

# Synthesis, Properties, and Self-Assembly of Poly(benzyl ether)-*b*-Polystyrene Dendritic–Linear Polymers

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**ABSTRACT:** Poly(benzyl ether)-*b*-polystyrene dendritic–linear polymers were successfully synthesized using a dendritic chloric poly(benzyl ether) ( $G_1$ -Cl,  $G_2$ -Cl, and  $G_3$ -Cl) as the macroinitiator through the atom transfer radical polymerization process. The structure and properties of the resultant polymers were characterized by gel permeation chromatography,  $^1\text{H-NMR}$ , Fourier transform IR, thermogravimetric analysis, and differential scanning calorimetry. It was found that the temperature, reaction time, molar ratio of the macroinitiator to styrene, and the generation number of the macroinitiator have significant effects on the molecular weights, conversion, and polydispersities of the resulting

polymers. These dendritic–linear block polymers had very good solubility in common organic solvents at room temperature. The terminal group (dendritic segments) of the polymers can affect their thermal stability. These dendritic–linear polymers after self-assembling in selective solvents (chloroform/acetone) formed core–shell micelles. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 98: 1106–1112, 2005

**Key words:** diblock polymer; atom transfer radical polymerization; dendritic poly(benzyl ether); dendritic–linear polymers; polystyrene

## INTRODUCTION

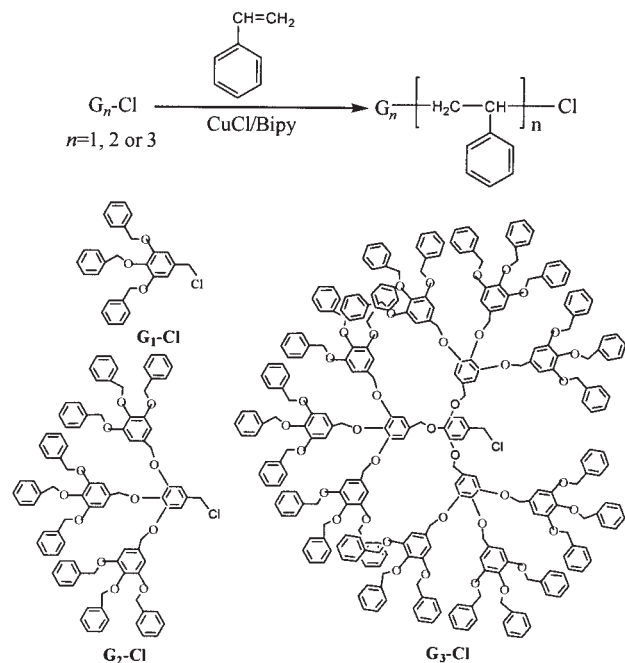
The synthesis of well-defined structure homopolymers or block polymers, which have a narrow molecular weight distribution, a predictable molecular weight, and a controlled architecture and composition, is one of the most meaningful and challenging works in the field of polymer chemistry.<sup>1,2</sup> Well-defined block polymers can show a series of excellent and special properties. In addition, they have been used to research the solution theory and have also been much advanced in industrial applications.<sup>2</sup> Meanwhile, rapidly increasing attention has been paid to dendrimers because of their highly regular three-dimensional structures and potential applications in fields such as diagnostics, host–guest chemistry, supramolecular chemistry, and catalysts. Furthermore, dendritic macromolecules are suitable building blocks for the design and synthesis of novel functional materials.<sup>3</sup> Polymers bearing dendritic and linear blocks might provide new properties for the polarity and high incompatibilities between dendritic and linear segments. For example, Cornelissen et al.<sup>4</sup> demonstrated the self-assembly behavior from rod–coil block

copolymers combining a flexible, bulky, apolar dendritic carbosilane segment and a rigid, helical polyisocyanide block. In chloroform these block copolymers respond to the addition of silver ions by the generation of nanowires consisting of [111] oriented crystalline silver. Zubarev et al.<sup>5</sup> reported dendron rod–coil molecules self-assemblies via hydrogen bonding and  $\pi$ – $\pi$  stacking interactions into well-defined nanoribbons. Zhao et al.<sup>6</sup> synthesized dendritic–linear diblock copolymers with a dendritic Fréchet type polyarylether head group. Until now, the methods of grafting reactions,<sup>7</sup> “living” free-radical approach,<sup>8</sup> “living” anionic approach,<sup>9</sup> cationic approach,<sup>10</sup> and atom transfer radical polymerization (ATRP)<sup>11–16</sup> have been applied to obtain these dendritic–linear diblock polymers. Among these approaches, ATRP offers a more portable way to form well-defined and predictable multicomponent polymer structures.<sup>17–21</sup> Dendritic–linear polymers with well-defined architectures are considered promising candidates for functional supramolecular materials. The incorporation of dendritic blocks into conventional linear polymers makes it possible to understand the self-assembly behavior of dendritic–linear block copolymers in selective solvents.<sup>22</sup>

Here, we report the synthesis of dendritic–linear diblock polymers based on strong cohesive linear polystyrene (PS) and strong steric effect dendritic poly(benzyl ether) blocks. In addition, their aggregation behaviors are investigated with transmission electron microscopy (TEM).

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**Scheme 1** The synthesis procedure of G<sub>n</sub>-b-PS diblock polymers.

## EXPERIMENTAL

### Materials

Styrene (St) from Shanghai No. 1 Chemical Reagent Factory was distilled under reduced pressure over calcium hydride. 2,2'-Bipyridyl (Bipy), an analytical reagent, was used as obtained from Shanghai No. 1 Chemical Reagent Factory. Copper(I) chloride (CuCl, analytical grade) was purified by stirring in acetic acid, washing with methanol, and then drying under reduced pressure. All other reagents were analytical grade and used as received.

### Synthesis of dendritic poly(benzyl ether) macroinitiator

The dendritic chloric poly(benzyl ether)s (G<sub>1</sub>-Cl, G<sub>2</sub>-Cl, and G<sub>3</sub>-Cl) were prepared by the literature method.<sup>23,24</sup>

### Synthesis procedure of G<sub>n</sub>-b-PS block polymers

In a typical synthesis, Bipy, CuCl, G<sub>n</sub>-Cl (2 : 1 : 1 molar ratio), and St with a certain molar ratio were added to a dry tube. The tube was cycled between a vacuum and nitrogen more than 10 times to remove the oxygen. Then, the tube was sealed and placed in a preheated, thermally regulated oil bath at a certain temperature. After a certain period of polymerization, the tube was removed from the oil bath and allowed to cool for a few minutes. Then, several milliliters of chloroform (CHCl<sub>3</sub>) were added to the tube, and the mixture was maintained for a period of time at room temperature to complete the dissolution of the polymer. The resulting solution was passed through a short column containing basic aluminum oxide to remove most of the Cu complex. The excess CHCl<sub>3</sub> was removed under reduced pressure and the product was precipitated from a large amount of methanol, filtered, and dried under a vacuum to constant weight.

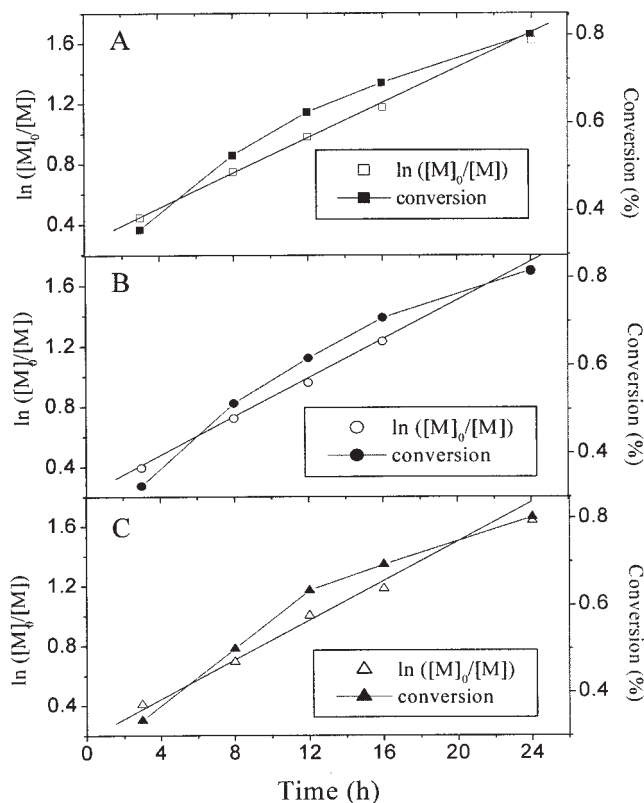
### Self-assembly of dendritic-linear block polymers

Extremely dilute solutions containing G<sub>n</sub>-b-PS (as low as 0.20 wt %) in chloroform were first prepared at room temperature (25°C). Then, acetone was added into these solutions with a 1 : 1 (v/v) ratio and without sonication, mechanical stirring, or other forms of applied energy. Samples for TEM measurements were prepared by aerosol spraying a dilute solution con-

**TABLE I**  
Results of Polymerization of St Using G<sub>n</sub>-Cl as Macroinitiator

Run	Macroinitiator	Time (h)	M <sub>n</sub> (calcd) × 10 <sup>-4</sup>	M <sub>n</sub> (GPC) <sup>c</sup> × 10 <sup>-4</sup>	M <sub>n</sub> (NMR) × 10 <sup>-4</sup>	PD	Conversion (%)	f <sup>f</sup>
1	G <sub>1</sub> -Cl	24	1.30	1.37	1.35	1.38	80.2	0.96
2	G <sub>1</sub> -Cl	12	1.02	1.14	1.07	1.32	62.4	0.95
3	G <sub>1</sub> -Cl	3	0.60	0.81	0.68	1.35	35.7	0.89
4	G <sub>2</sub> -Cl	24	1.41	1.44	1.54	1.39	81.6	0.92
5	G <sub>2</sub> -Cl	12	1.10	1.16	1.21	1.42	61.6	0.91
6	G <sub>2</sub> -Cl	3	0.65	0.95	0.73	1.35	32.5	0.88
7	G <sub>3</sub> -Cl	24	1.68	1.97	1.77	1.21	80.5	0.92
8	G <sub>3</sub> -Cl	12	1.45	1.48	1.58	1.36	63.3	0.90
9	G <sub>3</sub> -Cl	3	0.95	1.10	1.09	1.40	33.5	0.87

Polymerization conditions [St]<sub>0</sub>/[G<sub>n</sub>-Cl]<sub>0</sub>/[CuCl]/[Bipy] = 150 : 1 : 1 : 2; 100°C, in bulk; M<sub>n</sub> (calcd) = ([St]<sub>0</sub> × conversion × MW<sub>St</sub>)/[macroinitiator]<sub>0</sub> + MW<sub>macroinitiator</sub> (g mol<sup>-1</sup>); M<sub>n</sub> (GPC) determined by GPC in THF at 25°C; M<sub>n</sub> (NMR), obtained by a comparison of the signal intensity of ArCH<sub>2</sub>O and PhCH<sub>2</sub>O protons in the macroinitiator (4.8–5.2 ppm) with that of CH and CH<sub>2</sub> protons in the styrene monomer unit (1.7–2.6 ppm); PD, (polydispersity) = M<sub>w</sub>/M<sub>n</sub> (GPC); f, the initiator efficiency was calculated from f = M<sub>n</sub> (calcd)/M<sub>n</sub> (NMR).<sup>5</sup>



**Figure 1** The time dependence of  $\ln([M]_0/[M])$  and conversion for the ATRP of St in bulk at 100°C. Polymerization conditions:  $[St]_0/[G_n-Cl]_0/[CuCl]/[Bipy] = 150 : 1 : 1 : 2$ , (A)  $n = 1$ , (B)  $n = 2$ , and (C)  $n = 3$ .

taining dendritic-linear polymers (ca. 50 mL, 1.0 mg/mL) onto 200-mesh gilder copper TEM grids.

### Characterizations

$^1\text{H-NMR}$  spectra were recorded with an AVANCE DMX-500 NMR spectrometer by using tetramethylsilane as an internal standard at room temperature. The gel permeation chromatography (GPC) measurements were carried out on a Waters 201 with a  $\mu$ -styragel column and tetrahydrofuran as an eluent, and the molecular weight was calibrated with standard PS. Differential scanning calorimetry (DSC) measurements were carried out on a DS822 with a 10°C/min heating rate from 40 to 200°C under a nitrogen atmosphere, relative to indium standards. The IR spectra were recorded on a Jasco IR-700 IR spectrophotometer. Thermogravimetric analysis (TGA) measurements were performed on a Netzsch STA 409 PG/PC instrument (Germany) at a 20°C/min heating rate from 40 to 500°C in a flow of nitrogen. TEM micrographs were obtained on a Jeol model 1200EX instrument operated at a 160-kV accelerating voltage. The molar conversion was obtained on the basis of the weight and copolymer composition.

## RESULTS AND DISCUSSION

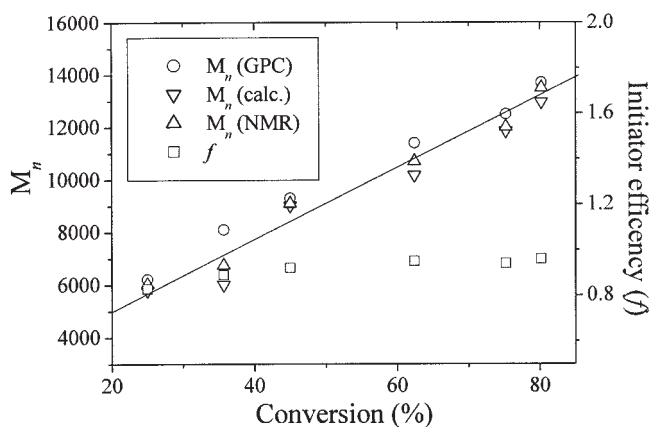
The macroinitiator and the synthetic route of well-defined dendritic-linear block polymers via ATRP are depicted in Scheme 1.

### Polymerization analysis

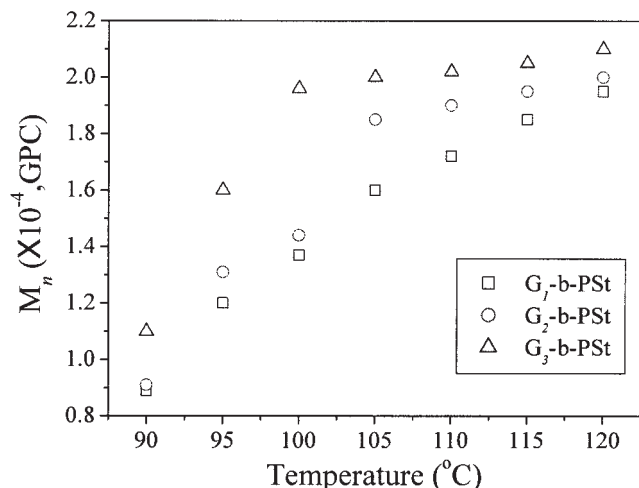
The polymerizations of St were carried out in sealed tubes using CuCl as a catalyst and Bipy as a ligand in the presence of a macroinitiator ( $G_1\text{-Cl}$ ,  $G_2\text{-Cl}$ , and  $G_3\text{-Cl}$ ) at a certain temperature. When using a dendritic as the macroinitiator, St was polymerized successfully by ATRP to give dendritic-linear diblock polymers with relatively narrow polydispersity ( $1.20 < M_w/M_n < 1.45$ ). The polymerization results of St using dendritic  $G_n\text{-Cl}$  as the macroinitiator at 100°C are provided in Table I. Using the CuCl/Bipy catalytic system, relatively high conversion and molecular weight were obtained.

For the ATRP of St initiated with the  $G_n\text{-Cl}$ /CuCl/Bipy catalytic system at 100°C and  $[St]_0/[G_n\text{-Cl}]_0/[CuCl]/[Bipy]$  ratio of 150 : 1 : 1 : 2, the time dependence of  $\ln([M]_0/[M])$  and conversion are shown in Figure 1. The plot of  $\ln([M]_0/[M])$  versus the polymerization time is a straight line until a higher conversion of ~80% is reached, which indicates the polymerization of St in bulk is first-order polymerization kinetics. This means that the concentration of propagating radicals is constant during the polymerization procedure.<sup>25</sup>

The effect of the polymerization conversion on the number-average molecular weight ( $M_n$ ) and the initiator efficiency ( $f$ ) is demonstrated in Figure 2. Note that the plots of the molecular weights determined by GPC [ $M_n(\text{GPC})$ ] or by  $^1\text{H-NMR}$  [ $M_n(\text{NMR})$ ] versus the polymerization conversion are linear and nearly consistent with the theoretical calculated values [ $M_n$



**Figure 2** The dependence of the  $M_n$  and initiator efficiency ( $f$ ) of  $G_1\text{-}b\text{-PS}$  on the polymerization conversion. Polymerization conditions:  $[St]_0/[G_1\text{-Cl}]_0/[CuCl]/[Bipy] = 150 : 1 : 1 : 2$ , 110°C, in bulk.



**Figure 3** The variation of the  $M_n$  with the polymerization temperature. Polymerization conditions:  $[St]_0/[G_n-Cl]/[CuCl]/[Bipy] = 350 : 1 : 1 : 2$ , 12 h, in bulk.

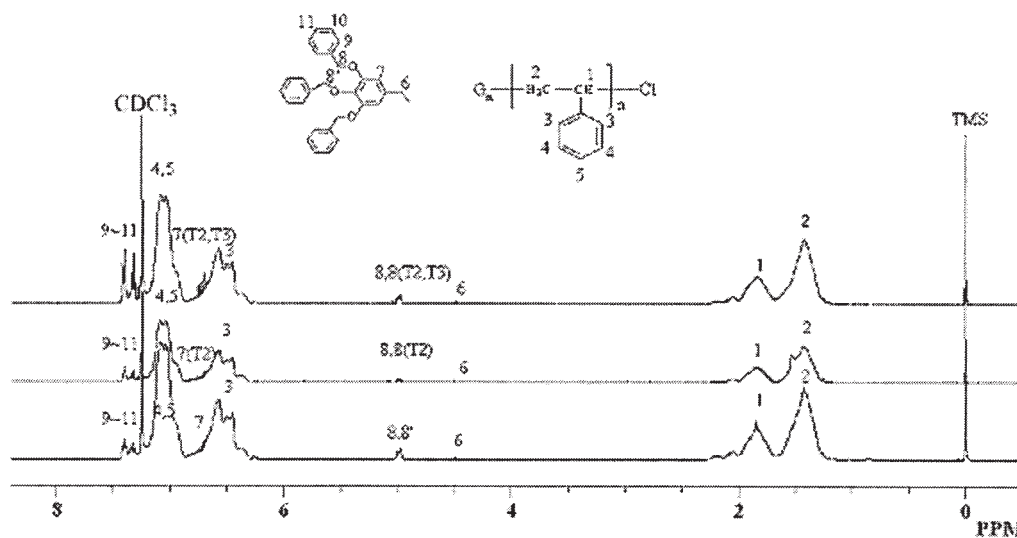
(calcd)]. This indicates that the average molecular weight of the resultant dendritic-linear polymer can be controlled through the polymerization conversion, and the contribution of chain breaking and transfer as well as the termination reaction during polymerization can be neglected until higher polymerization conversions. The  $M_n$ (GPC) values are higher than  $M_n$ (NMR) and  $M_n$ (calcd). The reason for this is dendritic-linear polymers have different hydrodynamic volumes compared to linear PS having the same molecular weight. Moreover, the  $f$ , which is calculated from the equation  $f = M_n(\text{calcd})/M_n(\text{NMR})$ , increases gradually when increasing the conversion. For example, the value of  $f$  increases from 0.89 to 0.96 for G<sub>1</sub>-Cl as the macroinitiator when the conversion varies from 30 to 85%. Similar results

are also obtained for the cases of G<sub>2</sub>-b-PS and G<sub>3</sub>-b-PS. This phenomenon can be attributed to the slow initiation of the dendritic initiators.<sup>25</sup>

The  $M_n$  plots for the reaction temperature are shown in Figure 3. It can be seen that the  $M_n$  values of three kinds of dendritic-linear copolymers increase quickly with the increase of the reaction temperature from 90 to 105°C and increase slowly with further increases in the reaction temperature to 120°C. This can be attributed to the fact that the speed of chain propagation increases with increasing the reaction temperature. However, the speed of chain transfer has a similar relationship. The  $M_n$  decreases at the relatively higher temperature can be attributed to the speed of chain transfer being higher than that of chain propagation.

### Characterization and thermal properties of polymers

<sup>1</sup>H-NMR, Fourier transform IR (FTIR), DSC, and TGA techniques were used for characterizing dendritic-linear diblock polymers. Figure 4 shows the <sup>1</sup>H-NMR spectra of G<sub>n</sub>-b-PS ( $n = 1-3$ ). These spectra show the expected resonances for the aromatic protons in PS (peaks 3, 4, and 5,  $\delta$  6.30–7.30) and methylene protons in dendritic poly(benzyl ether) (peak 6,  $\delta$  4.49; peaks 8 and 8',  $\delta$  4.9 and 5.01) and PS (peaks 1 and 2,  $\delta$  1.25–2.0).<sup>26</sup> These spectra are in agreement with the expected structure. The IR spectra of G<sub>n</sub>-b-PS ( $n = 1-3$ ) are compared in Figure 5. They are almost completely the same; the IR spectrum of the G<sub>n</sub>-b-PS diblock copolymer showed characteristic absorption at 1100  $\text{cm}^{-1}$ , which is due to the C—O—C stretching band of



**Figure 4** <sup>1</sup>H-NMR spectra of G<sub>n</sub>-b-PS;  $n = 1-3$ ; T2 and T3, generations 2 and 3 of the dendritic segment, respectively (CDCl<sub>3</sub>, tetramethylsilane, 25°C).

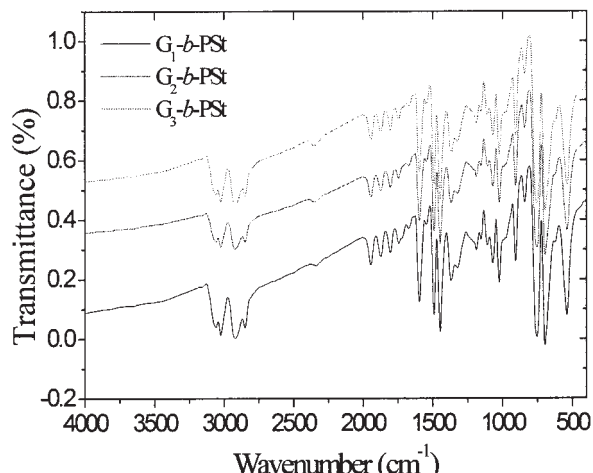


Figure 5 FTIR spectra of  $G_n$ -*b*-PS ( $n = 1-3$ ).

the dendritic segment; the absorptions at 2840–2920 and 3000–3100  $\text{cm}^{-1}$  correspond to the C—H stretching of methylenes and the phenyl groups at the same time; the absorption peaks for the phenyl ring are at 1400, 1500, and 1599  $\text{cm}^{-1}$ ; and the peaks at 710 and 770  $\text{cm}^{-1}$  result from a single-substituted phenyl ring.<sup>27</sup>

The curves of the loss weight rate of  $G_n$ -*b*-PS diblock polymers in a nitrogen stream from room temperature to 500°C were ascertained using TGA. Figure 6 gives the results of the analysis for  $G_n$ -*b*-PS ( $n = 1-3$ ). We found that there is no clear difference in the thermal stability of the three types of polymers ( $G_1$ -*b*-PS,  $G_2$ -*b*-PS, and  $G_3$ -*b*-PS) when changing the dendrimeric fragments. As indicated in Figure 6, the initial decomposition temperatures ( $T_{\text{init}}$ ) of these polymers are 386, 388, and 389°C, respectively, and the decompositions of the polymers are almost complete at 450°C. However, the weight loss of the three polymers before

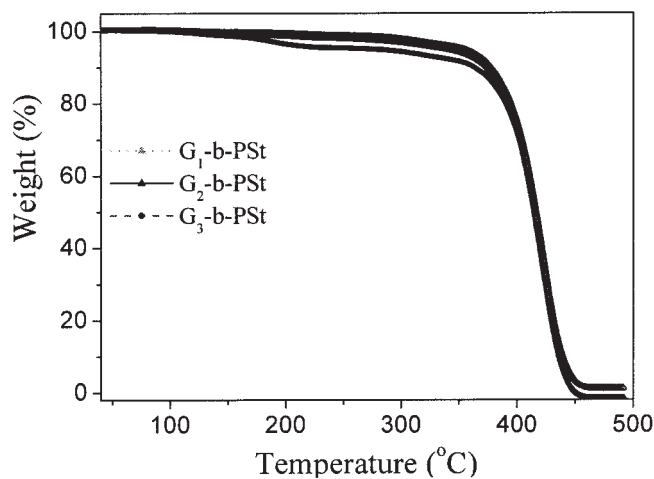


Figure 6 TGA curves of  $G_n$ -*b*-PS ( $n = 1-3$ ).

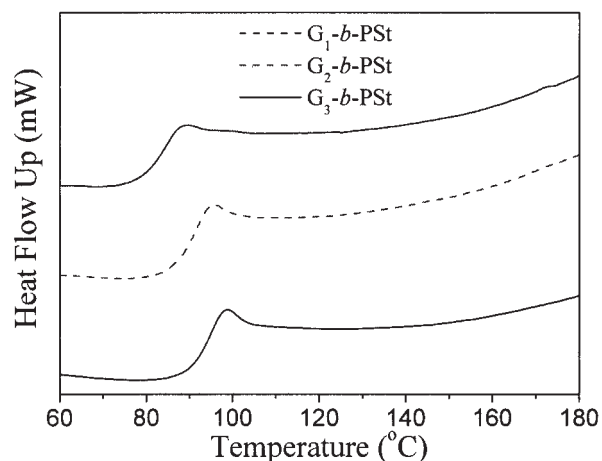


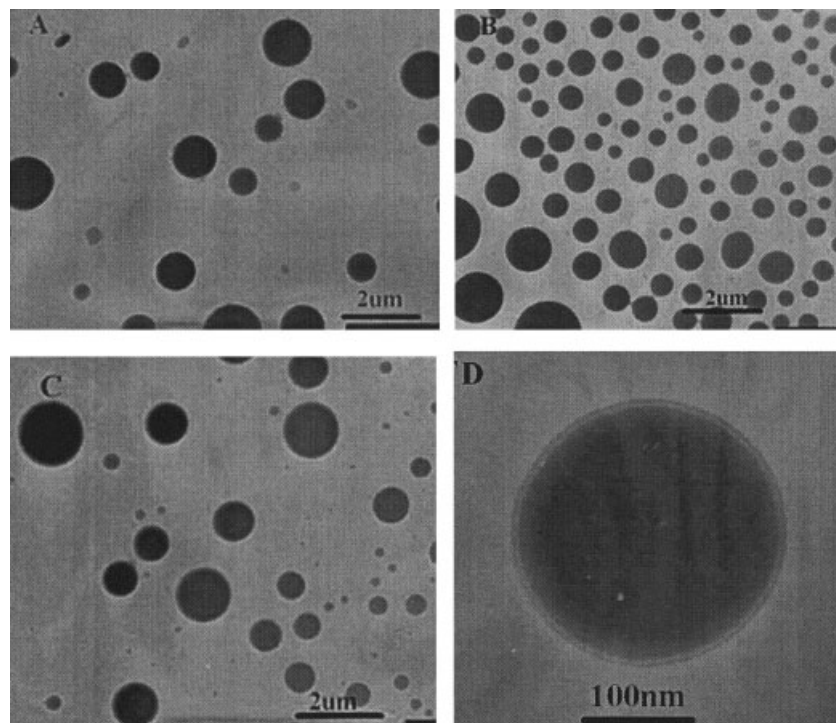
Figure 7 DSC curves of  $G_n$ -*b*-PS ( $n = 1-3$ ) polymers.

400°C is inappreciably different (<5%) with increasing the generation number of the macroinitiator. Usually, polymers with bulky side groups are considered to lose their side groups at the first stage of thermal decomposition. In our case, the polymer with the bulky dendritic side group can lose the dendron at a lower temperature than the measured  $T_{\text{init}}$ ; but it does not lose weight because of the low vaporability of the dendrons, resulting in a higher  $T_{\text{init}}$  value for the polymer with the higher generation dendritic side groups.<sup>28</sup>

Figure 7 indicates the DSC trace of block polymers. The only glass-transition temperature ( $T_g$ ) was observed throughout the whole scan range from 60 to 180°C. As expected, the generation number of the dendritic blocks affects the  $T_g$ . The changes in heat capacity ( $\Delta C_p$ ) of  $G_1$ -*b*-PS,  $G_2$ -*b*-PS, and  $G_3$ -*b*-PS are 0.23, 0.24, and 0.27 J/g °C. The  $C_p$  of the polymer is related to the magnitude of vibration and other molecular motions (e.g., pendent group rotation). When two polymers with identical chemical compositions are compared, a larger  $\Delta C_p$  is thus expected when a larger increase in chain mobility is achieved at  $T_g$ .<sup>29</sup> The higher the generation number of the dendritic block is, the lower the  $T_g$ . The  $T_g$  value systematically decreases when increasing the generation number of the dendritic block. All the  $T_g$  values of the three polymers are lower than that of the PS homopolymer.

#### Self-assembly behaviors of $G_n$ -*b*-PS in selected solvents

Different morphologies of self-assembly systems are basically related to the chemical composition of the copolymer and to the relative volume ratio of the two blocks. Among the possible morphologies, spherical micelles are of special interest because they have the lowest surface energies<sup>30</sup> and can lead to encapsulating active molecules such as DNA, enzymes, or drugs.



**Figure 8** TEM photographs of the micelles formed by (A) G<sub>1</sub>-b-PS, (B) G<sub>2</sub>-b-PS, (C) G<sub>3</sub>-b-PS, and (D) a high TEM magnification of G<sub>2</sub>-b-PS in the mixed solvent (CHCl<sub>3</sub>/acetone = 1:1, v/v) with a concentration of 0.1 wt %.

The dendritic poly(benzyl ether) segments have a fairly strong steric effect, so they will encounter strong repulsive forces when trying to accommodate the microsphere's aggregates.<sup>22</sup> Figure 8 shows a micrograph obtained from a 0.1 wt % solution of the dendritic-linear polymers in chloroform/acetone (1:1, v/v) cast onto a TEM grid. The unstained sample clearly shows spherical micelles with a diameter of 0.3–1.2 μm [Fig. 8(A–C)]. Both dendritic and linear blocks can be dissolved in chloroform; however, acetone is a good precipitator for a PS block. Therefore, it is assumed that the excess dendritic poly(benzyl ether) molecules are encapsulated in the aggregates and PS tails are hidden in the center of the micelles. This assumption is further supported by high TEM magnification observation [Fig. 8(D)]. We observed that isolated spherical micelles have a core-shell structure; thus, their inner core diameters can be as long as 0.28–1.18 μm and their outer shells can be as thick as about 15 nm. The average core diameter and shell thickness of the other kinds of G<sub>n</sub>-b-PS (*n* = 1 and 3) have no evident difference. These phenomena can be attributed to the fact that the repulsive forces from dendritic segments could counteract the favorable association of long flexible chain blocks and prevent the infinite aggregation of diblock polymers. A balance of attractive and repulsive forces among blocks could mediate the formation of supramolecular units.

## CONCLUSIONS

Dendritic-linear block polymers possessing well-defined molecular weights and low polydispersities were prepared using a CuCl/Bipy complex catalyst. These dendritic-linear block polymers, which consist of linear and dendritic segments, had very good solubility in common organic solvents at room temperature. However, the terminal group (dendritic segments) of the polymers affected their thermal stability. These dendritic-linear polymers self-assembled in selective solvents (chloroform/acetone) formed core-shell micelles.

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