# **Properties and Radiation Resistance of Aromatic Polymer-Based Polyblends**

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**ABSTRACT:** Blends of either of two different poly(phenylene oxide) (PPO) derivatives with poly(2-vinylnaphthalene) (P2VN) were prepared by casting from chloroform. The content of P2VN in the blends ranged from 0 to 25 wt % for each PPO derivative. Two kinds of PPO derivatives, *p*-*t*-butylbenzoyl poly(phenylene oxide) (*p*-*t*-BB-PPO) and benzoyl poly(phenylene oxide) (B-PPO), were used. The effects of the addition of P2VN to PPO derivatives were investigated by their thermal stability, light-resistance, and tensile properties. Even though the addition of P2VN to the PPO derivatives decrease the mechanical properties of B-PPO and its blends with P2VN were higher than those of the *p*-*t*-BB-PPO and its blends with P2VN. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 73: 1697–1705, 1999

**Key words:** radiation resistance; poly(phenylene oxide) (PPO); benzoyl poly(phenylene oxide) (B-PPO), *p*-*t*-butylbenzoyl poly(phenylene oxide) (*p*-*t*-BB-PPO); poly(2-vinylnaphthalene) (P2VN); blend

# **INTRODUCTION**

Aromatic polymers have attracted much interest because of their high thermal stability and good mechanical properties. The application of such aromatic polymers has been also recommended for the aerospace or medical industries in the 1990s because of their good radiation-resistance properties. Recently, the Seguchi group<sup>1-3</sup> reported that the aromatic polymers possessing good radiation resistance include polystyrene, polyimide, poly(ether ether ketone) (PEEK), and poly(ether ketone) (PEK) (Fig. 1). Aromatic polymers were expected to possess high thermal and radiation resistance since they were stabilized by a conjugated system in both the main chain and the side chain in the molecule.<sup>1-3</sup>

Poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) is one of such aromatic families and has been widely used for three decades. The use of PPO has expanded after the commercialization of its blend with high-impact polystyrene. PPO has many outstanding properties such as gas permeability, high thermal stability, and good mechanical strength.<sup>4-7</sup>

In this work, the radiation-resistant properties of two kinds of PPO derivatives were tested: p-tbutylbenzoyl poly(phenylene oxide) (p-t-BB-PPO) and benzoyl poly(phenylene oxide) (B-PPO). The PPO derivatives were selected because they exhibit different thermal and mechanical properties

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Figure 1 Aromatic polymers of good radiation-resistance materials.

depending on their structure,<sup>8</sup> and it may be expected that the radiation resistance also depends on the structure. The PPO derivatives were blended with poly(2-vinylnaphthalene) (P2VN). Blends containing P2VN have been one of the main subjects in this laboratory for years.<sup>9–11</sup> The purpose of this work was to reveal the properties and the radiation resistance of the PPO derivatives and the PPO derivative/P2VN blends.

# **EXPERIMENTAL**

# **Materials**

PPO (Aldrich, Milwaukee, WI), benzoyl chloride (Aldrich), and p-t-butyl benzoic acid (Aldrich) were used without purification. Carbon dioxide was dried in CaCl<sub>2</sub> under a nitrogen atmosphere. AlCl<sub>3</sub> was used without purification. MeOH and chloroform were purified by distillation from CaCl<sub>2</sub>. 2-Vinylnaphthalene was used as received without further purification.

#### Synthesis of P2VN

P2VN was obtained in a glass tube by polymerizing 2-vinylnaphthalene with AIBN as an initiator at 70-80°C in toluene for 72 h (Fig. 2). After the reaction, the mixture was poured into excess ethanol. The product of P2VN recovered from the ethanol was dried under a vacuum at 25°C to remove all volatiles.

#### Syntheses of PPO Derivatives<sup>12–17</sup>

B-PPO was obtained by polymerizing 0.02 mol of benzoyl chloride with PPO at the reflux temperature in 100 mL of carbon disulfide for 24 h. After the reaction, 4N HCl (60 mL) was poured into the reaction mixture. The carbon disulfide was evaporated and then circulated for 2 h in 100 mL methanol. The resultant polymer was filtered from this process and recrystallized from methanol. *p*-*t*-BB-PPO was prepared by a method similar to that for B-PPO (Fig. 3). The filtered PPO derivatives were dried *in vacuo* at room temper-



Figure 2 Synthesis of P2VN.



Figure 3 Syntheses of PPO derivatives.

ature. Details of the syntheses of the PPO derivatives were described elsewhere. $^8$ 

#### **Preparation of Blends**

Blends were prepared by dissolving the component polymers in chloroform. The solutions were cast onto a glass plate and most of the solvent was allowed to evaporate slowly in air at room temperature. The resulting films were then dried *in vacuo*. The compositions of P2VN in the blends ranged from 0 to 25 wt %.

## **Characterization of Polymers**

<sup>1</sup>H-NMR spectra were recorded using a Varian Unity Plus-300 spectrometer. Chemical shifts were reported in parts per million relative to  $Me_4Si$  as an internal standard. FTIR spectra were recorded on a Mattson IR-10410E spectrometer.

## Measurements

#### Molecular Weight

Molecular weight was determined by gel permeation chromatography (GPC) (Waters-Water, R-401). The measurements were conducted in chloroform with polystyrene as a standard.

#### **Thermal Stability**

The thermal stability of the polymers was examined with a Shimadzu TGA 50 thermogravimetric analyzer (TGA) at a heating rate of 10°C/min under a  $N_2$  atmosphere. Three measurements were averaged.

# **Light Resistance**

The light resistance of the samples was determined using a Fade-o-meter (Atlas) (at 60°C and 65% RH) and a color difference meter (NO-101DP). The sample films were cast from a solution of 1 g of polymer in 2 mL chloroform on a nonyellowing urethane-coated hiding paper. The films were dried slowly at room temperature and then under a vacuum to constant weight ratios. The areas of the films were  $5 \times 10 \text{ cm}^2$  and the thickness was  $35 \ \mu\text{m}$ . After the films had been exposed in the tester to UV for a given time, the color difference ( $\Delta E$ ) of the samples was measured using a color difference meter.

# **Tensile Tests**

Tensile tests were carried out using a Tinius Olsen tensile tester (Model 1000) for the samples prepared according to ASTM D-1822. The crosshead speed was 100 mm/min and the gauge length was 25 mm. Five measurements were averaged.

# Morphology

Scanning electron microscopy (SEM) was done using a JEOL JSM35-CF SEM. Samples were metallized by gold coating prior to the installation in the SEM chamber.

#### **Radiation Resistance**

The radiation resistance of the samples was in terms of the changes of the measured properties and the morphology mentioned above after the samples were exposed to 5 or 20 Mrad of  $^{60}$ Co  $\gamma$ -ray irradiation. For measurement of the radiation resistance, the  $^{60}$ Co  $\gamma$ -ray source was applied by the Korea Atomic Energy Research Institute (Atomic Energy of Canada Ltd. Radiochemical Company, Industrial Irradiation Division  $^{60}$ Co).

# **RESULTS AND DISCUSSION**

#### Characterization of Polymers

The molecular weight of the polymers are listed in Table I. P2VN was identified by the characteristic peaks at 6.5–7.8 ppm (m, aromatic, 7H) and 1.2–2.0 ppm (m,  $-CH_2-CH_2-$ , 4H) on the <sup>1</sup>H-NMR spectrum (solvent:  $CDCl_3$ ). The FTIR spectrum showed several characteristic peaks at 1506 cm<sup>-1</sup> (C=C, aromatic), 1621 cm<sup>-1</sup> (C=C, conjugated),

| Compounds                           | $M_n$                      | $M_w$                      | $M_w/M_n$           | $T_g$ (°C)          |
|-------------------------------------|----------------------------|----------------------------|---------------------|---------------------|
| P2VN<br><i>p–t-</i> BB-PPO<br>B-PPO | 5800<br>109,600<br>173,800 | 8600<br>182,000<br>207,200 | $1.7 \\ 1.6 \\ 1.1$ | $125 \\ 296 \\ 250$ |

Table IMolecular Weight Characteristics ofPPO Derivatives and P2VN

2920 cm<sup>-1</sup> (CH, aliphatic), and 3051 cm<sup>-1</sup>(CH, aromatic). *p*–*t*-BB-PPO was characterized by <sup>1</sup>H-NMR and FTIR spectroscopies: The <sup>1</sup>H-NMR (solvent: CDCl<sub>3</sub>) showed several characteristic peaks at 1.8–2.4 ppm [—(CH<sub>3</sub>)<sub>2</sub>, 6H], 6.4–6.6 ppm (aromatic, 2H), and 7.4–8.0 ppm (aromatic, 5H). The FTIR spectrum exhibited the characteristic peak at 1672 cm<sup>-1</sup> (C=O stretching). Also, B-PPO was identified by <sup>1</sup>H-NMR (solvent: CDCl<sub>3</sub>) spectroscopy with the characteristic peaks at 1.3 ppm [—C(CH<sub>3</sub>)<sub>3</sub>, 9H], 1.8–2.2 ppm [—(CH<sub>3</sub>)<sub>2</sub>, 6H], 6.4–6.6 ppm (aromatic, 2H), and 7.4–7.8 ppm (aromatic, 4H). The FTIR spectrum of the B-PPO also exhibited the characteristic peak of the C=O stretching peak at 1670 cm<sup>-1</sup>.

#### **Thermal Stabilities**

Table II shows typical TGA thermogram data of the *p*-*t*-BB-PPO/P2VN blends with different weight ratios. The average decomposition temperatures of the homopolymers are given as follows: 415 and 460°C for *p*-*t*-BB-PPO and B-PPO, respectively. It is seen that the thermal stability of *p*-*t*-BB-PPO was generally decreased by adding P2VN. The temperature exhibiting a 50% weight loss was decreased with increasing P2VN weight ratios. The average weight residue at 600°C was about 7–15%. In the B-PPO/P2VN blends, also shown in the same table, the initial decomposition temperatures were almost constant with increasing B-PPO content except at 85 : 15 weight ratios. This result means that the thermal stability of B-PPO is not much affected by a small amount of P2VN. The temperature showing a 50% weight loss was almost constant at 470– 480°C. At 600°C, the weight of the residue was given at 5–18%.

After *p*–*t*-BB-PPO/P2VN as well as the B-PPO/ P2VN blends were exposed under 5- and 20-Mrad  $\gamma$ -ray irradiation, not much change was observed in the thermal decomposition temperatures in comparison to those before irradiation (Table III). This shows that the thermal stability of the blends was not significantly affected by the  $\gamma$ -ray irradiation below 20 Mrad.

#### **Light Resistance**

The light resistance of the samples was semiquantitatively described in terms of the color difference with a National Bureau of Standards Unit. The color difference theories imply that the smaller  $\Delta E$  means better light resistance.<sup>11</sup> The samples were exposed in the Fade-o-meter for measurements of the light resistance. Table IV shows the light resistances of the various blend samples. The B-PPO film was colorless and  $\Delta E_1$ was 5.2 before irradiation. But after 5-Mrad irradiation,  $\Delta E_2$  was 9.3 and the film was pale yellow. The color difference change was 4.1. After 20-Mrad irradiation,  $\Delta E_2$  was 12.0 and the color dif-

Table II TGA Data of PPO Derivatives and P2VN Blends

| Compositions<br>(wt %)    | Initial Decomposition<br>Temperature (°C) | Temperature at Which<br>50% Weight Loss | Weight (%) Residue<br>at 600°C |  |
|---------------------------|---|---|--------------------------------|--|
| <i>p–t-</i> BB-PPO : P2VN |   |   |                                |  |
| 80:20                     | $420\pm3$                                 | $465\pm2$                               | $15\pm2$                       |  |
| 87:13                     | $420\pm3$                                 | $470 \pm 4$                             | $17\pm1$                       |  |
| 90:10                     | $420\pm5$                                 | $470\pm3$                               | $10\pm2$                       |  |
| 93:7                      | $415\pm5$                                 | $475\pm4$                               | $15\pm2$                       |  |
| 100:0                     | $415\pm4$                                 | $500\pm5$                               | $7\pm2$                        |  |
| B-PPO : P2VN              |   |   |                                |  |
| 85:15                     | $395\pm5$                                 | $470\pm5$                               | $5\pm2$                        |  |
| 88:12                     | $460\pm4$                                 | $480\pm4$                               | $8\pm1$                        |  |
| 92:8                      | $460\pm5$                                 | $480 \pm 4$                             | $5\pm 1$                       |  |
| 95:5                      | $460\pm5$                                 | $480 \pm 4$                             | $18\pm2$                       |  |
| 100:0                     | $460 \pm 4$                               | $470\pm5$                               | $18 \pm 1$                     |  |

|                           | Initial Dee<br>Tempera | Initial Decomposition<br>Temperature (°C) |            | Temperature at Which<br>50% Weight Loss |            | Weight (%) Residue at 600°C |  |
|---------------------------|------------------------|---|------------|---|------------|-----------------------------|--|
| Compositions              | 5 Mrad                 | 20 Mrad                                   | 5 Mrad     | 20 Mrad                                 | 5 Mrad     | 20 Mrad                     |  |
| <i>p–t</i> -BB-PPO : P2VN |                        |   |            |   |            |                             |  |
| 80:20                     | $413\pm5$              | $410\pm 6$                                | $474\pm2$  | $471\pm3$                               | $13\pm2$   | $16\pm2$                    |  |
| 87:13                     | $450 \pm 4$            |   | $478\pm3$  | _                                       | $12\pm1$   |                             |  |
| 90:10                     | $450\pm 6$             | $452\pm5$                                 | $480\pm3$  | $482\pm3$                               | $12 \pm 3$ | $13\pm2$                    |  |
| 93:7                      | $452\pm3$              | $456\pm2$                                 | $487\pm4$  | $485\pm5$                               | $18\pm2$   | $12\pm2$                    |  |
| 100:0                     | $450\pm5$              | $454\pm5$                                 | $481\pm5$  | $490 \pm 4$                             | $10\pm2$   | $18 \pm 4$                  |  |
| B-PPO : P2VN              |                        |   |            |   |            |                             |  |
| 85:15                     | $445\pm3$              | $444\pm3$                                 | $475\pm3$  | $475\pm4$                               | $1\pm3$    | $17\pm5$                    |  |
| 88:12                     | $450\pm4$              | $445\pm5$                                 | $480\pm3$  | $471\pm5$                               | $4\pm2$    | $0\pm 1$                    |  |
| 92:8                      | $450\pm4$              | $447\pm5$                                 | $478\pm 6$ | $477\pm4$                               | $17\pm5$   | $2\pm 2$                    |  |
| 95:5                      | $460 \pm 3$            | $449\pm5$                                 | $479\pm5$  | $479\pm4$                               | $0\pm 1$   | $1\pm 1$                    |  |
| 100:0                     | $460\pm3$              | $451\pm4$                                 | $479\pm4$  | $477\pm5$                               | $0\pm 1$   | $1\pm3$                     |  |

Table III TGA Data of PPO Derivatives and P2VN Blends After y-ray Irradiation (5 and 20 Mrad)

ference change was 6.8. These results imply that the yellowing was faster when 20-Mrad irradiation was applied to the samples. Similarly, the film was almost colorless before  $\gamma$ -ray irradiation for p-t-BB-PPO and  $\Delta E_1$  was 2.3, but after 5-Mrad irradiation,  $\Delta E_2$  was 10.7 and the color became pale yellow. The color difference change was 3.3. After 20-Mrad irradiation, however,  $\Delta E_2$ was 10.7 and the color difference change was 8.5. As the weight ratios of P2VN to p-t-BB-PPO were increased, the color became more yellowish and the color difference value had a tendency to increase. This shows that more P2VN led to the faster yellowing. The result, however, does not merely imply that the light resistance was decreased with increasing P2VN contents: The result may be, rather, due to the inherent yellowish color of P2VN. Table IV shows that B-PPO possesses more stable light resistance than that of p-t-BB-PPO when one compares the  $\Delta E_2 - \Delta E_1$ for the blends having the same 5 wt % of P2VN.

# **Tensile Properties**

The tensile properties of blends are shown in Figures 4 and 5. At a given weight ratio of the p-t-BB-PPO/P2VN blends, the tensile strength and elongation at break of the blends decreased with increasing P2VN weight ratios. The same result

| Table IV | <b>Color Difference</b> | <b>Determined by</b> | Fade-o-meter   | for PPO | Derivatives | and Thei | r Blends w | vith |
|----------|-------------------------|----------------------|----------------|---------|-------------|----------|------------|------|
| P2VN Bef | ore and After γ-ra      | y Irradiation (§     | 5 and 20 Mrad) |         |             |          |            |      |

|                                  |              |             |              | After $\gamma$ -ray Irradiation |                           |              |             |                           |
|----------------------------------|--------------|-------------|--------------|---------------------------------|---------------------------|--------------|-------------|---------------------------|
| Before $\gamma$ -ray Irradiation |              |             |              | 5 Mrad                          |                           |              | 20 Mrad     |                           |
| Sample                           | $\Delta E_1$ | Color       | $\Delta E_2$ | Color                           | $\Delta E_2 - \Delta E_1$ | $\Delta E_2$ | Color       | $\Delta E_2 - \Delta E_1$ |
| B-PPO : P2VN                     |              |             |              |                                 |                           |              |             |                           |
| 100:0                            | 5.2          | Colorless   | 9.3          | Pale yellow                     | 4.1                       | 12.0         | Pale yellow | 6.8                       |
| B-PPO : P2VN                     |              |             |              | ·                               |                           |              | ·           |                           |
| 95:5                             | 8.0          | Pale yellow | 12.2         | Yellow                          | 4.2                       | 15.8         | Yellow      | 7.8                       |
| B-PPO : P2VN                     |              |             |              |                                 |                           |              |             |                           |
| 85:15                            | 8.5          | Pale yellow | 12.8         | Yellow                          | 4.3                       | 15.5         | Yellow      | 7.0                       |
| p-t-BB-PPO : P2VN                |              |             |              |                                 |                           |              |             |                           |
| 100:0                            | 2.3          | Colorless   | 5.6          | Pale yellow                     | 3.3                       | 10.7         | Pale yellow | 8.5                       |
| p-t-BB-PPO : P2VN                |              |             |              |                                 |                           |              |             |                           |
| 95 : 5                           | 3.5          | Pale yellow | 13.28        | Yellow                          | 9.78                      | 14.2         | Yellow      | 10.7                      |



wt. ratios of P2VN in PPO derivative/P2VN blends

**Figure 4** Tensile strength of PPO derivatives and P2VN blends before and after  $\gamma$ -ray irradiation. (Closed symbols) B-PPO: (- $\bullet$ -) before radiation; (- $\blacksquare$ -) 5 Mrad; (- $\blacktriangle$ -) 20 Mrad. (Open symbols) *p*-*t*-BB-PPO: (- $\bigcirc$ -) before radiation; (- $\square$ -) 5 Mrad; (- $\triangle$ -) 20 Mrad.

was observed after  $\gamma$ -ray irradiation (Fig. 4). For the p-t-BB-PPO/P2VN blends, as the amount of  $\gamma$ -ray irradiation was increased, the decrease of the tensile properties was larger. The decrease in the tensile strength of the blends with added P2VN might be due to the low degree of polymerization of P2VN in this work. When the weight ratio of P2VN is over 15 wt %, the films could not be obtained due to the too brittle nature. In the case of the B-PPO/P2VN blends, the tensile strength of B-PPO decreased as the weight ratio of P2VN was increased. The same results were observed after 5- and 20-Mrad irradiation. Figure 5 shows that the elongation at break of the p-t-BB-PPO/P2VN blends exhibits the same trend as for the tensile strength. Of interest, however, is the unexpectedly increasing elongation at break for the B-PPO/P2VN blends as the weight ratios of P2VN was increased regardless of the  $\gamma$ -ray irradiation. The result may be due to stress-concentration sites which lead to shear banding and/or craze initiation, which has been observed sometimes in immiscible blends such as polycarbonate/nylon, polycarbonate/polyethylene, or PPO/polyethylene blends.<sup>18-20</sup> Comparison of Figures 4 and 5 also shows that B-PPO exhibits better tensile properties than those of *p*-*t*-BB-PPO.

#### Morphology

The morphology was observed for the p-t-BB-PPO/P2VN and B-PPO/P2VN blends with various

weight ratios after  $\gamma$ -ray irradiation. In Figure 6, gross phase separation was observed in the p-t-BB-PPO/P2VN blends, due to the immiscibility between *p*-*t*-BB-PPO and P2VN. Here, the matrix is *p*–*t*-BB-PPO and the domain is P2VN. One can see easily, however, the effect of  $\gamma$ -ray irradiation on the blend morphology in Figure 6. Regardless of the P2VN weight ratios, larger pinholes in the p-t-BB-PPO matrix were observed after  $\gamma$ -ray irradiation. More pinholes were observed in the case of 20-Mrad rather than in the case of 5-Mrad irradiation. The trend is more prominent when the weight ratio of P2VN is lower. The result clearly exhibits that P2VN has better radiation resistance than that of p-t-BB-PPO. The same result was observed in the case of the B-PPO/ P2VN blends. B-PPO showed less damage under 5 than 20 Mrad at the weight ratio of 85:15 of B-PPO/P2VN (Fig. 7). Careful inspection of Figures 6 and 7 indicates that B-PPO is more radiation-resistant than is *p*-*t*-BB-PPO, since no appreciable pinhole by the  $\gamma$ -ray attack is observed.

#### **CONCLUSIONS**

It was found that as the weight ratios of P2VN and the amount of  $\gamma$ -ray irradiation increased yellowing was faster. The tensile strength of the



**Figure 5** Elongation at break of PPO derivatives and P2VN blends before and after  $\gamma$ -ray irradiation. (Closed symbols) B-PPO: (- $\bullet$ -) before radiation; (- $\blacksquare$ -) 5 Mrad; (- $\blacktriangle$ -) 20 Mrad. (Open symbols) *p*-*t*-BB-PPO: (- $\bigcirc$ -) before radiation; (- $\square$ -) 5 Mrad; (- $\triangle$ -) 20 Mrad.



p-t-BB-PPO : P2VN 80 : 20 (B)



p-t-BB-PPO : P2VN 80 : 20 (A)





| p-t-BB-PPO : P2VN | p-t-BB-PPO : P2VN |
|-------------------|-------------------|
| 90 : 10           | 90 : 10           |
| (A)               | (B)               |

**Figure 6** SEM micrographs of *p*-*t*-BB-PPO and P2VN blends after 5- and 20-Mrad irradiation: (A) after 5-Mrad irradiation; (B) after 20-Mrad irradiation.

PPO derivatives/P2VN blends were measured before and after  $\gamma$ -ray irradiation. The tensile strength and elongation at break of *p*-*t*-BB-PPO decreased with increasing P2VN weight ratios. After 5- and 20-Mrad irradiation, the tensile strength was decreased. Unexpectedly, however, the elongation at break of B-PPO was slightly increased when the weight ratios of P2VN were increased. But the tensile strength showed the reverse trend. After irradiation for the B-PPO/



B-PPO : P2VN 100 : 0 (A)



B-PPO : P2VN 100 : 0 (B)







**Figure 7** SEM micrographs of B-PPO and P2VN blends after 5- and 20-Mrad irradiation: (A) after 5-Mrad irradiation; (B) after 20-Mrad irradiation.

P2VN or p-t-BB-PPO/P2VN blends, the morphology showed a severe  $\gamma$ -ray attack after 5- or 20-Mrad radiation. It may be concluded that even though the addition of P2VN to the PPO derivatives decrease the mechanical properties the radiation resistance was improved. The radiation resistance and the tensile properties of B-PPO and its blend with P2VN were higher than those of the p-t-BB-PPO and its blend with P2VN. This work was financially supported by the Ministry of Education, Korea (via the Advanced Materials Grant #95-33-0032).

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