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Supplementary Material

AuNPs synthesised *in situ* from self-assembled peptide hydrogels modulating peptide secondary structure

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Experimental section

Chemicals and materials

Tetrachloroauric acid ($HAuCl_4$), purchased from Shanghai Maclean Biochemical Technology Co., Ltd. Fmoc-FFCKK-OH peptide was synthesised with Fmoc-Asp(OtBu)-Wang resin (0.75 mmol, 1000 mg) and 4 times more amino acids (3.0 mmol) by standard Fmoc procedure with 98% purity. Ultrapure water was prepared by a ULUPURE unit (UPH-I-40L) with a resistivity of 18.25 MΩ cm.

Preparation of peptide hydrogels

Fmoc-FFCKK-OH was dissolved in ultrapure water and the prepared 100 mM of $HAuCl_4$ mother liquor was added to obtain solutions with Fmoc-FFCKK-OH and $HAuCl_4$ concentrations of 2 and 3 mM respectively. The hydrogels were formed after standing for 45 min.

Characterisations

FT-IR spectroscopy

The FT-IR spectra of the samples were recorded at room temperature using a NEXUS B70 FT-IR spectrometer in the wavenumber range of 4000 to 400 cm^{-1}. The FT-IR samples were prepared by freeze-drying hydrogel in a lyophiliser (LGJ-10N, Yaxingyike Technology Development Co., Ltd, China).

^{1}H NMR (DMSO-d6)

The hydrogels with a concentration of 2 mM of Fmoc-FFCKK-OH and 3 mM of Au^{3+} was lyophilised and redissolved in DMSO-d6. The solution was transferred into a 5-mm sample tube ^{1}H NMR spectra were recorded on a Bruker Advanced 500-MHz spectrometer.

Rheological characterisations

The rheological experiments were operated on a DHR-2 with a cone−plate system (diameter, 25 mm; cone angle, 2°1′5″). In the oscillation measurements, in order to ensure that the selected stress was in the linear viscoelastic region, an amplitude sweep was performed at a fixed frequency of 1 rad s^{-1} before the following frequency sweep.

TEM observations

The hydrogel was dipped by the copper mesh and the excess hydrogel was removed by blotting paper. Then, the copper mesh was put into lyophiliser. After freeze-drying with a lyophiliser, a small amount of sample was deposited on the copper mesh. Observations were performed on a JEOL Jam-1400 transmission electron microscope with an operating voltage of 120 kV, and photographs were obtained on a Gatan multi-scan CCD.

CD spectra

CD spectra were obtained by rinsing nitrogen during operation with a Chirascan V100 polarisation spectrophotometer (Applied Photophysics, UK). The test was done at room temperature, and the measurement

range was 190–260 nm.

SERS analysis

In order to reduce the damage to the samples, the 633-nm laser excitation was used in the experiments with the French HORLBA JY microconfocal Raman spectrometer.

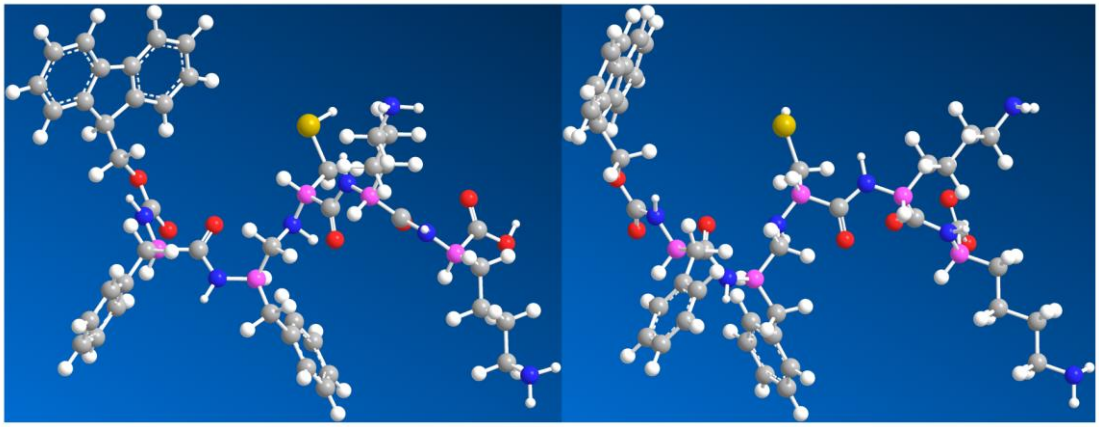

Figure S1. The molecular structure model of Fmoc-FFCKK-OH is shown from two different angles, highlighting the α-carbon of the amino acid in pink.

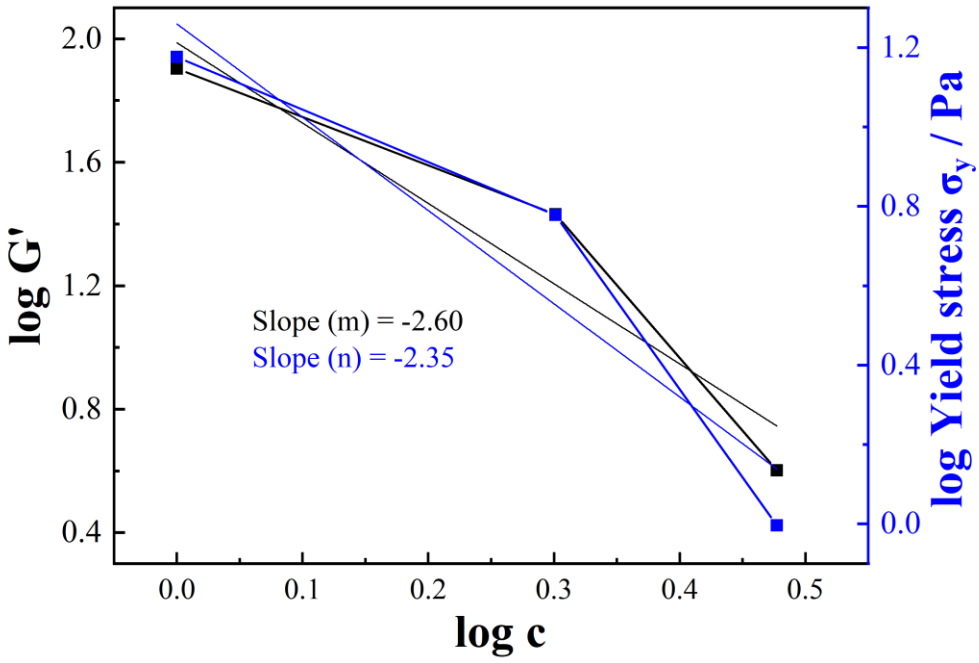

Figure S2. A typical plot of log G' *v.* log c and log σy *v.* log c to show the power law behavior for the Fmoc-FFCKK-OH in $HAuCl_4$ solution.

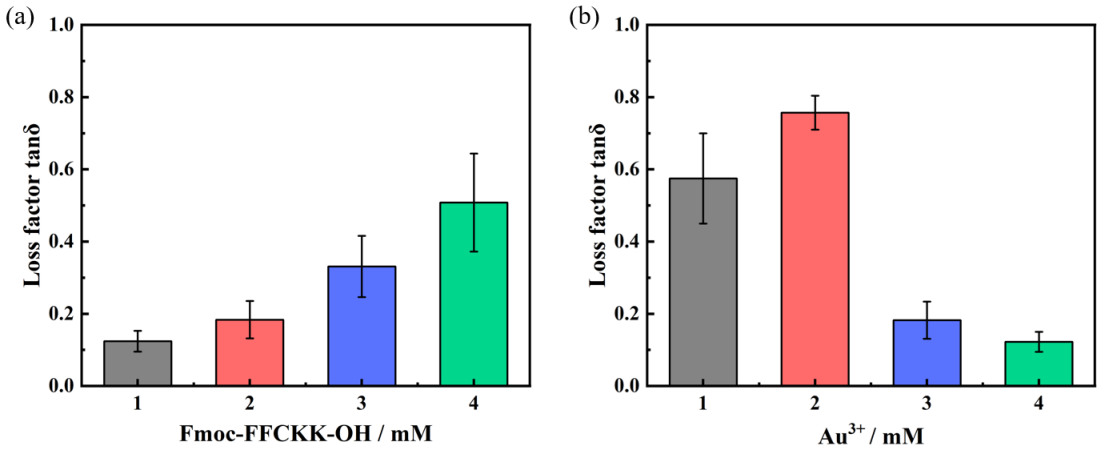

Figure S3. Average loss factor of angular frequency scanning rheograms for (a) different concentrations of Fmoc-FFCKK-OH (Au^{3+} concentration of 3 mM) and (b) different concentrations of Au^{3+} (peptide concentration of 2 mM).