Electronic Supplementary Information for

**3D assembly of Ti₃C₂-MXene directed by water/oil interfaces**

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Experimental Section

Raw materials: cetyltrimethylammonium bromide (CTAB, 99%), Hydrochloric acid (36%-38%), and Sodium hydroxide (96%) were purchased from Shanghai Linfeng Chemical Reagent CO. Ltd, Lithium fluoride (99%). n-Dodecane (98%) were obtained from Shanghai Aladdin Chemistry CO. Ltd. Nile Red and Rhodamin B (fluorescent dye) was provided by Nanjing Oddfoni Biological Technology CO. Ltd and Tianjin Chemical Reagent Research Institute, respectively. Titanium aluminum carbide (98%) was purchased from Beijing Forsman Technology CO. Ltd.

Preparation of $\text{Ti}_3\text{C}_2\text{T}_x$-MXene: LiF (1.98g) was firstly dissolved in 30ml HCl solution (6M), and then 3g $\text{Ti}_3\text{AlC}_2$ powder was added slowly. The etching process lasted for 100 hours at 40 °C and was terminated by rinsing the mixture several times. The sediment was dried for use at 70 °C following centrifugation.

Preparation of Pickering emulsions stabilized by CTAB-doped MXene: the pH of the MXene dispersion was firstly adjusted by the addition of NaOH (1M) or HCl solution (1M). Then, the MXene dispersion was loaded with CTAB solution (0.01mM, millimoles per liter (mM) relative to the aqueous phase) prior to adding dodecane. Finally, the mixture was subjected to vortex mixing at 2500 rpm to form the emulsions directly. Making Pickering HIPEs also followed this procedure with increasing the volume fraction of the dodecane phase to 70vol% (a typical recipe contains 2.1 ml oil, 0.9 ml water, 72 mg MXene and 16 mg CTAB).

Preparation of HIPE templated porous materials: 2-Hydroxyethyl methacrylate (HEMA) and the initiator AIBN were selectively diffused into the continuous phase of the HIPE with 70vol% of oil droplets. Polymerization was carried out at 60 °C for 4h, and the liquids were dried off at 70 °C to obtain a porous material.

Characterization: An FTIR spectrometer (Thermo Fisher Scientific, USA) was used to determine the spectra of MXene and CTAB-doped MXene over the range 400-4000 cm$^{-1}$ also using a pellet of powdered potassium bromide and a sorbent. The morphology of the solid foam-like structures was observed using a Phenom Pro desktop SEM (Phenom-World Company, China) operating at 10 kV. The images of Pickering emulsions/HIPEs were captured on a NIKON fluorescence microscope equipped with Xe lamp combined with filters. The aqueous and dodecane phases were labelled by Nile Red (excited by a 488 nm laser line) and Rhodamine B (excited by a 507 nm laser line). The mechanical strength of porous material was performed in an universal test machine (CMT 5254). Thermal grayimetric analysis of porous material was measured by using a Mettler Toledo (TGA2) thermal analyzer with Al$_2$O$_3$ pans under nitrogen flow with a heating rate of 10 °C/min from room temperature (RT) to 700 °C.
Supporting Figures

**Figure S1.** SEM images of as-prepared Ti$_3$C$_2$-MXene.

**Figure S2.** XPS spectrum of as-prepared Ti$_3$C$_2$-MXene.

**MOVIE S1.mov**

**Figure S3.** The video (Movie S1) shows the phase separation process of the mixture (water, dodecane and CTAB doped MXene) at pH=5.
**Figure S4.** The photograph showing (a) the mixture of 1.5ml water, 1.5ml dodecane and CTAB (0.045mM). (b) the mixture of 1.5ml water, 1.5ml dodecane and unmodified Ti$_3$C$_2$-MXene at different pH volume (the MXene concentration=5mg/ml).

**MOVIE S2.mov**

**Figure S5.** The video (Movie S2) shows the destabilization of a emulsion with the addition of acid.

**MOVIE S3.mov**

**Figure S6.** The video (Movie S3) shows the effect of load on a porous material prepared from a HIPE template.

**Figure S7** (a) mechanical resistance and (b) thermal gravimetric analysis of a porous material.