



# Article Effect of SW-CNT Diameter on Polymer Degradation and Resistance of Polystyrene/SW-CNTs Composites Induced by $\gamma$ -Irradiation

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**Abstract:** The outstanding electrical, mechanical, and thermal properties of carbon nanotubes (CNTs) make them promising materials for a wide range of applications. Numerous theoretical and experimental studies on the diameter-dependent properties of single-walled carbon nanotubes (SW-CNTs) exist. The incorporation of carbon nanotubes into commercial polymers can alter the properties of both materials. Herein, we demonstrate polystyrene/SW-CNT composites with different diameters to check property changes, including radiation-protective characteristics under various doses of gamma radiation. The intrinsic glassy state of polystyrene disappeared owing to the introduction of SW-CNTs into the polymer-polymer chain. In addition, when 1.3 nm diameter SW-CNTs were introduced, the Fourier-transform infrared spectroscopy peaks for alkyl aryl ethers were observed at 10–60 kGy of gamma irradiation. In this study, the different phenomena that occur when polystyrene/SW-CNT composites are formed with different SW-CNT diameters (0.78 nm and 1.3 nm) were investigated via systematic analyses.

**Keywords:** polymer/CNT composites; radiation resistance; diameter-dependent; radiation protective characteristics; gamma radiation

### 1. Introduction

In present times, commercial polymers have gained immense importance in industries and our daily lives. They have many advantages, such as diverse functionality, low cost, light weight, ease of processing, and excellent chemical stability [1]. Over the past few decades, the effects of irradiation on the structural, optical, thermal, and electrical characteristics of commercial polymers have been reported [2]. Polystyrene (PS) is a commercial polymer that is affected by gamma radiation. Gamma radiation can induce styrene polymerization [3]. After polymerization, radiation induces crosslinking of the polymer main chain [4].

The main consequence of gamma irradiation is the generation of radicals. These radicals can give rise to: chain scission, which proceeds with a decrease in molecular weight; and chain branching, wherein molecular weight increases up to crosslinking. Usually, under vacuum conditions, gamma-ray irradiation of PS leads to crosslinking. However, under atmospheric conditions, chain scission reactions predominate at the expense of crosslinking [5–7]. Some of the radicals react with oxygen to form peroxides and subsequently degrade the main polymeric backbone. This indicates that the main chain degradation of PS occurs exclusively with oxygen. Thus, it is called oxidative degradation [8,9].

Specifically, some changes in the PS properties observed in the low-irradiation range of 0–70 kGy were reported by C. Albano et al. Owing to the glassy properties of polystyrene, the radicals generated upon low gamma irradiation doses are trapped and do not change the



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). progress of decomposition. Furthermore, the double bond of the aromatic ring disperses or absorbs energy, preventing further reactions [10–12]. Therefore, the oxidative degradation of polymers may be related to radical and oxygen diffusion.

In addition, when single-walled carbon nanotubes (SW-CNTs) are combined with polymers, the unique properties of the two materials, such as mechanical stability, thermal stability, and radiation resistance, can be modified [13–19]. The evaluation of radiation effects on SW-CNTs is also being studied, which is necessary to understand the changes in the unique properties of the composites [20–23]. In the case of SW-CNTs, when irradiated with gamma rays, functional groups such as –OH, –COOH can be generated on the sidewalls. The average length of the SW-CNTs tends to decrease upon gamma irradiation, and as the irradiance increases, the straight SW-CNT bundles may become curved or wavy. Also, theoretical and experimental studies have been reported on SW-CNTs with various diameter-dependent properties such as chiral wrapping, stability, molecular dynamics, strength, and stiffness [24,25]. Thus, composites of PS and SW-CNTs with different diameters can be applied in many radiation fields such as radiation shielding, radiation sensors, and oil-absorbing membranes [26–29].

Herein, we have systematically studied the changes in material properties when SW-CNTs with two different diameters are introduced into polystyrene. We have investigated properties such as polymer degradation and radiation resistance characteristics, under various doses of gamma radiation.

#### 2. Materials and Methods

#### 2.1. Materials

Polystyrene (average Mw~192,000; catalog no. 430102), SW-CNTs ((6,5) chirality,  $\geq$ 95% carbon basis,  $\geq$ 95% as carbon nanotubes) of 0.78 nm average diameter (catalog no.: 773735), SW-CNTs ( $\geq$ 90% carbon basis,  $\geq$ 80% as carbon nanotubes) of 1.3 nm diameter (catalog no.: 724777), and tetrahydrofuran (THF, ReagentPlus<sup>®</sup>,  $\geq$ 99.0%, containing 250 ppm BHT as the inhibitor, catalog no.: 178810) were obtained from Sigma-Aldrich Inc. (Milwaukee, WI, USA) Ethanol (99.5%, catalogue no.: E0236) was purchased from Samchun Chemical Co., Ltd. (Seoul, Korea).All the reagents and solvents were used without further purification.

#### 2.2. Preparation of Samples and Gamma Irradiation

The PS (1 g) was dissolved in 3 mL of the THF solvent in each vial. Fifteen polymer solution vials were then prepared. In another flask, 10 mg of SW-CNTs and 5 mL of methanol were mixed into a solution. To blend with the PS, 1 mL (2 mg, 0.2 wt%) of the SW-CNT solution was added and stirred for one day to thoroughly mix the two solutions. The vial containing the two mixed solutions was placed in a shaker and dried naturally while maintaining well-mixed conditions.

Gamma irradiation was performed using a gamma <sup>60</sup>Co source on the dried composite samples with different radiation doses (10, 30, 60, and 120 kGy) at a dose rate of 10 kGy/h. The <sup>60</sup>Co source (MDS Nordion, Ottawa, ON, Canada, IR 221 n wet storage type C-188) was located at the Korea Atomic Energy Research Institute (KAERI) in Jeongeup, Republic of Korea.

#### 2.3. Thermal Analysis

The thermal properties of the polymers were determined via thermogravimetric analysis (TGA, TGA2, Mettler Toledo, Seoul, Korea) and differential scanning calorimetry (DSC, Q100, TA Instruments, New Castle, DE, USA) at a heating rate of 10 °C/min under a nitrogen atmosphere. To confirm the decomposition temperature of the composite, TGA was performed at 30–800 °C. The DSC thermograms of the samples were measured from 30–300 °C under a nitrogen atmosphere at a heating rate of 10 °C/min.

#### 2.4. Polymer Molecular Weight

The number-average molecular weight ( $M_n$ ) and weight-average molecular weight ( $M_w$ ) were determined via gel permeation chromatography (GPC, PL-GPC 110, Polymer Laboratories, Church Stretton, UK) in CHCl<sub>3</sub> at a flow rate of 1.0 cm<sup>3</sup>/min at 40 °C.

#### 2.5. Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR)

The FT-IR spectra of the gamma-ray-irradiated polystyrene/SW-CNT composites were obtained using an ATR-FTIR spectrophotometer (Bruker TENSOR 37, Bruker Corporation, Billerica, MA, USA). The spectra were measured in the wavenumber range of  $800-4000 \text{ cm}^{-1}$  in the ATR mode and recorded using the Bruker OPUS software (version 8.5, Bruker Corporation, Billerica, MA, USA) at a resolution of 4 cm<sup>-1</sup>.

#### 3. Results

Two types of SW-CNTs with different diameters, viz. 0.78 nm and 1.3 nm were added to PS to prepare the composite samples (Figure 1). Owing to its simple and well-known main backbone structure, PS was selected. The composite containing 0.2 wt.% of the SW-CNTs was irradiated with different radiation doses (0, 10, 30, 60, and 120 kGy) at a rate of 10 kGy/h to evaluate the effect of the diameter of SW-CNTs on the material properties of the composite.



**Figure 1.** Overview of PS/SW-CNT composites with different diameters of 0.78 and 1.3 nm and various gamma-irradiation conditions.

After gamma irradiation, TGA and DSC analyses were performed to explore the thermal characteristics of the as-prepared composite. The TGA analysis confirms that the high thermal decomposition temperature of 415 °C does not change (Figure 2). Thus, the intrinsic high thermal stability of the basic PS polymer is not inhibited by the addition of SW-CNTs.

In addition, DSC analysis was performed to analyze the thermodynamic changes upon formation of the composite. The DSC results confirm that the glass transition peak of intrinsic PS (102 °C) shifts to an increasingly higher temperature range with increasing total irradiation dose (Figure 3). Moreover, as the total irradiation dose increases, the glass transition temperature changes into two phases (105 °C and 120 °C at 60 kGy); when it was further irradiated, it appeared again as a single peak (120 kGy at 120 °C). However, this intrinsic glass transition phenomenon of PS completely disappears when SW-CNTs are added to form composites, regardless of their diameter. Thus, the crystalline parts that can be formed by polymer-polymer interactions disappear, and SW-CNTs are added therebetween to form an amorphous state [30]. The existing glassy state of PS traps radicals and also affects oxygen diffusion [10]. Thus, in the case of composites containing SW-CNTs, the amorphous phase may be strengthened and can affect the radicals and oxygen diffusion rate.



**Figure 2.** TGA (**a**) and DTG (**b**) profiles of PS and PS/SW-CNTs with diameters of 0.78 and 1.3 nm under various gamma-radiation conditions.



**Figure 3.** The effect of gamma irradiation on the DSC thermograms (second heating) of PS (**a**), PS/SW-CNT composites with a diameter of 0.78 nm (**b**) and 1.3 nm (**c**), at different integral doses.

Molecular weight analysis was performed to directly determine whether the polystyrene/ SW-CNT composites were decomposed and/or cross-linked upon the gamma irradiation. The molecular weights of the two composites are not significantly different from those of polystyrene without SW-CNTs (Figure 4). Some studies report that at low irradiation (0–70 kGy), the high resistance to degradation by PS is due to the presence of an aromatic ring in its molecular structure [10]. This phenomenon of radiation resistance was confirmed in both, the PS reference and PS/SW-CNTs composites.



**Figure 4.** Changes in number-average molecular weight (Mn) of PS (**a**) and PS/SW-CNT composites with diameters of 0.78 nm (**b**) and 1.3 nm (**c**) under gamma irradiation.

The FT-IR technique was used to track the changes in the chemical bonding between polystyrene and SW-CNTs (Figure 5). There is no significant change in chemical bonding when a 0.78-nm SW-CNT is introduced, but two peaks centered at 1250 cm<sup>-1</sup> and 1050 cm<sup>-1</sup> appear from 10–60 kGy when 1.3-nm SW-CNTs were introduced. These peaks appear in alkyl aryl ethers, and the oxidation reaction occurs in the case of the 1.3-nm SW-CNTs composites through radiation energy.



**Figure 5.** Fourier-transform infrared spectroscopy spectra for PS (**a**), and PS/SW-CNT composites with an SW-CNT diameter (0.78 nm (**b**) and 1.3 nm (**c**)) upon gamma irradiation.

The reaction by which PS decomposes into benzoic acid is harsh and complex, even under photooxidation conditions [31]. In addition, the decomposition products, such as acetic acid, benzyl alcohol, and benzoic acid, do not appear in their natural state and can only be obtained through treatments such as UV irradiation [32]. Contrarily, in the polystyrene/SW-CNT composite having a SW-CNT diameter of 1.3 nm, an intermediate of the decomposition product is formed even at a low gamma irradiation dose of 60 kGy or less [32]. Figure 6 shows the general decomposition products of PS under irradiation conditions. Interestingly, in this study, oxidation intermediates such as alkyl aryl ethers were formed, which were not the general decomposition products. When SW-CNTs of 0.78 nm diameters are used, this phenomenon occurs with an extremely low probability; thus, different internal environments may be created in the polymer/SW-CNTs composites depending on the diameter. Therefore, when the number of irradiated radicals is the same, the oxygen diffusion into polystyrene/SW-CNT may be the most effective factor inducing the oxidation reaction.



Figure 6. Schematic representation of formation of typical PS radiolysis products on irradiation.

In previous studies, polymer-CNT complexes involved a dispersion of the CNT bundles between the polymer, thereby increasing the effective surface area. For this reason, in a gas sensor, when SW-CNT was introduced rather than the polymer itself, the sensitivity was amplified more than 10 times [33].

In addition to the large effective surface area, oxygen diffusion into the composites can also be a key factor in the oxidation reaction. Commercially available SW-CNTs (diameter of 0.78 nm) have a (6,4) chirality and an effective diameter of ~4 Å (nominally 6.8 Å, but shrunken due to the van der Waals diameter of carbon atoms). This diameter allows water molecules to pass through while restricting the passage of chloride (Cl<sup>-</sup>). In addition, studies on the possibility of diffusion and permeability of various small ions and molecules through SW-CNTs are actively being conducted [34–37]. Results of a previous simulation and experiment on water permeability using 0.78-nm SW-CNTs exist. [34] Based on this, as shown in Figure 7a, in the case of oxygen (O<sub>2</sub>) having a larger volume than water (H<sub>2</sub>O). Thus, passing through the 0.78-nm SW-CNTs may be more difficult for oxygen than water. From this point of view, more oxygen can permeate through SW-CNTs with a large diameter (1.3 nm) than those with a small diameter (0.78 nm).



**Figure 7.** Illustration of the difference in volume between water and oxygen molecules (**a**), and the passage of oxygen inside the SW-CNTs (**b**).

Thus, when introducing SW-CNTs into commercial polymers, the diameter of the SW-CNTs may affect the properties of the composite, which can be tuned to achieve the desired effect. If SW-CNTs with a diameter <0.78 nm are selected, it is possible to obtain a robust material, mainly by controlling its glassy properties without being decomposed by irradiation. However, if SW-CNTs having diameters >1.3 nm are included in the composites, the oxidative intermediate can be formed through an oxidation reaction.

## 4. Conclusions

In summary, we have prepared PS/SW-CNTs composites with two different diameters of the SW-CNTs (0.78 nm and 1.3 nm) to check the gamma-irradiation effects on the asprepared composites. There was no significant change in the thermal stability or molecular weight of the composites after gamma irradiation. However, the intrinsic glassy state of polystyrene disappeared when the polymer and SW-CNTs were mixed. Interestingly, when SW-CNTs of 1.3 nm diameter were introduced, the chemical bonding was affected; this was evidenced by the oxidative intermediate. When SW-CNTs of diameters <0.78 nm were introduced into PS, it was possible to maintain radiation resistance while suppressing the brittle tendency of the polymer. Moreover, when SW-CNTs with diameters >1.3 nm were introduced, the oxidative reactions were accelerated, which may find immense utility as an eco-friendly waste plastic treatment method.

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