40 kV; current: 40 mA; wavelength: 0.154 nm). Bragg's angle (2θ) was scanned from 5° to 90° at 5°/min. Average crystallite size (L) and interplanar spacing (D) were calculated using Scherrer's and Bragg's equations, respectively [30]:

$$L = \frac{k\lambda}{\Delta W \cos \theta}$$

$$D = \frac{\lambda}{2 \sin \theta}$$

where k is a constant (0.94), λ is the Cu-K α radiation wavelength (0.154 nm), ΔW is the full width at half maximum (FWHM) of the peak, and θ is Bragg's angle. ΔW and θ values were obtained by fitting XRD peaks using the Lorentz function.

G. Gel Permeation Chromatography (GPC)

Relative molecular mass and distribution were determined by GPC (Agilent 1260 Infinity II) with a differential refractometer. Chromatography was performed using two Agilent PL gel MIXED-B LS columns (300 mm \times 7.5 mm, 10 μ m) with dichloromethane mobile phase at 1 mL/min flow rate. Column temperature was 30 °C with 100 μ L injection volume. Polystyrene served as the calibration standard. PC film (0.002 g) was completely dissolved in 2 mL dichloromethane and filtered through a 0.45- μ m membrane before injection. Molecular weight and distribution of PC films irradiated at different doses were analyzed using Agilent GPC software.

H. Thermal Deformation and Thermogravimetric Analysis

PC films irradiated at different doses were placed in a TLD-2000B far-infrared precision annealing furnace, heated at 15 °C/min to 135 °C, and held for 1 h. Samples were gradually cooled to 22 °C before removal for shape change observation. Simultaneously, TGA was performed on irradiated and pristine PC films using NETZSCH TG 209 F3. Temperature was increased from room temperature to 700 °C at 20 °C/min under 60 mL/min nitrogen flow. TGA provides quantitative weight change data, enabling assessment of thermal stability. The temperature corresponding to maximum weight loss rate was identified as the PC decomposition temperature, and the relationship between degradation temperature and absorbed dose was explored.

III. Results and Discussion

Figure 1 [Figure 1: see original paper] shows PC films irradiated at different EB doses (0–1000 kGy). The unirradiated PC film (0 kGy) appears colorless and transparent, while irradiated films exhibit varying degrees of coloration. With increasing absorbed dose, PC film color darkens progressively from colorless to light yellow to brownish yellow. Yellowing of PC after irradiation is a well-documented phenomenon [1]. High-energy EB can break certain chemical bonds in polycarbonate molecules, such as C–H bonds. When these bonds break, molecular fragments recombine or react with surrounding material to form chromophores that absorb specific wavelengths, producing a yellow appearance. Free radicals generated during PC irradiation react with hydrogen atoms on the polycarbonate main chain, deforming the chain and altering PC color. Additionally, polycarbonate undergoes oxidation and decomposition reactions during irradiation, with reaction products changing polycarbonate configuration and creating color differences between unirradiated and irradiated samples.

A. CIE Chromaticity Diagram

Figure 2 [Figure 2: see original paper]-a presents the CIE chromaticity diagram of pristine and irradiated PC films, with enlarged local images shown in Figure 2-b. Colors perceivable by an average human occupy a region of the chromaticity diagram, with X and Y values as projected coordinates determining sample chromaticity. Different sample colors appear at different diagram locations. As shown in Figure 2-b, PC film color shifts linearly toward the yellow region of the chromaticity diagram with increasing absorbed dose, enabling dose detection through chroma analysis. A three-dimensional graph of absorbed dose, X value, and Y value was established (Figure 3 [Figure 3: see original paper]-a), with projections on X–Y, X–Z, and Y–Z planes shown in Figures 3-b, c, and d, respectively.

Fitting revealed that PC film chromaticity value parameters (X and Y values) increase with absorbed dose (Z). Both X and Y values exhibit two-stage linear relationships with absorbed dose, with 400 kGy as the turning point. When absorbed dose ranges from 0 to 400 kGy, chrominance value parameters show a linear relationship with dose; when dose is 400–1000 kGy, they follow a different linear relationship. Moreover, at doses above 400 kGy, X and Y values increase more rapidly with dose. Correlation coefficients ranged from 0.994 to 0.997. Absorbed dose detection across 0–1000 kGy (EB) can be achieved through chromaticity analysis of irradiated PC films combined with fitting curves in Figures 3-a, c, and d. Therefore, PC films demonstrate potential for high-dose, wide-range dose detection. PC films are readily available and inexpensive, and chrominance measurement is convenient and rapid, making them simpler and more cost-effective as radiochromic dosimeters compared to other methods.

Transmittance of irradiated PC films to visible light at different wavelengths was analyzed, with results shown in Figure 3-e. Pristine PC film transmittance

across the entire visible wavelength range is approximately 90%, consistent with production specifications. EB irradiation alters PC film transmittance at different wavelengths. Transmittance decreases slowly at first, then rapidly as wavelength decreases from 700 nm to 400 nm. Larger absorbed doses produce lower transmittance at the same wavelength. Additionally, transmittance decreased sharply as dose increased from 0 to 400 kGy, then decreased more slowly as dose continued increasing to 1000 kGy. Optical transmittance of PC film at 450, 500, 550, and 600 nm varies with absorbed dose, as shown in Figure 3-f. Fitting curves reveal that transmittance of PC films to 500 nm and 550 nm light shows a relatively good linear relationship with absorbed dose. A single linear relationship exists between PC film absorbance and absorbed dose, with linear correlation coefficients above 0.98, enabling dose measurement through absorbance values.

B. Fading Characteristic Analysis

Signal stability is a crucial index in radiation measurement. This study investigated fading characteristics of PC film chromaticity values. Changes in chromaticity values (X and Y values) for PC films irradiated at 200, 600, and 1000 kGy over 60 days are shown in Figures 4-a and b. Both X and Y values decrease only slightly (no more than 0.3%) in the first 5 days, then remain stable. Therefore, the radiation signal (chromatic value) formed in PC film is stable. Color decline relates to environmental factors such as light, UV, temperature, humidity, and oxygen content. Thus, storing irradiated PC films in a suitable environment maintains radiation signal stability. Compared with PC photoluminescence signals (which attenuate significantly in the short term), long-term stability of chromatic value signals represents a major advantage.

C. UV–Visible Spectrum

Original absorption spectra of irradiated and unirradiated PC films were measured by UV–visible absorption spectrometry, with unirradiated PC film as the blank control. Net absorbance was obtained by subtracting the unirradiated PC film spectrum from the irradiated PC film spectrum, as shown in Figure 5 [Figure 5: see original paper]-a. Irradiated PC film shows a strong absorption peak at 290–500 nm, with greater absorbed dose producing higher absorbance values. The relationship between PC film absorbance at specific wavelengths (340–450 nm) and absorbed dose is shown in Figure 5-b. A relatively good linear relationship exists between net absorbance of PC films at 350, 370, 390, 410, 430, and 450 nm and absorbed dose (linear correlation coefficient > 0.995).

D. XRD Spectrum

XRD patterns of PC films irradiated at doses ranging from 0 to 600 kGy are shown in Figure 6 [Figure 6: see original paper]. The primary peak appears in the 12° – 30° range. The broad diffraction peak indicates small crystal size, characteristic of a partially crystalline polymer dominated by amorphous phase

[4]. Additionally, diffraction peak intensity decreases with increasing absorbed dose, indicating reduced crystallinity. Free radicals likely form in the PC crystal region during irradiation, reacting with crystal fragments and destroying crystal structure.

Table 1 specifies crystal size (L) and interplanar spacing (D) values after different irradiation doses. These parameters change with EB irradiation dose. As absorbed dose increases from 0 to 600 kGy, crystal size (L) decreases from 1.29 nm to 1.15 nm. Polymers typically undergo cross-linking and degradation reactions during irradiation, causing local order changes reflected in crystal structure modifications. EB irradiation induces local temperature increases, causing atomic rearrangement and introducing various defects (vacancies, interstitial atoms, dislocations) that affect crystal growth and stability. Furthermore, electron beam irradiation may trigger oxidation or reduction reactions on material surfaces, altering surface energy and consequently affecting crystal size and morphology.

E. Molecular Weight Analysis

GPC was employed to determine PC molecular weight. Figure 7 [Figure 7: see original paper] displays the molecular weight distribution of polycarbonate after different irradiation doses. PC film molecular weight primarily distributes between 1×10^3 g/mol and 2×10^5 g/mol. With increasing absorbed dose, molecular weight distribution and cumulative integral curves shift leftward, with decreasing high molecular weight fraction and increasing low molecular weight fraction. This indicates that degradation dominates under electron beam irradiation, resulting in overall molecular weight reduction. Peak average molecular weight (Mp), number average molecular weight (Mn), and weight average molecular weight (Mw) were analyzed, with results summarized in Table 2.

Mp indicates the predominant molecular weight region, Mn reflects small molecule characteristics, and Mw reflects macromolecule properties. Since macromolecules better represent polymer material characteristics, this study focused on Mw changes. Mp generally decreased with increasing irradiation dose. When absorbed dose reached 1000 kGy, Mw decreased from 44,585 g/mol to 27,494 g/mol. Equation (3) was used to calculate PC polymerization degree after different irradiation doses:

$$n = \frac{M_w}{M}$$

where M is PC monomer molecular weight (254) and n is polymerization degree. Results showed PC polymerization degree decreased from 176 to 108, confirming that PC undergoes degradation under electron beam irradiation below 1000 kGy. The primary degradation mechanisms include main chain scission caused

by electron beam energy and oxidation degradation accelerated by irradiation-generated free radicals.

F. Thermal Deformation and Thermogravimetric Analysis

Material thickness in this study did not meet thermomechanical analysis (TMA) requirements, so only qualitative thermal deformation analysis was performed. The deformation degree of PC films heated at 130 °C for 1 h is shown in Figure 8 [Figure 8: see original paper]-a. The pristine unirradiated sample showed no deformation, while the 1000 kGy irradiated sample was completely bent. Bending degree increased with absorbed dose from 0 to 1000 kGy, consistent with PC degradation under irradiation. As demonstrated above, PC molecular weight decreases under irradiation, reducing thermal deformation resistance. Future studies could investigate thermal deformation temperatures of PC films treated with different radiation doses using thermomechanical analysis (applying specific loads in a heating environment; the temperature at which deformation reaches a preset value defines the thermal deformation temperature).

Figure 8-b shows PC film degrades in two stages. The first stage occurs at 420–558 °C, with approximately 66% mass loss. The second stage occurs at 558–700 °C, with 20.2% mass reduction. Therefore, the main degradation temperature range is 420–558 °C. In the second stage, PC mass decreases more slowly than in the first stage. Jang et al. (2004) concluded that H₂O and CO₂ evolution accompanies the entire PC thermal decomposition process [31]. The first stage primarily detects alcohols, ethers, and carbonates, while the second stage involves carbonate rearrangement forming cross-linked structures that ultimately carbonize.

This study used the temperature (T_m) corresponding to the DTG curve peak point to analyze PC film thermal stability, with higher T_m indicating greater heat resistance. Figure 8-c displays T_m for PC films irradiated at different doses. At 50 kGy electron beam irradiation, T_m increased from 517.2 °C to 531.2 °C compared to unirradiated PC film, likely due to enhanced thermal stability from irradiation polymerization at lower doses. However, at 100 kGy, the irradiation degradation process gradually dominated, causing T_m decrease. This result aligns with Jaleh et al. (2007) [7]. With further absorbed dose increases, irradiation degradation became dominant, generating heat-resistant molecular structures during the degradation process that enhanced PC thermal stability. Irradiation may introduce new functional groups or modify existing ones, improving polycarbonate antioxidant properties and slowing high-temperature degradation rates, thereby altering intermediate and final products during PC thermal degradation and affecting degradation behavior.

IV. Conclusion

This study investigated optical and structural properties of PC films after EB irradiation. The chroma and absorbance values of PC film show good correlation

with absorbed dose, enabling dose detection through chroma or absorbance measurements. The radiation signal (chroma value) stored in PC remains essentially unchanged for 60 days. Irradiation breaks PC main chains and alters molecular structure. Crystallinity, crystal size, weight-average molecular weight, and polymerization degree decrease with increasing dose, while irradiated PC thermal stability exceeds that of unirradiated PC. Subsequent studies can further develop other dosimetry properties of PC radiochromic film dosimeters, including repeatability, intra-batch uniformity, dose rate dependence, annealing characteristics, and effects of temperature and humidity on radiation detection stability and signal storage.

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