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Emission enhancement and color adjustment of silicon-cored structure in tetraphenyl benzene with aggregation-enhanced emission[†]

Hua Wang,^a Yan Liang,^b Huanling Xie,^a Linglong Feng,^a Haifeng Lu^{*a} and Shengyu Feng^{*a}

In this paper, we report the synthesis and optical behavior of tetraphenyl benzene and its two derivatives. All the three compounds exhibited aggregation-enhanced emission (AEE) properties at a low concentration. The AEE mechanism was investigated and is due to restricted intramolecular rotation and unique packing structure. Furthermore, this kind of dendritic benzene derivatives exhibited interesting optical properties with increased concentration. Furthermore, we found a new phenomenon that the silicon-cored structure could efficiently enhance emission intensity and adjust the emission colors of dendritic benzene. The phenomenon was called "silicon-cored effect." This effect may give some guidance to the design of new luminescent materials with AIE and AEE properties.

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Introduction

The derivatives of benzene with multiple contiguous phenyl substituents have many potential applications in many areas of science and technology, such as modification of luminescent materials for optoelectric devices, template materials for supramolecular assembly, and the preparation of analogues of graphene.1-22 Aryl substituents are not constrained by additional bonds in the plane of the aromatic core, and thus, the aryl substituents exhibit large, twisted angles with the central benzene.⁶⁻⁹ Therefore, the derivatives of benzene with multiple contiguous phenyl substituents have much more complex nonplanar topologies than planar molecules.15 These nonplanar topologies could efficiently limit conjugation and inhibit extensive aromatic π - π and C-H··· π interactions.^{6-9,15} Therefore, these compounds typically show greater highestoccupied molecular orbital lowest-unoccupied molecular orbital gaps, lower degrees of self-association, less efficient packing, and higher solubility than planar analogues with similarly high thermal stability.15,23

Currently, these dendritic benzene derivatives are mostly used to modify the plane conjugate chromophore in optical applications.⁶⁻¹⁰ The introduction of the dendritic benzene could efficiently restrain the aggregation of luminescent compounds, which can broaden their emission bands, shift the frequencies emitted, and cause quenching. These potential effects allow thin films to be processed from solution as long-lived amorphous phases with high values of T_{g} , excellent thermal stability, efficient luminescence, and other attractive properties. Most studies focused on the assistant function of dendritic benzenes in optical application, but information on their self-optical properties is lacking. Tang's group found that benzene-cored luminophors exhibited aggregation-induced emission properties and the mechanism could be attributed to the restriction of intramolecular rotations.²⁴⁻²⁶ Based on this result, we designed and synthesized two derivatives of benzene with multiple contiguous phenyl substituents, 1,2,3,4-tetraphenyl benzene (TPB) and 1,2,3,4,5-pentaphenyl benzene (PPB) (see Scheme 1). The synthesis process is provided in ESI.[†] We found that the two compounds exhibited common aggregation-enhanced emission (AEE) at low concentration. However, these compounds exhibited distinctive optical

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Scheme 1 The molecular structures of TPB, PPB and BTPB-DPS.

^eKey Laboratory of Special Functional Aggregated Materials& Key Laboratory of Colloid and Interface Chemistry (Shandong University), Ministry of Education, School of Chemistry and Chemical Engineering, Shandong University, Jinan, 250100, People's Republic of China. E-mail: fsy@sdu.edu.cn; Fax: +86-531-88564464; Tel: +86-531-88364866

^bCollege of Food and Bioengineering, Qilu University of Technology, Jinan, Shandong 250353, People's Republic of China

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properties, which were caused by aggregation at high concentration.

The silicon-cored aromatic molecules are appropriate luminescent materials because of their high values of T_{g} , excellent thermal stability, efficient luminescence, and other attractive properties that can be attributed to the non-planar topologies of the silicon-cored tetrahedron structure.27-31 When the siliconcored structure is introduced to non-planar phenyl-substituted benzenes, the product would have much more complicated topologies. These unique topologies may generate some new significant properties in many areas of science and technology. In this paper, we also report the synthesis of bis(1,2,3,4-tetraphenyl benzene-yl) diphenylsilane (BTPB-DPS) (see Scheme 1). The synthesis process is provided in ESI.[†] BTPB-DPS exhibits an interesting AEE phenomenon. Furthermore, this silicon-cored derivative shows higher fluorescence emission and larger emission wavelength than the pure dendritic benzene derivatives.

Results and discussion

Optical properties of tetraphenyl benzene derivatives at low concentration

The PL behavior of 1,2,3,4-tetraphenyl benzene (**TPB**) as the simplest model in solution and aggregation states was initially examined. Fig. 1 shows that its maximum emission wavelength was 343 nm in tetrahydrofuran (THF) solution and the PL intensity was very weak. When large amounts of water were added to their THF solutions (with final concentrations constant at 2×10^{-5} mol L⁻¹), PL intensity gradually increased with increased water content in THF. Therefore, this simple molecule exhibited aggregation-enhanced emission (AEE). The PL peak intensity remained almost unchanged when less than



Fig. 1 (a) PL spectra of the dilute solutions of TPB (2×10^{-5} mol L⁻¹) in water/THF mixtures with different water contents. (b) PL spectra of the dilute solutions of PPB (2×10^{-5} mol L⁻¹) in water/THF mixtures with different water contents. (c) PL spectra of the dilute solutions of BTPB-DPS (10^{-5} mol L⁻¹) in water/THF mixtures with different water contents. The excitation wavelength was 280 nm. (d) Changes in the PL peak intensities of TPB, PPB and BTPB-DPS with different water fractions in the water/THF mixtures.



Fig. 2 The absorption spectra of TPB (a), PPB (b) and BTPB-DPS (c) with water/THF content at 0% and 99%.

70% water was added to the THF solutions, but intensity rapidly increased thereafter because the solvation power of the mixture had lowered to such an extent that the dyes had begun to aggregate. When the water fraction was increased to 99%, the PL peak intensities increased by up to elevenfold. The fluorescent quantums in solution and aggregate were also measured. The fluorescence quantum was just 4.3% in THF solution. When the water fraction was increased to 99%, the fluorescence quantum increased to 10.3%.

PPB and **BTPB-DPS** were also tested to further investigate the optical behavior. The two compounds exhibited AEE properties at low concentration. When the water fraction was increased to 99%, the PL peak intensities were increased by up to eleven and twelvefold, and the fluorescent quantums increased from 3.9% to 7.6% for **PPB** and 4.5% to 12.7% for **BTPB-DPS**. According to Tang's work on the AIE mechanism of benzene-cored luminophors, the AEE of the three compounds was due to the restricted intramolecular rotation.^{24,25}

As shown in Fig. 2, the experimental UV data of **TPB** in THF and water were tested to further investigate the aggregationenhanced emission properties. In solution, **TPB** exhibited the maximum peak absorption spectra at 244 nm. The 244 nm band is associated with the π - π transition of the phenyl group of **TPB** molecule in dilute THF solution. When the volume of water fractions increased to 99%, the maximum peaks in the absorption spectra of **TPB** are red shifted and a new shoulder band appeared at around 293 nm. The absorption spectra of **PPB** and **BTPB-DPS** also exhibited a bathochromic shift of maximum UV absorption when the water content was 99% in contrast to those in pure THF solution (from 245 nm to 284 nm and 246 nm to 280 nm, respectively).

Optical properties of tetraphenyl benzene derivatives in different concentrations

After discussing the AEE properties of the three compounds in water/THF systems, we further investigated the optical properties in pure THF solutions at different concentrations. The results were interesting.

TPB was dissolved in THF at concentration of 2×10^{-5} mol L^{-1} , 2×10^{-4} mol L^{-1} , 2×10^{-3} mol L^{-1} and 2×10^{-2} mol L^{-1} . As the concentration increased in THF, the emission bands of **TPB** exhibited a gradual red-shift tendency from 341 nm to 377 nm when the emission intensity increased (see Fig. 3 and Table 1). Similarly, the excitation bands also exhibited a bathochromic shift from 276 nm to 323 nm (Fig. 4 and Table 1). Meanwhile, the fluorescence emission intensity finally increased by ninefold. At low concentration, the



Fig. 3 (a–c) PL spectra of the THF solutions of TPB, PPB and BTPB-DPS with different concentrations; (d) PL spectra of the THF solutions of TPB, PPB and BTPB-DPS, 2×10^{-2} mol L⁻¹ for TPB and PPB, 10^{-2} mol L⁻¹ for BTPB-DPS.

 Table 1
 The fluorescence emission and excitation data of three compounds in different concentrations

Concentration/ mol L ⁻¹		2 imes 1	0^{-5}	2×1	10^{-4}	$2 \times$	10^{-3}	$2 \times$	10^{-2}
ТРВ	λ_{em}/nm	341		347		357		377	
	λ _{ex} /nm	276		290		309		323	
PPB	λ_{em}/nm	343		347		359		378	
	λ_{ex}/nm	273		290		310		324	
Concentration/			=	-5	–4		3		2
mol L			10	0	10 .		10 °		10 -
BTPB-DPS	$\lambda_{\rm em}/{\rm nm}$		346		349		378		408
	λ_{ex}/nm		272		281		323		336

aggregation of **TPB** molecules only enhanced the intensity of fluorescence emission with no red-shift (Fig. 1). At higher concentration, the aggregation of **TPB** molecules exhibited



Fig. 4 The excitation spectra of (a) TPB, (b) PPB and (c) BTPB-DPS with different concentrations.

not only fluorescence emission intensity enhancement, but also a bathochromic shift.

Encouraged by this result, we also tested **PPB** and **BTPB-DPS** to further investigate the optical properties of the compounds in this series. The concentrations in THF were set at 2×10^{-5} mol L⁻¹, 2×10^{-4} mol L⁻¹, 2×10^{-3} mol L⁻¹ and 2×10^{-2} mol L⁻¹ for **PPB**, and 10^{-5} mol L⁻¹, 10^{-4} mol L⁻¹, 10^{-3} mol L⁻¹ and 10^{-2} mol L⁻¹ for **BTPB-DPS**. The two derivatives also exhibited the fluorescence enhancement and wavelength red-shift properties in the THF solution (see Fig. 3 and Table 1).

As the concentration increased in THF, the emission bands of **PPB** exhibited a gradual red-shift tendency from 343 nm to 378 nm, and the excitation bands exhibited a bathochromic shift from 273 nm to 324 nm. Meanwhile, the fluorescence emission intensity also increased by sixfold. The optical property of **PPB** was similar to the **TPB** molecules. It indicated that the presence of an additional phenyl group did not have a significant influence on the optical properties of **TPB**.

For the silicon-cored derivative **BTPB-DPS**, the results were distinctive and exciting. **BTPB-DPS** exhibited a gradual red-shift tendency from 346 nm to 408 nm, and the excitation bands exhibited the bathochromic shift from 272 nm to 336 nm (Fig. 4 and Table 1). Meanwhile, the fluorescence emission intensity also increased by ninefold.

We found an interesting phenomenon in the optical properties of tetraphenyl benzene and silicon-cored derivative. As shown in Fig. 5, emissions were all colorless for **TPB** at different concentrations, and the same phenomenon was observed for **PPB** under a 365 nm UV lamp. However, **BTPB-DPS** exhibited obvious blue emissions in 10^{-3} and 10^{-2} mol L⁻¹ THF solution. We dropped the THF solution of **TPB**, **PPB** and **BTPB-DPS** on thin-layer chromatography (TLC) plate to further investigate the solid state fluorescent properties. After solvent evaporation, fluorescence was observed under UV lamp. As shown in Fig. 3,



Fig. 5 Images of TPB and BTPB-DPS in THF solutions with different concentrations under 365 nm UV lamp. For TPB: (a) 2×10^{-5} mol L⁻¹, (b) 2×10^{-4} mol L⁻¹, (c) 2×10^{-3} mol L⁻¹, (d) 2×10^{-2} mol L⁻¹; for BTPB-DPS: (A) 10^{-5} mol L⁻¹, (B) 10^{-4} mol L⁻¹, (C) 10^{-3} mol L⁻¹, (D) 10^{-2} mol L⁻¹.

under the UV lamp (365 nm), **TPB** on TLC plate showed no luminescence and **PPB** showed the same phenomenon, whereas **BTPB-DPS** exhibited obvious blue light emission.

According to the above-mentioned phenomenon, **BTPB-DPS** is an interesting kind of AIE system. The fluorescence variation from colorless to blue emission of **BTPB-DPS** is due to the emission bathochromic shift and intensity enhancement. It is different from other molecules, which are only based on the variation of fluorescent intensity.^{25,32-36}

Furthermore, when the concentration of tetraphenyl benzene groups were the same at 2×10^{-2} mol L⁻¹, **BTPB-DPS** exhibited higher fluorescence emission and blue emission in contrast to the colorless emission of **TPB** and **PPB** (see Fig. 2d). Therefore, the silicon-cored derivative **BTPB-DPS** exhibited the emission enhancement and color adjustment effect of tetraphenyl benzene. Because the silicon-cored structure did not enlarge the conjugation of tetraphenyl benzene, the reason may be due to the molecular packing ways in aggregate states. To our knowledge, this enhancement and adjustment effect of silicon-cored structure was the first report in the investigation of AIE systems. Particularly, when the silicon-cored structure was introduced to the dendritic benzene, the silicon-cored

derivative exhibits higher fluorescence and larger emission wavelength than the dendritic benzene. It means that the silicon-cored structure could efficiently enhance the fluorescence and adjust the emission wavelength of dendritic benzene. This phenomenon can be called the "silicon-cored effect" in this series of dendritic benzene derivatives.

The crystal structures of tetraphenyl benzene derivatives

The crystal structures of the three compounds were also further investigated to explain the above-mentioned unique optical phenomenon.

The molecules of **TPB** exhibited a symmetrical structure. With four contiguous phenyl substituents, **TPB** is forced to adopt a conformation with large torsional angles (50.3° and 68.7°) between the central and peripheral aromatic rings. In one direction, each molecule engages in two C–H··· π interactions with two neighbors (C–H··· π centroid distances of 3.5 Å) to define chains that pack to form corrugated sheets (Fig. 6a). The molecules were packed side by side. In the other direction, adjacent sheets are joined by four additional C–H··· π interactions per molecule (C–H··· π centroid distances of 3.2 and 3.7 Å),



Fig. 6 The packing modes of TPB (a and b), PPB (c and d), BTPB-DPS (e and f); (hydrogen atoms are omitted for clarity in e and f).

with each central aromatic **TPB** ring serving as a double acceptor (Fig. 6b). The molecules were packed head to tail.

As expected, the average torsional angles between the central aromatic ring and the five phenyl groups of **PPB** are slightly larger than those of **TPB**. **PPB** formed sheets with prominent embraces, which define dimers joined by two C-H··· π interactions (C-H··· π centroid distances of 3.3 Å). Adjacent sheets are joined by four additional C-H··· π interactions per molecule (C-H··· π centroid distances of 3.6 and 4.0 Å), with each central aromatic ring of **PPB** serving as a double acceptor (Fig. 6c and d). In one direction, the molecules were packed side by side. In the other direction, the molecules were packed head to tail. According to the above mentioned discussion, **PPB** has similar molecular packing ways to **TPB**. These molecular packing modes made molecules of **TPB** and **PPB** have efficient fluorescence without π ··· π quenching in their aggregate states.³⁷⁻⁴⁴

BTPB-DPS exhibited a different crystal structure. The whole molecule shows a tetrahedral structure. The two tetraphenyl benzene groups attached on the Si atom exhibited different topological structures. The torsional angles between the central and peripheral aromatic rings are 43.8°, 69.3°, 75.4°, and 76.3° for one and 66.1° , 68.0° , 68.8° , and 83.5° for the other. The torsional angles in silicon-cored derivatives of BTPB-DPS are larger than those of TPB and PPB. Larger torsional angles resulted in weaker intermolecular interactions. However, it was interesting that the intramolecular C-H \cdots π interaction was found in BTPB-DPS molecules. For the intermolecular interactions, in one direction, only the C-H $\cdots\pi$ interaction (centroid distances of 3.3 Å) between the H atom of one phenyl group in four peripheral phenyl groups and the phenyl group on the Si atom was found (Fig. 6e). In the other direction, the distance to neighbouring molecules was greater than 10 Å and the molecular adhesion only depends on weak van der Waals' forces (Fig. 6f). To sum up, BTPB-DPS shows weaker intermolecular interactions than TPB and PPB. The reason that BTPB-DPS exhibited higher fluorescence than TPB and PPB could also be attributed to this. Furthermore, the weak intermolecular interactions resulted in the disorderly arrangement of BTPB-DPS molecules in the aggregated states in contrast to the other "pure" aromatic hydrocarbon compounds (TPB and PPB). The larger bathochromic shift could be attributed to the intramolecular interactions and the disorderly arrangement in concentrated solution.

According to the above-mentioned data, the AEE phenomenon mechanism of the three compounds is the synergistic effect of the restriction of intramolecular rotations and unique molecular packing modes in the aggregate state, which could avoid the fluorescence quenching.³⁷⁻⁴⁴

Conclusions

In conclusion, we investigated the optical behavior of **TPB** and the other two derivates. All these compounds exhibited AEE properties at low concentration with increased water content. The AEE could be due to intramolecular restriction and unique molecular packing modes. Furthermore, this kind of benzene with multiple contiguous phenyl substituent derivatives exhibited the gradual bathochromic shift and emission enhancement in THF solution with increased concentration. The silicon-cored derivative **BTPB-DPS** showed a unique AIE property and exhibited better optical properties than the hydrocarbon compounds (**TPB** and **PPB**). This phenomenon of emission enhancement and color adjustment of the siliconcored structure is called the "silicon-cored effect", which may be due to the unique silicon-cored structure that results in a highly complicated topological structure in aggregated states. This class of highly fluorescent dendritic benzene compounds can lead to new applications, such as optoelectronics, sensors, and biological devices.

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