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Synthesis of pentamethyldisilanyl-substituted starlike molecule with triazine core and its application to dye-sensitized solar cells

Akinobu Naka [a, *], Kiyoshi Fujishima [a], Erika Okada [a], Mao Noguchi [a], Joji Ohshita [b], Yohei Adachi [b], Yousuke Ooyama [b], Mitsuo Ishikawa [a]

[a] Department of Life Science, Kurashiki University of Science and the Arts, 2640 Nishinoura, Tsurajima-cho, Kurashiki, Okayama 712-8505, Japan
[b] Department of Applied Chemistry, Graduate School of Engineering, Hiroshima University, Higashi-Hiroshima 739-8527, Japan

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ABSTRACT

Two-dimensional starlike molecule **7** consisting of benzothiadiazole-thiophenylphenyltriazine groups and pentamethyldisilanyl substituted thienylene units that extend to three directions was synthesized by the reactions of 2,4,6-tris[4-(4-hexyl-5-(7-(3-hexyl-5-iodothiophen-2-yl)benzo[c][1,2,5]thiadiazol-4-yl)-thiophen-2-yl)phenyl]-1,3,5-triazine (**5**) with the pentamethyldisilanylthienyl substituted stannyl derivative (**6**). UV-visible absorption and fluorescence properties of starlike molecules **4** and **7** have been investigated in dioxane and in the solid state. The application of TiO_2-electrode coated on the surface with the use of **7** to dye-sensitized solar cells has been discussed.

1. Introduction

Three-dimensional organosilicon dendrimers containing π-conjugated units have attracted a great deal of attention to their potential application in organic photo- and electroluminescent materials [1–13]. We have reported synthesis and optical properties of various types of three-dimensional starlike silicon compounds [14,15], and also two-dimensional ones, whose arms extended to three or four directions [16,17] (see Chart 1). They show interesting properties, depending on their unique molecular structure, and may be used as functionality materials. For example, the compounds bearing the arms consisting of a regular alternating arrangement of a silicon-silicon bond and bithienylene unit, which extend to three directions reveal high fluorescence quantum yields and long lifetimes of the excited state [15–17].

Recently, a considerable attention has been focused on low-band gap materials as active layer in photovoltaic cells for light absorption [18,19]. The alternating arrangements of electron-donating and electron-accepting moieties in the materials give rise to mutual interaction between HOMO of the donor and LUMO of the acceptor, and reduction of the band gap in these systems. Recently, Ohshita et al. prepared disilanylene polymers with conjugated donor–acceptor systems and used them as sensitizing dyes [20–24]. In this paper we report synthesis of a new donor-acceptor type of the disilanyl substituted starlike molecule bearing a triazine core, and its application to dye-sensitized solar cells (DSSC)s.

2. Results and discussion

The synthesis of the arms having benzothiadiazole and pentamethyldisilanyl substituted thienylene units were carried out as shown in Schemes 1–3. Treatment of the lithio derivative prepared by the reaction of 2,4,6-tris(4-bromophenyl)-1,3,5-triazine (**1**) and *n*-butyllithium, with 2-isopropyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane afforded compound **2** in 18% yield (Scheme 1) [25].

Suzuki-Miyaura cross coupling reaction of **2** with 4-(5-bromo-3-hexylthiophen-2-yl)-7-(3-hexylthiophen-2-yl)benzo[c][1,2,5]thiadiazol (**3**) in THF afforded 2,4,6-tris(4-(4-hexyl-5-(7-(3-hexylthiophen-2-yl)benzo[c][1,2,5]thiadiazol-4-yl)thiophen-2-yl)phenyl)-1,3,5-triazine (**4**) in 24% yield.

Compound **5** was prepared by treating **4** with 3 quiv. of *N*-iodosuccinimide (NIS) in chloroform in 47% yield. The starlike

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* Corresponding author.
E-mail address: anaka@chem.kusa.ac.jp (A. Naka).

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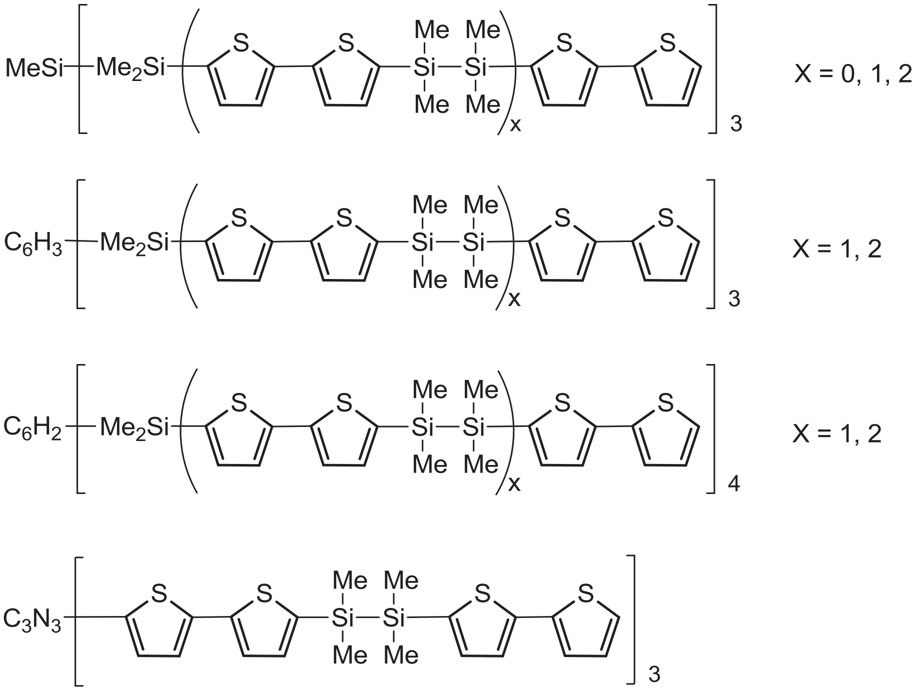
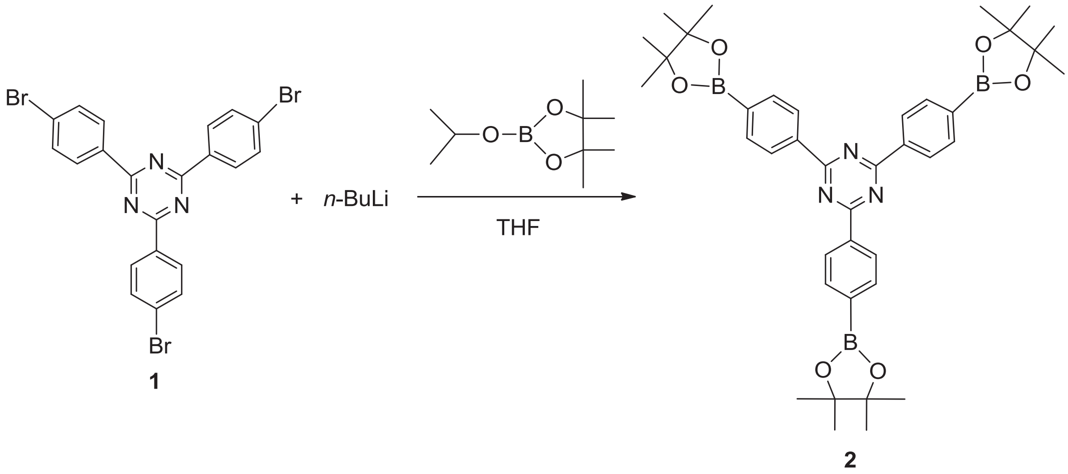


Chart 1. Various types of three-dimensional starlike silicon compounds.



Scheme 1. Synthesis of compound **2**.

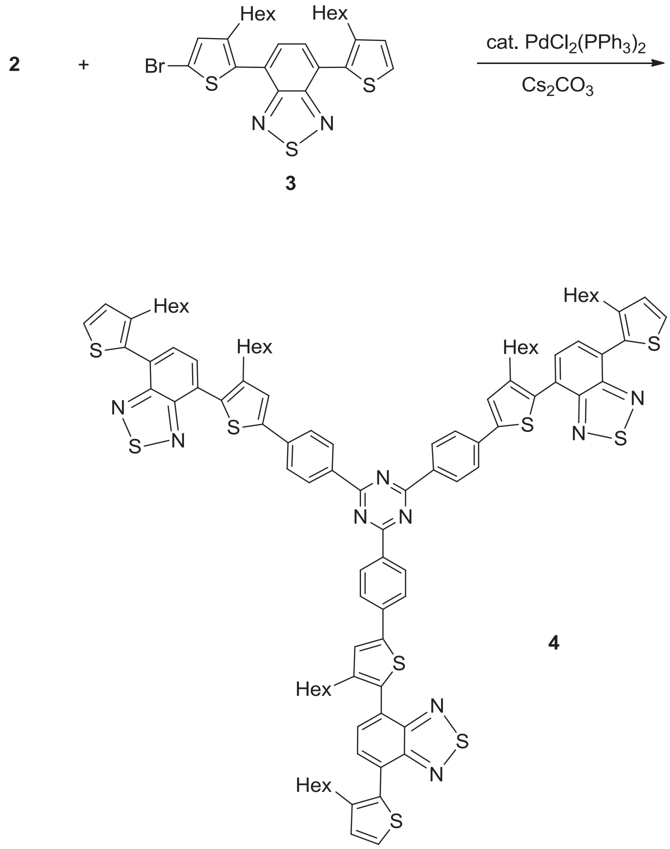
molecule, 2,4,6-tris(4-(4-hexyl-5-(7-(4-hexyl-5'-(1,1,2,2,2-pentamethyldisilyl)-2,2'-bithiophen-5-yl)benzo[c][1,2,5]thiadiazol-4-yl)thiophen-2-yl)phenyl-1,3,5-triazine (**7**) was prepared by Stille coupling reaction of 1,1,2,2,2-pentamethyl-2-(5-(tributylstannyl)thiophen-2-yl)disilane (**6**) with **5**. The product **7** was isolated from the resulting reaction mixture, using a silica gel column in 24% yield.

The structure of **7** was verified by mass and ^{1}H, ^{13}C, and ^{29}Si NMR spectrometric analysis (see Experimental Section).

In fact, mass spectrometric analysis for **7** indicates the presence of a parent ion at m/z 2343, which is consistent with the calculated value for $C_{126}H_{153}N_9Si_6S_{12}$. The ^{1}H NMR spectrum of **7** shows two signals at 0.13, 0.40 ppm due to the methylsilyl protons, and the signals at 7.10, 7.21, 7.31, 7.52 ppm attributed to thienyl ring protons, along with hexyl protons and phenylene protons. The ^{13}C NMR spectrum of **7** reveals two signals at −2.9 and −2.4 ppm attributed to the methylsilyl carbons, and twenty three signals due to the aromatic carbons, as well as the signals due to the hexyl carbons. The ^{29}Si NMR spectrum reveals three signals at −23.5 and −18.8 ppm, due to two different kinds of the silicon atoms. These results are wholly consistent with the structure proposed for **7**.

*2.1. Optical properties of starlike molecules **4** and **7***

The UV–visible absorption spectra and fluorescence spectra of compounds **4** and **7** are shown in Figs. 1 and 2, respectively, and their spectral data are summarized in Table 1. Compound **4** in dioxane exhibits two bands at 373 nm ($\varepsilon = 74000$ M^{-1} cm^{-1}) and 414 nm ($\varepsilon = 56000$ M^{-1} cm^{-1}). The fluorescence spectrum of **4** excited at 373 nm displays the emission maximum at 560 nm. In the case of excitation at 373 nm, the fluorescence quantum yield of



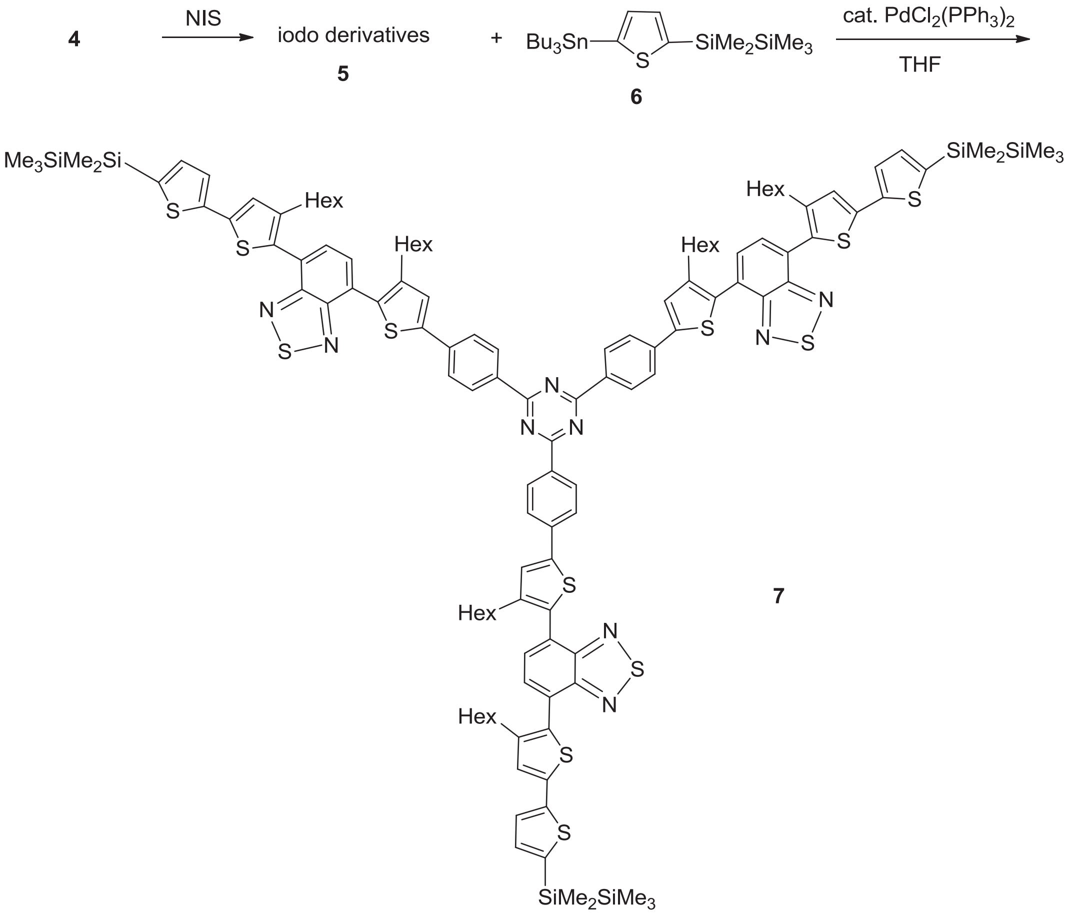
Scheme 2. Synthesis of compound **4**.

4 in dioxane is found to be $\phi_F = 17\%$. While compound **7** with the pentamethyldisilanyl unit also reveals two absorption maxima at $\lambda_{max} = 361$ nm ($\varepsilon = 81000$ M^{-1} cm^{-1}) and 441 nm ($\varepsilon = 57000$ M^{-1} cm^{-1}). The PL spectrum of **7** exhibits a broad band with emission maximum of 596 nm. The fluorescence quantum yield of **7** is 43%, which is much higher than that of **4**, having no disilanylene groups. The σ-π conjugation consisting of disilanyl units and oligothiophenes would be an important factor for a great enhancement in the fluorescence for the starlike molecule [26].

Emission spectra of **4** and **7** in the solid state were recorded at room temperature. Considerable changes are observed in the fluorescence maxima (605 nm for **4**, 643 nm for **7**) and the quantum yields (8% for **4**, 11% for **7**) compared to those in dioxane. The λ_{max} of **4** and **7** in the solid state is red-shifted from that in solution. The red-shift in the solid spectra may be attributed to the formation of aggregate sites derived from the strong intermolecular interaction [27,28]. The fluorescence quantum yields of **4** and **7** in the solid state become smaller than those in solution [29]. The aggregation-induced quenching would be caused by π-π stacking contacts of fluorophore in the solid state.

2.2. *Applications of **7** to dye sensitizer of DSSC*

The starlike molecule **7** was coated on the surface of the TiO_2-electrode, according to the following method. Namely, the TiO_2-electrodes prepared on FTO plates was immersed in chloroform containing compound **7** (2 g/L) in argon for 40 min under irradiation at >400 nm (photochemical condition) or in the dark for 24 h (thermal condition). The resulting orange-colored TiO_2-electrode



Scheme 3. Synthesis of compound **7**.

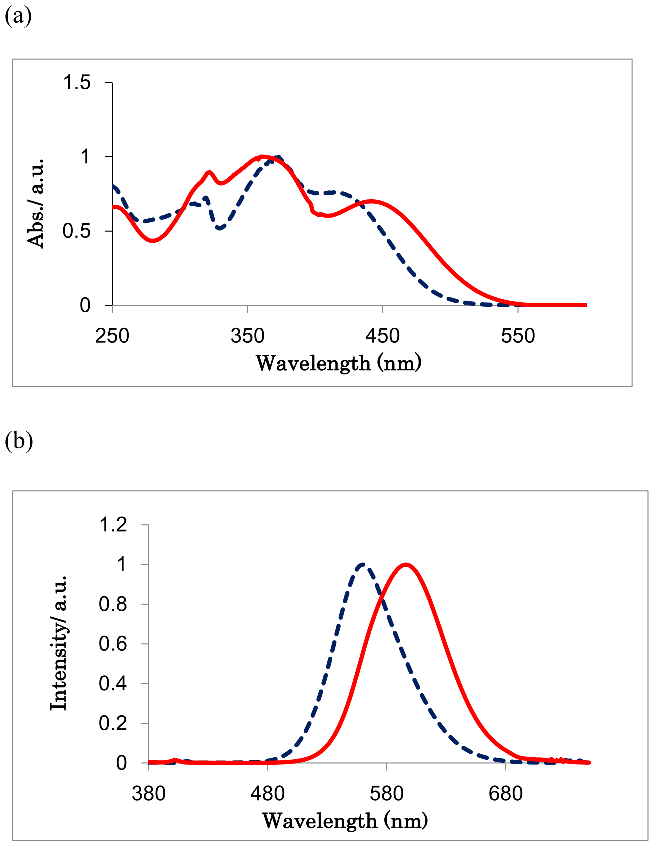


Fig. 1. (a) UV–vis of compound **4** and **7** in dioxane. (b) FL spectra of compound **4** and **7** in dioxane. ······ **4** and ▬ **7**.

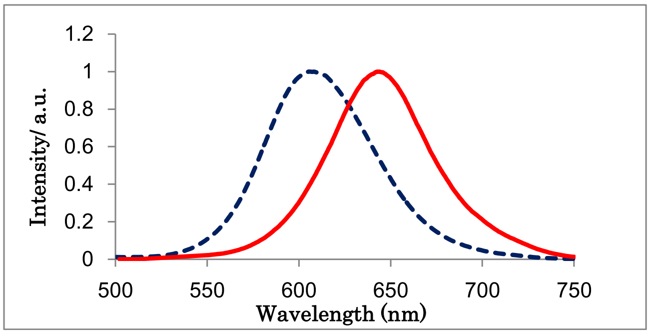


Fig. 2. FL spectra of compound **4** and **7** in powder. ······ **4** excited at 373 nm and ▬ **7** excited at 361 nm.

was rinsed thoroughly with chloroform. Thus, the starlike compound **7** with a triazine unit could readily coat on the surface of the TiO_2 electrode under both photochemical and thermal conditions [30,31]. DSSCs (FTO/**7**-attached $TiO_2/I^- \cdot I_3^-/Pt$) were fabricated, and then the performance was evaluated. The IPCE (incident photon to current efficiency) spectra and the *I-V* curves of the cells are shown in Fig. 3 and the DSSC parameters are summarized in Table 2.

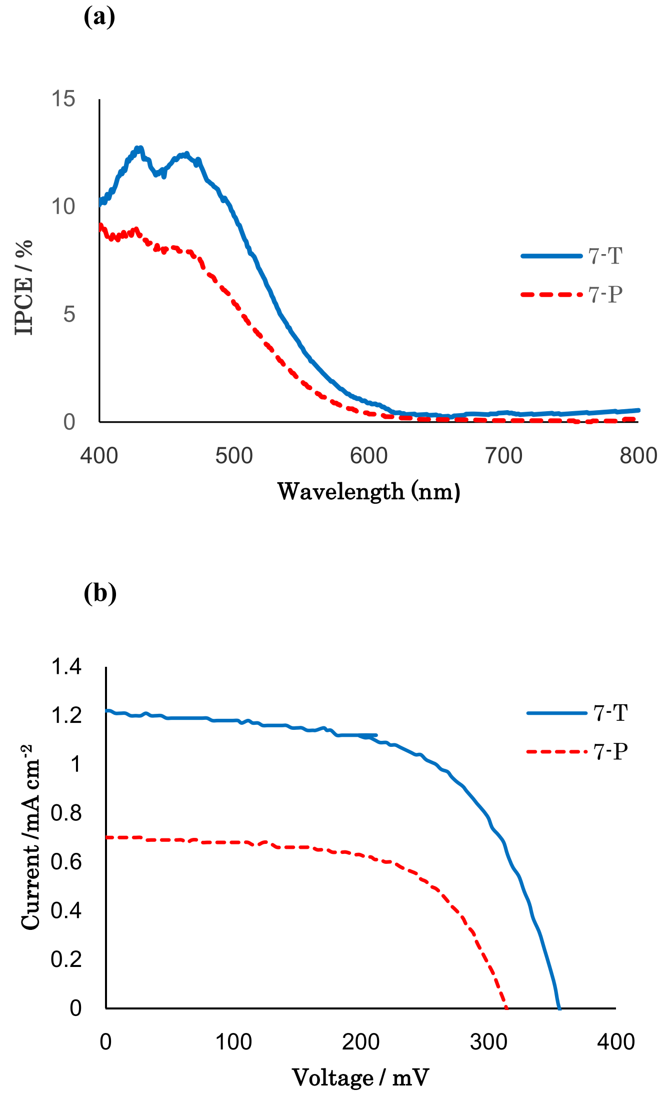


Fig. 3. (a) IPCE spectra of TiO_2 electrodes modified compound **7** (**7**-P: photochemical condition, **7**-T: thermal condition). (b) I-V characteristics of DSSCs based on TiO_2 electrodes modified compound **7** (**7**-P: photochemical condition, **7**-T: thermal condition).

Compound **7** exhibited clear sensitizing effects and the PCEs (power conversion efficiencies) were 0.14% (**7**-P; photochemical condition) and 0.27% (**7**-T; thermal condition) for the DSSCs. Thermally modified cell showed higher performance. Though the photochemical condition might form Si—O—Ti bonds, in which direct reaction of Ti-OH surface with the photo-excited Si—Si bonds would be involved, the longer immersing time in the thermal condition seems to affect the performance due to the higher degree of compound **7** attached.

In conclusion, we prepared the starlike molecule consisting of a benzothiadiazole- thiophenylphenyltriazine group and pentamethyldisilanyl substituted thienylene units. The compound was attached to TiO_2 electrodes under photochemical and thermal

Table 1
Absorption and fluorescence data for compounds **4** and **7**.

Compound	$\lambda_{max, Abs}$ (in dioxane) (nm)	$\lambda_{max, F}$ (in dioxane) (nm)	$\lambda_{max, F}$ (powder) (nm)	Φ_F (in dioxane)	Φ_F (powder)
4	373, 414	560	605	0.17	0.08
7	361, 441	596	643	0.43	0.11

Table 2
DSSC performance for compound **7**.

Compound	V_{OC}/mV	J_{SC}/mAcm^{-2}	FF	PCE (η)%
7-P	315	0.70	0.63	0.14
7-T	363	1.21	0.61	0.27

condition and the resulting TiO_2 electrodes were applied to DSSCs. Studies to explore starlike molecules other than DSSC applications are in progress.

3. Experimental

3.1. General procedures

All reactions were performed under an atmosphere of dry nitrogen. NMR spectra were recorded on JNM–ECS400 spectrometer and JNM–LA500 spectrometer. Low–resolution mass spectra were measured on a JEOL Model JMS–700 instrument. High-resolution mass spectra were measured using LTQ Orbitrap XL at the Natural Science Center for Basic Research and Development (*N*-BARD), Hiroshima University. UV–visible absorption spectra were measured with a JASCO V–560 spectrometer in dioxane. Fluorescence spectra were measured with a JASCO FP–8200 spectrometer and JASCO FP–8300 spectrometer. Melting point was measured with a Yanaco–MP–S3 apparatus. Column chromatography was performed by using Wakogel C–300 (WAKO). THF used as a solvent was distilled from sodium/benzophenone ketyl, just before use. Compounds **2** and **3** were synthesized following a procedure described in literature [25,32]. Fabrication and evaluation of DSSCs were performed as reported in the literature [22,23].

3.1.1. Synthesis of compound **4**

In a 200 mL two–necked flask was placed 0.6270 g (0.912 mmol) of **2** and 2.4078 g (4.40 mmol) of 4-(5-bromo-3-hexylthiophen-2-yl)-7-(3-hexylthiophen-2-yl)benzo[c][1,2,5]thiadiazol (**3**) and 0.8927 g (2.74 mmol) of cesium carbonate and 0.0676 g (0.0963 mmol) of bis(triphenylphosphine)dichloropalladium in 75 mL of THF and 15 mL of H_2O. After the mixture was heated to reflux for 3 days, it was hydrolyzed with dilute hydrochloric acid. The organic layer was separated, washed with water, and dried over anhydrous magnesium sulfate. After the solvent was evaporated, compound **4** (0.3743 g, 24% yield) was isolated by a silica gel column eluting with hexane-ethyl acetate (50:1) as a red solid: M.p. 75–76 °C. HRMS (ESI) exact mass calcd for $C_{99}H_{106}N_9S_9$ ($M^+ + H$): 1708.60521, found 1708.60650. MS *m/z* 1707 (M^+); ^{1}H NMR (δ $CDCl_3$) 0.83 (t, 9H, CH_3, J = 7.4 Hz), 0.85 (t, 9H, CH_3, J = 7.4 Hz), 1.21–1.32 (m, 36H, CH_2), 1.65 (quint, 6H, CH_2, J = 7.4 Hz), 1.72 (quint, 6H, CH_2, J = 7.4 Hz), 2.69 (t, 6H, CH_2, J = 7.4 Hz), 2.74 (t, 6H, CH_2, J = 7.4 Hz), 7.12 (d 3H, thienylene protons, J = 5.2 Hz), 7.46 (d, 3H, thienylene protons, J = 5.2 Hz), 7.52 (s, 3H, thienylene protons), 7.69 (d, 3H, phenylene protons, J = 7.6 Hz), 7.73 (d, 3H, phenylene protons, J = 7.6 Hz), 7.90 (d, 6H, phenylene protons, J = 8.8 Hz), 8.83 (d, 6H, phenylene protons, J = 8.8 Hz); ^{13}C NMR (δ $CDCl_3$) 13.9 (2C) (CH_3), 22.4, 22.5, 29.0, 29.1, 29.3, 29.6, 30.5 (2C), 31.4 (2C) (CH_2), 125.2, 125.8, 126.3, 126.9, 127.4, 129.1, 129.3, 129.5, 129.7, 132.1, 132.9, 135.0, 137.6, 141.5, 142.9, 143.5, 153.8, 154.1, 170.4 (thienylene and phenylene carbons).

3.1.2. Synthesis of compound **5**

In a 100 mL two–necked flask was placed 0.2020 g (0.118 mmol) of **4** in 18 mL of chloroform and 3 mL of acetic acid. To this was added 0.0997 g (0.443 mmol) of *N*-iodosuccinimide with ice cooling and the mixture was stirred over night. The mixture was hydrolyzed with dilute hydrochloric acid. The organic layer was separated, washed with water, and dried over anhydrous magnesium sulfate. After the solvent was evaporated, compound **5** (0.1157 g, 47% yield) was isolated by a silica gel column eluting with hexane-chloroform (2:1) as a red solid: M.p. 92–93 °C. HRMS (ESI) exact mass calcd for $C_{99}H_{103}N_9S_9I_3$ ($M^+ + H$): 2086.29514, found 2086.29744. MS *m/z* 2085 (M^+); ^{1}H NMR (δ $CDCl_3$) 0.83 (t, 9H, CH_3, J = 7.4 Hz), 0.84 (t, 9H, CH_3, J = 7.4 Hz), 1.16–1.34 (m, 36H, CH_2), 1.62 (quint, 6H, CH_2, J = 7.4 Hz), 1.71 (quint, 6H, CH_2, J = 7.4 Hz), 2.65 (t, 6H, CH_2, J = 7.4 Hz), 2.72 (t, 6H, CH_2, J = 7.4 Hz), 7.26 (s 3H, thienylene protons), 7.50 (s, 3H, thienylene protons), 7.63 (d, 3H, phenylene protons, J = 7.0 Hz), 7.70 (d, 3H, phenylene protons, J = 7.0 Hz), 7.87 (d, 6H, phenylene protons, J = 8.6 Hz), 8.80 (d, 6H, phenylene protons, J = 8.6 Hz); ^{13}C NMR (δ $CDCl_3$) 14.02, 14.04 (CH_3), 22.47, 22.53, 29.0, 29.1, 29.2, 29.7, 30.5, 30.6, 31.47, 31.54 (CH_2), 125.4, 126.1, 126.4, 127.3, 129.5 (3C), 129.6, 132.8, 135.1, 137.8, 138.2, 139.0, 143.1, 143.5, 143.7, 153.8, 153.9, 170.7 (thienylene and phenylene carbons).

3.1.3. Synthesis of compound **6**

In a 50 mL three–necked flask was placed 9.2435 g (31.5 mmol) of 2-bromo-5-pentamethyldisilanylthiophene in 150 mL of THF. To this was added 20.0 mL (32.0 mmol) of a 1.6 M *n*-butyllithium–hexane solution at −80°C. After the mixture was stirred for 2 h at −80°C, the resulting mixture was slowly added to 10.8589 g (33.4 mmol) of tributyltin chloride at −80°C. After the mixture was stirred for 1 h at −80°C, it was warmed to room temperature and stirred over night. After the mixture was hydrolyzed with dilute hydrochloric acid. The organic layer was separated, washed with water, and dried over anhydrous magnesium sulfate. After the solvent was evaporated, compound **6** (10.4707 g, 66% yield) was isolated by a silica gel column eluting with hexane: MS *m/z* 504 (M^+) ^{1}H NMR (δ $CDCl_3$) 0.09 (s, 9H, $SiMe_3$), 0.37 (s, 6H, $SiMe_2$), 0.89 (t, 9H, CH_3, J = 7.2 Hz), 1.08–1.12 (m, 6H, CH_2), 1.34 (sext, 6H, CH_2, J = 7.2 Hz), 1.53–1.64 (m, 6H, CH_2), 7.25 (d 1H, thienylene proton, J = 3.6 Hz), 7.33 (d, 1H, thienylene proton, J = 3.6 Hz); ^{13}C NMR (δ $CDCl_3$) −2.5 (Me_2Si), −2.4 (Me_3Si), 10.9, 13.7, 27.2, 29.0 (Bu), 134.7, 136.2, 142.1, 144.5 (thienyl ring carbons).

3.1.4. Synthesis of compound **7**

In a 100 mL two–necked flask was placed 0.1031 g (0.0493 mmol) of **5** and 0.1668 g (0.331 mmol) of **6** and 0.0041 g (0.0058 mmol) of bis(triphenylphosphine)dichloropalladium in 23 mL of THF. After the mixture was heated to reflux for 4 days, it was hydrolyzed with dilute hydrochloric acid. The organic layer was separated, washed with water, and dried over anhydrous magnesium sulfate. After the solvent was evaporated, compound **7** (0.0274 g, 24% yield) was isolated by a silica gel column eluting with hexane-chloroform (2:1) as a dark red solid: M.p. 86–87 °C. HRMS (ESI) exact mass calcd for $C_{126}H_{154}N_9Si_6S_{12}$ ($M^+ + H$): 2344.75858, found 2344.75747. MS *m/z* 2343 (M^+); ^{1}H NMR (δ $CDCl_3$) 0.13 (s, 27H, $SiMe_3$), 0.40 (s, 18H, $SiMe_2$), 0.84 (t, 9H, CH_3, J = 7.0 Hz), 0.85 (t, 9H, CH_3, J = 7.0 Hz), 1.20–1.34 (m, 36H, CH_2), 1.69 (quint, 6H, CH_2, J = 7.6 Hz), 1.73 (quint, 6H, CH_2, J = 7.6 Hz), 2.69 (t, 6H, CH_2, J = 7.6 Hz), 2.74 (t, 6H, CH_2, J = 7.6 Hz), 7.10 (d, 3H, thienylene protons, J = 3.2 Hz), 7.21 (s 3H, thienylene protons), 7.31 (d, 3H, thienylene protons, J = 3.2 Hz), 7.52 (s, 3H, thienylene protons), 7.70 (d, 3H, phenylene protons, J = 7.6 Hz), 7.74 (d, 3H, phenylene protons, J = 7.6 Hz), 7.90 (d, 6H, phenylene protons, J = 8.4 Hz), 8.83 (d, 6H, phenylene protons, J = 8.4 Hz); ^{13}C NMR (δ $CDCl_3$) −2.9 (Me_2Si), −2.4 (Me_3Si), 14.1 (2C) (CH_3), 22.5 (2C), 29.2 (2C), 29.7 (2C), 30.6, 30.7, 31.64 (2C) (CH_2), 125.2, 125.6, 126.0, 126.6, 126.9, 127.2, 129.5, 129.6, 129.8, 131.0, 133.1, 134.8, 135.2, 137.9, 138.1, 139.1, 142.2, 142.7, 143.1, 143.6, 154.0 (2C), 171.0 (thienylene and phenylene carbons); ^{29}Si NMR (δ $CDCl_3$) −23.5, −18.8.

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