Synthesis and Properties of Poly[(1,1-dihexyl-3,4-diphenyl-2,5-silolene)-*co*-(methylphenylsilylene)]

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ABSTRACT

Polycarbosilane conjugated with 1,1-dihexyl-3,4-diphenyl-2,5-silolene and methylphenylsilylene, such as poly[(1,1-dihexyl-3,4-diphenyl-2,5-silolene)-*co*-(methylphenylsilylene)] has been prepared. The characteristic diene stretching frequency appears at 1696-1600 cm⁻¹ in the Fourier-transform infrared spectrum of the polymer. The obtained material is highly soluble in organic solvents such as tetrahydrofuran and chloroform. The molecular weight of the polymer was determined as $1436/1084 (M_w/M_n)$ with polydispersity of 1.32 by gel permeation chromatography. The prepared material in THF solvent show strong absorption maximum peaks at 249, 261, and 336 nm with molar absorptivities of 2.74×10^4 , 2.57×10^4 , and 3.16×10^3 cm⁻¹ M⁻¹, respectively, in the UV-visible absorption spectrum, strong excitation maximum peak at 336 nm, and strong emission maximum peak at 398 nm in the fluorescence emission spectrum. Thermogravimetric analysis shows that the polymer is thermally stable up to 200°C with a weight loss of 3% only in nitrogen.

Key words : Poly[(1,1-dihexyl-3,4-diphenyl-2,5-silolene)-*co*-(methylphenylsilylene)], 2,5-Dibromo-1,1-dihexyl-3,4-diphenylsilole, Lithiation, Electronic properties, Thermal stability

Introduction

Conjugated organosilicon polymeric materials have received considerable attention due to their potentially useful applications [1,2]. Organosilicon polymers composed of fluorescent groups are also of great interest for use in electroluminescence and organic semiconductor materials [3,4].

Siloles or silacyclopenta-2,4-dienes are a series of fivemembered silacyclic dienes that contain unique π -electron system of low-lying LUMO level associated with σ^* - π^* conjugation arising from the interaction between the σ^* orbital of two exocyclic σ -bond on the silicon atom and the π^* orbital of butadiene group [5,6]. π -Conjugated polymers containing siloles have also received much attention for potential optoelectronic, photoluminescence [7,8], and sensor applications [9]. Endcapped silole dendrimers on an ethenyl-phenyl-carbosilane skeleton show green to greenish-blue fluorescence and have potential applications as electroluminescent materials [10]. However, few syntheses of conjugated polycarbosilane containing 1,1-dihexyl-3,4-diphenyl-2,5-silolene and methylphenylsilylene along the polymer back bone such as poly[(1,1dihexyl-3,4-diphenyl-2,5-silolene)-*co*-(methylphenylsilylene)] have been reported [11].

Aryl- and heteroaryl-conjugated polymers are usually synthesized through metal-catalyzed polycondensation reactions, based on Yamamoto, Suzuki, or Stille coupling reactions [12, 13], and the Grignard metathesis (GRIM) method [14]. Recently, to expand the use of GRIM, "turbo-Grignard" reagents such as LiCl plus a Grignard reagent have been used for the synthesis of various aryl and heteroaryl conjugated polymers [15]. Very recently, I have reported the syntheses of poly(1,1disubstituted-3,4-diphenyl-2,5-silole)s *via* GRIM polymerizations of 1,1-disubstituted-2,5-dibromo-3,4-diphenylsiloles using a "turbo-Grignard" reagent [16].

I have previously reported the synthesis of polycarbosilanes

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conjugated with diacetylene and aromatic groups of thiophene or phenylene simultaneously present in the polymer backbone using n-butyllithium [17] as well as their molecular energy dynamics using ultrafast fluorescence spectroscopy [18].

With these results in mind, I focused on the preparation of fluorescent poly(carbosilane) containing aromatic chromophores such as 3,4-diphenyl-2,5-silolene and methylphenylsilylene in the polymer main chain through copolymerization using *n*-butyllithium and evaluated their photoelectronic properties.

Here, I report the syntheses of new conjugated polycarbosilane of poly[(1,1-dihexyl-3,4-diphenyl-2,5-silolene)-*co*-(methylphenylsilylene)] (6) *via* copolymerization of 1,1dihexyl-2,5-dibromo-3,4-diphenyl-2,5-silolene (3) with methylphenyldichlorosilane (5) using *n*-butyllithium. The material obtained was characterized using Fourier-transform infrared (FTIR) and ¹H, ¹³C, and ²⁹Si nuclear magnetic resonance (NMR) spectroscopies in the solution state. We have investigated the photoelectronic and thermal properties of the prepared polymer **6** using absorption, excitation, and fluorescence emission spectrophotometry in solution state as well as thermogravimetric analysis (TGA).

Materials and Methods

1. General Procedures

All glassware was assembled and then flame-dried while being swept with argon before reaction. All synthetic manipulations were carried out by standard vacuum-line Schlenk techniques under argon. Chemicals were purchased from Aldrich Aldrich Chemicals Inc. and used as received. Tetrahydrofuran was distilled from sodium benzophenone ketyl before use [19]. ¹H and ¹³C nuclear magnetic resonance (NMR) spectroscopy was performed on a Varian Mercury Plus 300 or a Bruker DRX Avance 400 MHz FT-NMR spectrometers using CDCl₂ as solvent. ²⁹Si nuclear magnetic resonance (NMR) spectroscopy was performed on Varian Unity INOVA 500 MHz FT-NMR spectrometer at the Daegu Center of the Korea Basic Science Institute using CDCl₃ as solvent. The chemical shifts are expressed in ppm relative to tetramethylsilane (TMS) as an internal standard or the residual proton signal of the solvent as standard. IR spectra were recorded by a Shimadzu IR 430 spectrometer or a Bruker IFS-48 FT-IR spectrometer. Gel permeation chromatography (GPC) analyses were performed on a Waters 1525 pump and Breeze software system with a Waters Styragel HR 3 column and refractive index detector at

40°C. The eluent was THF at a flow rate of 1.0 mL/min. Calibration was done with a series of monodispersed polystyrene standards: M_p 580, 3250, 10100, and 28500 whose a M_w/M_n value is less than 1.2. UV-vis absorption spectra were obtained on a Hewlett Packard 8453 spectrophotometer. Excitation and fluorescence emission spectra were taken on a Spex Fluorolog-3-11 fluorescence spectrophotometer. Thermogravimetric analysis (TGA) of polymer sample was performed on a TGA-50 Shimadzu thermal analysis system. The temperature was increased at the heating rate of 10°C/min from room temperature to 900°C with nitrogen flow rate of 20 mL/min.

2. Synthesis of Dihexylbis(phenylethynyl)silane (2)

A three-necked flask equipped with a thermometer, rubber septa, and magnetic stirring bar was charged with phenylacetylene (25.5 mL, 232 mmol) and THF (180 mL). The solution was cooled to -70° C in a dry ice/acetone bath. *n*-Butyllithium (2.5 M, 78.6 mL, 197 mmol) was added to the stirred solution, using a syringe, under a stream of argon. The acetone/dry ice bath was replaced with an ice/water bath. When the reaction temperature reached approximately -5° C, dichlorodihexylsilane (1) (26.0 mL, 92.8 mmol) was added dropwise using a syringe. The solution was allowed to warm to room temperature with stirring. The reaction mixture was poured into aqueous ammonium chloride solution (200 mL). The organic phase was separated using a separating funnel and the aqueous layer was extracted with diethyl ether. The combined organic layer was washed with water and brine, dried over anhydrous magnesium sulfate, filtered, and concentrated by rotary evaporation under vacuum. Scarlet liquid of product 2 (37.0 g, 99.7%) were obtained by removal of solvents under high vacuum [6]. ¹H NMR (300 MHz, CDCl₃, δ): 0.84-1.62 (m, 26H), 7.28-7.33 (m, 6H), 7.48-7.52 (m, 4H). ¹³C NMR (75 MHz, CDCl₃, δ): 14.34, 15.06, 22.81, 23.90, 31.73, 32.92, 89.71, 106.81, 123.10, 128.37, 128.47, 132.33. ²⁹Si NMR (99.36 MHz, CDCl₃, δ): -34.69.

Synthesis of 2,5-Dibromo-1,1-dihexyl-3,4diphenylsilole (3)

A three-necked flask equipped with a thermometer, rubber septa, and magnetic stirring bar was charged with lithium (0.53 g, 75.6 mmol), naphthalene (10.12 g, 79.0 mmol) and THF (50 mL) under a stream of argon. The reaction mixture was stirred at room temperature for 3 h to dissolve the lithium. A solution of 2 (6.73 g, 16.8 mmol) in THF (120 mL) was added dropwise, through a cannula, to the lithium naphthalenide over 20 min.

Once the addition was complete, the solution was cooled to -10° C using an acetone bath with a small amount of dry ice. A solution of anhydrous ZnCl₂ (11.45 g, 84.0 mmol) in THF (70 mL) was added to the naphthalenide/silane mixture, through a cannula, over 20 min. The acetone/dry ice bath was removed and the resulting solution was allowed to warm to room temperature. A three-necked, foil-wrapped, round-bottomed flask equipped with a thermometer, stirring bar, and rubber septa was charged with NBS (7.45 g, 41.9 mmol) and THF under an argon atmosphere. The resulting solution was cooled to -70°C using a dry ice/acetone bath. The room-temperature silane solution was added to the cold NBS solution through a cannula, ensuring that the internal temperature did not exceed -50° C. After the addition was complete, the resulting mixture was stirred for a further 30 min at -70° C. The reaction mixture was poured into aqueous ammonium chloride solution (200 mL). The organic phase was separated using a separating funnel and the aqueous laver was extracted with diethyl ether. The combined organic layer was washed with aqueous sodium thiosulafate solution, water, and brine, dried over anhydrous magnesium sulfate, filtered, and concentrated by rotary evaporation. The crude naphthalene-silole mixture was sublimed under vacuum at 35°C. The residual material from the sublimation was dissolved in hot hexane, followed by cooling in a freezer. A pale brownish powder product 3 (4.03 g, 42.8%) were obtained by vacuum filtration [6]. ¹H NMR (300 MHz, CDCl₃, δ): 0.89-1.54 (m, 26H), 6.93-6.97 (m, 4H), 7.14-7.18 (m, 6H). ¹³C NMR (75 MHz, CDCl₃, δ): 9.96, 14.36, 22.81, 23.11, 31.66, 32.91, 122.11, 127.59, 127.76, 129.19, 137.46, 157.08. ²⁹Si NMR (99.36 MHz, CDCl₃, δ): 6.18. UV-vis (THF) $\lambda_{max} nm (\epsilon cm^{-1} M^{-1}): 243 (1.28 \times 10^4).$

Synthesis of Poly[(1,1-dihexyl-3,4-diphenyl-2,5silolene)-co-(methyl-phenylsilylene)] (6)

A flame dried 100 mL three-neck round bottom flask equipped with a reflux condenser, a pressure equalizing addition funnel, and Teflon covered magnetic stirring bar was cooled to -78° C using a dry ice/acetone bath under argon atmos-

phere and then n-BuLi (2.5 M/hexane, 1.57 mL, 3.92 mmol) and THF (10 mL) were added. Monomer 3 (1.00 g, 1.78 mmol) and THF (10 mL) were placed in the addition funnel. This solution was slowly added to the flask with well stirring. The reaction mixture was allowed to stir at -78° C for 2 h, and then warm to room temperature. The reaction mixture was again cooled to -78° C and (CH₃)(C₆H₅)SiCl₂ (5) (0.340 g, 1.78 mmol) was added with well stirring at -78° C for 2 h. The reaction mixture was allowed to warm to room temperature with stirring for 5 h. The crude product was precipitated by the addition of methanol and 10% aqueous HCl (20-30 mL) and isolated by decantation. The crude polymer was redissolved in THF (40-50 mL), washed with 10% aqueous HCl, deionized water, and concentrated at reduced pressure. The polymer was reprecipitated by addition of methanol (50-100 mL) and dried under reduced pressure. Polymer 6, 0.750 g, 80.4% was obtained as a viscous yellowish liquid. ¹H NMR (300 MHz, CDCl₃, δ): 0.091-0.68 (m, 3H), 0.75-1.44 (m, 26H), 7.09-7.52 (m, 10H). ¹³C NMR (75 MHz, CDCl₃, δ): 0.00, 13.61, 14.55, 22.92, 24.19, 31.84, 33.44, 123.69, 128.48, 132.23, 133.51, 138.97, 152.02. ²⁹Si NMR (99.36 MHz, CDCl₃, δ): -6.10, -12.10, -21.03. IR (KBr disk) v_{max}: 3055, 3018, 2955, 2922, 2852, 2159, 1696, 1600, 1489, 1463, 1422, 1407, 1377, 1259, 1185, 1125, 1074, 1022, 996, 955, 837, 759, 737, 696 cm⁻¹. UV-vis (THF) λ_{max} , nm (ϵ , cm⁻¹ M⁻¹): 249 (2.74 × 10⁴), 261 (2.57 × 10⁴), 336 (3.16 × 10³). GPC (THF) M_w =1,436, M_n =1,085, PDI=1.32. The excitation and fluorescence emission spectra in THF solution and TGA thermogram for polymer 6 are described in the Results and Discussion.

Results and Discussion

1. Synthesis of Monomer

2,5-Dibromo-1,1-dihexyl-3,4-diphenylsilole (**3**) as a monomer was prepared by a two-step reaction, which was reported previously [5,6,16], as shown in Scheme 1. In the first step, dichlorodihexylsilane (**1**) was reacted with phenylethynylli-







thium, which was prepared *in situ* by a reaction of phenylacetylene with *n*-butyllithium in tetrahydrofuran (THF) at -70° C, to yield dihexylbis(phenylethynyl)silane (2) quantitatively. In the second step, intramolecular reductive cyclization of 2 was performed by the treatment of 4 mol of lithium naphthalenide followed by anhydrous ZnCl₂ and N-bromosuccinimide (NBS), to afford compound 3 in moderate yield [5, 6,16].

The monomer **3** was characterized using various spectroscopic methods, such as ¹H, ¹³C, and ²⁹Si NMR spectroscopies in the solution state. As shown in Fig. 1, the ¹H NMR spectrum of **3** exhibits multiplet resonances at 0.89-1.54 and 6.93-7.18 ppm, ascribed to hexyl and phenyl groups of the monomer **3**, respectively [20,21]. In the ¹³C NMR spectrum of **3**, six carbon peaks from the hexyl group bonded to the silicon is observed at 9.96, 14.36, 22.81, 23.11, 31.66, and 32.91 ppm [20,21]. In the ²⁹Si NMR spectrum of **3**, the silicon resonance is observed at 6.18 ppm. The intramolecular reductive cyclization reaction of **2** was completed under this reaction condition to yield the monomer **3**.

I examined the absorption spectrum of the monomer 3 in

THF solution. The maximum absorption band for **3** is observed at 243 nm, with a molar absorptivity of 1.28×10^4 cm⁻¹ M⁻¹. The strong absorption band in the ultraviolet-visible (UV-vis) spectra of **3** may result from chromophores such as phenyl and diene groups in the 3,4-diphenylsiole monomer [3].

2. Synthesis of Polymer

The synthetic route for polycarbosilane contained with 1,1dihexyl-3,4-diphenyl-2,5-silolene and methylphenylsilylene along main chain is outlined in Scheme 2, which is similar to the copolymerization reactions of 1,4-bis(4-chlorophenyl or 5chlorothiophenyl)-buta-1,3-diyne using *n*-butyllithium reported previously [17].

The monomer **3** was treated with *n*-butyllithium at -78° C to give the dilithiated intermediate (**4**) of 1,1-dihexyl-3,4-diphenylsilolene, followed by copolymerization of **4** with (CH₃)(C₆H₅)SiCl₂ (**5**) *in situ* was performed to yield polycarbosilane such as poly[(1,1-dihexyl-3,4-diphenyl-2,5-silolene)*co*-(methylphenylsilylene)] (**6**).



Fig. 2. Absorbance (dashed line, λ_{max} =249, 261, 336 nm), excitation (solid line, $\lambda_{ex,max}$ =336 nm, λ_{del} =398 nm), and fluorescence emission (dotted line, $\lambda_{em,max}$ =398 nm, λ_{ex} =336 nm) spectra of polymer **6** in THF solution.

The prepared polymeric material **6** is obtained in good yield as a dark yellowish viscous liquid, and is soluble in common organic solvents such as CHCl₃ and THF. The observed molecular weight of **6** was 1436/1084 (M_w/M_n) with polydispersity of 1.32, indicating that the obtained material **6** is oligomeric. This phenomenon can be explained by the intrinsic-low reactivities of the functionalities at the 2,5-position of the silole monomer [7] and severe steric congestion [22].

I characterized polymer **6** using various spectroscopic methods such as by ¹H, ¹³C, and ²⁹Si NMR spectroscopies in the solution state and FTIR spectroscopy. The FTIR spectrum of **6** show characteristic C=C stretching frequencies at 1696-1600 cm⁻¹, suggesting that the conjugated diene groups were kept intact during polymerization reaction [23]. The ²⁹Si NMR spectrum of polymer **6** in CDCl₃ solution shows major resonance peaks at -6.10, -12.10, and -21.03 ppm, indicating that the silicon atoms in the polymer main chain has the structural environments composed of 1,1-dihexyl-3,4-diphenylsilolene and methylphenylsilylene groups in the form of oligomeric low molecular weight, suggesting structures consistent with the backbone shown in Scheme 2 [24].

3. Properties of Polymer

I examined the absorption, excitation, and fluorescence emission spectra of polymer 6 in THF solution as shown in Fig. 2.

The maximum absorption bands λ_{max} for **6** are observed at 249, 261, and 336 nm with molar absorptivities of 2.74×10^4 , 2.57×10^4 , and 3.16×10^3 cm⁻¹ M⁻¹, respectively, which the



Fig. 3. TGA thermogram of polymer 6 in nitrogen.

band extends from 249 to 400 nm. The strong absorption band in the UV-vis spectra of **6** is attributed to chromophores such as phenyl and diene groups along the polymer backbone [3]. A comparison of maximum the absorption bands of polymer **6** with that of the monomer **3** in THF solution shows a red shift of 6-18 nm, probably because of strong intermolecular interactions through the polymer backbone.

The excitation spectrum for **6** at a detection wavelength of 398 nm shows a strong excitation peak at $\lambda_{ex,max}$ of 336 nm, as shown in Fig. 2 as a dotted line. The strong excitation band in the excitation spectra of polymer **6** is attributed to the phenyl and diene groups along the polymer backbone [3].

The fluorescence emission spectrum of **6** at an excitation wavelength of 336 nm in THF solution has a strong emission peak at $\lambda_{em,max}$ of 398 nm, as shown in Fig. 2 as a solid line. The strong emission band in the fluorescence spectrum of the polymer is ascribed to fluorophores such as phenyl and diene groups along the polymer backbone [3]. The fluorescence emission FWHM (full width at half maximum) value of **6** is observed at 77 nm. The excitation and fluorescence emission spectra of the polymer implys that **6** has chromophores containing phenyl and diene groups along the polymer main chains [3].

The thermal stability of **6** in a nitrogen atmosphere was examined using TGA, with a heating rate of 10° C/min and the result is shown in Fig. 3. Polymer **6** is stable up to 200° C, with only 3% loss of the initial weights in nitrogen. Polymer **6** rapidly lost weight on heating above 200° C. Rapid weight losses of 82% of the initial weights occurred between 200 and 600°C. Additionally, weight losses of 9% of the initial weights occurred between 600 and 900°C. When polymer **6** was heated to 900°C, the polymers lost 94% of their initial weights, and char yields of about 6% was observed.

Conclusion

I have successfully prepared a new conjugated polycarbosilane contained with 1,1-dihexyl-3,4-diphenylsilolene and methylphenylsilylene groups in the polymer main chain such as poly[(1,1-dihexyl-3,4-diphenyl-2,5-silolene)-co-(methylphenylsilylene)] (6) via copolymerization of 1,1-dihexyl-2,5dibromo-3,4-diphenyl-2,5-silolene (3) with methylphenyldichlorosilane (5) using *n*-butyllithium. The prepared material is soluble in common organic solvents such as CHCl₃ and THF. The polymer was characterized in solution, using various spectroscopic methods. The FTIR spectrum of the polymeric material shows characteristic diene stretching frequency at 1696-1600 cm⁻¹. In THF solution, the material has strong maximum absorption bands at 249-336 nm, with red shifts of 6-18 nm relative to that of the monomer, strong maximum excitation peak at 336 nm, and strong maximum fluorescence emission band at 398 nm. All the absorption, excitation, and emission spectra suggest that the obtained material contains chromophores such as phenyl and diene groups in the polymer main chains. TGA shows that the polymer is thermally stable up to 200°C with a weight loss of 3% in nitrogen.

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