

Novel dendritic light-emitting materials containing polyhedral oligomeric silsesquioxanes core

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Abstract

A new series of star-like light-emitting materials (POSS1, POSS2 and POSS3) were synthesized by hydrosilylation of the polyhedral oligomeric silsesquioxane with 4,4'-bis(4-(di-(4-methylphenyl)amino)styryl)-2-(hexan-1-yloxy)-5-(10-undecen-1-yloxy)benzene (C1), 4,4'-bis[(E)-2-(*N*-ethylcarbazoyl)ethenyl]-2-(hexan-1-yloxy)-5-(10-undecen-1-yloxy)benzene (C2), and Iridium(III) bis(2-phenylpyridine-*C*²-*N'*) (13-teradecenyl acetonate) (C3), respectively. All synthesized materials are soluble in common organic solvents, such as chloroform, toluene and 1,2-dichloroethane, and exhibit good film-forming properties. Therefore, they can be used to fabricate devices by spin-coating. The aggregation of peripheral chromophores can be prevented by the rigid POSS core. A double-layer, light-emitting diode with the configuration of indium–tin oxide/poly(ethylene 3,4-dioxythiophene)/POSS1/Ca/Al was fabricated using POSS1 as the active layer. The device emitted green light with a maximum brightness of 115 cd/m² and a current yield of 0.07 cd/A. When the light-emitting layer is blended with 0.8% electron-transport material, 2-(4'-*tert*-butylphenyl)-5-(4'-diphenyl)-1,3,4-oxadiazole, the maximum brightness and current yield of the device can reach 1469 cd/m² and 0.8 cd/A, respectively. The performance of a POSS2 device with the same device structure can reach 1102 cd/m² and 0.88 cd/A. POSS3 is a kind of triplet material and blended with 4,4-*N,N'*-dicarbazole-biphenyl as host material, to fabricate the light-emitting device. The maximum brightness and current yield reach 1008 cd/m² and 1.04 cd/A, respectively. Blending with 1,3,5-tris(2-*N*-phenylbenzimidazolyl)benzene as the hole-blocking material yields a maximum brightness and current yield of 1172 cd/m² and 3.99 cd/A.

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1. Introduction

Organic light-emitting diodes (OLEDs) have attracted a great deal of attention not only in academic circles, but also for their application as full-color and white-light flat-panel displays [1,2]. Extensive investigations of OLEDs have been sought to achieve high brightness, high luminous efficiency and multi-color emission, and in particular, to improve the durability of devices. Thin films of small molecules are formed by thermal evaporation and large-area flat films are usually difficult to obtain. Solution-processing is generally thought to be a lower cost approach to manufacture large-area full-color displays, especially for polymer materials. Conjugated polymers have so far dominated solution-processing OLEDs, and devices with

good efficiencies have been reported [3]. More recently solution-processible dendrimers have been developed for use as the light-emitting layer in OLEDs. Light-emitting dendrimers consist of cores, dendrons and linking groups. The materials fall into two main classes — those in which the chromophores are attached to dendrons that contain non-conjugated moieties [4,5], and those in which the dendrons are fully conjugated [6,7]. The attached dendrimers have a number of potential advantages. First, the dendrons can be chosen as light-emitting groups or functional groups, such as hole-transport material (HTM) or electron-transport material (ETM). Second, the cores can also be chosen as light-emitting chromophores, HTM or ETM. Third, different cores and dendrons can be collocated to achieve balanced hole/electron mobility which is critical for highly efficient light-emitting devices.

Hybrid organic–inorganic polymers that contain segments of polyhedral oligomeric silsesquioxanes (POSS) exhibit a number

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of potentially useful properties, including high thermal stability in air and good adhesion to a number of substrates [8,9]. They can also be used as building blocks of organic–inorganic nanocomposite materials for various applications, owing to the ease with which functional groups can be attached to POSS [10–14]. For instance, POSS can be used as a monofunctional or graftable monomer, a difunctional comonomer, a surface modifier or a polyfunctional crosslinker to prepare miscellaneous polymers. In the application of semiconducting polymers, the brightness and quantum efficiency of poly(*p*-phenylene vinylene) and polyfluorene (PF) can be improved by incorporating bulky POSS into the end of the chain [15]. Recently POSS were also incorporated into PF as the center core [16,17] or side chains [18,19] to reduce aggregation as well as enhance light-emitting properties.

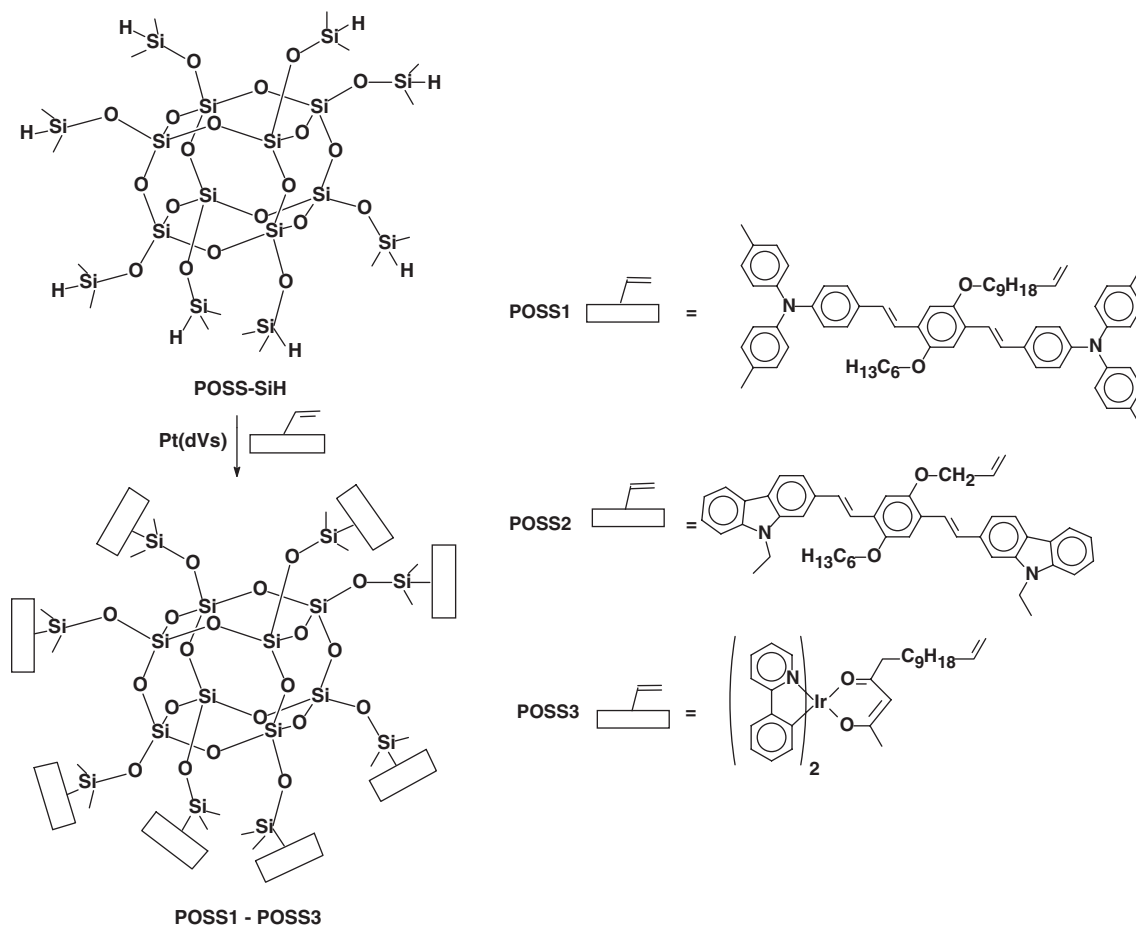
So far POSS were incorporated into polymers to enhance their thermal and/or luminescent properties. In this study, star-like light-emitting materials based on a silsesquioxane core attached with different chromophores were synthesized. The star-like materials should be round and bulky to reduce aggregation, and the thermal stability of the synthesized materials is enhanced by the inorganic and rigid POSS core [8,9]. Besides, a conventional spin-coating process can be applied to deposit these light-emitting materials into thin films, which reduces the complexity and cost by thermal evaporation process. The chemical structures, thermal

properties and optoelectrical characteristics of these materials are elucidated in this study.

2. Experimental section

2.1. Synthesis of materials

The chromophores C1, C2 and C3 were synthesized as described in the literature [20,21]. The overall yields were in the range of 40–60%. All of the chromophores link with a terminal vinyl bond as the reactive group. POSS1–POSS3 were prepared by hydrosilylation between C1–C3 and POSS–SiH, as shown in Scheme 1. The synthesis of POSS1 is given as follows. POSS–SiH (0.1 g, 0.098 mmol) and C1 (0.59 g, 0.86 mmol) was dissolved in dry toluene (3 mL) in a 10 mL flask. Three drops of platinum(0)-1,3-divinyl-1,1,3,3-tetramethyl disiloxane complex [Pt(dvs)] were then added. The solution was heated at 50 °C for 6 h in an atmosphere of nitrogen. After cooling to the ambient temperature, the reaction mixture was poured into methanol with stirring and filtered. The crude product was dissolved in THF and then re-precipitated in methanol. The above purification procedure was repeated three times. The obtained product was then dried at 60 °C under vacuum for 12 h. A yellow-greenish solid was obtained with a yield of 20.1% (0.12 g). POSS2 and POSS3 were synthesized by the same procedure. POSS2 was a yellow



Scheme 1. Synthetic route for POSS1–POSS3.

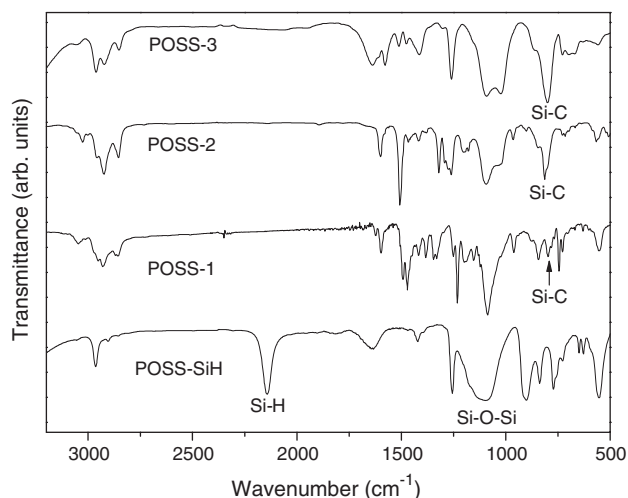


Fig. 1. FT-IR spectra of C1–C3 and POSS1–POSS3.

solid with a yield of 53.5% (0.16 g). POSS3 was also a yellow solid with a yield of 33% (0.2 g).

2.2. Characterization methods

The synthesized materials were characterized by the following techniques. FT-IR spectra were recorded on a Perkin-Elmer Spectrum One FT-IR spectrometer with KBr pellets. Nuclear magnetic resonance spectra (300 MHz) were recorded on a Varian VXR-300 spectrometer. Gel permeation chromatography (GPC) data assembled from Viscotek with an RI detector and three columns in series were used to measure the molecular weight distribution relative to polystyrene standards at 40 °C. Differential scanning calorimetry was performed on a SEIKO DSC 6200 unit at a heating rate of 20 °C min⁻¹ and a cooling rate of 50 °C min⁻¹. Thermal gravimetric analysis (TGA) was undertaken on a Perkin-Elmer Pyris 1 TGA instrument with a heating rate of 10 °C min⁻¹. UV–Vis spectra were obtained with an HP 8453 diode array spectrophotometer. Photoluminescence (PL) spectra were obtained using ARC SpectraPro-150 luminescence spectrometer.

A degassed CHCl₃ solution was used to determine the PL quantum yields (ϕ_f). The concentration was adjusted to reduce the absorbance of the solution below 0.1, and the excitation was performed at the corresponding $\lambda_{\text{ex, max}}$. A solution of quinine sulfate in 1 M H₂SO₄, which has a ϕ_f of 0.546 ($\lambda_{\text{ex}} = 365$ nm), was

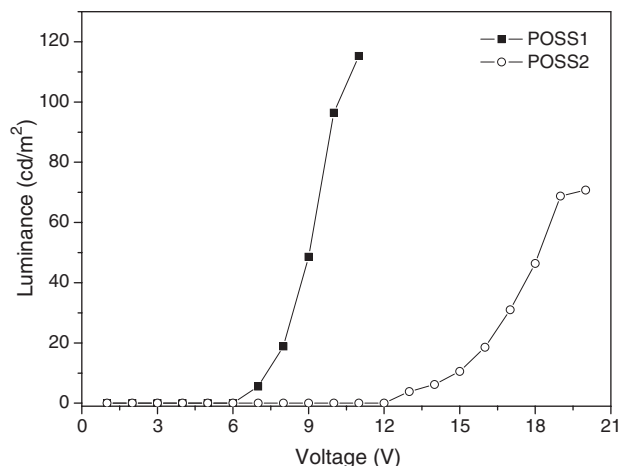


Fig. 2. Luminance–voltage characteristics of LED from POSS1 and POSS2.

used as a standard. Cyclic voltammetric measurements of the material were made in acetonitrile with 0.1 M tetrabutylammonium hexafluorophosphate as the supporting electrolyte at a scan rate of 80 mV/s. Platinum wires were used as both the counter and working electrodes, and silver/silver ions (Ag in 0.1 M AgNO₃ solution, from Bioanalytical Systems, Inc.) was used as the reference electrode. Ferrocene was used as an internal standard, and the potential values were obtained and converted to vs SCE (saturated calomel electrode). The corresponding highest-occupied molecular orbital (HOMO) and lowest-unoccupied molecular orbital (LUMO) energy levels were estimated from the onset redox potentials.

2.3. LED fabrication

Double-layer LEDs were fabricated as sandwich structures between aluminum (Al) cathodes and indium–tin oxide (ITO) anodes. ITO-coated glass substrates were cleaned sequentially in ultrasonic baths of detergent, 2-propanol/deionized water (1:1 volume) mixture, toluene, deionized water and acetone. A 50 nm-thick hole injection layer of poly(ethylene 3,4-dioxythiophene) (PEDOT) doped with poly(styrenesulfonate) was spin-coated on top of ITO from a 0.7 wt.% dispersion in water and dried at 150 °C for 1 h in a vacuum. Thin films of POSS1, POSS2 and their blends with 0.8 wt.% 2-(4'-*tert*-butylphenyl)-5-(4'-diphenyl)-1,3,4-

Table 1
Thermal and optical properties of the materials

Material	T_g^a (°C)	T_d^b (°C)	$\lambda_{a, \text{max}}^c$			$\lambda_{\text{em, max}}^d$			ϕ_f^e	E_g^f (eV)
			CHCl ₃ (nm)	Toluene (nm)	Film (nm)	CHCl ₃ (nm)	Toluene (nm)	Film (nm)		
C1	85	300	430	432	430	496	486	502	0.51	2.48
C2	81.2	328	409	411	413	464	461	496	0.39	
C3	43.9	314	457	463	465	534	535	539	0.36	
POSS1	—	386	404	405	393	495	493	492	0.66	2.49
POSS2	88.8	384	408	410	413	471	468	496	0.46	
POSS3	128.4	336	457	464	466	534	534	539	0.58	

^a T_g : glass transition temperature determined by the DSC curve. ^b T_d : thermal degradation temperature at 5 wt.% loss. ^c $\lambda_{a, \text{max}}$: the absorption maxima from the UV–Vis spectra in CHCl₃ solution or in thin film. ^d $\lambda_{\text{em, max}}$: the PL maxima in CHCl₃ solution or in thin film. ^e ϕ_f : PL quantum yield. ^f E_g : the optical energy gap calculated from the UV–Vis spectra in thin film.

Table 2
Electrochemical properties of the materials

	E^{ox} (V) vs Ag/Ag ⁺	E^{red} (V) vs Ag/Ag ⁺	HOMO (eV)	LUMO (eV)	EC E_g (eV)
C1	1.22	-1.27	5.16	2.62	2.54
C2	1.15	-1.42	5.09	2.47	2.62
C3	1.27	-1.06	5.21	2.83	2.38
POSS1	1.24	-1.26	5.18	2.63	2.55
POSS2	1.11	-1.46	5.05	2.43	2.62
POSS3	1.32	-1.01	5.26	2.88	2.39

oxadiazole (PBD) were spin-coated from the 1,2-dichloroethane solutions onto the PEDOT layer and dried at 50 °C overnight in a vacuum. The POSS3 device was prepared by blending it with 18 wt.% 4,4-*N,N'*-dicarbazole-biphenyl (CBP) and 50 wt.% 1,3,5-tris(2-*N*-phenylbenzimidazolyl)benzene (TPBI) as a light-emitting layer. The thickness of the active layer was ca. 50 nm. Finally, 30 nm Ca and 120 nm Al electrodes were made through a shadow mask onto the polymer films by thermal evaporation using an AUTO 306 vacuum coater (BOC Edwards, Wilmington, MA). Evaporations were carried out typically at base pressures lower than 2.66×10^{-4} Pa. The active area of each electroluminescent (EL) device was 4 mm² and the device was characterized following a published protocol [22].

3. Results and discussion

3.1. Structures and thermal properties

Fig. 1 shows FT-IR spectra of POSS-SiH, POSS1, POSS2 and POSS3. Several characteristic absorption bands were found at, for example, 2143 cm⁻¹ (Si-H bending), 1203 cm⁻¹ (Si-CH₃ stretching) and 1087 cm⁻¹ (Si-O-Si stretching) [23]. The strong Si-H absorption was observed in POSS-SiH, while it was diminished in POSS1-POSS3. Meanwhile, a new Si-C stretching band was found at 790–800 cm⁻¹ which showed that chromophores were linked to the POSS core. From GPC results, the molecular weights (*M_w*, *M_n*) of POSS1 were estimated to be 9200 and 8310. The molecular weights (*M_w*, *M_n*) of POSS2 are 4140 and 4290. The molecular weights (*M_w*, *M_n*) of POSS3 are 5840 and 7411. The average arm numbers of three POSS-containing materials were calculated according to the following equation:

$$\# \text{ arms} = (\overline{M}_n - M_w \text{ of POSS core}) / (M_w \text{ of the chromophore})$$

resulting in 7.7, 4.6 and 6.4 for POSS1, POSS2 and POSS3, respectively, based on the weight of the silsesquioxane core, 1017. Sellinger et al. claimed that nearly complete substitution of eight arms was observed. Even uncommon 9 and 10 substitutions were found; this is quite surprising result considering steric factors [17]. For the chemical structure that they proposed, those chromophores were attached to the POSS core via the elimination of terminal bromo group. Chromophores were considerably spread outward. For the chemical structure that we proposed, the chromophores were laterally attached to the POSS core, and larger steric hindrance was brought. Eight arms were not totally replaced in our

case. These results suggest that POSS1-POSS3 are star-like dendrimers that consist of arms on the silsesquioxane core. Although POSS is a rigid core, the synthesized dendrimers are soluble in common organic solvents, such as chloroform, toluene and 1,2-dichloroethane. Transparent and self-standing films of the dendrimers can be cast using these solvents.

Table 1 presents the thermal properties of the materials. The thermal decomposition temperatures (*T_d*) of C1 and POSS1 are 300 and 386 °C, respectively. The increased *T_d* of POSS1 suggests that the incorporation of the silsesquioxane core enhanced the thermal stability of C1. POSS2 and POSS3 yielded the same results. Table 1 also presents the glass transition temperatures (*T_g*) obtained from the DSC curve. The *T_g* values of POSS2 and POSS3 are increased. The *T_g* of POSS3, is considerably increased by about 80 °C using silsesquioxane as the core. These results suggest that the incorporation of POSS enhanced the thermal properties of chromophores.

3.2. Optical properties

Table 1 presents the UV-Vis absorption and PL emission spectroscopic data. The UV-Vis absorption spectra of C1 in different solvents and film include only one absorption peak with a maxima between 430 and 432 nm, which is attributed to the π - π^* transition along the conjugated backbone. C2 also exhibits one absorption peak between 409 and 413 nm. In contrast, the absorption spectrum of POSS1 shows one peak with maxima between 393 and 403 nm in different solvents and the film state, which is blue-shifted from that of C1. The reason to this observation is explained as follows. The rigid and bulky POSS is incorporated among the chromophores, and the aggregation of surrounding chromophores is effectively suppressed. The blue-shifted and narrower UV-Vis absorption spectrum is consistent with this observation. However, the peak of POSS2 is almost at the same wavelength as that of C2, perhaps because the carbazole group is more planar than the triphenylamine group and the aggregation is not so easily eliminated. C3 and POSS3 are triplet materials and the absorption peak of ³MLCT is between 457 and 466 nm in the

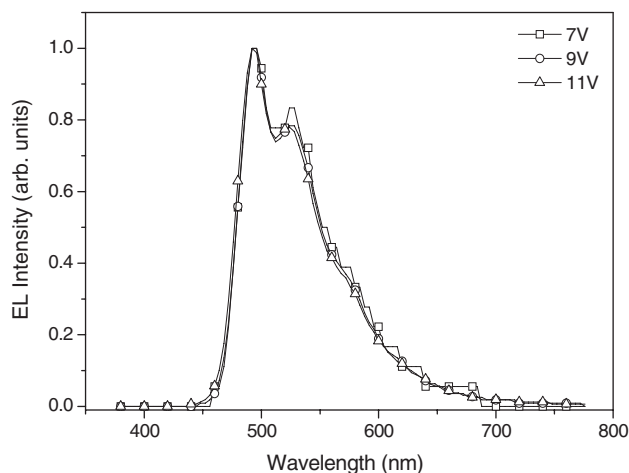


Fig. 3. EL spectra from POSS1 under different voltages.

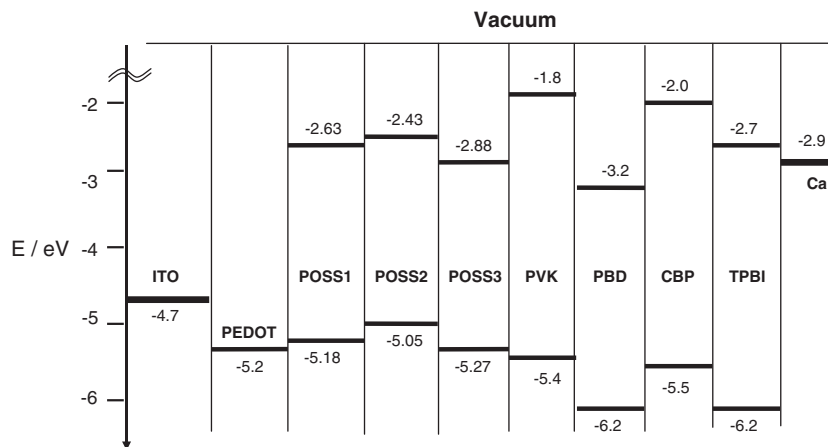


Fig. 4. Energy level of the organic and electrode materials.

solution or film state. The $^3\text{MLCT}$ absorption bands thus are associated with high oscillator strengths, and strong spin-orbit coupling leads to efficient phosphorescence [24].

The peak maximum (λ_{max}) of the PL spectra of the chromophore C1 varies very little among solvents. The red-shift due to the aggregation is observed when the material is dissolved in a poor solvent or in the film state [25]. The shoulder band around 520 nm also increased in this case. This is evident that aggregation also occurs in a poor solvent. However, the PL λ_{max} values of POSS1 in different solvents are all around 493 nm without significant change. Moreover, the aggregation at around 520 nm is less clear in POSS1 than that in C1, because a rigid POSS molecule was incorporated to prevent aggregation. The PL λ_{max} of POSS2 is the most red-shift in thin film state among three POSS materials. The reason could be due to the C2 chromophore which is more planar than the other two C1 and C3 chromophores. Sellinger et al. claimed that thin film and solution (toluene) PL spectra have nearly identical shapes and somewhat blue-shift (~ 4 nm) in films, suggesting no tendency towards aggregation in the solid state [17]. Those chromophores were spread outward. In our experiments some extent of red-shift was observed (~ 5 nm).

This could be the effect of aggregation since chromophores were laterally attached to the POSS core.

The PL quantum yields (Φ_f) of the materials in CHCl_3 solution were determined [26,27] using quinine sulfate as a standard. POSS1 shows a higher Φ_f value 0.66 than of C1 (0.51). The Φ_f value of POSS2 is 0.46, which exceeds that of C2 (0.39). The Φ_f value of POSS3 is 0.58, which exceeds that of C3 (0.36). These results suggest that the incorporation of the POSS core suppresses the formation of aggregates and reduces self-quenching. These phenomena are consistent with earlier observation in previous literatures [15–17].

3.3. Electrochemical properties

Table 2 shows the electrochemical properties of C1–C3 and POSS1–POSS3. For POSS1, a reversible oxidation is shown with an onset potential of ca. 1.24 V vs SCE, whereas the reduction with an onset of ca. -1.26 V vs SCE for POSS1. This suggests that the presence of the triphenylamine group makes the material more suitable for p-doping than n-doping. Based on the onset redox potentials, the ionization potential (HOMO

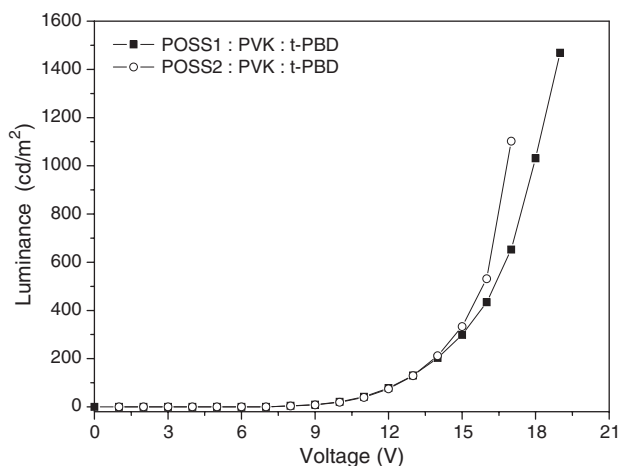


Fig. 5. Luminance–voltage characteristics of LED from POSS1 and POSS2 blending with PBD and PVK.

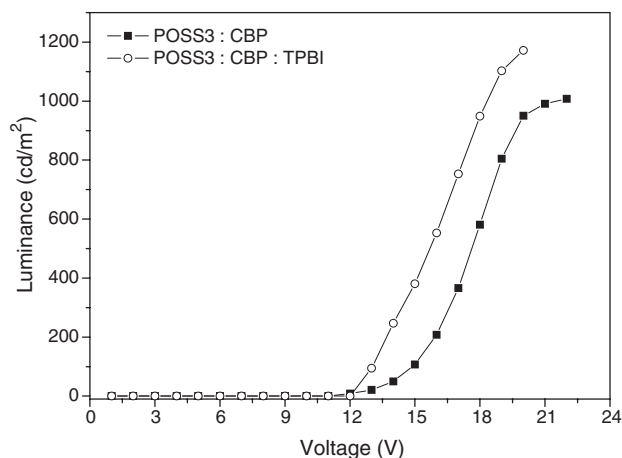


Fig. 6. Luminance–voltage characteristics of LED from POSS3 with different device structures.

level) and electron affinity (LUMO level) of POSS1 are estimated to be 5.62 and 3.12 eV, respectively, assuming a ferrocene (FOC) value of -4.8 eV below the vacuum level ($E_{\text{FOC}}=0.41$ V vs Ag/Ag^+). The corresponding electrochemical band gap is 2.5 eV, which matches closely the optical band gap observed from the UV–Vis absorption data. POSS2 is also more suitable for p-doping because of the presence of the carbazole group; however, the energy barrier between PEDOT and POSS2 is higher than that with POSS1. The electrochemical band gaps of POSS2 and POSS3 are also highly consistent with the UV–Vis absorption results. The band gaps of POSS1–POSS3 do not differ from those of C1–C3 when POSS is the center core.

3.4. EL properties of LED devices

Fig. 2 plots the luminance–voltage characteristics of the double-layer devices with a configuration of ITO/PEDOT/POSS1 or POSS2/Ca/Al. The turn-on voltage of the POSS1 device is 7 V and $\lambda_{\text{EL-max}}$ is 496 nm. The maximum brightness of 115 cd/m^2 at 11 V and the maximum external quantum efficiency of 0.07 cd/A were obtained at 10 V. The turn-on voltage of the POSS2 device is 13 V and $\lambda_{\text{EL-max}}$ is 476 nm. The maximum brightness of 70 cd/m^2 and the maximum external quantum efficiency of 0.02 cd/A are obtained at 20 V. Fig. 3 shows EL spectra of the POSS1 at various voltages. No significant peak shift or new band formation is observed at a high driving voltage. Therefore, the device exhibits stable luminescence because of the star-like molecule which prevents the close packing of the chromophores, and the excimer or aggregation in this case. The POSS2 device yields similar results.

Blends of POSS1 or POSS2 were made with 0.8 wt.% PBD to enhance the electron transport through the device to improve the performance and efficiency of the device. Poly(*N*-vinyl carbazole) (PVK) (1 wt.%) was also added to increase the HOMO level which is close to that of PEDOT. Therefore the hole injection is improved and the device performance would be driven higher. Fig. 4 shows the energy diagram and Fig. 5 plots the luminance–voltage characteristics of the corresponding EL device. The turn-on voltage of the blended POSS1 device is 8 V and $\lambda_{\text{EL-max}}$ is 496 nm. A maximum brightness of 1469 cd/m^2 at 19 V and a maximum current yield of 0.8 cd/A at 14 V were obtained. The turn-on voltage of the blended POSS2 device is 8 V and $\lambda_{\text{EL-max}}$ is 476 nm. A maximum brightness of 1102 cd/m^2 at 17 V and a maximum current yield of 0.88 cd/A at 8 V were observed. These values represent ten-fold enhancements in performance over pristine POSS1 or POSS2 devices. With reference to an investigation in 1996, Hosokawa et al. applied the vacuum evaporation process to fabricate OLED devices using small molecules similar to C1 and C2. The performance of the device was 210 and 162 cd/m^2 at 7 V, respectively [20]. Using POSS as the center core in POSS1 and POSS2 also enhanced the performance of the device. The modest efficiencies of the EL device were further improved by optimizing the blend composition.

POSS3 is a triplet light-emitting material. The compound CBP is used as the host to prevent the concentration quenching [21]. Fig. 6 plots the luminance–voltage characteristics of the POSS3

devices. The turn-on voltage of the device is 12 V and $\lambda_{\text{EL-max}}$ is 524 nm. A maximum brightness of 1008 cd/m^2 at 22 V and a maximum current yield of 1.04 cd/A at 17 V were observed. TPBI was added to the light-emitting layer as the hole-blocking material to improve further [28]. The turn-on voltage of the device is 13 V and $\lambda_{\text{EL-max}}$ is 524 nm. A maximum brightness of 1172 cd/m^2 at 20 V and a maximum external quantum efficiency of 3.99 cd/A at 14 V were observed. However, the performance of the device was not as good as that of the conventional triplet OLED device, because the purity of triplet materials is very important. Notably, blending with PBD, PVK or CBP does not change the EL spectra, because the POSS core reduces close packing or aggregation, and further blending with those materials has or no effect on the emission spectra.

4. Conclusion

New light-emitting materials were synthesized with POSS as the core to link the chromophores. The synthesized dendrimers exhibit good thermal, electro-oxidative stabilities and film-forming properties. Bright green and green-bluish emission from POSS1 and POSS2 can be observed in the device structure that is blended with PVK as the hole-transporting material and PBD as the electron-transporting material; the maximum brightness and current yield of the device were 1469 cd/m^2 and 0.8 cd/A for POSS1, and 1102 cd/m^2 and 0.88 cd/A for POSS2, respectively. POSS3 is a triplet material. Its maximum brightness and current yield were 1172 cd/m^2 and 3.99 cd/A when CBP was used as the host and TPBI used as the hole-blocking material.

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