# **Supporting Information**

#### **1. Experimental Section**

*Preparation of Peptide Microtubes and Nanofibers*: The lyophilized diphenylalanine peptide (Bachem, Switzerland) was dissolved in acetonitrile-water solvent (2 mg/mL) at 95 °C for 4 min, which yields a transparent solution. The acetonitrile content in solvent mixture was varied from 0% to 100%. The resulting peptide solutions were 1) in-situ cooled to room temperature; 2) deposited on microscopic glass coverslips and dried at ambient temperature. When the acetonitrile content increased to 90%, the self-assembly rate was dramatically improved and a vast number of peptide assemblies were formed within several minutes. All the self-assembly processes on glass surface were very quick and required only several seconds, due to the simultaneous cooling and evaporation of solvent.

*Scanning Electron Microscopy:* All the samples were sputter-coated with platinum using E1045 Pt-coater (Hitachi, Japan), and then imaged by an S-4800 field emission scanning electron microscope (Hitachi High-technologies CO., Japan) at the acceleration voltage of 5 kV.

*X-Ray Diffraction:* X-ray diffraction measurements were performed on a D8 Focus powder diffractometer (Bruker, German). The diffracted intensity of CuKa radiation (wavelength of 1.5418 nm, under a condition of 40 kV and 40 mA) was measured with a scan rate of 2  $^{\circ}$ /min in a 20 range between 3  $^{\circ}$  to 50  $^{\circ}$ .

### 2. Supporting Figures



**Fig. S1** SEM images of diphenylalanine assemblies (Route I) with different acetonitrile content (a-5%; b-10%; c-30%; d-70%; e-90%; f-95%).



Fig. S2 SEM images of hollow diphenylalanine nanotubes (Route I) with 90% acetonitrile content.



Fig. S3 Macroscopic image of diphenylalanine assemblies formed in acetonitrile-water solvent mixture with 90% acetonitrile content (Route I).



Fig. S4 SEM images of diphenylalanine assemblies (Route II) on glass surface with different acetonitrile content (a- 50%; b-70%).



Fig. S5 Diameter distribution of nanofibers assembled on glass surfaces with various acetonitrile contents (a-90%; b-95%; c-100%).



Fig. S6 Powder XRD patterns of diphenylalanine nanofibers prepared on a glass surface with 90% acetonitrile.

## 3. Supporting Tables

| Table 51 The property parameters of organic solvents |                |             |             |                 |                                     |
|--|----------------|-------------|-------------|-----------------|-------------------------------------|
| Solvent  | Polarity index | HBD ability | HBA ability | Surface tension | Assemblies morphology               |
| Water  | 10.20          | 1.17        | 0.47        | 72.80           | Nanotubes/Microtubes <sup>a,b</sup> |
| Acetonitrile   | 5.80           | 0.19        | 0.40        | 19.10           | Nanofibers <sup>b</sup>             |
| Methanol   | 5.10           | 0.98        | 0.66        | 22.55           | Nanofibers <sup>b</sup>             |
| Ethanol  | 4.3            | 0.86        | 0.75        | 22.27           | Microcrystals <sup>c</sup>          |
| Chloroform   | 4.10           | 0.20        | 0.10        | 27.16           | Organogels <sup>d</sup>             |
| Toluene  | 2.40           | 0.00        | 0.11        | 28.53           | Organogels <sup>d</sup>             |

Table S1 The property parameters of organic solvents

Note: a-ref.<sup>1, 2</sup> and this work; b-this work; c-Ref.<sup>3</sup>; d-Ref.<sup>4</sup>.

### Reference

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