Supplementary information
Rational control of interlayer space inside two-dimensional titanium carbides for highly efficient uranium removal and imprisoning

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Experimental Section

Materials.

Synthesis of pristine and intercalated Ti$_3$C$_2$T$_x$ MXenes. Ti$_3$AlC$_2$ powders with purity >99 wt % and particle size < 19 μm (i.e., 800 mesh) were purchased from Beijing Jinhezhi Materials Co., Ltd. 0.5 g Ti$_3$AlC$_2$ powders were first slowly added in portions into 10 mL 15 wt % HF (diluted from 40 wt % HF, Sinopharm Chemical Reagent Co., Ltd.). The mixture was then stirred and held at 40 ºC for 48 h, after which the product was centrifuged at 5000 rpm and washed with deionized water 6 times to obtain a pristine Ti$_3$C$_2$T$_x$ water suspension (labeled as Ti$_3$C$_2$T$_x$-hydrated). In order to prepare intercalated Ti$_3$C$_2$T$_x$, Ti$_3$C$_2$T$_x$-hydrated was first filtered and immediately dispersed in 1 M NaOH (Sinopharm Chemical Reagent Co., Ltd.) solution at room temperature for 2h, or dispersed in dimethyl sulfoxide (DMSO, Sinopharm Chemical Reagent Co., Ltd.) at 37 ºC for 48 h. The products were then centrifuged and washed with deionized water 6 times to obtain the NaOH activated Ti$_3$C$_2$T$_x$ water suspension (labeled as Ti$_3$C$_2$T$_x$-NaOH-hydrated) and the DMSO intercalated Ti$_3$C$_2$T$_x$ water suspension (labeled as Ti$_3$C$_2$T$_x$-DMSO-hydrated). The corresponding dry samples (labeled as Ti$_3$C$_2$T$_x$-dry, Ti$_3$C$_2$T$_x$-NaOH-dry and Ti$_3$C$_2$T$_x$-DMSO-dry, respectively) were prepared by filtering various Ti$_3$C$_2$T$_x$ water suspensions on 0.45 μm nylon membranes and drying in a vacuum oven at room temperature for 12 h. Generally, the concentrations of the studied hydrated Ti$_3$C$_2$T$_x$ samples were in the range of 10-15 mg mL$^{-1}$. The actual concentrations of the
hydrated samples were obtained by measuring the mass of the dry sample on the filtering membrane for a fixed volume of suspension.

*Calcination of Ti$_3$C$_2$Tx after uranium sorption.* 200 mg of Ti$_3$C$_2$Tx-hydrated was first used to uptake U(VI) at the condition of pH=5.0, [U]$_{\text{initial}}$=100 mg L$^{-1}$, m/V=0.4 g MXene L$^{-1}$ and t=360 min, and then the sorbent was collected by filtering and was dried in a vacuum oven. The dry U(VI)-sorbed MXene sample was transferred into a quartz tube furnace and was calcined at the desired temperature for 10 min, using a heating rate of 20 °C min$^{-1}$ to the calcination temperature, under an air or nitrogen atmosphere.

*Characterization.*

The morphologies and chemical compositions of the MXene samples were measured by a Hitachi S-4800 field-emission scanning electron microscope (SEM) equipped with Horiba 7593-H energy-dispersive X-ray spectroscopy (EDS). A Bruker D8 Advance diffractometer (Cu Kα radiation, $\lambda$ = 1.5406 Å) was used to collect powder X-ray diffraction (XRD) patterns. The step size for XRD pattern scanning was 0.02°. Hydrated samples were obtained by rapidly filtering the MXene suspensions with 0.45 μm nylon filtration membranes, and then immediately subjected them to XRD measurements in wet conditions. A Bruker Tensor 27 spectrometer was used to record the fourier transform infrared (FTIR) spectra of samples by a potassium bromide pellet method. The residual concentrations of uranium and other metal
elements were determined by an inductively coupled plasma optical emission spectrograph (ICP-OES, Horiba JY2000-2, Japan). The low concentrations of uranium at ppb levels in the uranium-contaminated water treatment test were measured by a Thermo Fisher Element XR high resolution inductively coupled plasma mass spectrometer (HR-ICP-MS). UV-vis spectra of solution samples were measured by a Hitachi U-3900 spectrophotometer to determine the residual concentrations of methylene blue (MB) in the dye sorption experiments. In order to predetermine the suitable calcination temperature for dry U(VI) sorbed MXene samples, a Q500 thermogravimetry (TA Instruments) was used to acquire thermogravimetric curves over the range of 20–1000 °C with a heating rate of 20 °C min⁻¹ in air. X-ray absorption near edge-structure (XANES) spectra of the U L₃-edge (17,166 eV) for samples and reference compounds were collected at the beamline 1W1B of the Beijing Synchrotron Radiation Facility (BSRF). The U L₃-edge XANES spectra of UO₂ and UO₂(NO₃)₂·6H₂O were measured in transmission mode. The U L₃-edge XANES spectra of U-sorbed Ti₃C₂Tx was measured in fluorescence mode using a lytle-type ion chamber detector. The incident X-ray beam was modulated to desired energies by using a silicon (111) double-crystal monochromator.

**Batch sorption experiments.**

*Removal of U(VI).* A series of sorption experiments for U(VI) uptake in aqueous solution by various Ti₃C₂Tx MXene samples was carried out as a function of contact
time, pH, initial U(VI) concentration ([U]_{initial}), ionic strength, other competing metal cations, solid-liquid ratio and storage time. A proper amount of UO$_2$(NO$_3$)$_2$·6H$_2$O (Sinopharm Chemical Reagent Co. Ltd.) was first dissolved in deionized water to prepare a 400 mg L$^{-1}$ U(VI) stock solution. The U(VI) stock solution was then diluted to initial concentrations ranging from 5 to 220 mg L$^{-1}$ to perform U(VI) uptake experiments by a batch method. Typically, 4 mg Ti$_3$C$_2$Tx MXene sample was mixed with an appropriate amount of deionized water to keep the total volume of suspension as 5 mL in a 20 mL beaker. Then 5 mL of solution containing U(VI) or multi-metal ions was added such that the concentration of sorbent was 0.4 g L$^{-1}$. The pH value of the solution was adjusted with small amounts of 0.1 M NaOH and 0.1 M HNO$_3$, and then the mixture solution was stirred at room temperature for a specified time. The solid extractant was removed after U(VI) sorption to obtain the supernatant sample by using a polyethersulfone syringe filter (0.22 μm, ANPEL Scientific Instrument Co., Ltd., Shanghai). The supernatant was diluted with 5 wt% HNO$_3$ before the final concentration was determined by ICP-OES or HR-ICP-MS. For the ionic strength tests, NaClO$_4$ (0.1-500 mmol L$^{-1}$), Ca(NO$_3$)$_2$ (1-200 mmol L$^{-1}$) and Mg(NO$_3$)$_2$ (1-200 mmol L$^{-1}$) were selected as representative electrolytes while [U]$_{initial}$ was fixed as 40 mg L$^{-1}$. In the selective sorption test, all initial concentrations of U(VI) and other competing metal ions, including Co$^{2+}$, Ni$^{2+}$, Zn$^{2+}$, Sr$^{2+}$, La$^{3+}$, Nd$^{3+}$, Sm$^{3+}$, Gd$^{3+}$ and Yb$^{3+}$, were 0.5 mmol L$^{-1}$. In the solid-liquid ratio test, 4 mg Ti$_3$C$_2$Tx-DMSO-hydrated and 0.6 mg U(VI) were mixed with different volumes of deionized water to change the
dosage of sorbents from 0.4 g L\(^{-1}\) to 0.02 g L\(^{-1}\). In the experiment for simulated uranium-contaminated water treatment, a stock solution of uranium contaminated water including 1 mg L\(^{-1}\) U(VI), 200 mg L\(^{-1}\) Na(I), 100 mg L\(^{-1}\) Ca(II) and 100 mg L\(^{-1}\) Mg(II) was first prepared. The desired volume of the stock solution from 2.5 mL to 125 mL was mixed with an equal volume of Ti\(_3C_2T_x\)-DMSO-hydrated aqueous solution which contained 25 mg sorbent to achieve the treated capacity in the range of 200~10000 kg water per kg sample.

**Removal of Cu(II) and Cd(II).** A 12 mg sample of Ti\(_3C_2T_x\)-DMSO-dry or Ti\(_3C_2T_x\)-DMSO-hydrated was mixed with an appropriate amount of water to keep the total volume of suspension as 15 mL. Then, 15 mL of solution containing 100 mg L\(^{-1}\) Cu(II) or Cd(II) (as Cu(NO\(_3\))\(_2\).3H\(_2\)O or Cd(NO\(_3\))\(_2\).4H\(_2\)O, Beijing Chemical Works) was added and the composite solution was stirred at room temperature. 0.1 mol L\(^{-1}\) NaOH was then used to adjust the solution pH to about 5. MXene samples were separated at different intervals from 5 to 360 min. The concentrations of Cu(II) or Cd(II) in the supernatant solution after the sorption process was determined using ICP-OES.

**Removal of MB(Methylene Blue).** A 12 mg sample of Ti\(_3C_2T_x\)-dry, Ti\(_3C_2T_x\)-hydrated or Ti\(_3C_2T_x\)-DMSO-hydrated was mixed with an appropriate amount of water to keep the total volume of suspension as 15 mL. Then, 15 mL of solution containing 100 mg L\(^{-1}\) MB (C\(_{16}\)H\(_{18}\)N\(_3\)ClS, Beijing Chemical Works) was added and the composite solution was stirred at room temperature. MXene samples were separated at different intervals from 5 to 360 min. The concentrations of MB in the supernatant
solution after the sorption process was determined by UV-vis spectroscopy with a linear calibration curve over the concentration range of 1-50 mg L$^{-1}$. In addition, MB-sorbed samples were filtered and dried in a vacuum oven at room temperature for 12 h to investigate the intercalation behavior of MB molecules inside galleries of various MXene samples by XRD.

For all batch sorption experiments, the uptake capacity $q_e$ (mg g$^{-1}$) and distribution coefficient $K_d$ (mL g$^{-1}$) were calculated using the following equations:

$$ q_e = \frac{(C_0 - C_e) \times V}{m} $$

(S1)

$$ K_d = \frac{C_0 - C_e}{C_e} \times \frac{V}{m} $$

(S2)

where $C_0$ and $C_e$ are initial and final equilibrium concentration of cations, respectively; and $V$ and $m$ are the volume of solution and the mass amount of solid sorbent in the batch sorption tests, respectively.

Desorption and leaching experiments.

*Desorption of U(VI) from U-sorbed Ti$_3$C$_2$T$_x$-DMSO-hydrated sample.* U(VI) sorption onto the Ti$_3$C$_2$T$_x$-DMSO-hydrated sample was first performed for pH=5.0, [U]$_{\text{initial}}$=100 mg L$^{-1}$, m/V=0.4 g L$^{-1}$ and t=360 min. After sorption, the solution was centrifuged under 8000 rpm for 30 min to decant the supernatant. Then 0.01, 0.1 and 0.2 mol L$^{-1}$ HNO$_3$ were used as eluents to treat the sediment for 30 min to evaluate the desorption efficiency of U(VI) under acidic conditions.

*Leaching of uranium from calcined U-sorbed Ti$_3$C$_2$T$_x$ samples.* 5 mg of calcined U-
sorbed Ti$_3$C$_2$Tx samples prepared under different temperatures and atmospheres was immersed in 15 mL aqueous solutions (deionized water or various concentrations of Na$_2$CO$_3$ and HNO$_3$) by shaking for 24 h. The supernatants were separated and diluted with 5 wt% HNO$_3$ for the uranium concentration measurements. The long term test procedure for leaching of uranium from simulated underground water, which contained 0.29 mmol L$^{-1}$ Ca(NO$_3$)$_2$, 0.31 mmol L$^{-1}$ CaBr$_2$, 0.53 mmol L$^{-1}$ MgSO$_4$, 0.45 mmol L$^{-1}$ Na$_2$SO$_4$, 0.60 mmol L$^{-1}$ NaHCO$_3$, 0.43 mmol L$^{-1}$ KHCO$_3$ and 0.011 mmol L$^{-1}$ Na$_2$CO$_3$, was similar, except that supernatant samples were separated at different intervals from 2 h to 10 days.

**Sorption data fitting by kinetics models.**

In order to clarify the sorption process of U(VI) by Ti$_3$C$_2$Tx-DMSO-hydrated, the pseudo-first-order kinetic model and the pseudo-second-order kinetic model were used to analyze the experimentally observed kinetic data. The linearized form of the two models are given as follows:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} \times t$$

(S3)

$$\frac{t}{q_t} = \frac{1}{k_2 q_e} + \frac{t}{q_e}$$

(S4)

where $q_e$ (mg/g) and $q_t$ (mg/g) are the quantities of the sorbed U(VI) at equilibrium time and time $t$ respectively, and $k_1$ (1/min) and $k_2$ (g/(mg·min)) are the pseudo-first-order and pseudo-second-order sorption rate constants, respectively. Plots of $\log(q_e - q_t)$ versus $t$, and $t/q_t$ versus $t$ give straight lines, and $k_1$ and $k_2$ are derived from the
slopes. The model parameters and correlation coefficients obtained by both the models are listed in Table S1.

**Sorption data fitting by isotherm models**

The Langmuir model and Freundlich model are two common models to describe sorption isotherms. The Langmuir model assumes that sorption occurs on a homogenous surface by a finite monolayer sorption and the sorption energy decreases as the distance from the surface increases. The Langmuir isotherm model can be described as follows:

\[
\frac{C_e}{q_e} = \frac{1}{q_mk_L} + \frac{C_e}{q_m}
\]  

(S5)

where \(q_m\) is the maximum sorption capacity corresponding to a complete monolayer coverage (mg/g) and \(k_L\) is a constant indirectly related to the sorption capacity and energy of sorption (L/mg), which characterizes the affinity of the adsorbate for the adsorbent. A linearized plot is obtained when \(C_e/q_e\) is plotted against \(C_e\); \(q_m\) and \(k_L\) are derived from the slope and intercept.

The Freundlich model is basically empirical, and can be applied to non-ideal sorption on heterogeneous surfaces as well as multilayer sorption, and can be expressed as:

\[
\ln q_e = \ln k_F + \frac{1}{n} \ln C_e
\]

(S6)

where \(q_e\) (mg/g) is the equilibrium sorption amount, and \(k_F\) and \(n\) are the Freundlich
constants related to the sorption capacity and the sorption intensity, respectively. A linear relationship was obtained by plotting $\ln q_e$ against $\ln C_e$, and the values of $k_F$ and $n$ were calculated from the slope and intercept of the straight line.

Another commonly used isothermal model is that of Dubinin-Raduskevich (D-R). It was also employed to fit the sorption data of U(VI) on Ti$_3$C$_2$T$_x$-DMSO-hydrated. The D-R model describes sorption on a single type of uniform pore. Its linear expression is expressed as follows:

$$\ln q_e = \ln q_m - \beta \varepsilon^2$$  \hspace{1cm} (S7)

where $q_m$ (mol/g) represents theoretical monolayer saturation capacity, $\beta$ (mol$^2$/kJ$^2$) is a constant correlated to sorption energy, and $\varepsilon$ is the Polanyi potential (kJ/mol) related to the equilibrium concentration, described as:

$$\varepsilon = RT \ln(1 + \frac{1}{C_e})$$  \hspace{1cm} (S8)

where $R$ is the universal gas constant (kJ/(mol·K)) and $T$ is the absolute temperature (K). The mean free energy $E$ (kJ/mol) which is used to estimate the sorption type can be calculated from the constant $\beta$:

$$E = (-2\beta)^{0.5}$$  \hspace{1cm} (S9)

where $q_m$ and $\beta$ for U(VI) sorption on Ti$_3$C$_2$T$_x$-DMSO-hydrated were obtained from the linear plot of $\ln q_e$ against $\varepsilon^2$, and accordingly $E$ was calculated.

The model parameters and correlation coefficient obtained by the above three models are listed in Table S2.
Fig. S1 Wide angle XRD patterns of pristine and intercalated Ti$_3$C$_2$Tx MXenes under dry and hydrated conditions. The XRD pattern of parent material (Ti$_3$AlC$_2$) before HF etching is also presented for comparison.

Fig. S2 SEM images and EDS analysis results of U(VI) sorbed hydrated MXenes after vacuum drying treatment. (a-c) Ti$_3$C$_2$Tx-DMSO-hydrated, (d-f) Ti$_3$C$_2$Tx-NaOH-hydrated, (g-i) Ti$_3$C$_2$Tx-hydrated.
Fig. S3 FTIR spectra of Ti$_3$C$_2$Tx-DMSO-hydrated and three U(VI) sorbed hydrated MXenes after vacuum drying treatment. The uranyl asymmetric stretch mode is assigned at 912 cm$^{-1}$.

Fig. S4 XANES spectra of U LIII-edge for sample of Ti$_3$C$_2$Tx-U and reference compounds.
Fig. S5 An illustration of enlarged $c$-$LP$ for U(VI) sorbed MXene in ideal condition, compared with the sample of Ti$_3$C$_2$Tx-dry. The dimension of intercalated hydrated uranyl ion can be estimated by calculating $1/2\Delta c$-$LP$.

Fig. S6 Zeta potentials of Ti$_3$C$_2$Tx, Ti$_3$C$_2$Tx-NaOH and Ti$_3$C$_2$Tx-DMSO water suspensions dependence on solution pH. The concentration of MXene is 100 mg L$^{-1}$. 
Fig. S7 (a-c) XRD patterns of dry MXene samples and corresponding wet samples with different immersion times in water. (a) Ti$_3$C$_2$Tx-dry, (b) Ti$_3$C$_2$T$_x$-NaOH-dry, (c) Ti$_3$C$_2$T$_x$-DMSO-dry. (d-f) XRD patterns of hydrated MXene samples and corresponding wet samples with different immersion times in 100 mg L$^{-1}$ U(VI) solution at pH 5.0±0.1. (d) Ti$_3$C$_2$Tx-hydrated, (e) Ti$_3$C$_2$T$_x$-NaOH-hydrated, (f) Ti$_3$C$_2$T$_x$-DMSO-hydrated.
Fig. S8 (a,b) Sorption kinetics of Cu(II) (a) and Cd(II) (b) by hydrated and dry MXenes. (c,d) Sorption kinetics of MB by different MXenes and XRD patterns of MB-uptaked samples after vacuum drying.

Fig. S7a and Fig. S7b displays that the uptake capacities of Cu(II) and Cd(II) by Ti$_3$C$_2$Tx-DMSO-hydrated have increased 91% and 62% relative to that of Ti$_3$C$_2$Tx-DMSO-dry. Figure S6c shows that sorption amounts of MB by Ti$_3$C$_2$Tx-dry and Ti$_3$C$_2$Tx-hydated are 8 mg g$^{-1}$ and 78 mg g$^{-1}$, respectively. Ti$_3$C$_2$Tx-DMSO-hydrated exhibited an excellent MB sorption capacity of 125 mg g$^{-1}$ and a high removal percentage of 99.8%, suggesting a superior dye removal performance compared to previously reported results. The featured intercalation peaks of MB are also be identified for hydrated MXenes, as shown in Fig. S7d. The (002) peak at 20 of 6.82° corresponds to a 1/2 Δc-LP of 2.91 Å, which is close to the smallest dimension of the MB molecule (3.25Å × 7.60Å × 17.0Å). Another broader peak centered at 20 of 5.91°
(1/2 $\Delta c$-LP = 4.91 Å) was found for Ti$_3$C$_2$T$_x$-DMSO-hydrated after MB sorption. It may be that the large interlayer space of Ti$_3$C$_2$T$_x$-DMSO-hydrated allows the co-intercalation of MB with two different dimensions (3.25Å and 7.60Å) in a c-LP of MXene, which would accommodate more guest molecules. On the contrary, no change in the XRD pattern was observed for Ti$_3$C$_2$T$_x$-dry, due to only exterior surface sorption.

![Fig. S9 Effect of pH on U(VI) sorption behavior in aqueous solution by Ti$_3$C$_2$T$_x$-DMSO-hydrated. [U]initial = 100 mg L$^{-1}$, m/V = 0.4 g L$^{-1}$.](image)
**Fig. S10** The linear fitting of kinetics data for U(VI) sorption by Ti$_3$C$_2$Tx-DMSO-hydrated with the pseudo-first-order kinetic (a) and the pseudo-second-order kinetic (b) models.

**Fig. S11** The linear fitting of sorption isotherm for U(VI) by Ti$_3$C$_2$Tx-DMSO-hydrated with the Langmuir (a), Freundlich (b) and Dubinin–Raduskevich (c) models.
**Fig. S12** Effect of ionic strength U(VI) sorption behavior in aqueous solution by Ti$_3$C$_2$Tx-DMSO-hydrated. [U]$_{\text{initial}}$ = 40 mg L$^{-1}$, m/V = 0.4 g L$^{-1}$, pH = 5.0±0.1.

**Fig. S13** Effect of divalent cation on uptake of U(VI) by Ti$_3$C$_2$Tx-DMSO-hydrated. (a) Ca(II), (b) Mg(II). [U]$_{\text{initial}}$ = 40 mg L$^{-1}$, m/V = 0.4 g L$^{-1}$, pH = 5.0±0.1.
Fig. S14 Effect of solid-to-liquid ratio on the sorption of U(VI) by Ti$_3$C$_2$Tx-DMSO-hydrated at pH of 5.0±0.1. Total amount of U(VI) is 0.6 mg and amount of sorbent is 4 mg in all solutions.

Fig. S15 The effect of storage time of Ti$_3$C$_2$Tx-DMSO-hydrated in air on U(VI) sorption capacity.
Fig. S16 Desorption efficiency of U(VI) sorbed Ti$_3$C$_2$T$_x$-DMSO-hydrated with different concentrations of HNO$_3$ as the eluent.

Fig. S17 XRD patterns of dry sample of U(VI) sorbed Ti$_3$C$_2$T$_x$-hydrated and corresponding calcined products under various temperatures and atmospheres. (a) U(VI) sorbed Ti$_3$C$_2$T$_x$-hydrated, (b) calcined at 200 ºC in air, (c) calcined at 400 ºC in air, (d) calcined at 500 ºC in air, (e) calcined at 450 ºC in N$_2$. ● peak position for Ti$_3$C$_2$T$_x$ MXene, ▲ anatase (JCPDF card no. 21-1272), ■ rutile (JCPDF card no. 21-1276).
Fig. S18 Thermogravimetric weight-loss curve and its derivative for U(VI) sorbed Ti$_3$C$_2$T$_x$-hydrated. The sharp increase of sample weight at 450 °C corresponds to the formation of anatase and rutile.

Fig. S19 SEM images of U(VI) sorbed Ti$_3$C$_2$T$_x$-hydrated calcined under various temperatures and atmospheres. (a) calcined at 200 °C in air, (b) calcined at 400 °C in air, (c) calcined at 500 °C in air, (d) calcined at 450 °C in N$_2$. 
**Fig. S20** Long term test of U(VI) leaching in simulated underground water by air-400 °C calcined sample.
Table S1. Kinetics model constants and correlation coefficients for U(VI) sorption by Ti$_3$C$_2$T$_x$-DMSO-hydrated.

<table>
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<tr>
<th>Kinetics model</th>
<th>Pseudo-first-order</th>
<th>Pseudo-second-order</th>
<th>Saturation capacity from experiment (mg/g)</th>
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<td>$k_1$ (min$^{-1}$)</td>
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Table S2. Comparison of isotherm parameters fitting by Langmuir, Freundlich and Dubinin–Raduskevich models for U(VI) sorption by Ti$_3$C$_2$T$_x$-DMSO-hydrated.

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<th>Dubinin–Raduskevich</th>
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