## Dimensional Control of Supramolecular Assemblies of Diacetylene-Derived Peptide Gemini Amphiphile: From Spherical Micelles to Foamlike Networks

Hao Jiang, a Martin Ehlers, a Xiao-yu Hu, a,b Elio Zellermanna and Carsten Schmuck\*a

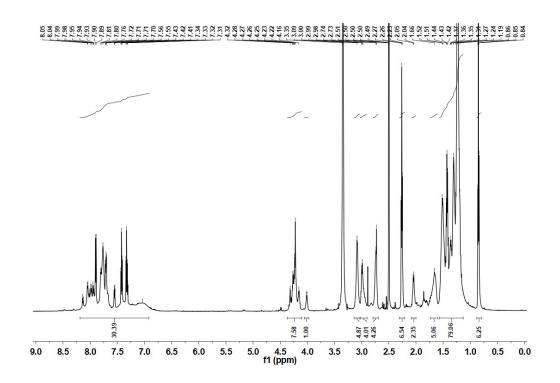


Figure S1. <sup>1</sup>H NMR of DA<sub>2</sub>P-Fmoc in DMSO-d<sub>6</sub> recorded at 600 MHz.

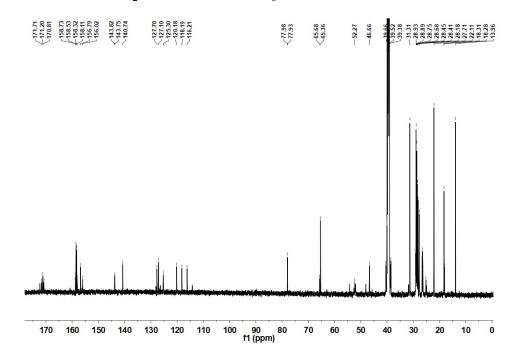
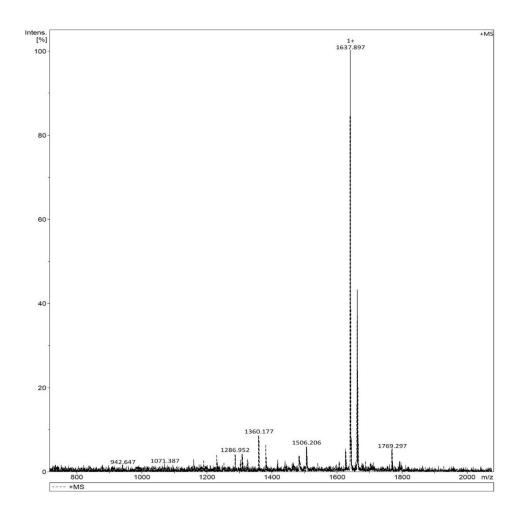


Figure S2. <sup>13</sup>C NMR of DA<sub>2</sub>P-Fmoc in DMSO-d<sub>6</sub> recorded at 151 MHz.

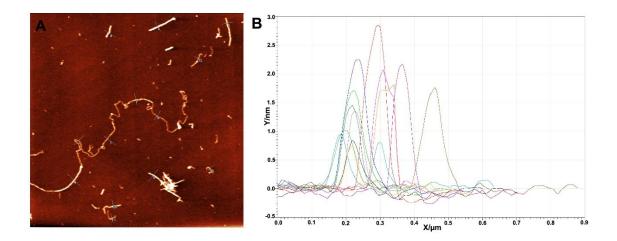


**Figure S3.** MALDI-TOF-MS spectrum of  $\mathbf{DA_2P\text{-}Fmoc}$  m/z calculated for  $C_{93}H_{150}N_{16}O_{9}$  1636.29, found  $[M + H]^+$  1637.90.

## Sample preparation for AFM and TEM measurements

AFM images were obtained in tapping mode using a NanoDrive Controller with an Innova Scanning Probe Microscope (Veeco Germany, Mannheim) and N-type silicon cantilever (AC 160TS OLYMPUS). A drop of prepared sample solution (6 μL, pure water or methanol) was placed on freshly cleaved mica and then spin-coated at a speed of 3000 rad/min for 2 min. The AFM data were analysed using Gwiddion-2.20 software. TEM images were obtained using a CM 200 FEG (Phillips Company). A drop of prepared sample solution was placed on 400-mesh formvar copper grid coated with carbon. After 2 min, the grid was tapped with a

filter paper to remove remaining solvent from the surface. Negative staining was performed by addition of a drop of an aqueous solution of uranyl acetate (2%) onto the copper grid. After 1 min, the liquid on the surface of the grid was removed by tapping it with a filter paper.



**Figure S4.** (A) Height image and (B) section analysis of AFM measurement of **DA<sub>2</sub>P-Fmoc** assemblies at the concentration of 0.2 mM with an aging time up to 1 month.

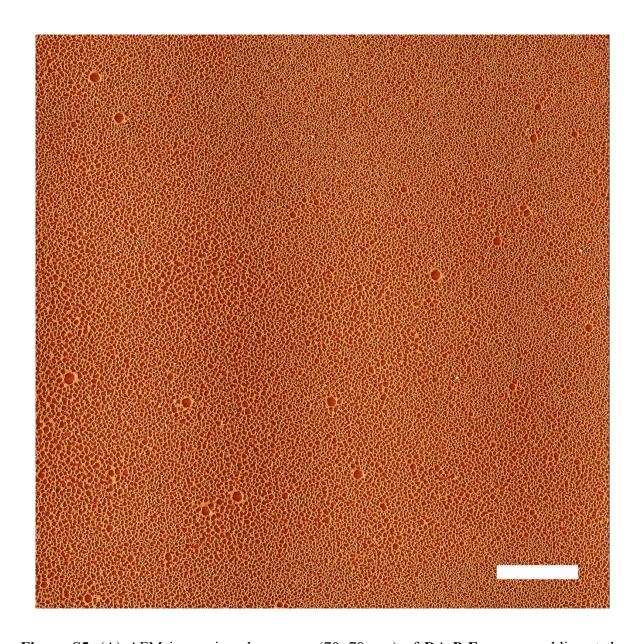
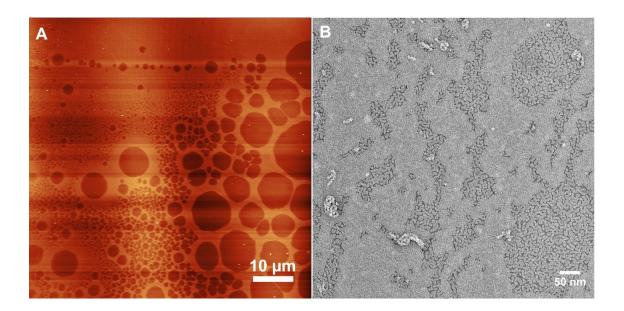


Figure S5. (A) AFM image in a large area (70×70  $\mu$ m) of **DA<sub>2</sub>P-Fmoc** assemblies at the concencration of 1 mM in methanol with an aging time of 10 days. Scale bar is 10  $\mu$ m.



**Figure S6.** (A) AFM image of **DA<sub>2</sub>P-Fmoc** assemblies in the outer region of spin-coated film and (B) TEM image of drop-casted film at the concentration of 1 mM in methanol with an aging time of 10 days.

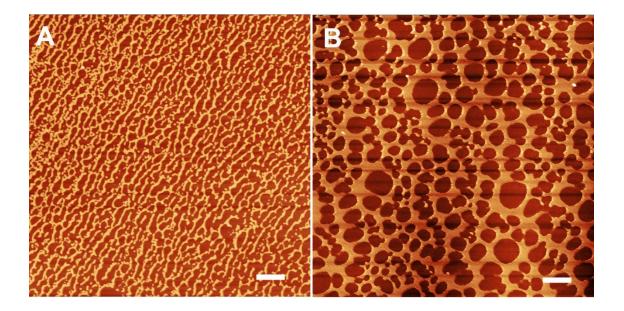


Figure S7. AFM images of  $DA_2P$ -Fmoc assemblies at the concentration of (A) 0.05 mM and (B) 0.2 mM in methanol with the aging effect. Scale bars are 1  $\mu$ m.