

Radiation Crosslinking of Poly(butylene succinate) in the Presence of Low Concentrations of Trimethallyl Isocyanurate and Its Properties

Meri Suhartini,¹ Hiroshi Mitomo,¹ Naotsugu Nagasawa,² Fumio Yoshii,² Tamikazu Kume²

¹Department of Biological and Chemical Engineering, Faculty of Engineering, Gunma University, Tenjin-cho, Kiryu, Gunma 376-8515, Japan

²Japan Atomic Energy Research Institute, Takasaki Radiation Chemistry Research Establishment, Watanuki-machi, Takasaki, Gunma 370-1292, Japan

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ABSTRACT: Poly(butylene succinate) (PBS) with different molecular weights was irradiated using an electron beam (EB) in the presence of polyfunctional monomers at ambient temperature. Five polyfunctional monomers were used in this work. The highest amount of gel fraction was achieved when PBS was blended with trimethallyl isocyanurate (TMAIC). The amount of TMAIC introduced strongly influenced the amount of resulting gel. It was determined that the crosslinked PBS sample containing 1% TMAIC had a higher glass-transition temperature than did the original

PBS. It was also observed that the presence of crosslinking bonds in the irradiated PBS greatly improved its heat stability and diminished its ability to biodegrade. Accordingly, it can be concluded that crosslinked PBS in the presence of TMAIC significantly improved its heat resistant properties. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 88: 2238–2246, 2003

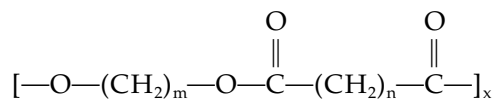
Key words: poly(butylene succinate); polyfunctional monomer; radiation crosslinking; heat stability; biodegradation

INTRODUCTION

Aliphatic polyesters such as poly(butylene succinate) (PBS), poly(ϵ -caprolactone) (PCL), and poly(lactic acid) (PLA) are considered the most economically competitive of the biodegradable polymers.¹ However, the insufficient properties of these polymers have prevented them from being utilized in diverse applications. To obtain more suitable biodegradable polymers with modified parameters, a variety of processing techniques such as blending and copolymerization have been used.²

PBS (Structure 1 with $m = 2$ and $n = 4$) is a biodegradable aliphatic thermoplastic polyester. Aliphatic polyesters are produced through a polycondensation reaction of glycols such as ethylene glycol and butanediol-1,4 with aliphatic dicarboxylic acids such as succinic and adipic acids as principal raw materials.^{3,4} PBS polymer is degraded by bacteria in soil, and it is a so-called environmentally friendly polymer.

Irradiation processing can modify the structure of aliphatic polyesters and affect their principal properties. It has been reported that irradiation of poly(ϵ -caprolactone) in the supercooled state improves its heat resistance and processibility.⁵ PBS [poly(butylene



Structure 1

succinate] has been studied as a means of producing a biodegradable foam by electron-beam irradiation.⁶ The results of that investigation suggested that PBS with desirable parameters can be synthesized if certain additives are blended with the original polymer matrix. With the help of suitable additives, the heat stability, mechanical properties, and processibility of plastics can be improved. A specific monomer distribution in the polymer chains of aliphatic polyesters can provide the polyesters with good material properties, while remaining biodegradable and reasonably priced to produce.^{7,8} Further, it has been observed that the physical properties of the aliphatic polyesters were improved as a result of adding aromatic units to the polymer.^{9,10} It is known that the biodegradation rate of PBS strongly depends on its degree of crystallinity and its morphology. Aliphatic polyesters with aromatic groups in their side chain should have properties and biodegradability different from those with aromatic groups in their main backbone chains.¹¹

Radiation crosslinking is a major application of radiation processing in industry. PBS has a melting temperature in the range of 90°C–120°C: its thermal prop-

Correspondence to: Fumio Yoshii (yoshii@taka.jaeri.go.jp).

TABLE I
Some Properties of PBS Samples Used in This Work

Sample type	M_w	Melting point (°C)	Density (g/cm ³)	ΔH (J/g)
PBS1	2.96×10^5	92	1.23	45
PBS2	1.56×10^5	93	1.23	44
PBS3	1.75×10^5	114	1.26	72

erties are similar to those of polyethylene. Unfortunately, very low resistance to thermal degradation is a serious limitation to the processing and application of PBS. Therefore, improving the heat resistance of PBS is essential for further expansion of its applicability. The well-known method for achieving such an improvement in polymer properties is to crosslink new compositions by using ionizing radiation. In this study the radiation crosslinking behavior of PBS in the presence of polyfunctional monomer and its properties was investigated.

EXPERIMENTAL

Materials

Some properties of PBS samples used in this study are shown in Table I. These polymers were produced at Showa High Polymer Co. Ltd., Japan. To prepare the mixed materials, five kinds of polyfunctional mono-

mers were used. The structures of these polyfunctional monomers are shown in Table II.

Preparation of samples and irradiation

Monomer content of a final composition is expressed as the ratio of the mass of monomer to the total mass of the sample: PBS and the additive were mixed in a laboratory plastomill, model 50C150 (Toyo Seiki). The mixing speed was 20 rpm at 150°C. Each sample was pressed in an Ikeda hot press at 150°C to form a 0.5-mm-thick sheet: samples were preheated for 3 min and then were heated at the same temperature for another 3 min at a pressure of 120 kgf/cm². After that the samples were cooled to room temperature by cold pressing using water as a coolant for 3 min. Irradiation of the samples was carried out in a plastic bag using an accelerator with energy of 2 MeV and current of 1 mA, at various doses, ranging from 10 to 100 kGy, at a dose rate of 10 kGy/pass.

Gel fraction measurements

Gel content in irradiated samples was estimated after removing the soluble part by extraction in boiling chloroform for 48 h. The gel fractions were calculated as the ratio of the weight of the dried gel to the initial weight of the polymer.

TABLE II
Structure of Polyfunctional Monomers Used in This Work

Monomer name	Structure	M_w
Trimethylallyl isocyanurate (TMAIC) (Nipon Kasei Co., Ltd.)		291
Poly(ethylene glycol dimethyl methacrylate) (2G) (Shin-nakamura chemical Co., Ltd.)	$\text{H}_2\text{C}=\text{C}(\text{R})-\text{C}(\text{O})-\text{O}-(\text{CH}_2\text{CH}_2\text{O})_n-\text{C}(\text{O})-\text{C}(\text{R})=\text{CH}_2$	242 ($n = 2$)
Poly(ethylene glycol dimethyl methacrylate) (4G) (Shin-nakamura chemical Co., Ltd.)	$\text{H}_2\text{C}=\text{C}(\text{R})-\text{C}(\text{O})-\text{O}-(\text{CH}_2\text{CH}_2\text{O})_n-\text{C}(\text{O})-\text{C}(\text{R})=\text{CH}_2$	330 ($n = 4$)
Trimethylolpropane trimethacrylate (TMPT) (Shin-nakamura chemical Co., Ltd.)	$\text{H}_3\text{C}-\text{C}(\text{H}_2)-\text{C}(\text{CH}_3)(\text{CH}_2\text{OOC}-\text{C}(\text{CH}_3)=\text{CH}_2)_3$	338
Tetramethylolmethane tetraacrylate (A-TMMT) (Shin-nakamura chemical Co., Ltd.)	$\text{C}-(\text{CH}_2\text{OOC}-\text{HC}=\text{CH}_2)_4$	352

$$\text{Gel fraction (\%)} = [(W_g/W_i)] \times 100\%$$

where W_g is the weight of the gel and W_i is the weight of the original sample.

Enzymatic degradation test

Enzymatic degradation of samples was carried out by determining the rate of their solubilization in a solution according to ASTM D 1924-63 and ASTM D 2676. The samples were immersed in phosphate buffer solution (pH 7.4) with the composition of the reaction mixture as follows:

0.2M phosphate buffer (pH 7.0) 4.0 mL
lipase AK enzyme (10 mg/mL) 1.0 mL
0.1% surfactant (MgCl_2) 1.0 mL

Thin PBS films, each with dimensions of $10 \times 10 \times 0.1$ mm and weighing about 9 mg, were dried in an oven at 40°C for 24 h. The films were put into test tubes, to which 5.0 mL of the reaction mixture was added, and were incubated at 55°C with shaking for various lengths of time. Then the films were taken out of the test tubes, washed with distilled water and methanol, and dried in an oven at 40°C to a constant weight. The progress of enzymatic degradation was expressed as the percent weight loss of the samples.

Soil burial test

The soil burial test was performed in a plastic trough that was 57 cm long, 17 cm wide, and 14 cm high. The soil consisted of composted topsoil, black garden soil, and pond sludge in equivalent ratios maintained at a pH of 7 and a moisture content of about 40%. The 0.50-mm-thick dumbbell-cut samples, were buried about 3 cm from the soil surface for burial durations from ranging from 1 to 6 months. After each interval the samples were removed from the soil and wiped by tissue paper, cleaned with distilled water, and dried at 40°C in an oven to a constant weight.

Analytical measurements

A thermomechanical analyzer (Shimadzu TMA-50) was used for evaluation of the heat stability. A thin PBS film $5 \times 2 \times 0.1$ mm in size was fixed to the sample holder and heated from 25°C to 200°C in a nitrogen atmosphere under a constant load of 0.5 g with a heating rate of $10^\circ\text{C}/\text{min}$.

The heat resistance of the samples was analyzed by a creep test using a burner. The PBS sheets, with sample dimensions of $0.5 \times 10 \times 50$ mm, were hung out with an applied stress of 0.23 MPa, and the deformation of the sample (in elongation) was measured at

different temperatures for 3 min. After measurement the stress was reduced to 0.02 MPa in order to estimate the recovery and rearrangement of the unloaded material.

Thermal stability of the samples was evaluated by a standard autoclave test using a Pasolina IST-150–type autoclave (Chioda, Japan). For measurement, the PBS dumbbell sheets were hung out without any applied load at 120°C for 1 h with a pressure of 0.18 MPa.

Dynamic viscoelastic properties were measured by using a torsion-pendulum-type viscoelastometer (Rhesca RD-1000AD) at 0.2–1 Hz in a temperature range of -150°C to 150°C . For analysis samples $100 \times 10 \times 0.5$ mm in size were used.

Melting temperatures were measured using a differential scanning calorimeter (DSC-7, Perkin–Elmer) in a nitrogen flow atmosphere with a heating rate of $10^\circ\text{C}/\text{min}$.

RESULTS AND DISCUSSION

Crosslinking behavior of PBS

Poly(butylene succinate) (PBS) is known to be a crosslinkable polymer. When the polymer is exposed to an electron beam from an accelerator, the molecular weight of the polymer increases and forms an insoluble fraction called a gel fraction. This process affects the mechanical, physical, and chemical properties of the polymer, thereby changing the quality of the original material.

The gel contents of PBS1 in the presence of different monomers (1% weight of monomer) versus dose of irradiation are shown in Figure 1. The gel fraction of irradiated pure PBS1 reached only 18.23% at an irradiation dose of 50 kGy. For mixed samples, however, the gel content increased significantly. By adding 1% TMAIC, PBS1 with a gel fraction of 82.9% was synthesized (at the same 50 kGy irradiation dose). Further, irradiation of PBS1 up to a dose of 100 kGy did not change the gel content. Compared with other monomers, the gel fraction of irradiated PBS1 containing A-TMMT, TMPT, 2G, or 4G (see Table II) did not reach a high value of irradiated PBS1–TMAIC blend.

TMAIC yielded a higher gel content in radiation crosslinking of PBS1 in comparison with the other monomers, perhaps because TMAIC had additional allylic hydrogens as α -methyl groups. Additional allylic hydrogens cause no steric hindrance, which may be a result of the transition-state formation of the monomer chain transfer reaction by the abstraction of allylic hydrogens of α -methyl groups, which are at the δ site away from the isocyanurate group.¹² Also, TMAIC has a cyclic unit that achieves a greater three-dimensional network by irradiation than that of aliphatic polyfunctional monomers. It is well known that, when bulk polymers are irradiated, the dominant

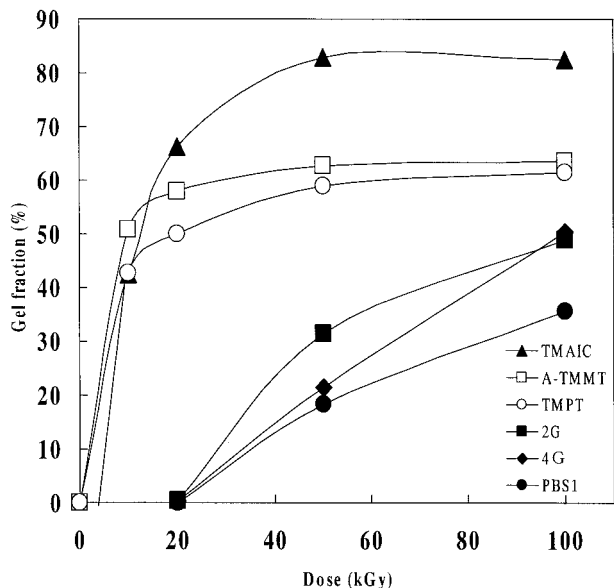


Figure 1 Gel fraction–dose curves for PBS1 with 1% weight of different monomers (TMAIC, 3.44×10^{-3} mol %; A-TMMT, 2.84×10^{-3} mol %; TMPT, 2.96×10^{-3} mol %; 2G, 4.13×10^{-3} mol %; 4G, 3.03×10^{-3} mol %).

reactions and the final products depend strongly on the transport of excitation energy (and charge), on the strength of individual chemical bonds, and on the geometrical arrangement of the polymer molecules. Usually, in most polymers, one of two general processes dominates: crosslinking or degradation.¹³

Figure 2 shows the gel contents of irradiated PBS1 with different amounts of TMAIC. It can be seen that, at irradiation doses of 10 and 20 kGy, PBS1 samples containing 0.2%, 0.5%, and 1% TMAIC had a higher

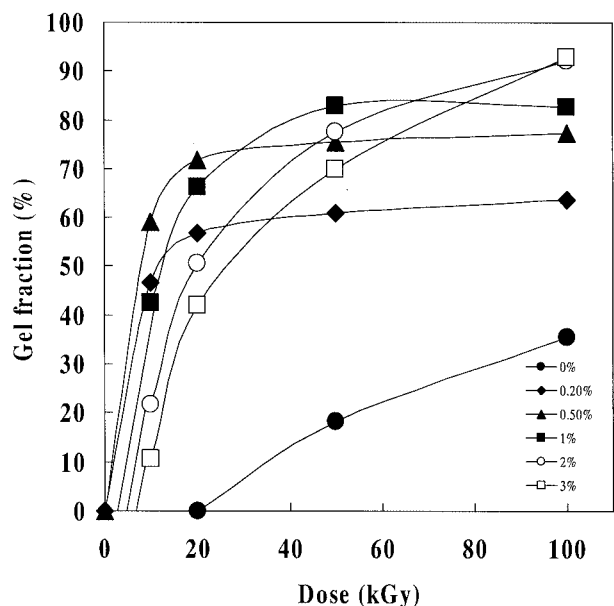


Figure 2 Gel fraction–dose curves for PBS1 with TMAIC in different concentrations (wt %).

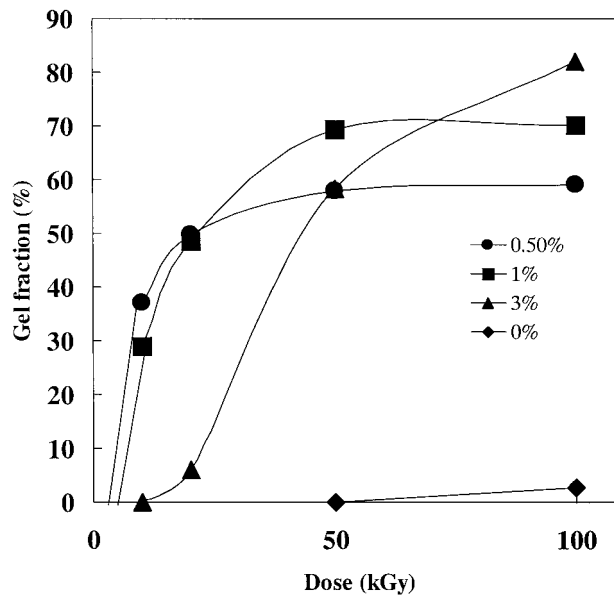


Figure 3 Gel fraction–dose curves for PBS2 with TMAIC in different concentrations (wt %).

gel content than those of PBS1 containing 2% and 3% TMAIC; whereas at an irradiation dose of 100 kGy, PBS1 containing 2% and 3% TMAIC had a higher gel content than those of PBS1 containing 0.2%, 0.5%, and 1% TMAIC. This is perhaps because of insufficient production of polymer radicals (reacting with a monomer to support the process of crosslinking) at a dose of 10–20 kGy for 2% or 3% TMAIC. Therefore, the monomer reacted only with one side of the layer of polymer. The phenomenon also might be stem from TMAIC having additional allylic hydrogens as α -methyl groups. The resulting propagation reaction that inversely may be sterically more hindered as a result of more steric crowding (which is typical for growing polymer radicals having a α -methyl group).¹² In contrast, an irradiation dose of 100 kGy with the addition of 2% and 3% TMAIC led to a gel content higher than with 0.2%, 0.5%, and 1% TMAIC. This is probably because PBS1 produced a sufficiently large amount of polymer radicals to react with monomers at a dose of 100 kGy. In this condition all monomers can be used to initiate effective crosslinking in the polymer. The yield of gel formation of PBS1 containing 0.2%; 0.5%, and 1% TMAIC at an irradiation dose of 50 kGy was not significantly different from that at an irradiation dose of 100 kGy. This might be because of all the monomer being used to form the crosslinking when the sample was irradiated at 50 kGy.

Figure 3 shows the gel contents of irradiated PBS2 with different concentrations of TMAIC. It clearly can be seen that TMAIC enhanced the yield of gel formation of the irradiated PBS2. At doses of 10 and 20 kGy, PBS2 containing 0.5% and 1% TMAIC resulted in a higher gel content than did PBS2 containing 3%

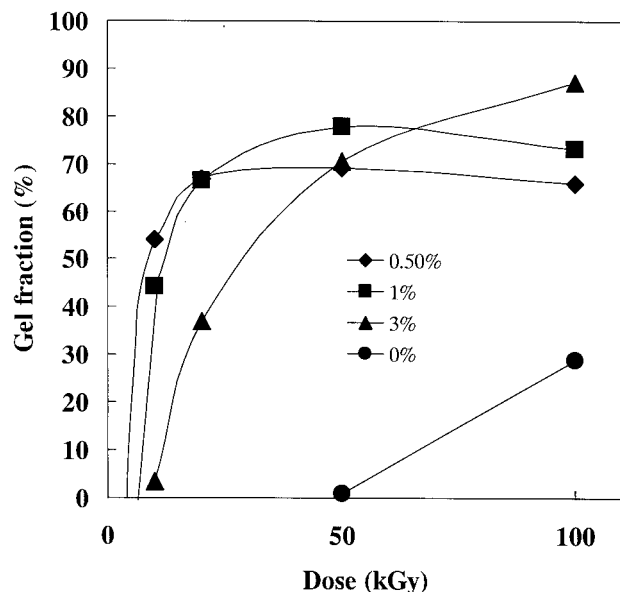


Figure 4 Gel fraction–dose curves for PBS3 with TMAIC in different concentrations (wt %).

TMAIC. However, at a dose of 100 kGy the addition of 3% TMAIC in PBS2 yielded a higher gel content than for that with 0.5% and 1% TMAIC. These phenomena are the same as that for irradiated PBS1 containing TMAIC, as mentioned above. Similar behavior was found for PBS3 (Fig. 4).

From Figures 2, 3, and 4 it can be deduced that irradiated PBS1 yielded more gel in the presence of TMAIC, as compared with those of PBS2 and PBS3. It is known that this phenomenon is affected remarkably by the crystallinity of the polymeric material. The recombination reactions that occurred during irradiation were predominantly in the amorphous regions of the sample. This is because of the higher mobility of the molecular chains in the amorphous part. PBS1 has a higher molecular weight ($M_w = 2.96 \times 10^5$) and lower melting point than do PBS2 and PBS3 (as shown in Table I). Irradiated PBS1 therefore exhibited more crosslinking than did PBS2 and PBS3.

Heat stability of crosslinked PBS1

To prevent flow of the polymer during processing at high temperatures, high melt strength is required. The heat stability of irradiated PBS1 containing 1% TMAIC was observed by measuring the deformation (in elongation) of the sample using a thermomechanical analyzer.

The plots of elongation of the irradiated PBS1, with and without 1% TMAIC and irradiated at 50 kGy, against temperature are shown in Figure 5. Irradiated PBS1 without TMAIC revealed high elongation its melting point, whereas PBS1 containing 1% TMAIC had low elongation, even at a high temperature, and

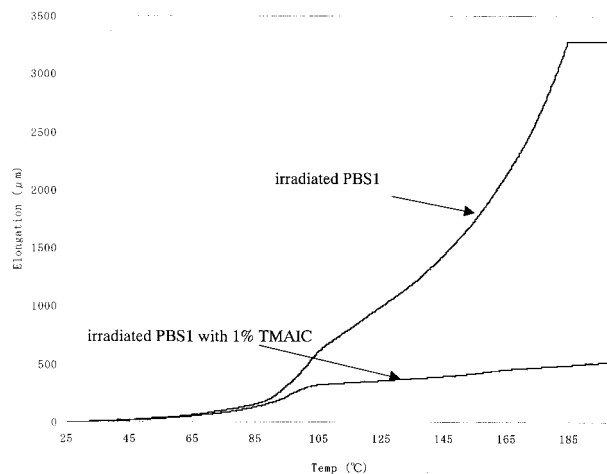


Figure 5 Thermomechanical analysis of irradiated PBS1.

was more stable at high temperatures. This is because TMAIC has a cyclic unit that strengthens the heat stability. It was reported by Ki et al. that both an aromatic unit and a cyclic unit enhance the thermal stability of aliphatic-aromatic random copolyesters.¹⁵

Heat resistance of irradiated PBS1 was determined by measuring the deformation of loaded PBS1 at different temperatures beyond the melting point. A stress of 0.23 MPa was evaluated as a load applied to a cross-sectional area of the sample. The relationship between time and deformation (in elongation) of irradiated PBS1 with and without TMAIC is shown in Figure 6. It can be seen that at 100°C, pure PBS1 irradiated at a dose of 50 kGy had a significantly higher elongation compared with irradiated PBS1 containing 1% TMAIC. The irradiated pure PBS1 ruptured immediately at a temperature of 120°C, whereas PBS1 containing TMAIC was slightly elongated, and after reducing the load (remaining stress was 0.02 MPa), it recovered. The recovery of the deformed sample is probably a result of rearrangement of the crys-

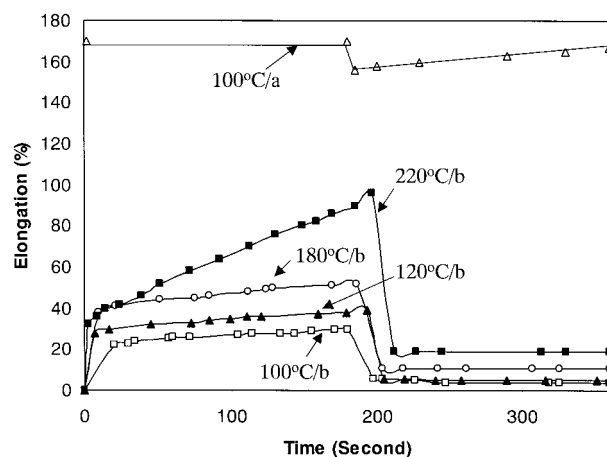


Figure 6 Creep test of irradiated PBS1 at irradiation dose of 50 kGy: (a) PBS1; (b) PBS1 containing 1% TMAIC.

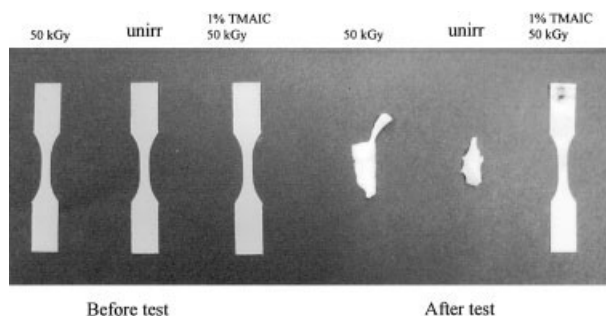


Figure 7 Autoclave test of irradiated PBS3 at temperature of 120°C with a pressure of 0.18 MPa for 1 h.

talline part of the polymer structure. A further increase in temperature also led to further deformation. In contrast, irradiated PBS1 containing 1% TMAIC did not break even at 220°C and remained complete for the duration of the test (6 min). This is also probably because the addition of cyclic units into the main chain enhances heat resistibility of random copolyesters after radiation-induced crosslinking.

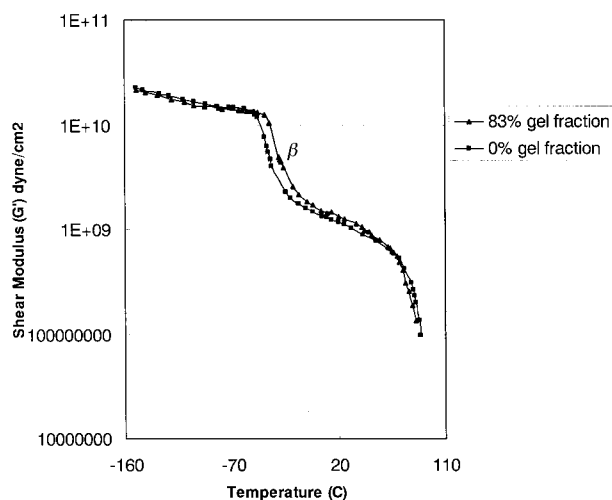
Heat stability was investigated by using an autoclave test at 120°C for 1 h with a pressure of 0.18 MPa. In Figure 7 the results of the autoclave test of PBS3 are shown: unirradiated PBS3 broke, pure PBS3 irradiated at 50 kGy melted, and irradiated PBS3 containing 1% TMAIC remained stable (no deformation was observed). From these experimental results it can be deduced that the heat stability of the sample depended strongly on the yield of gel formation. The addition of TMAIC, which is a polyfunctional monomer into PBS, produced a network structure in the main chains, resulting in an increase of sample melt strength. Similar results have been obtained for polyethylene and polypropylene, and it has been reported that sufficiently high melt strength of polyethylene and polypropylene is achieved by a adding long branch structure into the main chains.¹⁶ Analogous with these data is the enhanced heat stability of PBS blended with 1% TMAIC after radiation-induced crosslinking.

Dynamic viscoelastic properties

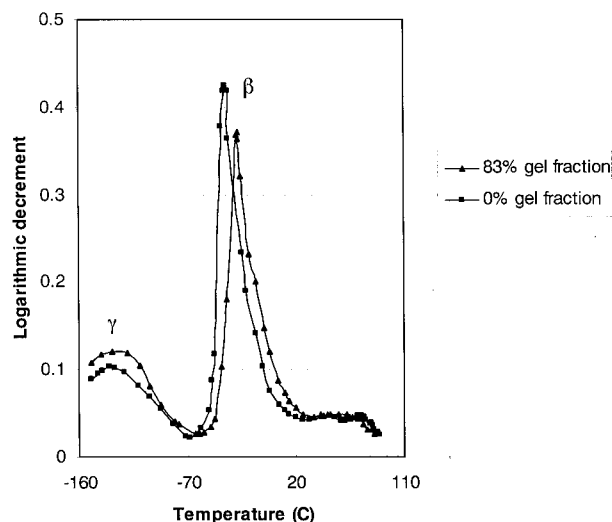
Dynamic viscoelastic properties were analyzed for both the uncrosslinked and crosslinked PBS1 samples. The difference in the deterioration mechanism between uncrosslinked and crosslinked PBS1 was investigated. The crosslinking effects on molecular motion, revealed by viscoelastic measurement of semicrystalline PBS1, are discussed. Figure 8(a) shows the temperature dependence of the shear modulus (G') of the irradiated PBS1 sample containing 1% TMAIC (83% gel) and the original PBS1 sample (without gel). At temperatures below the β relaxation, the shear moduli of uncrosslinked and crosslinked samples were not

significantly different, whereas at the β relaxation the crosslinked sample shifted to a temperature higher than the uncrosslinked sample. The decrease in shear modulus in the β -relaxation region was scarcely affected by the degree of crosslinking in the sample. It was found that the onset temperature for the fall in shear modulus of the crosslinked PBS1 sample occurred slightly lower than that of the uncrosslinked PBS1 sample because of the decrease in the melting point.

Figure 8(b) shows the logarithmic decrease (mechanical loss) in the crosslinked (83% gel) and uncrosslinked samples. Two discrete mechanical loss peaks, γ relaxation and β relaxation, can be observed. The γ relaxation of polymers such as polyethylene, poly(methyl methacrylate), and polystyrene can be attributed to the molecular motions of short branching or the local motion of chains existing on the crystallite surface, or both. For rigid aromatic polymers the low-



(a)



(b)

Figure 8 (a) Temperature dependence of shear modulus of PBS1 sample; (b) Temperature dependence of mechanical loss of PBS1 sample.

temperature mechanical relaxation can be attributed to the local motion of aromatic units in the main chain.^{17–19} The γ -relaxation peak of the crosslinked sample broadened, which also could be a result of the local motion of cyclic units in the main chain, and the β -relaxation shifts to a higher temperature could be from an increasing glass-transition temperature (T_g). It is known that, in highly crosslinked materials, the T_g and the modulus at the rubbery plateau are related to the crosslink density.²⁰ The glass-transition temperature shifts higher, and the modulus increases with an increase in crosslink density.

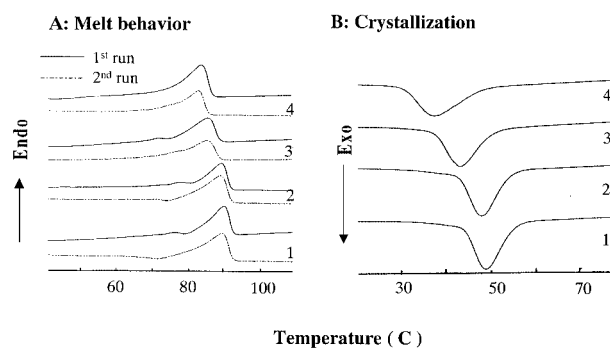
An increasing glass-transition temperature indicates improved heat stability resulting from the need for more heat to increase the molecular motion of the sample. These facts show that the thermal resistance of the PBS1 sample was greatly improved by irradiation in the presence of 1% TMAIC.

Melting and crystallization behaviors of crosslinked PBS

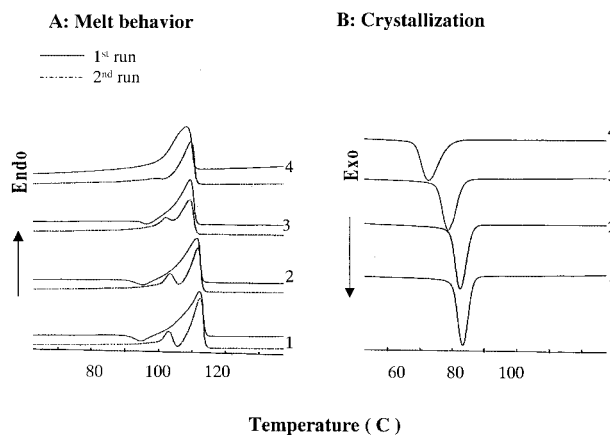
The presence of TMAIC in PBS samples enhanced the yield of gel formation by irradiation. To elucidate the mechanism, an effect of crosslinking yield on a sample's morphology was investigated. Figure 9(a) shows the melting and crystallization behaviors of PBS1 and its crosslinked form. DSC second-run heating was performed directly after the cooling of the first run. The melting point of PBS1 samples in the second run was slightly lower than that in the first run because of the short time available to form crystallites (T_c : 49°C), so that the crystallization was not completed. The increasing crosslinking yield led to a lowering of the melting point of PBS1, as shown in (A) of Figure 9(a).

Figure 9(b) shows melting and crystallization behaviors of PBS3 and its crosslinked form. The melting point of PBS3 samples in the second run was slightly higher than that in the first run as a result of having sufficient time to form crystallites (T_c : 83°C); crystallization was completed in this case. In a manner similar to PBS1 (and also to crosslinked polyethylene), increasing the crosslinking yield led to a lowering of the melting point of PBS3, as shown in (A) in Figure 9(b). Melting temperature is often discussed on the basis of crystallite size, so that a decrease in melting temperature is considered an indication of a decrease in crystallite size.²¹ A decrease in crystallite size without a change in distortion parameter and unit cell dimension possibly suggests that crystals are degraded in the vicinity of the crystalline–noncrystalline interface. The crystallization temperature becomes broader and shifts lower with increasing gel content, as shown in Figure 9(a,b). It is understood that crosslinking has a remarkable influence on crystallization.

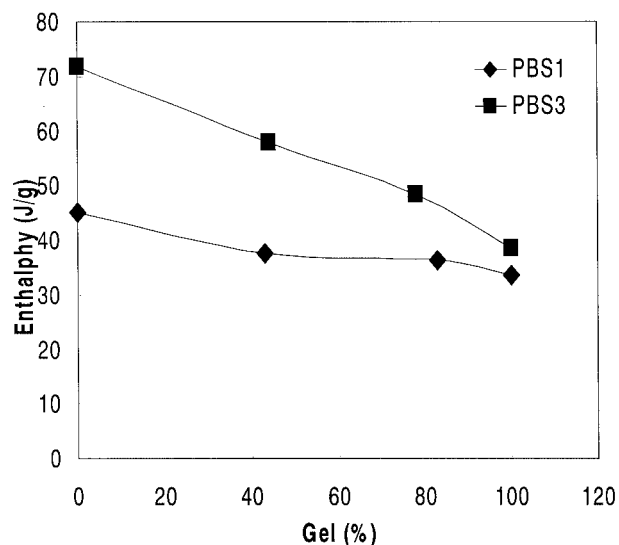
Figure 9(c) shows the enthalpy of PBS1 and PBS3 versus gel contents. It can be seen that a decrease in



(a)



(b)



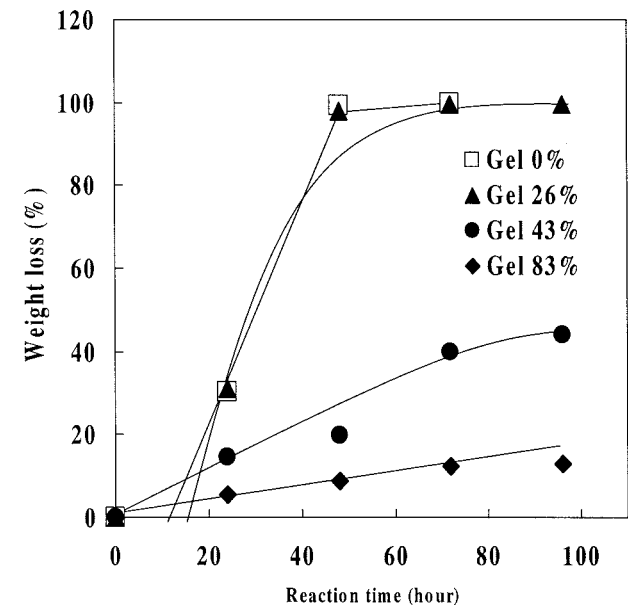
(c)

Figure 9 (a) DSC curves of irradiated PBS1 in the presence of 1% TMAIC: 1—unirradiated, 2—43% gel fraction, 3—83% gel fraction, 4—100% gel fraction; (b) DSC curves of irradiated PBS3 in the presence of 1% TMAIC: 1—unirradiated, 2—44% gel fraction, 3—78% gel fraction, 4—100% gel fraction; (c) enthalpy–gel fraction curves for PBS1 and PBS3.

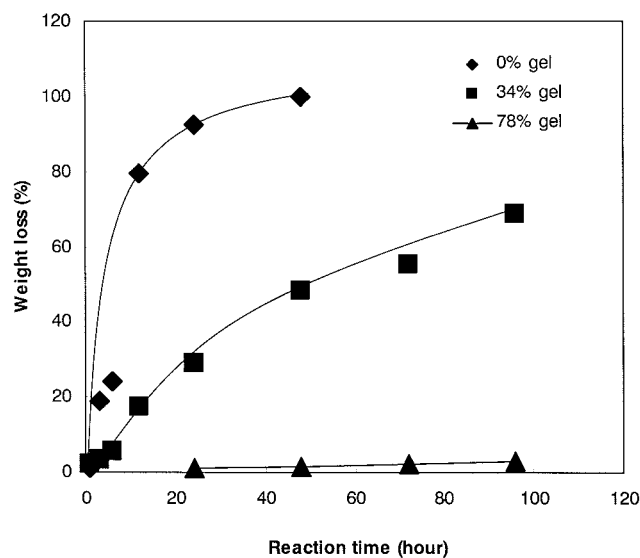
the sample enthalpy occurred when the gel content was increased. The enthalpy of the polymer depended on the amount of crystallite. Evidently, increasing the yield of gel formation decreased the amount of crystallites in the PBS samples.

Biodegradation test of crosslinked PBS

An enzymatic test is a convenient method for evaluating the rate of biodegradability of polymers over a



(a)



(b)

Figure 10 (a) Weight loss of PBS1 in enzymatic degradation test (reaction temperature = 55°C, enzyme concentration = 1.67 mg/mL, sample thickness = 0.1 mm); (b) weight loss of PBS3 in enzymatic degradation test (reaction temperature = 55°C, enzyme concentration = 1.67 mg/mL, sample thickness = 0.1 mm).

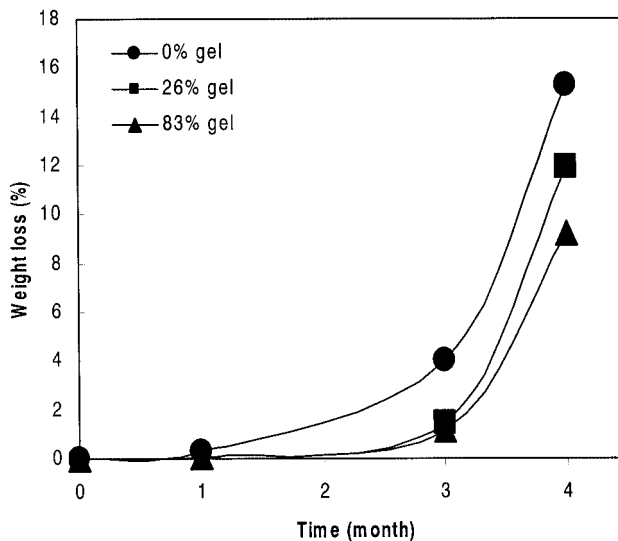


Figure 11 Weight loss of PBS1 in soil burial degradation test.

short time. It has been reported that esterase and lipase enzymes cause degradation of poly(butylene succinate).²² In this study we used lipase AK for analyzing the biodegradation of PBS.

Figure 10(a) shows that PBS1 samples without gel fraction degraded completely at 48 h of immersion in the enzyme bath, PBS1 containing 26% gel degraded completely at 72 h of immersion, whereas PBS1 containing 43% and 83% gel degraded 44.08% and 12.9%, respectively, at 96 h of immersion in the enzyme bath. It can be deduced that PBS1 with a high gel fraction degraded more slowly than did PBS1 with a low gel fraction, indicating that the increase in weight loss was caused by the more soluble fraction of the sample. The important factors affecting the biodegradability of polymers are structure, physical properties such as the type and degree of crystallinity, orientation, and mechanical properties.²³⁻²⁸ Consequently, the observed decrease in the biodegradation rate in the sample with increasing gel content could be tentatively attributed to the parallel steady increase of the overall crystallinity and/or mechanical properties. From the experimental results it can be deduced that the biodegradability of irradiated PBS1 containing 1% TMAIC was strongly affected by the yield of gel formation in the sample. Similar behavior was noted for PBS3 [Fig. 10(b)].

A soil burial test is a standard test for biodegradability of polymer samples by bacteria in soil. The effect of gel content on biodegradability is shown in Figure 11. It can be seen that the weight loss of the samples was strongly affected by the gel content in the samples. This behavior is similar to that of enzymatic degradation (see above). However, there are several important factors that may affect the results of the soil burial test, such as temperature, humidity, pH, oxygen

concentration, number of active microorganisms, and availability of mineral nutrients. To evaluate their effects is a complex task, and a set of coordinated parameters needs to be followed. This was not performed in this work.

CONCLUSION

Radiation crosslinking of poly(butylene succinate) of different molecular weights in the presence of five polyfunctional monomers was investigated. Of the polyfunctional monomers, it was found that trimethylalyl isocyanurate (TMAIC) was better at enhancing the formations of gel in the irradiated PBS than did the other polyfunctional monomers. The amount of TMAIC blended with PBS influenced the amount of gel resulting after irradiation. Crosslinking took place most efficiently for PBS that contained 1% TMAIC. It was found that crosslinked PBS in the presence of TMAIC exhibited considerably improved heat stability. The biodegradation rate of the modified polymers was slightly diminished with an increasing gel fraction; however, the polymers were still biodegradable.

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