Supporting Information

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Experimental Section

Materials:

The diphenylalanine peptide (FF) was purchased from Bachem (Budendorf, Switzerland). 1,1,3,3,6,6-hexafluoro-2-propanol (HFIP), toluene and hexane were obtained from Sigma-Aldrich. All the reagents above were used without further purification.

Preparation:

Unilocular hollow spheres: In a typical synthesis, 10 μL gluaraldehyde aqueous solution was first added into 100 μL toluene at 30 ^{o}C with ultrasound treatment in 10 min. Then 30 μL HFIP solution of FF with the concentration of 100 mg/mL was added into the above emulsion with ultrasound treatment in another 15 min. The emulsion was kept still and aging overnight. After washing with water twice, the final product could be obtained.

Multicamerate hollow spheres: The preparation of multicamerate hollow spheres was the same with the above procedure except changing toluene into hexane.

Characterization:

The SEM images of the samples were taken by S-4800 (HITACHI, Japan) scanning electron microscopy. Then the sample was dried under vacuum and coated by a thin layer of platinum. The TEM images of the samples were carried out using a Philips CM200-FEG transmission electron microscope. The FTIR spectroscopy was recorded

by Bruker TENSOR-27 spectrophotometer. The precipitate was dried under vacuum and then pressed into KBr pellet for FTIR spectroscopy measurement. The PL spectra of samples were measured by an F-4500 spectrofluorometer (HITACHI). The XPS spectra were obtained by VG ESCA-LAB 220i-XL apparatus. Ultrasound treatment was performed by Kunshan KQ-100TDV ultrasonic instrument with the power kept at 80 W.

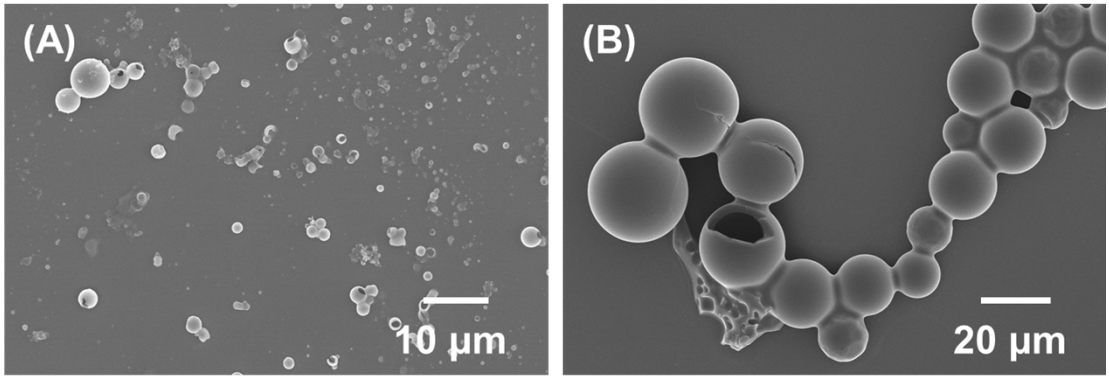

Figure S1. SEM images of unilocular FF hollow spheres obtained with the ultrasonic power kept at 100 W (A) and 50 W (B).

Ultrasound treatment was performed by Kunshan KQ-100TDV ultrasonic instrument (ultrasonic frequency: 80 KHz, ultrasonic power: 40~100 W). The unilocular and multilocular FF hollow spheres mentioned in the text were obtained with the ultrasonic power kept at 80 W. Besides, controlled experiments were also conducted at 100 W and 50 W, as shown in Figure S1. When the ultrasonic power was set at 100 W, FF hollow spheres could also be obtained, but with low yield. The sizes of FF hollow spheres obtained at 100 W were smaller than those obtained at 80 W. This may due to the smaller emulsion droplet formed at higher ultrasonic power (100 W). When the ultrasonic power was set at 50 W, larger FF hollow spheres could be obtained due to the larger emulsion droplet formed at lower ultrasonic power (50 W). However, the yield of hollow spheres was low. This may attribute to the difficulty in emulsion droplet formation at lower ultrasonic power.

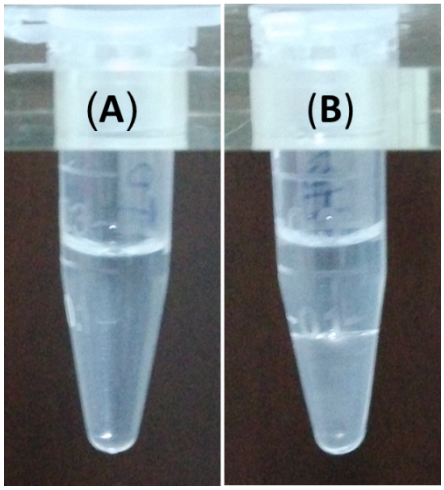

Figure S2. Optical images of solubilities of HFIP with toluene (A) and hexane (B).

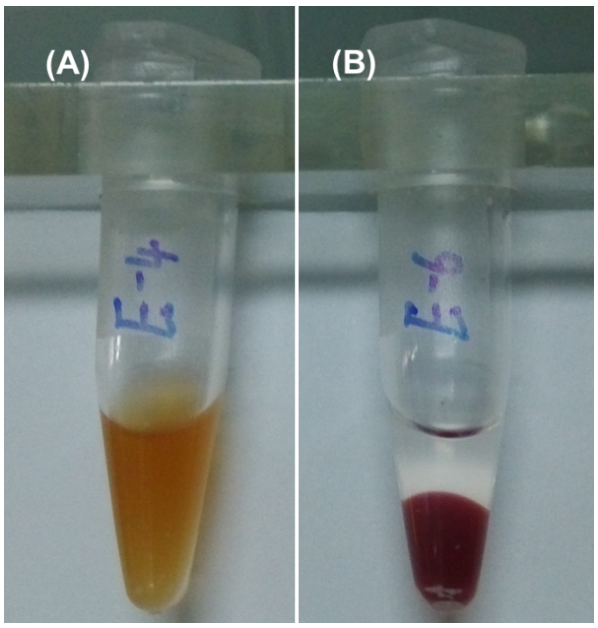

Figure S3. Optical images of emulsion formed when adding FF/HFIP solution to GA/toluene emulsion (A) and GA/hexane emulsion (B) with ultrasound treatment.