Large-scale visualization of dispersion of liquid-exfoliated two-dimensional

nanosheets

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Experimental Section

Chemicals and Materials. 4-Bromobenzaldehyde and palladium diacetate (Pd(OAc)₂) were purchased from SA EN Chemical Technology Co., Ltd. (Shanghai, China). 4-vinylpyridine and anhydrous potassium phosphate were obtained from Alfa Aesar, Thermo Fisher Scientific Inc. (Shanghai, China). Poly(sodium-p-styrenesulfonate) (PSS), anhydrous dimethyl formamide (DMF) and trifluoroacetic acid (TFA) were purchased from J&K Scientific Ltd. Tetrahydrofuran (THF), dichloromethane (DCM), petroleum ether (PE), methanol, ethanol, acetonitrile, anhydrous sodium sulfate, dimethyl sulfoxide (DMSO) and glycerol were purchased from Beijing Chemical Reagent Company (Beijing, China). Deionized water was freshly prepared from a Millipore Milli-Q system with 18.2 MU/cm resistivity (Barnstead, CA, USA). Laponite XLG clay and sodium montmorillonite clay were supplied from Nanocor, PGW grades. All the reagents in this work were used without further purification.

Preparation of Laponite and Montmorillonite Nanosheets Post-labelled by FSPH. Firstly, 100 mg of laponite powder was exfoliated into 0.1% nanosheets by adding PSS in aqueous solutions. Moreover, FSPH was mixed with 0.1% laponite nanosheets at a constant weight percentage (keeping the concentration of FSPH increased from 10 μ M to 2 mM, for example, the 50 μ M of FSPH-laponite nanosheets hybrid solution, add 100 uL of 500 μ M FSPH into 900

uL of 0.1% laponite nanosheet solution). 100 mg of montmorillonite powder was exfoliated into 0.1% nanosheets under ultrasonication for 1 h. The montmorillonite nanosheets by post-labelled FSPH were prepared according to the same method.

The Titration Experiment of Isothermal Titration Calorimetry (ITC). FSPH (5 × 10⁻⁴ M,

 $60 \ \mu$ L) solutions were titrated into 1% laponite nanosheets solutions (300 μ L). All the data were fitted by independence model. The temperature of the cell was set at room temperature.

Sample Characterizations. The UV-vis absorption spectra were recorded by a Shimadzu UV-3600 spectrophotometer (Japan). Fluorescence spectra were measured by a Hitachi F-7000 fluorescence spectrophotometer (Japan). The excitation and emission slits were both set at 5.0 nm with a scanning rate of 1200 nm/min. The quantum yield values were obtained from the reconvolution fit analysis (Edinburgh F980 analysis software) equipped with an integrating sphere. TEM photographs were performed on a Tecnai G220 TEM (FEI Company) at an accelerating voltage of 200 kV. Dynamic light scattering (DLS) and zeta potential were determined by using a Malvern Zetasizer 3000 HS nano-granularity analyzer. ITC was implemented on a PEAQ ITC system (China). Fluorescence microscope images were recorded on a CLSM (Leica, TCS SP8).

Supporting Figures



Fig. S1. TEM image of (a) laponite nanosheets and (b) montmorillonite nanosheets.



Fig. S2. Fluorescence spectra of FSPH labelled-laponite nanosheets, pure FSPH solutions (50 μ M) and laponite nanosheets.



Fig. S3. UV-vis absorption spectra of the FSPH labelled-laponite nanosheets in the different concentrations of FSPH (10 μ M to 80 μ M).



Fig. S4. UV-vis absorption spectra of FSPH (10 μ M) in aqueous solution and in glycerolmethanol mixtures containing 0.5% DMSO solution, respectively.



Fig. S5. CIE chromaticity diagram of the FSPH labelled-laponite nanosheets.



Fig. S6. Fluorescence spectra for FSPH labelled-laponite nanosheets in the different weight percentage of laponite nanosheets (1% to 10%).



Fig. S7. Fluorescence spectra for FSPH labelled-montmorillonite nanosheets in the different concentrations of FSPH (10 μ M to 200 μ M).



Fig. S8. 2D fluorescence microscopy image of FSPH labelled-montmorillonite nanosheets (50 μ M).



Fig. S9. 3D-representation of fluorescence confocal microscopy images of FSPH labelledmontmorillonite nanosheets (50 μ M), the green colored patterns represented the stained montmorillonite nanosheets.