

# Supporting information

## Facile Construction of Dendritic Amphiphiles with Aggregation-Induced Emission Characteristics for Supramolecular Self-Assembly

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## Materials and Methods

**Materials:** All reagents were commercially available were purchased from J&K, Meryer, Titan, TCI and Aladdin. All the chemicals used as supplied without further purification. The solvent including tetrahydrofuran (THF), dichloromethane ( $\text{CH}_2\text{Cl}_2$ ), chloroform ( $\text{CHCl}_3$ ), Triethylamine (TEA) were further dried by distillation.

**Characterization:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker AVANCE III 500MHZ spectrometer using Deuterated solvents (including  $\text{CDCl}_3$ ,  $\text{DMSO-d}_6$ ,  $\text{CDCl}_2$ ,  $\text{D}_2\text{O}$ ) as internal reference. High-resolution mass spectra (HRMS) were obtained on a Finnigan MAT TSQ 7000 Mass Spectrometer operating in a MALDI-TOF mode. Quantum yield was determined by a Quanta-integrating sphere. Absorption spectra were measured on a Milton Ray Spectronic 3000 array spectrophotometer. Steady-state photoluminescence (PL) spectra were recorded on a Perkin Elmer LS 55 spectrometer and Edinburgh FS5 fluorescence spectrophotometer. CLSM images were taken on a confocal laser scanning microscopy (ZEISS-LSM880, Germany), Field Emission Transmission Electron Microscope-F200 (JEM-F200). Fluorescence images were collected on Stimulated Emission Depletion Nanoscopy (STED Nanoscopy) by TCS SP8 STED 3X. Size analyses were implemented using a Zetasizer Nano ZSP (Malvern NanoZSP+MPT-2) and Mastersizer 2000.

## Synthesis and Characterization

**ROS Detection in Aqueous Solution:** A commonly used ROS indicator DCFH-DA was utilized to detect the ROS generation of AIEgens in aqueous solution under white light irradiation ( $22.1 \text{ mW/cm}^2$ ). DCFHDA was firstly prehydrolyzed into DCFH. Then, the ROS indicator ( $40 \times 10^{-6} \text{ mol/L}$ ) in PBS was further diluted to  $5 \times 10^{-6} \text{ mol/L}$  in the sample solution of AIE-den-3 ( $2 \times 10^{-6}$

mol/L) for measurement by PL instrument. The fluorescence of 2',7'-dichlorofluorescein triggered by AIEgen-sensitized ROS under white light irradiation was measured at different time intervals. The PL spectra were measured with excitation at 488 nm and emission was collected from 500 to 620 nm. The fluorescence intensity at 525 nm was recorded to indicate the generation rate of overall ROS.

**Cytotoxicity study:** In vitro cytotoxicity of the materials against HeLa cells were assessed by CCK-8. HeLa cells were seeded (5000 cells per well) in 96-well plate. Then after 24 h, the cells were exposed to series of 200 $\mu$ L of medium containing the materials with tested concentrations, respectively. Each series was incubated for 24 h at 37 °C in a humidified atmosphere. After co-incubation, 1% volume of CCK-8 dye solution was added to each well. After additional incubation for 2 h, the absorbance of the products was measured at a wavelength of 450 nm. The results were expressed as the viable percentage of cells after different treatments relative to the control cells without any treatment.

**Cell uptake and imaging:** HeLa cells were incubated in DMEM medium containing 10% FBS and 1% antibiotics (penicillin-streptomycin) at 37 °C under 5% CO<sub>2</sub> for 24 h. Then, the media was replaced by fresh complete media containing AIE-den-2 and AIE-den-3 with further incubation for 0.5 h, 1.0 h, 1.5 h and 2 h in 37 °C, respectively. The concentration of each materials was 20  $\mu$ g/ mL. After that, the cells were gently washed with PBS for 3 times, and then observed by CLSM. All experiments were performed with three parallel samples.

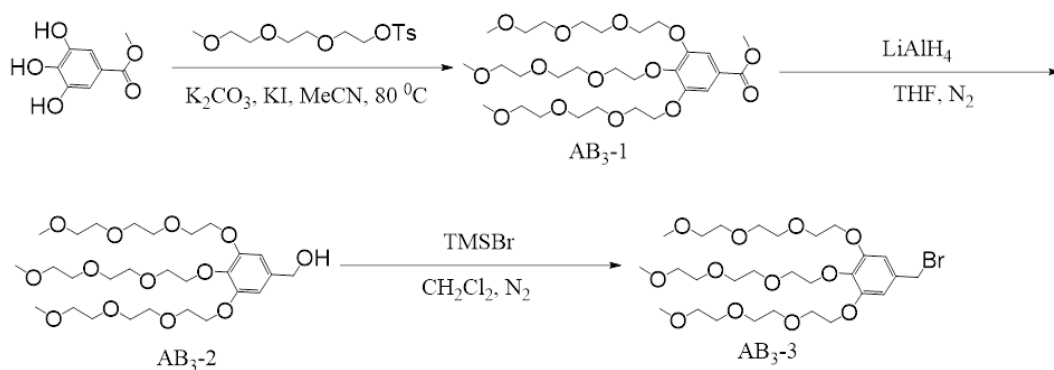
**Confocal Colocalization:** HeLa cells were seeded and cultured in glass bottom dish for 24 h, and then, the media were replaced by fresh complete media containing AIEgens with further incubation for 3 h in 37 °C. Further, the cells were stained with LysoTracker Green (for AIE-den-3)

or LysoTracker Red (for AIE-den-2) at 37 °C for 20 min. The medium was then removed and the cells were washed with PBS for three times and then imaged CLSM.

**Intracellular ROS Generation:** HeLa cells were seeded and cultured in glass bottom dish for 24 h. Then, the cells were further treated in fresh media containing AIE-den-3 micelles (40 µg/mL determined by TTFMN) for 24 h. Afterward, the cells were incubated with 1 mL fresh serum-free media containing  $10 \times 10^{-6}$  mol/L DCFH-DA at 37 °C for 5 min. After washing, the cells irradiated with white light irradiation (22.1mW/cm<sup>2</sup>) for 5min, followed by CLSM imaging. The cells treated with AIE-den-3 micelles without laser irradiation or PBS with/without laser irradiation served as the controls. The CLSM images were captured with excitation at 488 nm and emission was collected from 500 to 550 nm.

**Live/Dead Cell Staining:** First, HeLa cells were seeded and cultured in glass bottom dish for 24 h, then exposed to different following treatments: 1) PBS; 2) irradiated with white light irradiation (22.1mW/cm<sup>2</sup>) for 5min; 3) incubated with AIE-den-3 micelles for 24 h; 4) incubated with AIE-den-3 micelles for 24 h and irradiated with white light irradiation (22.1 mW /cm<sup>2</sup>) for 5min. The concentration of AIE-den-3 was 40 µg/mL. After different treatments, the cells were incubated at 37 °C for another 24 h, then successively stained with PI (60 µg/mL) and FDA (100 µg/mL) in PBS for 10 min. Subsequently, the cells were gently washed and then imaged by CLSM. Conditions: excitation wavelength: 488 nm for FDA and 543 nm for PI; emission filter: 500–550 nm for FDA and 550–650 nm for PI.

**Synthesis of hydrophilic dendritic groups**



**Scheme S1.** Synthesis routes to AB<sub>3</sub>-1, AB<sub>3</sub>-2, AB<sub>3</sub>-3.

### Synthesis of AB<sub>3</sub>-1

AB<sub>3</sub>-1 was synthesized according to previous work.<sup>1</sup> Methyl gallate (0.92 g, 0.005 mol), 2-(2-(2-methoxyethoxy)ethoxy)ethyl 4-methylbenzenesulfonate (6.36 g, 19 mmol), KI (500 mg), anhydrous potassium carbonate (5.6 g, 33.25 mmol), and 30 mL acetonitrile were added into 250 mL flask. The resulting mixture was stirred at 78 °C for 27 h under nitrogen atmosphere. After the reaction was cooled down to room temperature, solvent was removed under reduced pressure. The desired solid was diluted with distilled water (50 mL) and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3×40 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and evaporated then purified with chromatography. The <sup>1</sup>H-NMR (500 MHz, Chloroform-d, 295 K, TMS) (Figure S1) δ: 7.29 (s, 2H), 4.21(m, 6H), 3.88 (s, 3H), 3.87 (dd, 4H) and 3.80 (m, 2H), 3.73 (m, 6H), 3.65 (m, 12H), 3.54 (m, 6H), 3.37 (d, 9H).

### Synthesis of AB<sub>3</sub>-2

AB<sub>3</sub>-2 was synthesized according to previous work.<sup>2,3</sup> Adding of LiAlH<sub>4</sub> (560 mg, 15 mmol) into a two-neck flask, and purged with nitrogen for three times. Then, adding 20 mL solution of AB<sub>3</sub>-1 (5.20 g, 0.01 mol) with 20 mL THF at 0 °C over a period of 10 min. The mixture was stirred at room temperature for 3 h and quenched by slow addition of isopropyl alcohol (2 mL), water (3 mL), and 15% aq. NaOH (2 mL). The insoluble salts were filtered off through a pad of Celite by using a suction funnel, and the filtrate was concentrated under reduced pressure. The

residue was dissolved in  $\text{CH}_2\text{Cl}_2$  (100 mL), washed with water and brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and concentrated under reduced pressure.  $^1\text{H-NMR}$  (500 MHz, Chloroform- $d$ , 295 K, TMS) (Figure S2)  $\delta$ : 6.68 (s, 2H), 4.57 (s, 2H), 4.15 (m, 6H), 3.83 (dd, 4H) and 3.80 (m, 2H), 3.73 (m, 6H), 3.65 (m, 12H), 3.54 (m, 6H), 3.37 (d, 9H).

### Synthesis of AB<sub>3</sub>-3

AB<sub>3</sub>-2 (3.06 g, 4.21 mol) was added into a 100 mL of two-neck flask, and purged with nitrogen for three times. Then anhydrous  $\text{CHCl}_3$  (10 mL) was added at 0 °C and TMSBr (0.8 mL, 6.31 mmol) was added over a period of 10 min and kept under argon. After 48 h stirring at room temperature, the reaction mixture was evaporated to dryness. No further purifications were necessary and directly used for the next step.  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ , 295 K) (Figure S3): 6.66 (s, 2H), 4.44 (s, 2H), 4.19 (m, 6H), 3.87 and 3.81 (m, 6H), 3.76 (m, 6H), 3.68 (m, 12H), 3.58 (m, 6H), 3.41 (d, 9H).

### Synthesis of TPE-1

TPE-1 was synthesized according to previous work.<sup>4</sup> Adding of 4,4'-Dihydroxybenzophenone (4.36 g, 19.76 mmol), 4,4'-dibromobenzophenone (5.60 g, 16.48 mmol) and zinc dust (9.06 g, 140.0 mmol) into to a three-neck flask, and purged with nitrogen for three times. Then anhydrous THF (100 mL) was added under nitrogen and the whole system was cooled to -20 °C.  $\text{TiCl}_4$  (8.0 mL, 54.8 mmol) was added slowly. The reaction mixture was stirred at room temperature for 20 min and then heated at 75 °C for another 24 h. The system was cooled down and the mixture was quenched by adding 1 M HCl to react with the residual zinc. The mixture was extracted by ethyl acetate (100 mL  $\times$  3), dried over anhydrous  $\text{Na}_2\text{SO}_4$  and then the solvent was removed to give a crude product which was purified by flash column chromatography.  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ,

295 K) (Figure S4): 7.25-7.20 (d, 4H), 6.88-6.83 (d, 8H), 6.61-6.58 (d, 4H). ESI-MS (Figure S5): calculated for  $C_{26}H_{18}Br_2O_2$ ,  $m/z$  519.97, found,  $m/z$  519.96 ( $[M]^+$ ).

### Synthesis of TPE-2

4-formylphenylboronic acid (1.49 g, 0.01 mol), TPE-1 (1.56 g, 0.003 mol),  $Pd(PPh_3)_4$  (200 mg), anhydrous potassium carbonate (2.6 g), were added into THF/H<sub>2</sub>O (45mL/15mL) solution. Then the system was stirred at 80 °C for 27 h under nitrogen atmosphere. After the reaction was cooled down to room temperature, solvent was removed under reduced pressure. The desired solid was diluted with distilled water (50 mL) and extracted with  $CH_2Cl_2$  (3×40 mL). The organic layer was dried over  $Na_2SO_4$  and evaporated then purified with chromatography.  $^1H$ -NMR (500 MHz, THF-8d, 295 K) (Figure S6): 10.04 (s, 2H), 7.92 (m, 4H), 7.73 (m, 4H), 7.44 (m, 4H), 7.15 (m, 4H), 6.95 (m, 4H), 6.61 (m, 4H).  $^{13}C$ -NMR (500 MHz, THF-8d, 295 K, TMS) (Figure S7)  $\delta$ : 190.65, 156.78, 146.04, 145.07, 142.47, 136.86, 136.39, 135.62, 134.66, 132.59, 132.10, 129.70, 126.91, 126.31, 114.41. ESI-MS (Figure S8): calculated for  $C_{40}H_{28}O_4$ ,  $m/z$  572.20, found,  $m/z$  572.19 ( $[M]^+$ ).

### Synthesis of TPE-3

TPE-1 (520 mg, 1 mmol), 5-formyl-2-thiopheneboronic acid (370 mg, 2.4 mmol),  $Pd(PPh_3)_4$  (150 mg), anhydrous potassium carbonate (1.6 g), were added into THF/H<sub>2</sub>O (45mL/15mL) solution. Then the system was stirred at 80 °C for 27 h under nitrogen atmosphere. After the reaction was cooled down to room temperature, solvent was removed under reduced pressure. The desired solid was diluted with distilled water (50 mL) and extracted with  $CH_2Cl_2$  (3×40 mL). The organic layer was dried over  $Na_2SO_4$  and evaporated then purified with chromatography.  $^1H$ -NMR (500 MHz, THF-8d, 295 K) (Figure S9): 9.87 (s, 2H), 8.33 (s, 2H), 7.82 (d, 2H), 7.55 (m, 6H), 7.14 (m, 4H),

6.90 (m, 4H), 6.55 (m, 4H).  $^{13}\text{C}$ -NMR (500 MHz, THF-8d, 295 K, TMS) (Figure S10)  $\delta$ : 181.78, 157.02, 152.84, 145.80, 143.23, 142.60, 137.16, 136.04, 134.44, 132.64, 132.11, 130.85, 125.46, 123.95, 114.44. ESI-MS (Figure S11): calculated for  $\text{C}_{36}\text{H}_{24}\text{O}_4\text{S}_2$ ,  $m/z$  584.11, found,  $m/z$  584.107 ( $[\text{M}]^+$ ).

### Synthesis of AIE-den-1

AB<sub>3</sub>-1 (6.22 g, 0.01mol) was dispersed in water solution (10 mL), followed by addition of KOH (4 g). The resulting mixture was stirred at 100 °C for 2 h. After the reaction was cooled down to room temperature, 2 mol/L HCl was dropped to switch the pH to 2.0. Then, white flocculent precipitate would generate. Collecting precipitate by filtration and recrystallization and the pure product could be achieved by completely removing water. The obtained product was used for the next step directly. In a 50mL of round bottom flask, AB<sub>3</sub>-1 acid (3.04 g, 0.005 mol) was dissolved in 10 ml of anhydrous chloroform. Then an excess of SOCl<sub>2</sub> was dropped slowly in 30 min. The solution was stirred under nitrogen at room temperature for 20 h. Finally, the solvent and excess of SOCl<sub>2</sub> were removed under reduced pressure to yield the AB<sub>3</sub>-1 dichloride. The AB<sub>3</sub>-1 dichloride was dissolved in 10 mL anhydrous chloroform. Then, the anhydrous TPE-2 (1.029 g, 0.008 mol) was added, after then, 1 mL Triethylamine was dropped. The solution was stirred under room temperature for 18 h. Then, adding of 50 mL H<sub>2</sub>O to extract the product. After that, the combined extracts were dried over anhydrous MgSO<sub>4</sub>, filtered, and the solvent was removed under reduced pressure to yield a translucent oily solid. Purification was carried out via column chromatography.  $^1\text{H}$ -NMR (500 MHz, CDCl<sub>3</sub>, 295 K) (Figure S12): 10.4 (s, 2H), 7.93 (m, 4H), 7.75 (m, 4H), 7.49 (m, 4H), 7.39-7.41 (m, 4H), 7.21 (dd, 4H), 7.17 (ddd, 4H), 7.02 (m, 4H), 4.29-4.15 (m, 12H), 3.86 (dt, 8H) and 3.81 (m, 4H), 3.72 (m, 12H), 3.64 (m, 24H), 3.53 (m, 12H), 3.36 (m, 18H). ESI-MS



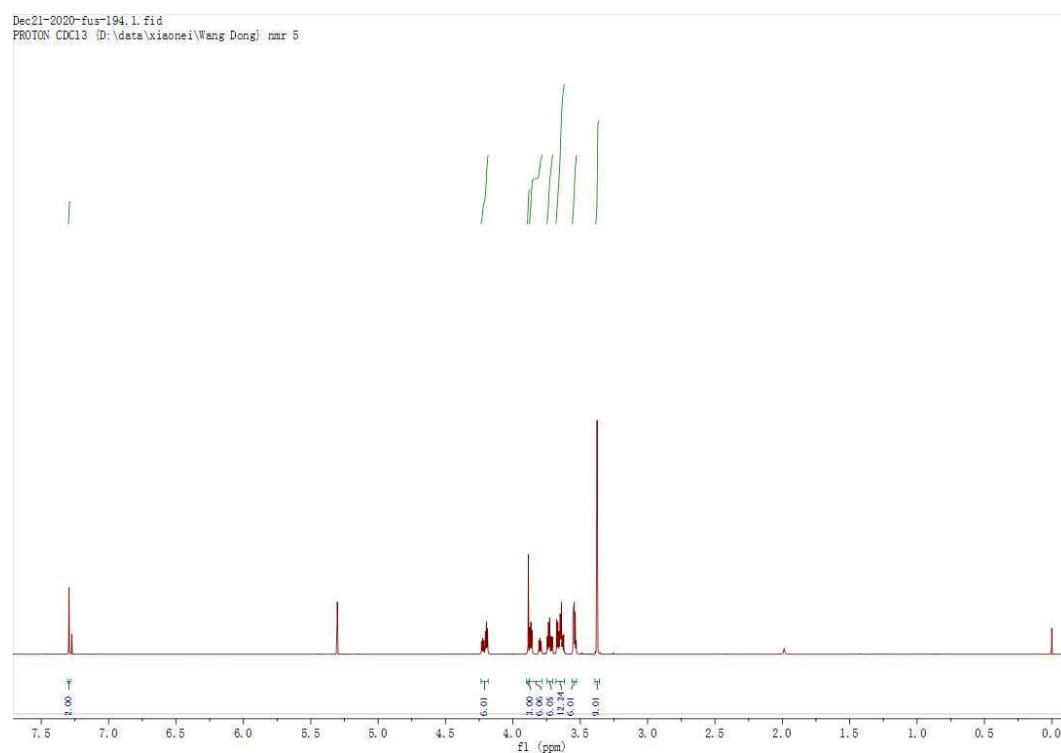
(**Figure S13**): calculated for  $C_{96}H_{120}O_{30}$ ,  $m/z$  1752.79, found,  $m/z$  1775.77 ( $[M+Na]^+$ ), 1791.77 ( $[M+K]^+$ ).

### Synthesis of AIE-den-2

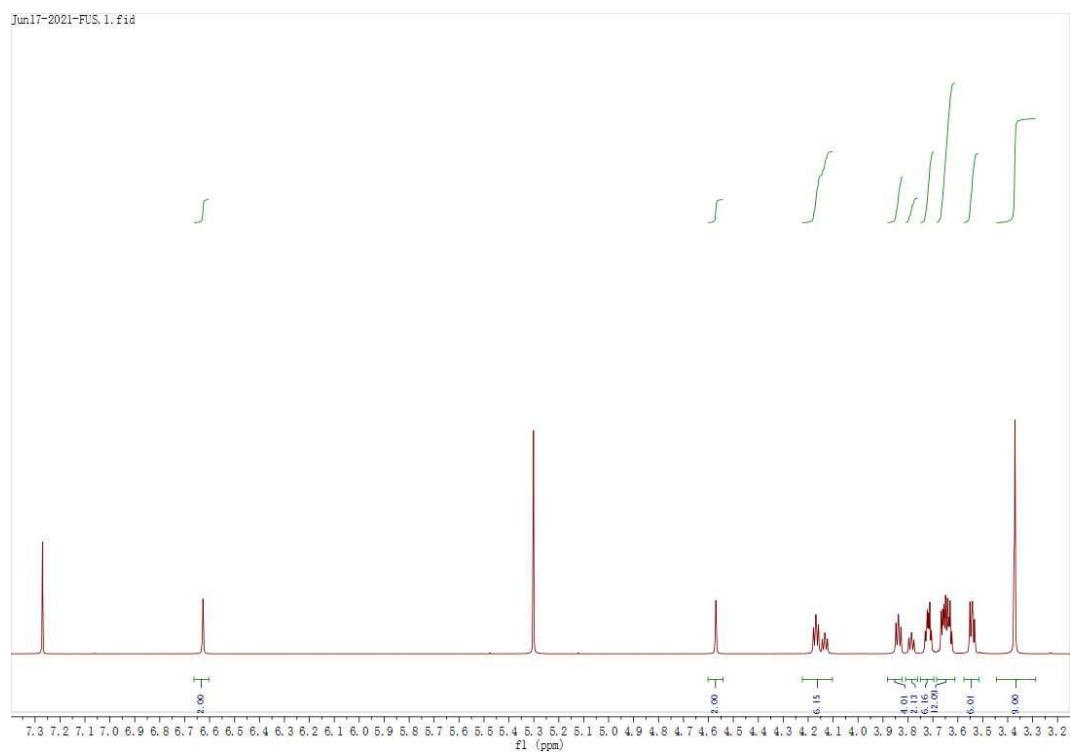
Tpe-2 (120 mg, 0.205 mmol), AB<sub>3</sub>-3 (539 mg, 0.821 mmol), KI (40 mg), anhydrous potassium carbonate (2.6 g), and 40 mL acetonitrile were added into 250 mL flask, the resulting mixture was stirred at 78 °C for 27 h under nitrogen atmosphere. After the reaction was cooled down to room temperature, solvent was removed under reduced pressure. The desired solid was diluted with distilled water (50 mL) and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3×40 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and evaporated then purified with chromatography. The <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>, 295 K, TMS) (**Figure S14**) δ: 9.80 (s, 2H), 7.64 (dd, 2H), 7.38 (m, 4H), 7.30 (m, 2H), 7.02 (m, 4H), 6.91 (m, 4H), 6.67 (m, 4H), 6.57 (m, 4H), 4.79 (t, 4H), 4.08 (tt, 12H), 4.78 to 4.71 (m, 12H), 3.64 (m, 12H), 3.58 (m, 24 H), 3.47 (m, 12H), 3.29 (m, 18H). ESI-MS (**Figure S15**): calculated for  $C_{92}H_{120}O_{28}S_2$ ,  $m/z$  1736.74, found,  $m/z$  1759.74 ( $[M+Na]^+$ ).

### Synthesis of AIE-den-3

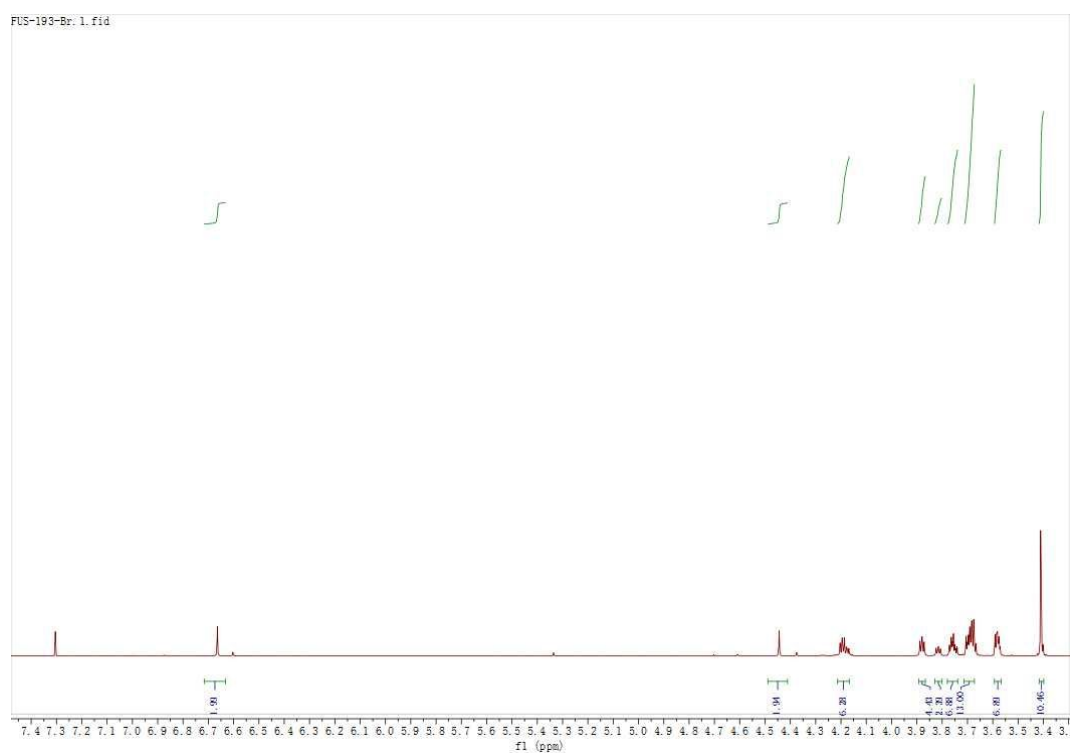
AIE-den-3 was synthesized according to previous work.<sup>5</sup> To a solution of AIE-den-2 (140 mg, 0.0799 mmol) in EtOH 20 mL, malononitrile (66 mg, 1.0 mmol) was added. The system was refluxed for 20 h with nitrogen atmosphere, followed by solvent removal. The residue was purified by chromatography. <sup>1</sup>H-NMR (500 MHz, CD<sub>2</sub>Cl<sub>2</sub>, 295 K, TMS) (**Figure S16**): 8.65 (s, 2H), 7.94 (dd, 2H), 7.78 (m, 2H), 7.63 (m, 4H), 7.08 (m, 4H), 6.93 (m, 4H), 6.83 (m, 4H), 6.74 (m, 4H), 4.89 (t, 4H), 4.08 to 4.00 (m, 12H), 3.72 to 3.67 (m, 12H), 3.58 (m, 12H), 3.51 (m, 24 H), 3.41 (m, 12H), 3.22 (m, 18H). ESI-MS (**Figure S17**): calculated for  $C_{98}H_{120}N_4O_{26}S_2$ ,  $m/z$  1832.76, found,  $m/z$  1855.76 ( $[M+Na]^+$ ).



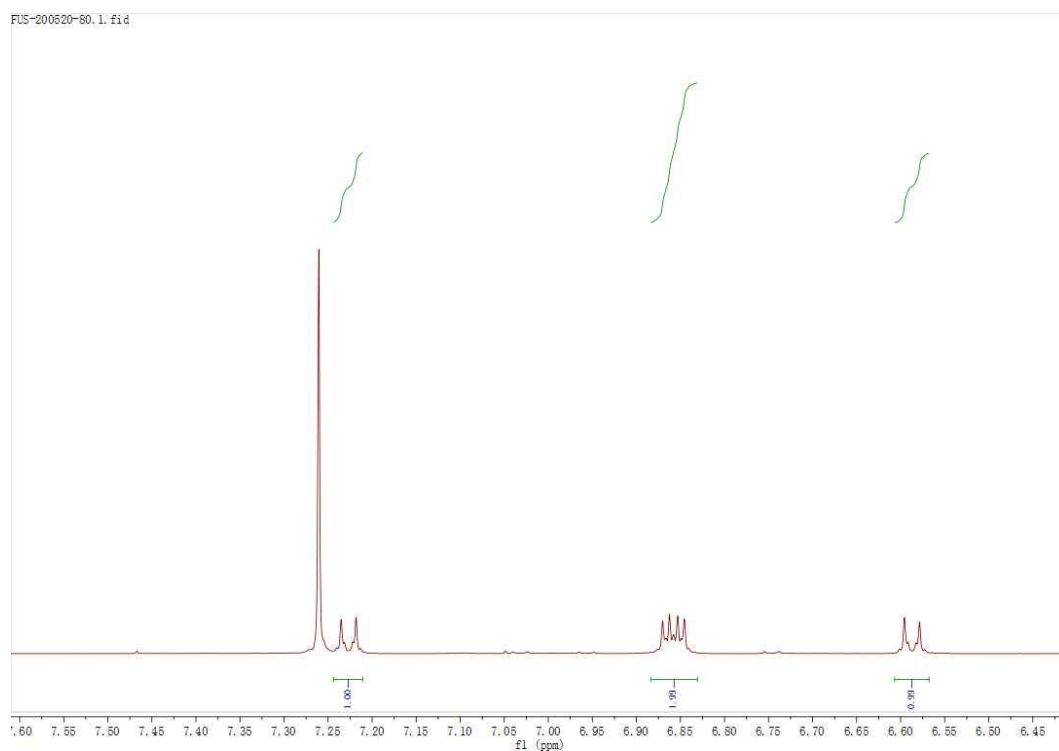
**Figure S1.** <sup>1</sup>H-NMR spectrum of AB<sub>3</sub>-1.



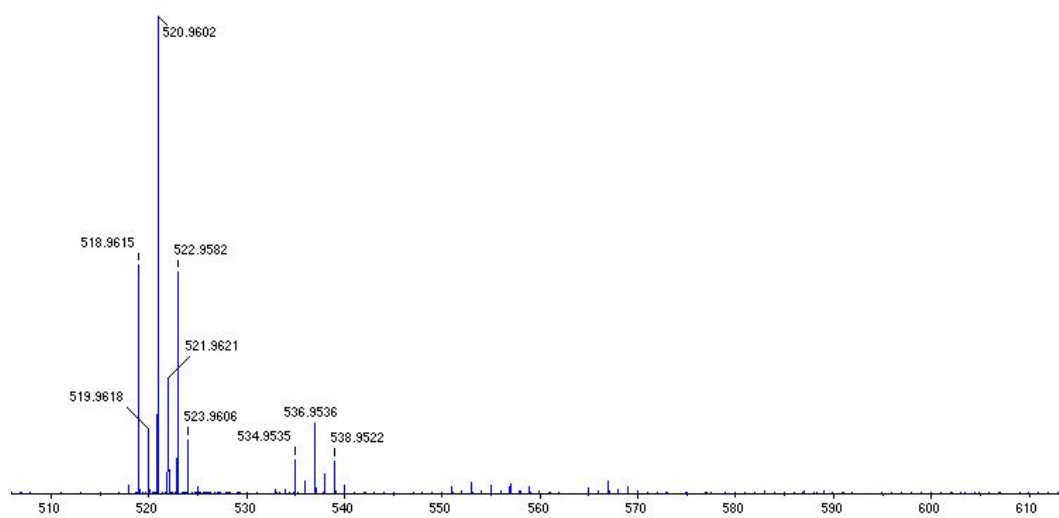
**Figure S2.**  $^1\text{H}$ -NMR spectrum of  $\text{AB}_3\text{-2}$ .



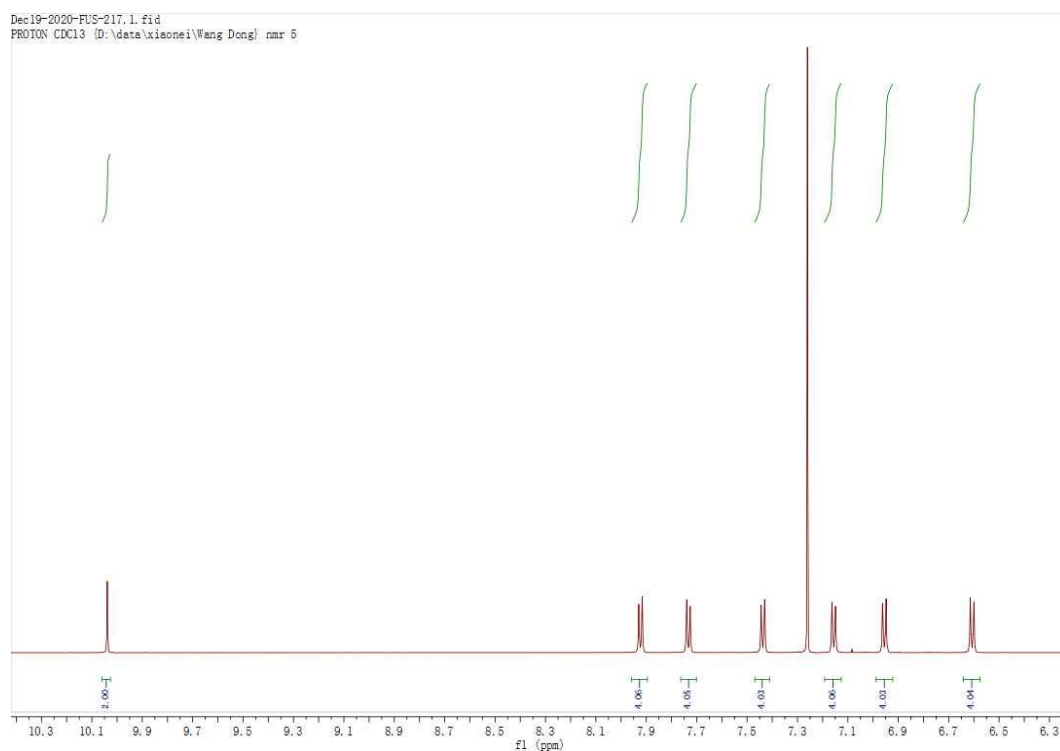
**Figure S3.**  $^1\text{H}$ -NMR spectrum of  $\text{AB}_3\text{-3}$ .



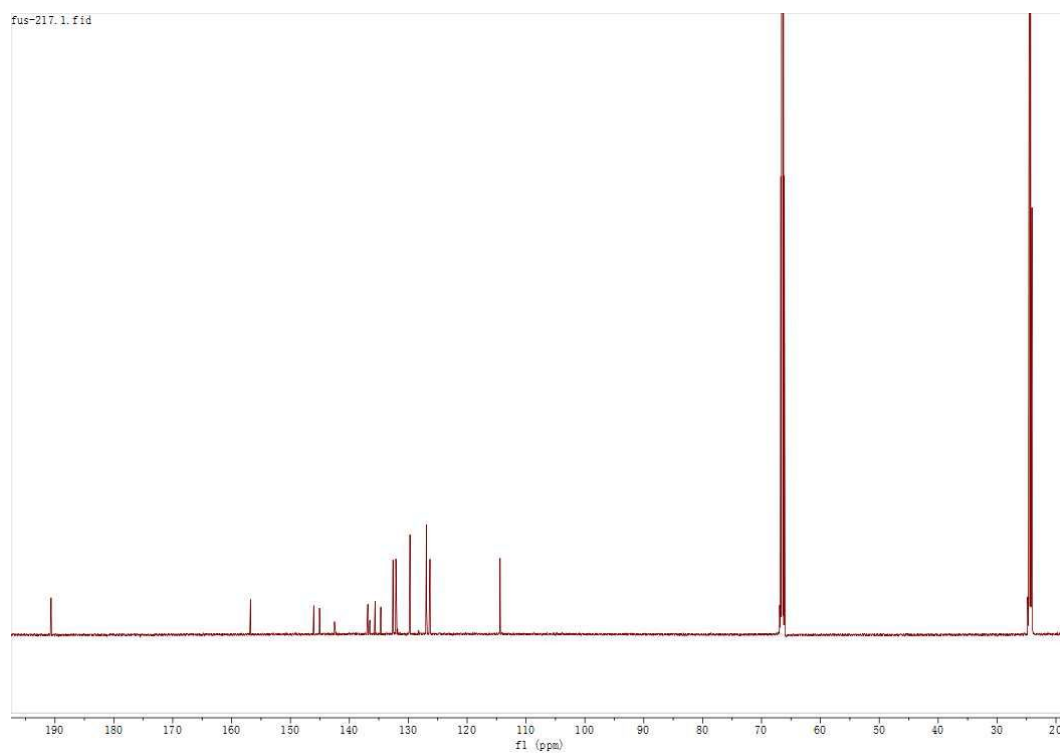
**Figure S4.**  $^1\text{H}$ -NMR spectrum of TPE-1.



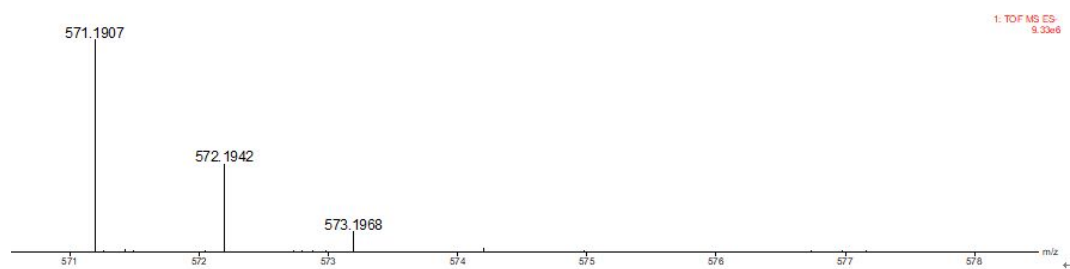
**Figure S5.** ESI-MS spectrum of TPE-1.



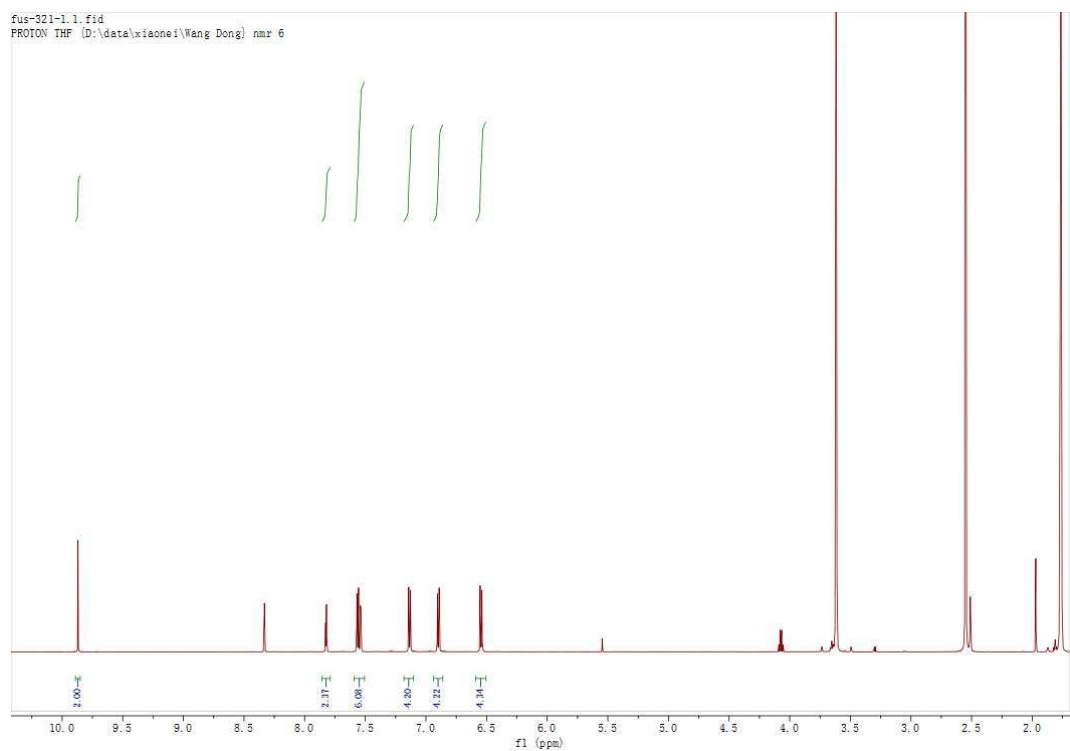
**Figure S6.**  $^1\text{H}$ -NMR spectrum of TPE-2.



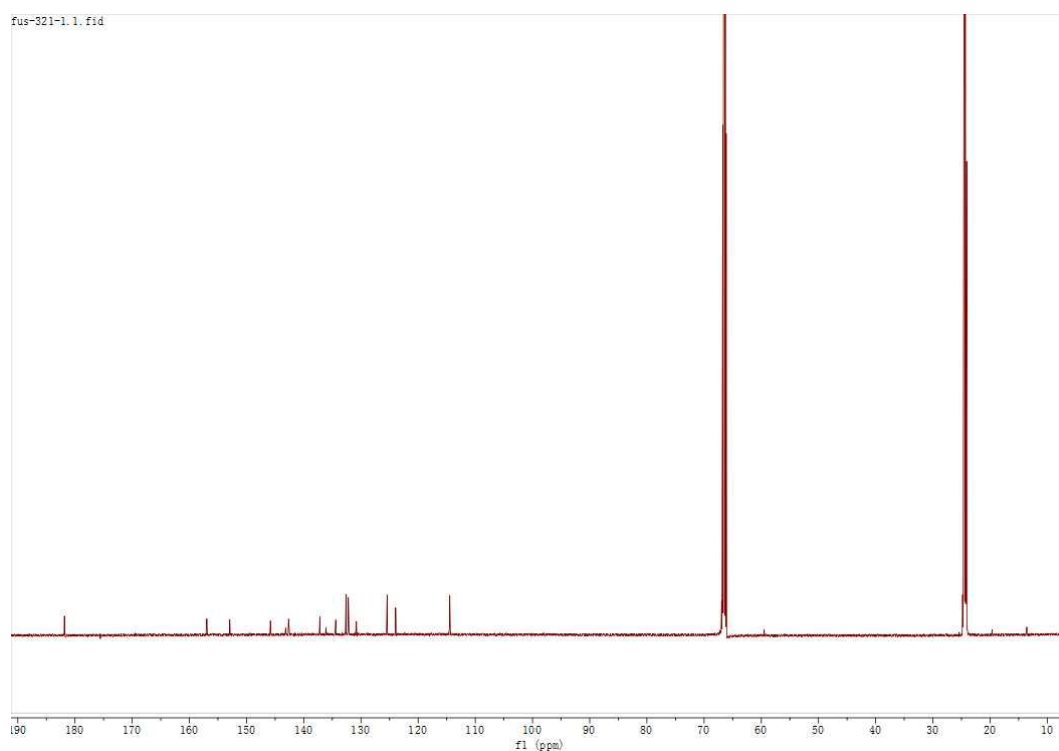
**Figure S7.**  $^{13}\text{C}$ -NMR spectrum of TPE-2.



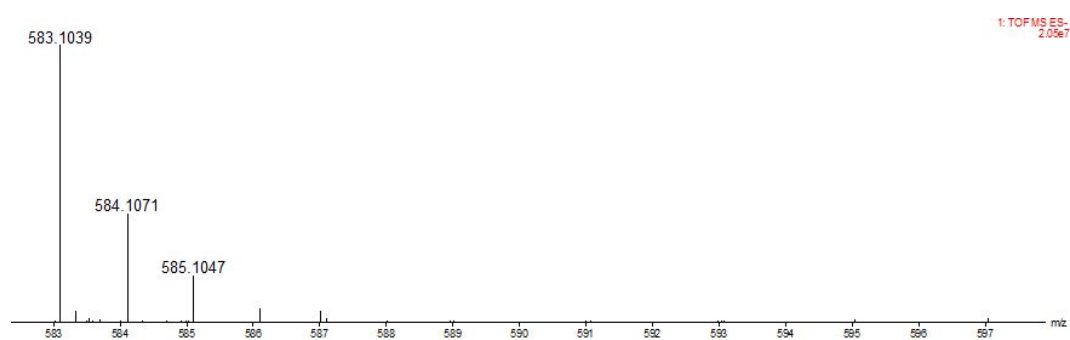
**Figure S8.** ESI-MS spectrum of TPE-2.



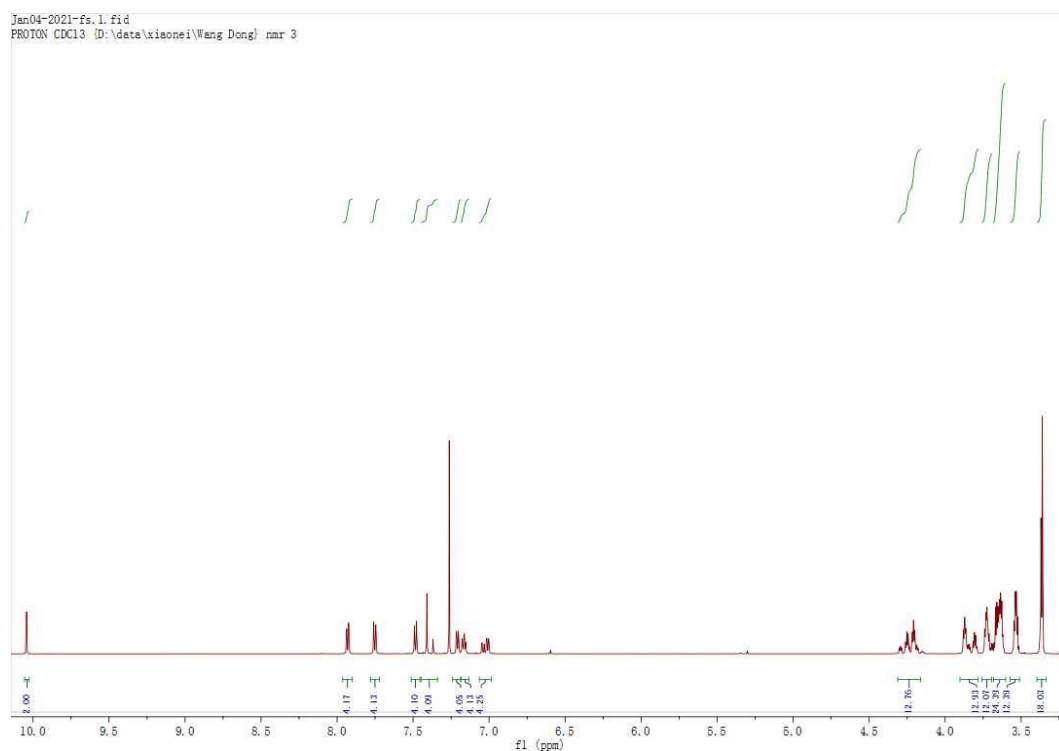
**Figure S9.**  $^1\text{H}$ -NMR spectrum of TPE-3.



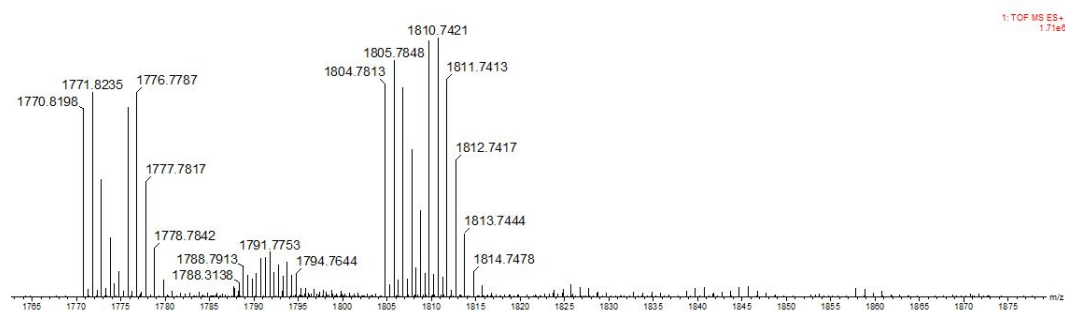
**Figure S10.**  $^{13}\text{C}$ -NMR spectrum of TPE-3.



**Figure S11.** ESI-MS spectrum of TPE-3.

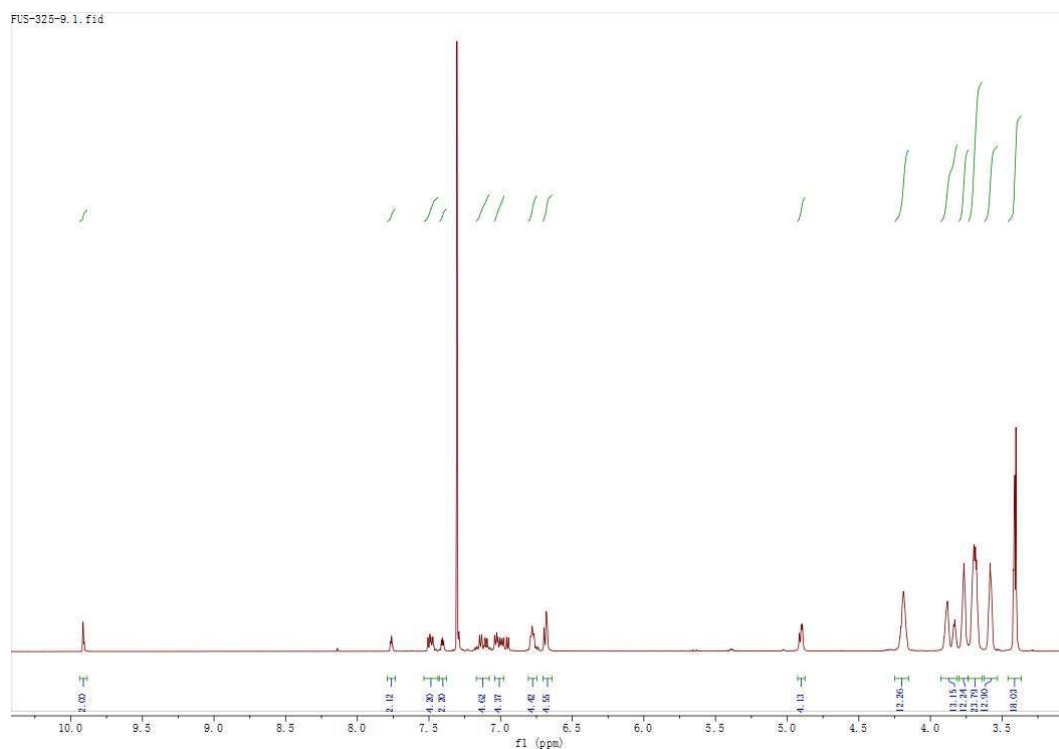


**Figure S12.**  $^1\text{H}$ -NMR spectrum of AIE-den-1.

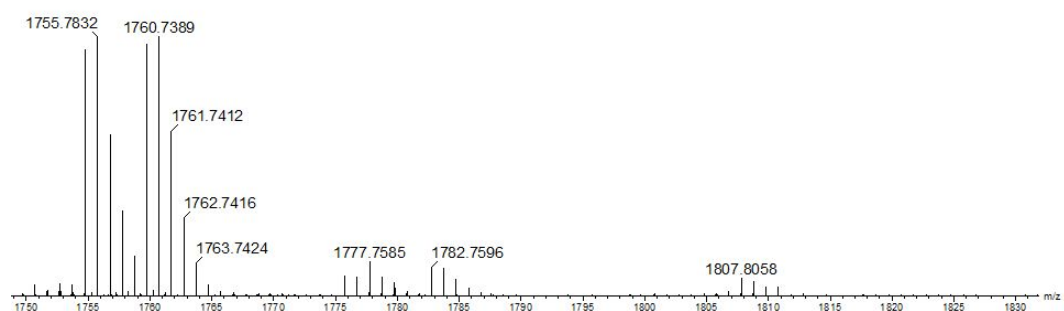


**Figure S13.** ESI-MS spectrum of AIE-den-1.

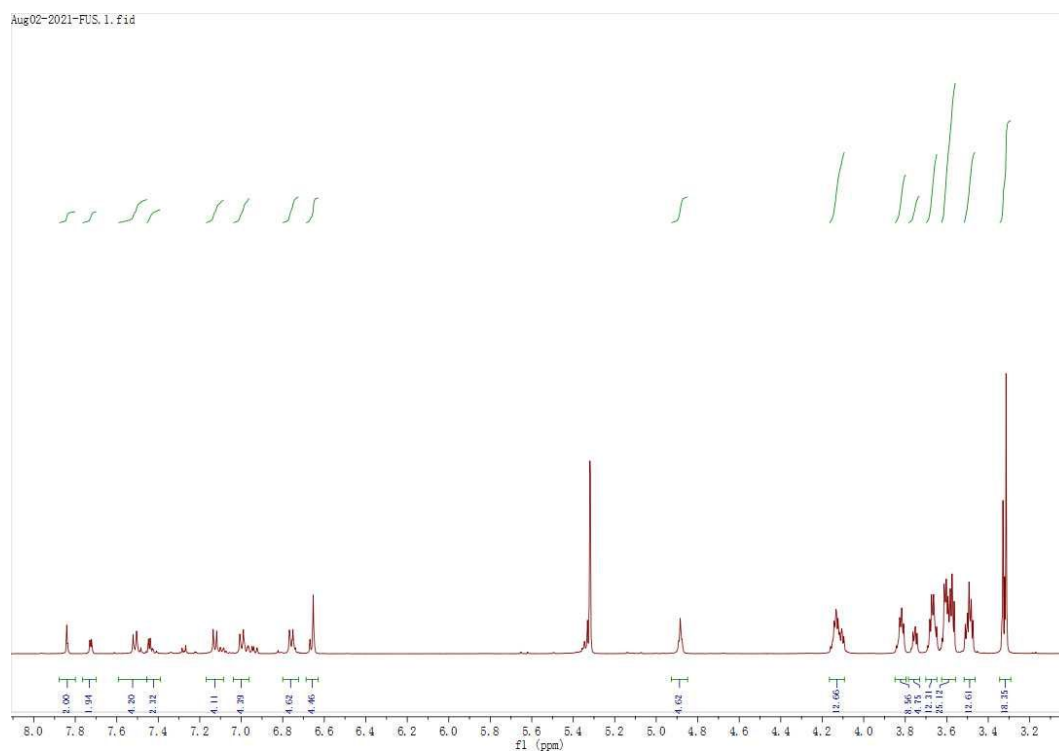




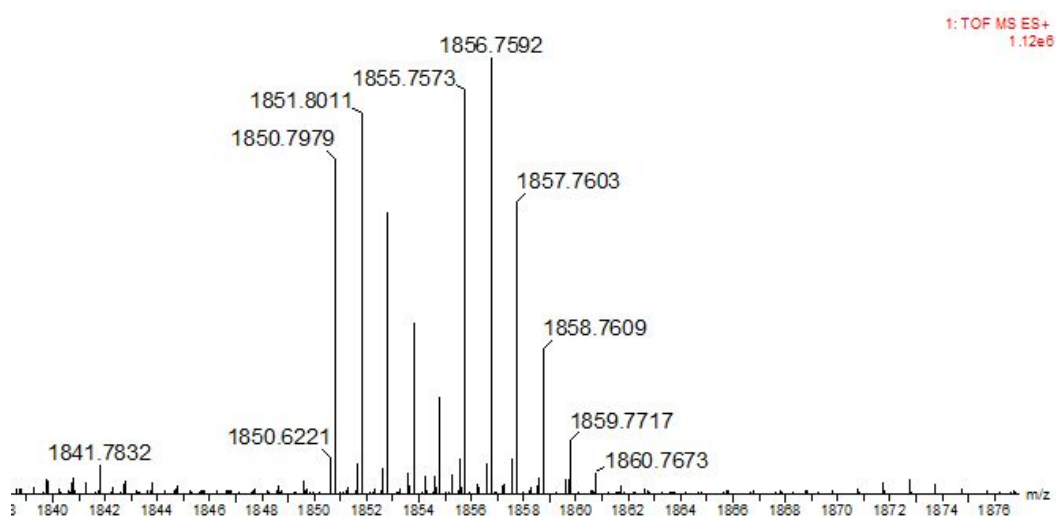
**Figure S14.**  $^1\text{H}$ -NMR spectrum of AIE-den-2.



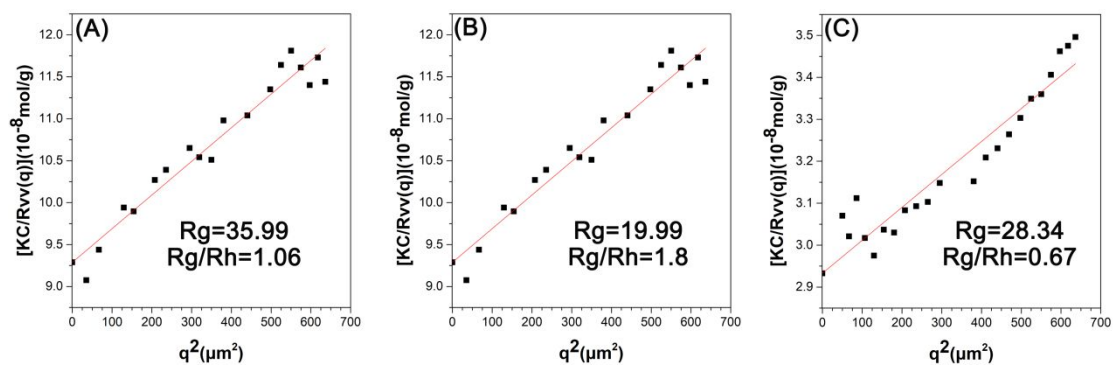
**Figure S15.** ESI-MS spectrum of AIE-den-2.



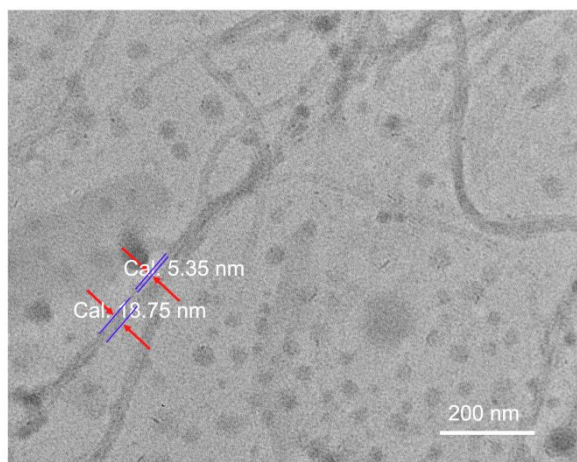
**Figure S16.**  $^1\text{H}$ -NMR spectrum of AIE-den-3.



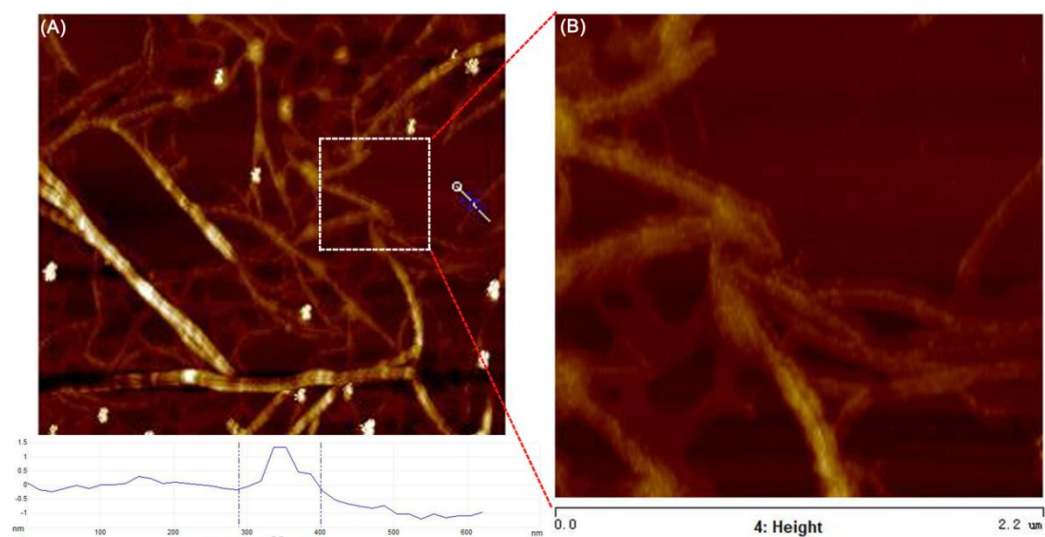
**Figure S17.** ESI-MS spectrum of AIE-den-3.



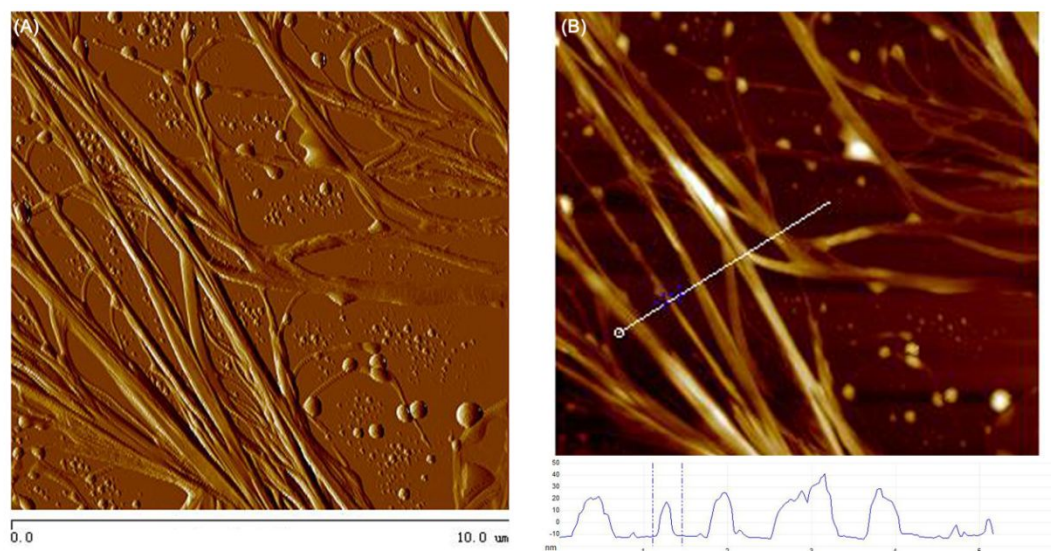
**Figure S18.** SLS analysis of self-assembly of (A) AIE-den-1, (B) AIE-den-2 and (C) AIE-den-3 in H<sub>2</sub>O solutions.



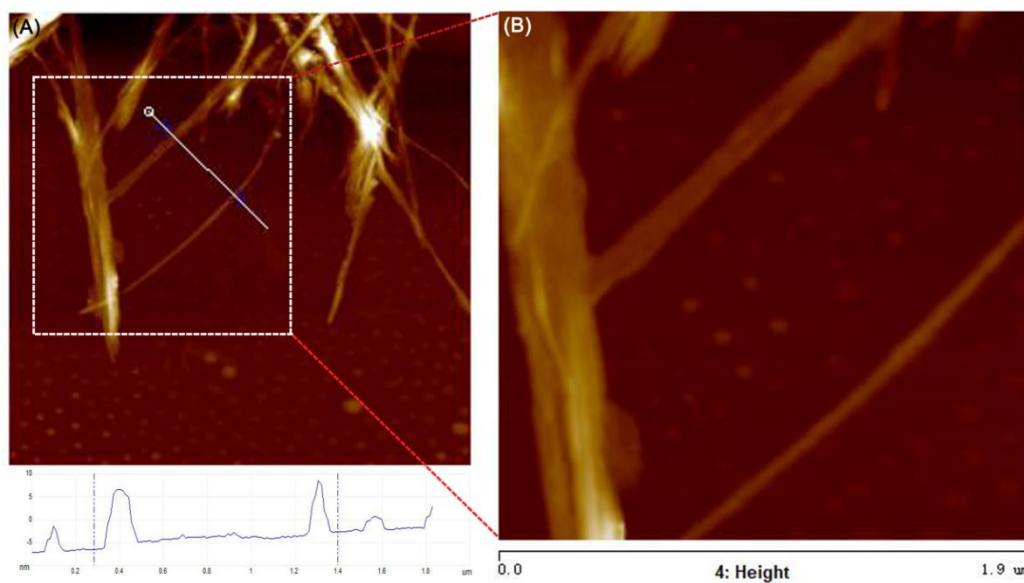
**Figure S19.** TEM images of nanotubes (self-assembly of AIE-den-1 in 30% THF of THF/H<sub>2</sub>O solutions).



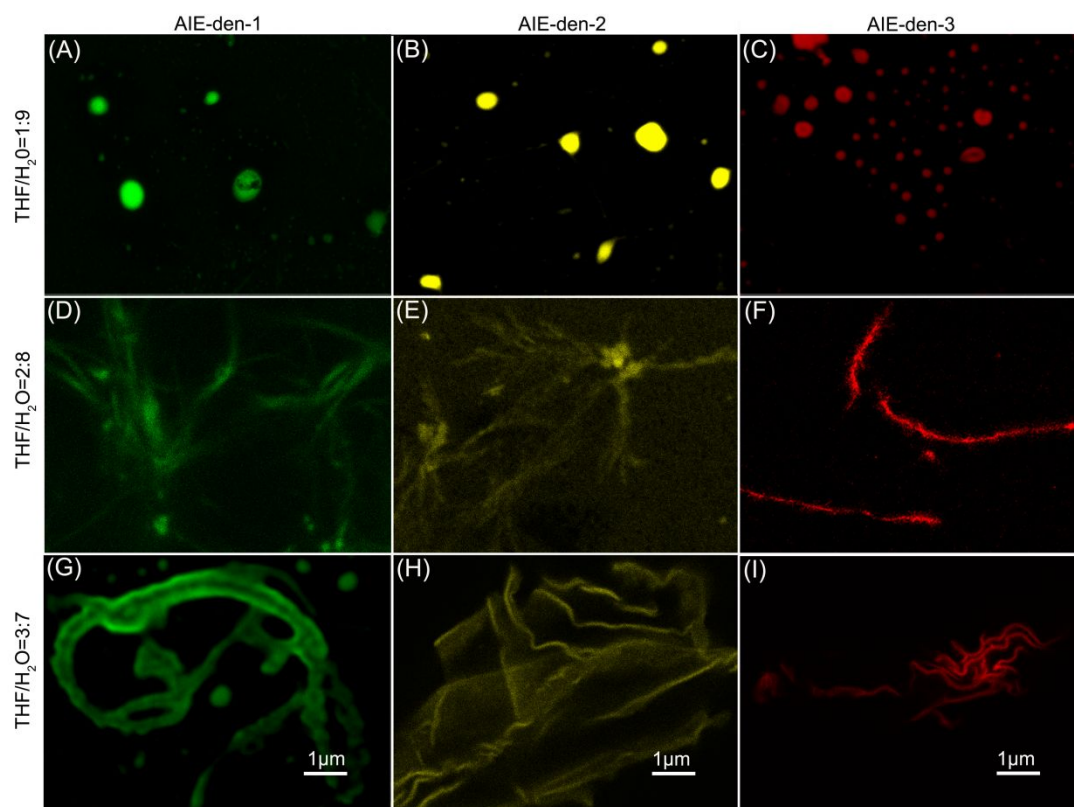
**Figure S20.** (A, B) AFM images of ribbon-like structure. The sample was prepared through self-assembly of AIE-den-1 in THF/H<sub>2</sub>O system (with 40% THF fraction).



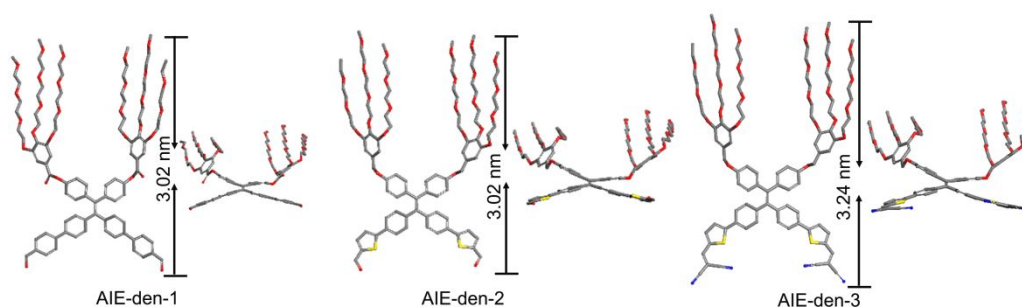
**Figure S21.** (A, B) AFM images of fibers. The sample was prepared through self-assembly of AIE-den-2 in THF/H<sub>2</sub>O system (with 40% THF fraction).



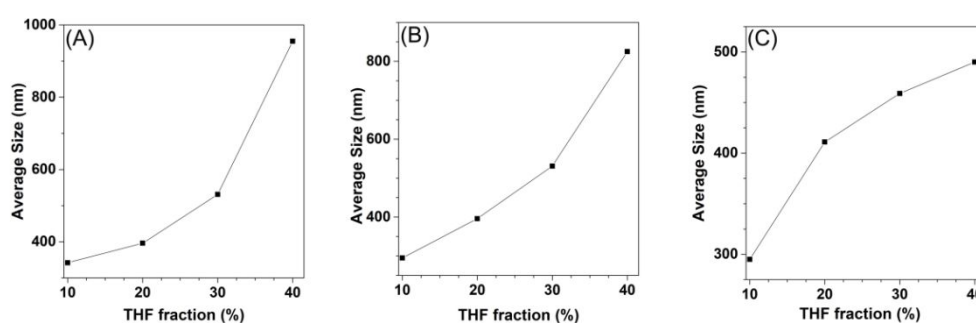
**Figure S22.** (A, B) AFM images of fibers. The sample was prepared through self-assembly of AIE-den-3 in THF/H<sub>2</sub>O system (with 40% THF fraction).



**Figure S23.** Fluorescence images of supramolecular topologies captured by STED nanoscopy. (A) and (B) and (C): Fluorescence images of micelles from self-assembly of AIE-den-1, AIE-den-2 and AIE-den-3 in THF/H<sub>2</sub>O=1:9 system. (D) and (E) and (F): Hybride of micelles and fibers from self-assembly of AIE-den-1, AIE-den-2 and AIE-den-3 in THF/H<sub>2</sub>O=2:8 system. (G): tubules from self-assembly of AIE-den-1 in THF/H<sub>2</sub>O=3:7 system. (H) and (I): fibers from self-assembly of AIE-den-2 and AIE-den-3 in THF/H<sub>2</sub>O=3:7 system.

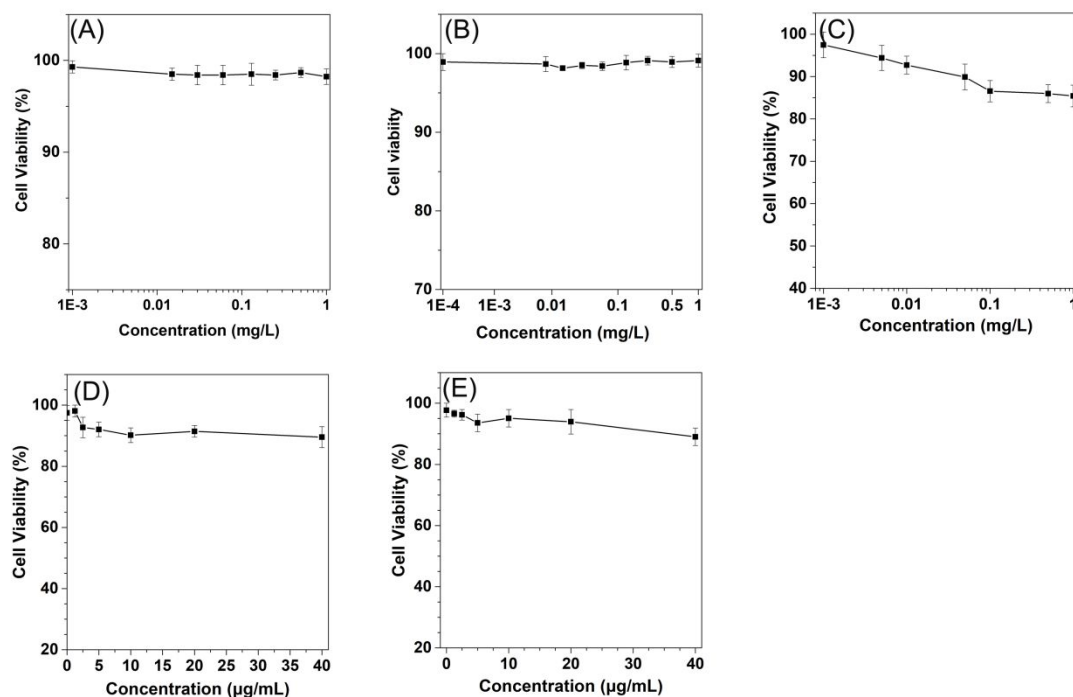


**Figure S24.** Molecular modals of AIE-den-1, AIE-den-2 and AIE-den-3.

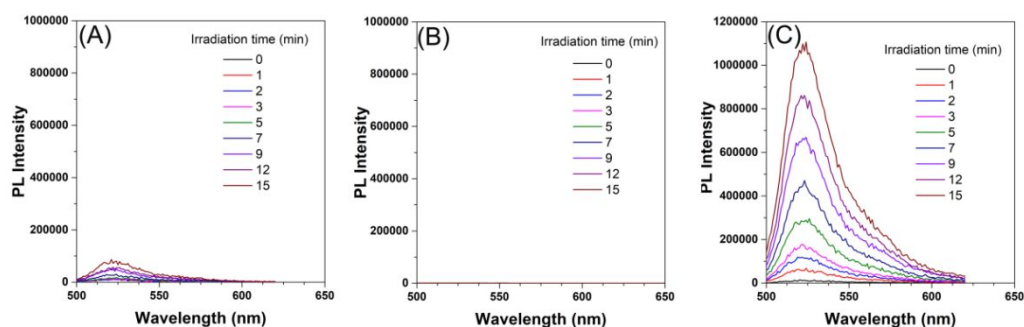


**Figure S25.** (A) and (B) and (C) Size distribution changes of self-assembly of AIE-den-1, AIE-den-2 and AIE-den-3 in different solvent system, characterized by DLS analysis.





**Figure S26.** (A) and (B) and (C) Cell toxicities of AIE-den-1, AIE-den-2 and AIE-den-3 for normal cells (3T3 cells) tested by CCK-8 assay. (D) and (E) Cell toxicities of AIE-den-1, AIE-den-2 for HeLa cells tested by CCK-8 assay.



**Figure S27.** PL spectra of DCFH in PBS in present of (A) DCFH alone, (B) AIE-den-3 alone, (C) TFMN+DCFH after exposure to white light irradiation with different time. The concentration of AIE-den-3 was  $10 \times 10^{-6}$  mol/L; Light power:  $22.1 \text{ mW/cm}^2$ .

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