# In-situ Generated of $Ti_3C_2T_x$ ($T_x$=F, O and OH) MXene Decorated CuO Nanocomposite with Extraordinary Catalytic Activity for TKX-50 Thermal Decomposition

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## Supporting Information

**Measurement conditions**

Powder X-ray diffraction (XRD) was performed using a Rigaku smartlab 9 with Rigaku's original cross beam optics system (rated voltage of 40 kV, rated current of 150 mA). Field emission scanning electron microscopy (FESEM) (ZEISS GeminiSEM 500, Germany) was used to observe GO, $Ti_3AlC_2$, $Ti_3C_2T_x$, CuO etc.. X-ray photoelectron spectroscopy (XPS) measurements were carried out on an ESCALAB 250Xi spectrometer (Thermo Scientific, USA) equipped with a pass energy of 30 eV with a power of 100 W (10 kV and 10 mA) and a monochromatized Al Kα X-ray (hν = 1486.65 eV) source. All samples were analyzed under a pressure of less than $1.0 \times 10^{-9}$ Pa. Spectra were acquired using Avantage software (Version 5.979) with a step of 0.05 eV. Raman spectra were obtained using a Raman spectrometer (LabRAM HR Evolution, HORIBA JobinYvon, France) at room temperature using the 532 nm line as the excitation source. The thermal analysis experiment was conducted using METTLER TOLEDO TGA/DSC3$^+$; the $N_2$ flow rate was 50 ml/min; the selected heating rate were 5, 10, 15, and 20°C/min and the program heated from 50°C to 350°C.

**Experimental Section**

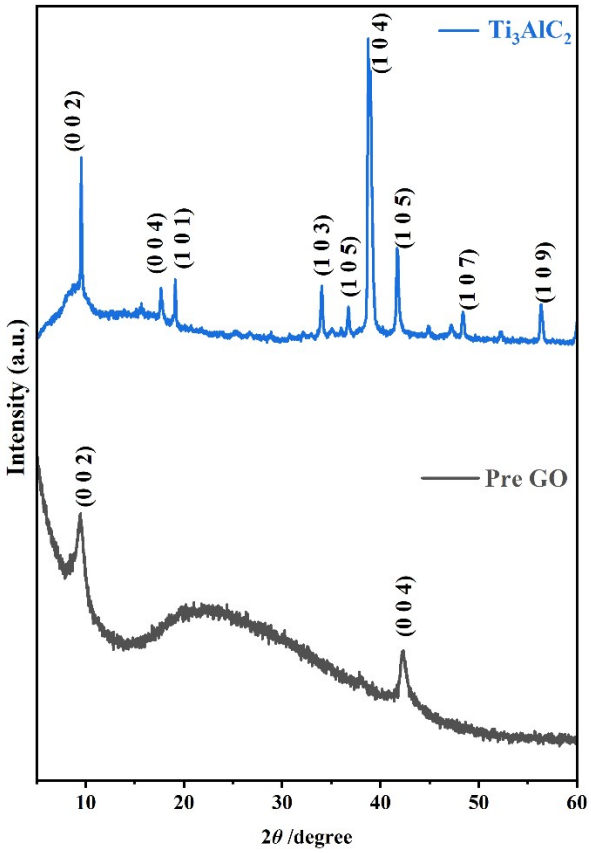



**Fig. S1.** XRD patterns of Pre GO (Graphite oxide) and $Ti_3AlC_2$

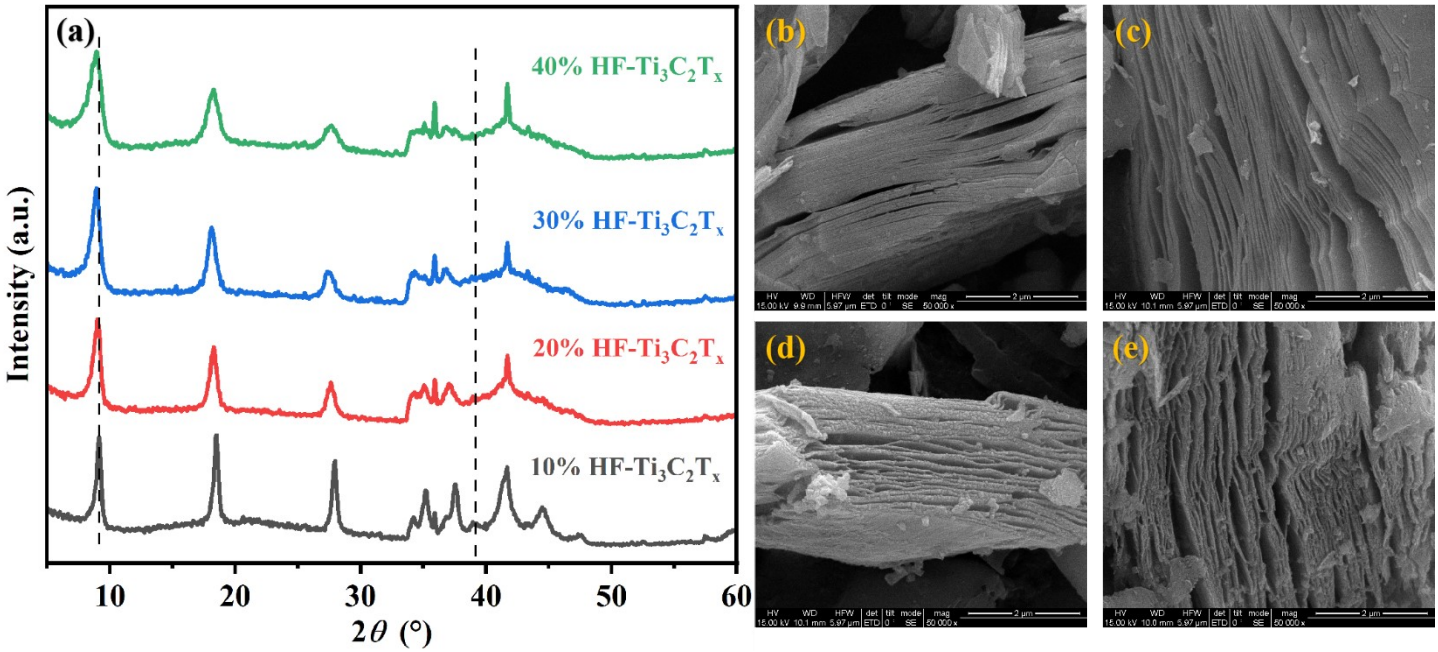
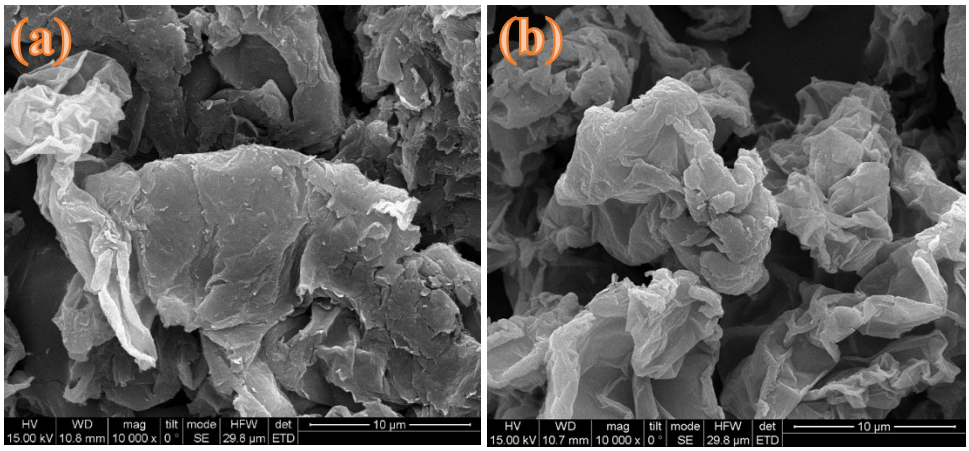
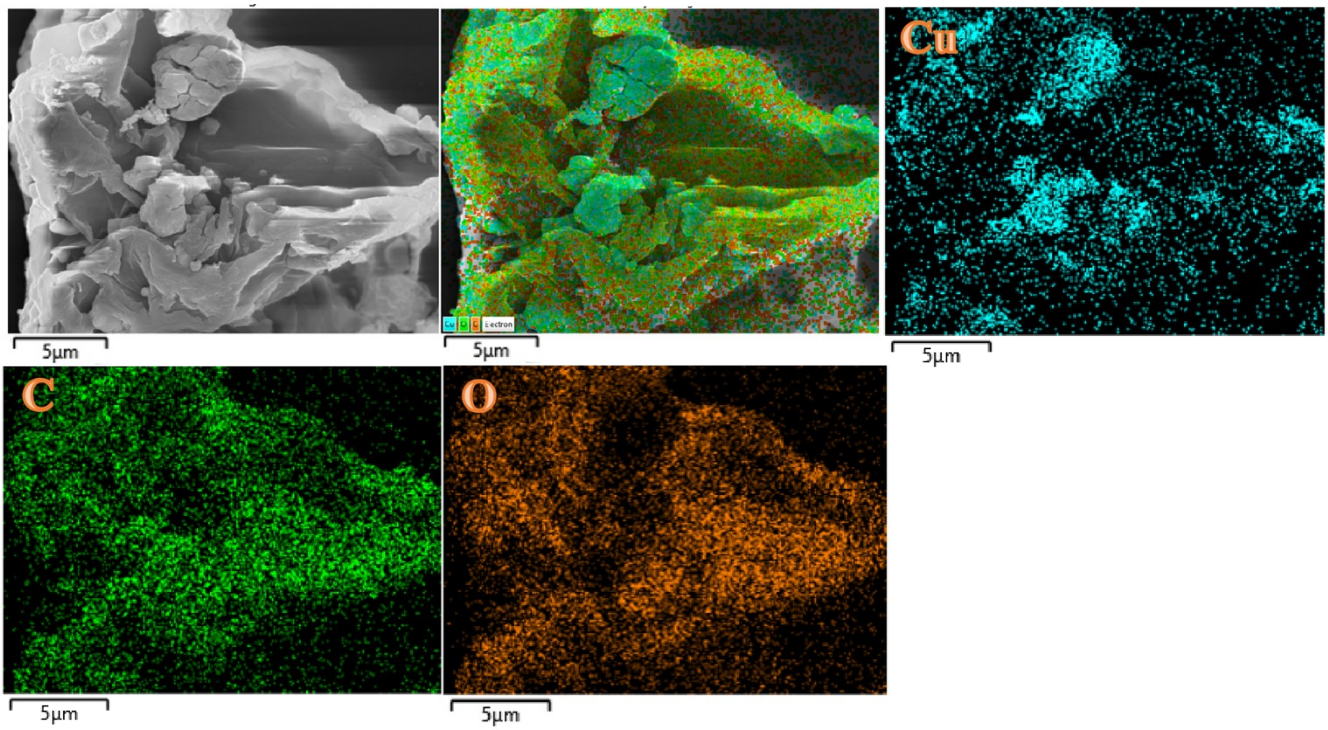



**Fig. S2.** (a) XRD patterns of 10%HF-$Ti_3C_2T_x$, 20%HF-$Ti_3C_2T_x$, 30%HF-$Ti_3C_2T_x$, 40%HF-$Ti_3C_2T_x$. SEM images of (b) 10%HF-$Ti_3C_2T_x$, (c) 20%HF-$Ti_3C_2T_x$, (d) 30%HF-$Ti_3C_2T_x$, (e) 40%HF-$Ti_3C_2T_x$.


(a)
(b)
HV 15.00 kV WD 10.8 mm mag 10 000 x tilt 0 ° mode SE HFW 29.8 µm det ETD 10 µm
HV 15.00 kV WD 10.7 mm mag 10 000 x tilt 0 ° mode SE HFW 29.8 µm det ETD 10 µm

(c)


Cu
5µm
5µm
5µm
C
O
5µm
5µm

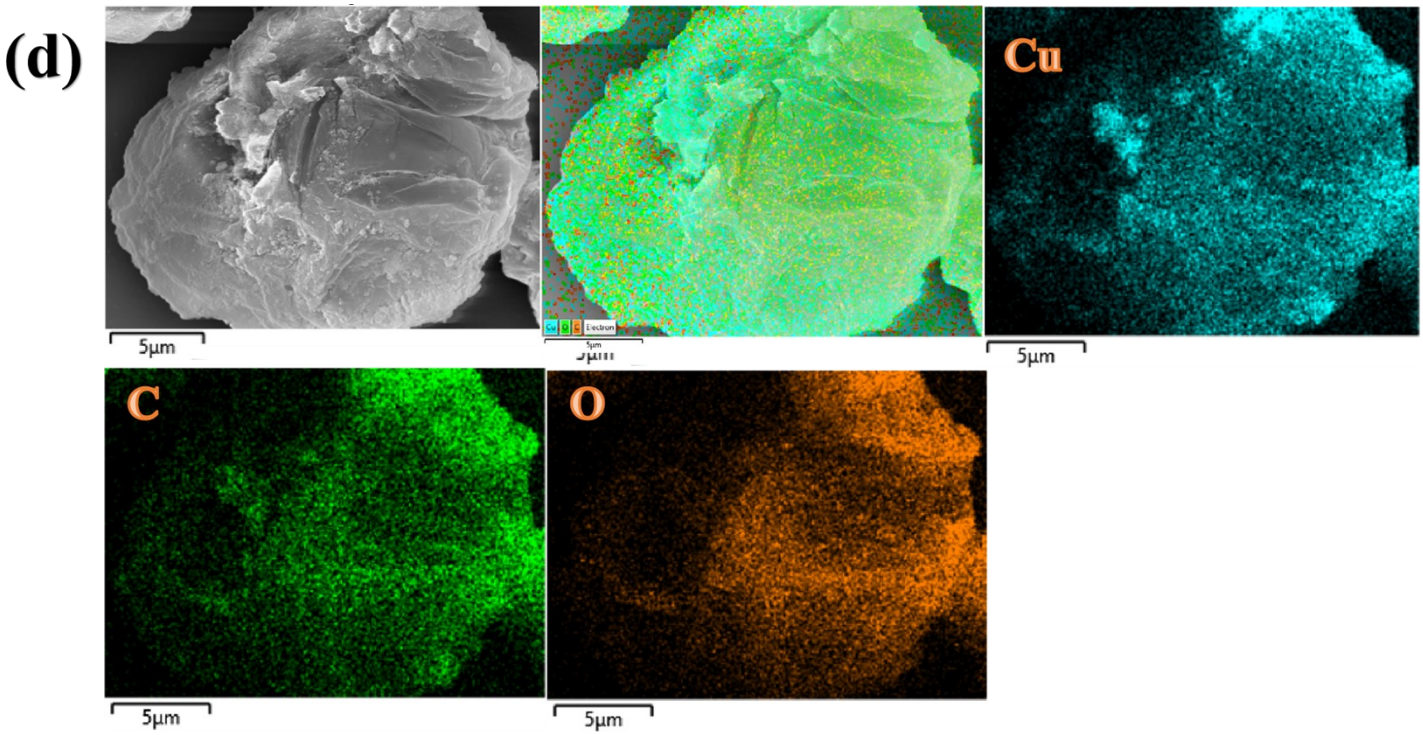
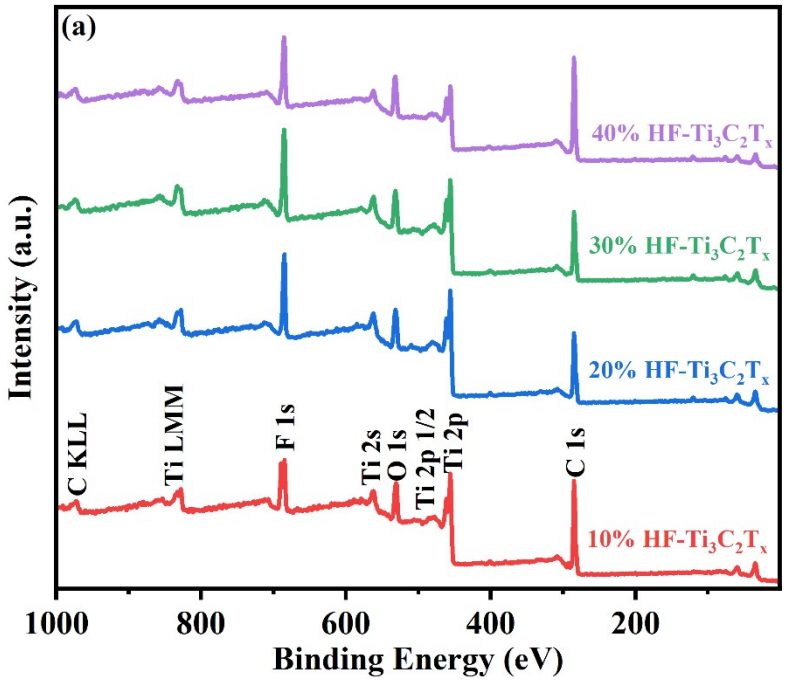



**Fig. S3.** SEM images of (a) Pre GO, (b) GO, (c) SEM images of GO/CuO composite and corresponding Cu, O, C elemental mapping. (d) SEM images of I-GO/CuO composite and corresponding Cu, O, C elemental mapping

As can be seen from **Fig. S**4, with the increase of HF etching concentration, the element distribution and valence states on the surface of $Ti_3C_2T_x$ MXene change little, thus exerting a little influence on it[1].



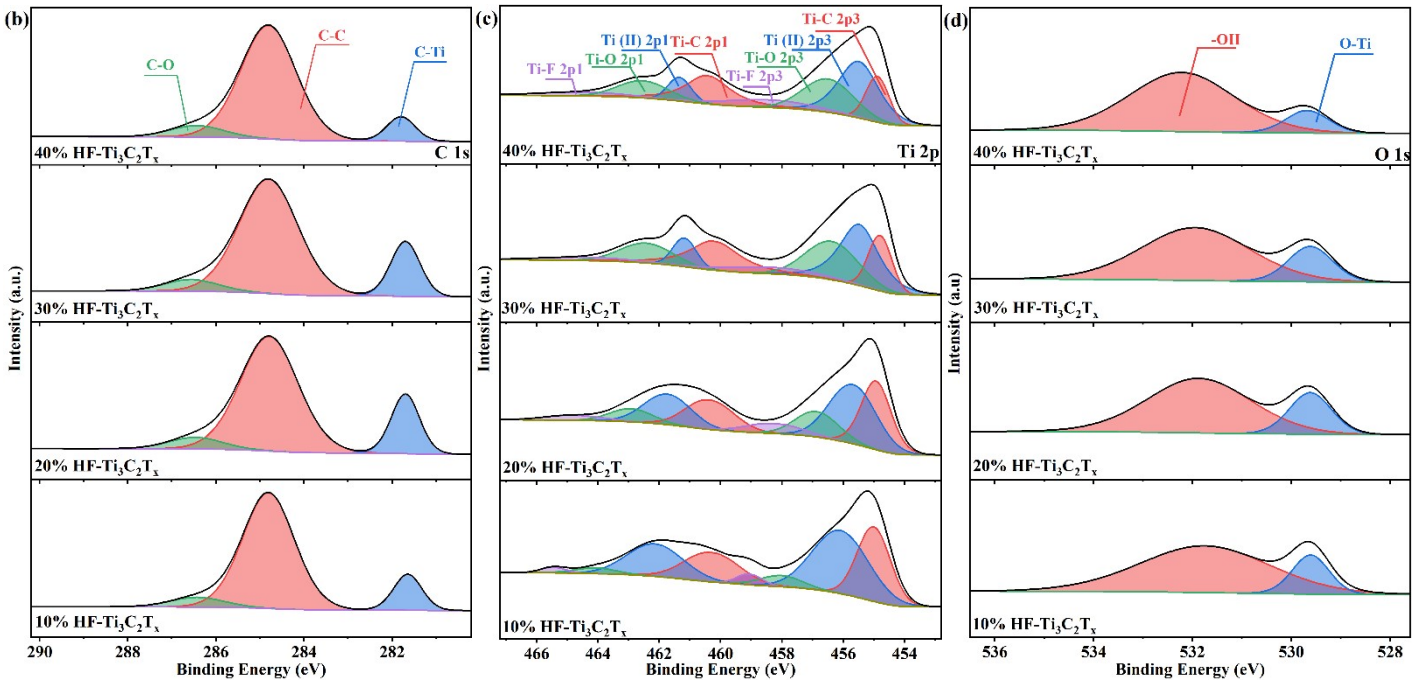



**Fig. S4.** XPS survey spectra of (a) 10%HF-$Ti_3C_2T_x$, 20%HF-$Ti_3C_2T_x$, 30%HF-$Ti_3C_2T_x$, 40%HF-$Ti_3C_2T_x$. High-resolution XPS spectra of (b) C 1s, (c) Ti 2p and (d) O 1s of 10%HF-$Ti_3C_2T_x$, 20%HF-$Ti_3C_2T_x$, 30%HF-$Ti_3C_2T_x$, 40%HF-$Ti_3C_2T_x$.

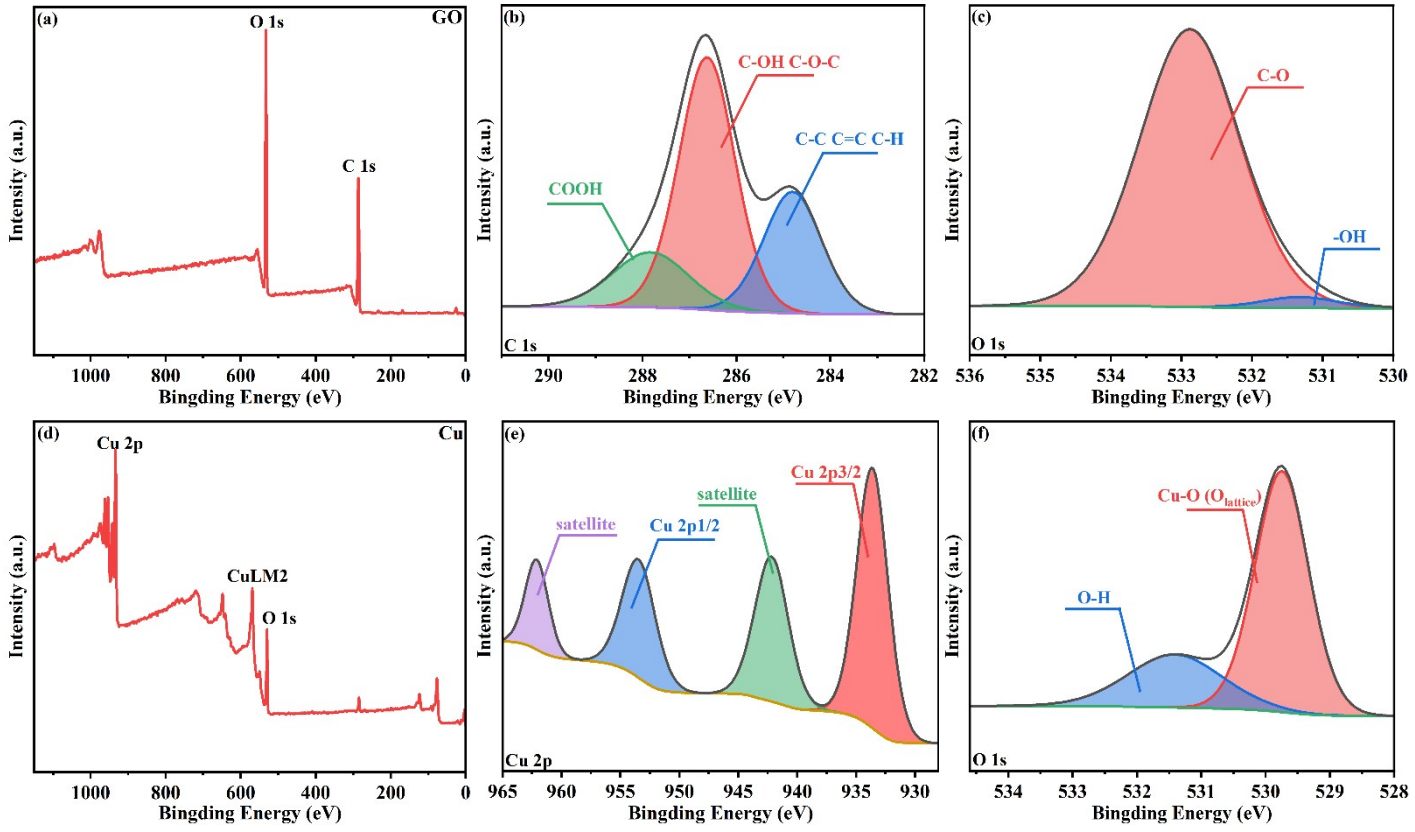



**Fig. S5.** XPS survey spectra of (a) GO. High-resolution XPS spectra of (b) C 1s, (c) O 1s of GO. XPS survey spectra of (d) CuO. High-resolution XPS spectra of (e) Cu 2p, (f) O 1s of CuO.

As shown in **Fig. S6**a, the characteristic peaks of D and G band near 1360 and 1590 $cm^{-1}$ are reflected in the GO and I-GO/CuO complex, while in **Fig. S6**b, each corresponding peak in $Ti_3C_2T_x$ and I-$Ti_3C_2T_x$/CuO is represented correspondingly, which indicates the generation of the complex. Meanwhile, the corresponding peak intensity of I-GO/CuO I-$Ti_3C_2T_x$/CuO and spectrum are much weaker than that of GO and $Ti_3C_2T_x$. This may be related to the encapsulation structure of I-GO/CuO I-$Ti_3C_2T_x$/CuO.

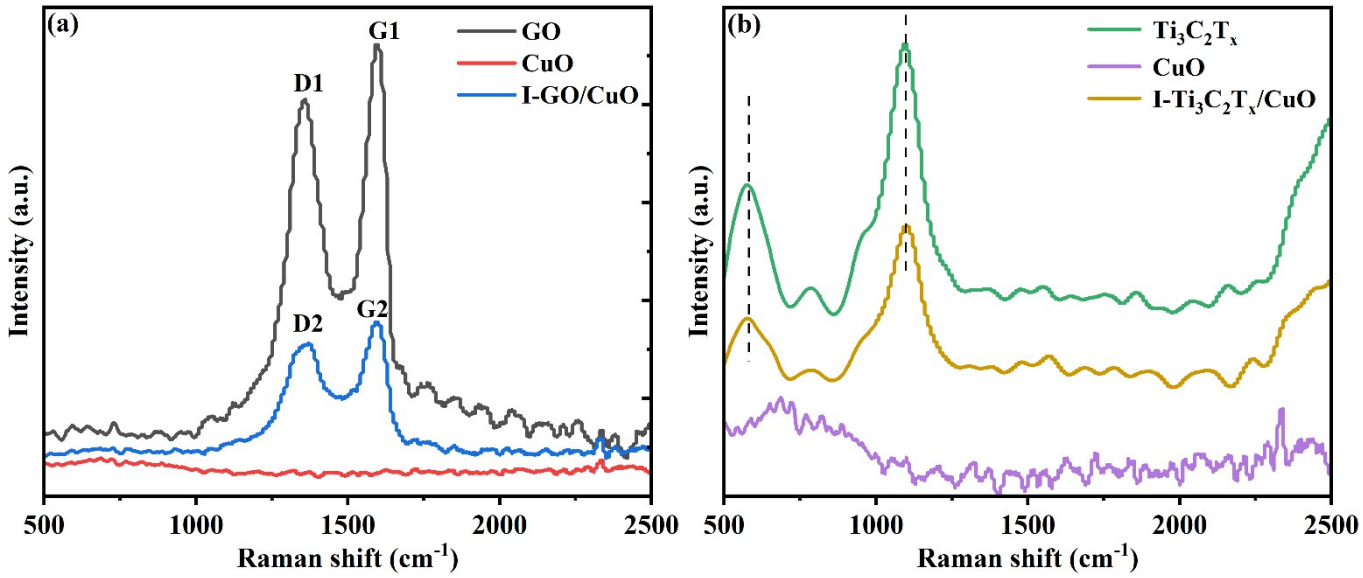



**Fig. S6.** Raman spectra of (a) GO, CuO, I-GO/CuO. (b) $Ti_3C_2T_x$, CuO, I- $Ti_3C_2T_x$/CuO.

## Catalysis performance

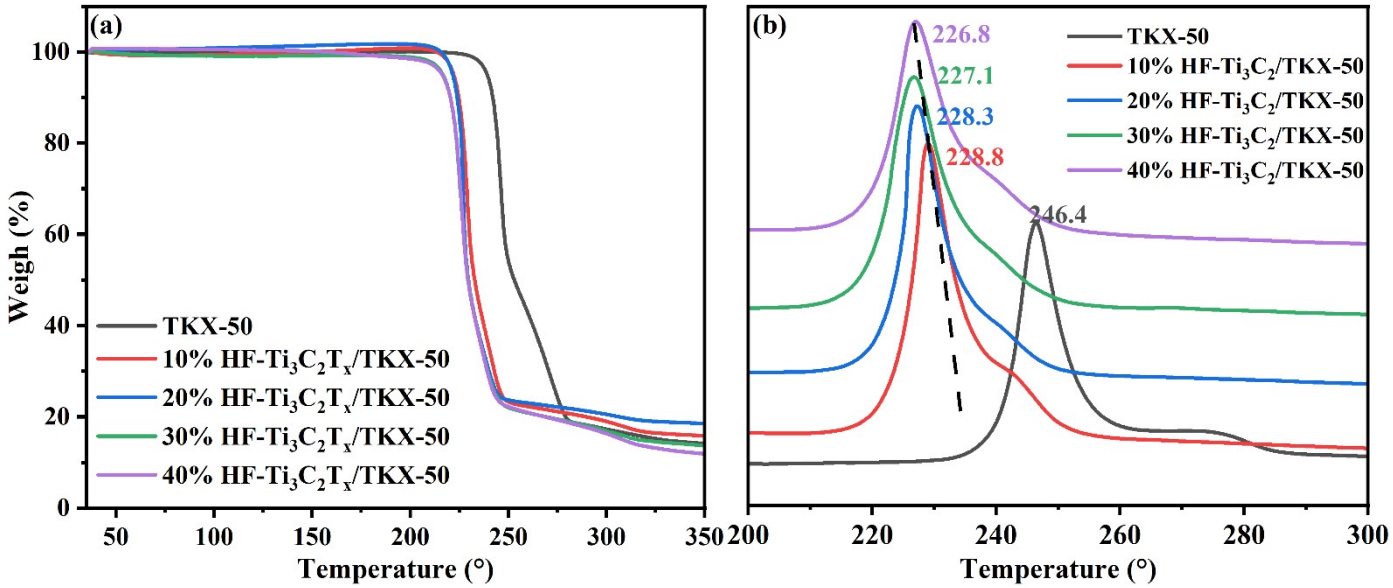
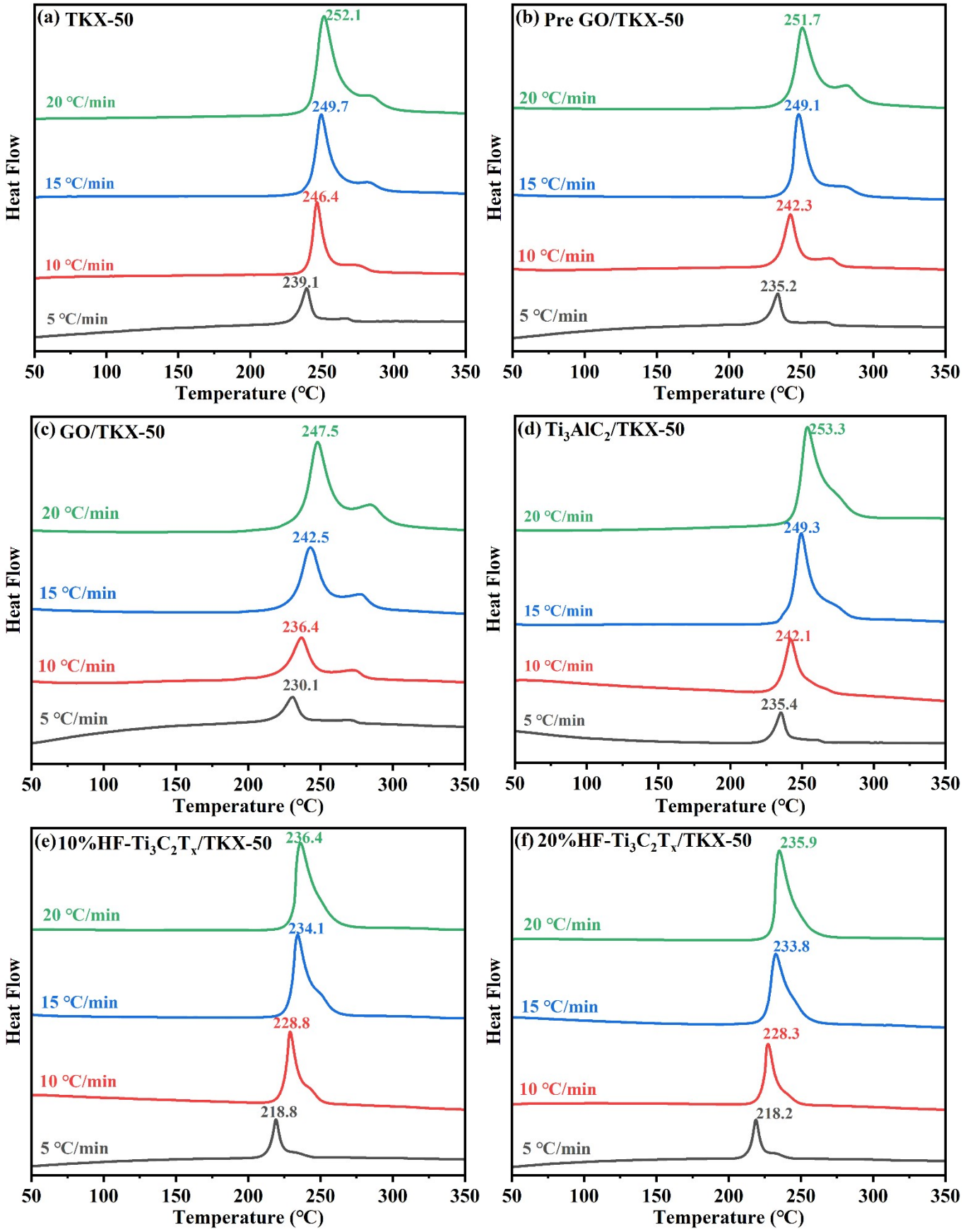
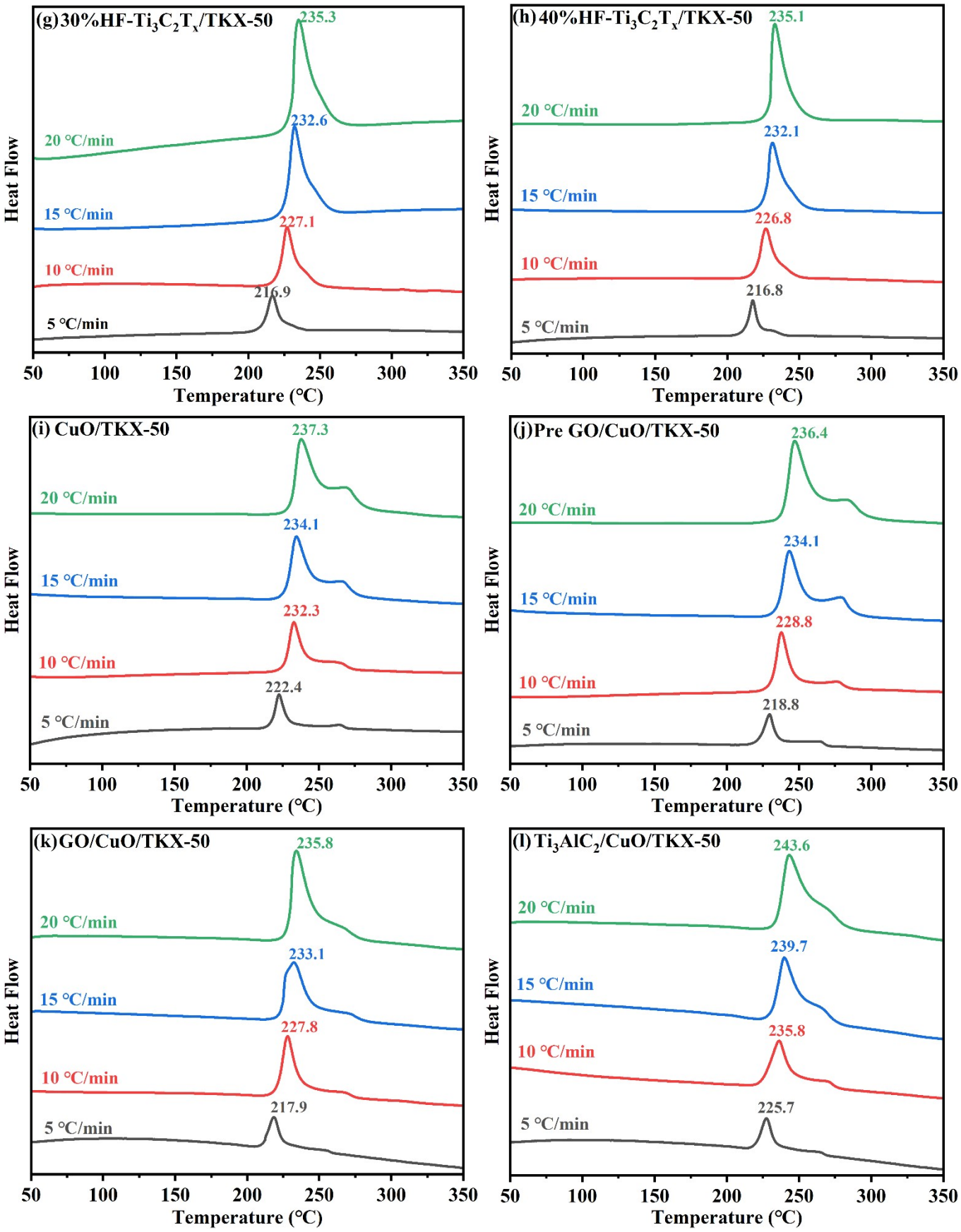



**Fig. S7.** 10°C/min hating rate (a) TG curves of 10%HF-$Ti_3C_2T_x$, 20%HF-$Ti_3C_2T_x$, 30%HF-$Ti_3C_2T_x$, 40%HF-$Ti_3C_2T_x$. (b) DSC curves of 10%HF-$Ti_3C_2T_x$, 20%HF-$Ti_3C_2T_x$, 30%HF-$Ti_3C_2T_x$, 40%HF-$Ti_3C_2T_x$.

## Catalysis mechanism




(g) 30%HF-Ti3C2Tx/TKX-50
235.3
232.6
227.1
216.9
20 °C/min
15 °C/min
10 °C/min
5 °C/min
Heat Flow
50 100 150 200 250 300 350
Temperature (°C)
(h) 40%HF-Ti3C2Tx/TKX-50
235.1
232.1
226.8
216.8
20 °C/min
15 °C/min
10 °C/min
5 °C/min
Heat Flow
50 100 150 200 250 300 350
Temperature (°C)
(i) CuO/TKX-50
237.3
234.1
232.3
222.4
20 °C/min
15 °C/min
10 °C/min
5 °C/min
Heat Flow
50 100 150 200 250 300 350
Temperature (°C)
(j) Pre GO/CuO/TKX-50
236.4
234.1
228.8
218.8
20 °C/min
15 °C/min
10 °C/min
5 °C/min
Heat Flow
50 100 150 200 250 300 350
Temperature (°C)
(k) GO/CuO/TKX-50
235.8
233.1
227.8
217.9
20 °C/min
15 °C/min
10 °C/min
5 °C/min
Heat Flow
50 100 150 200 250 300 350
Temperature (°C)
(l) Ti3AlC2/CuO/TKX-50
243.6
239.7
235.8
225.7
20 °C/min
15 °C/min
10 °C/min
5 °C/min
Heat Flow
50 100 150 200 250 300 350
Temperature (°C)

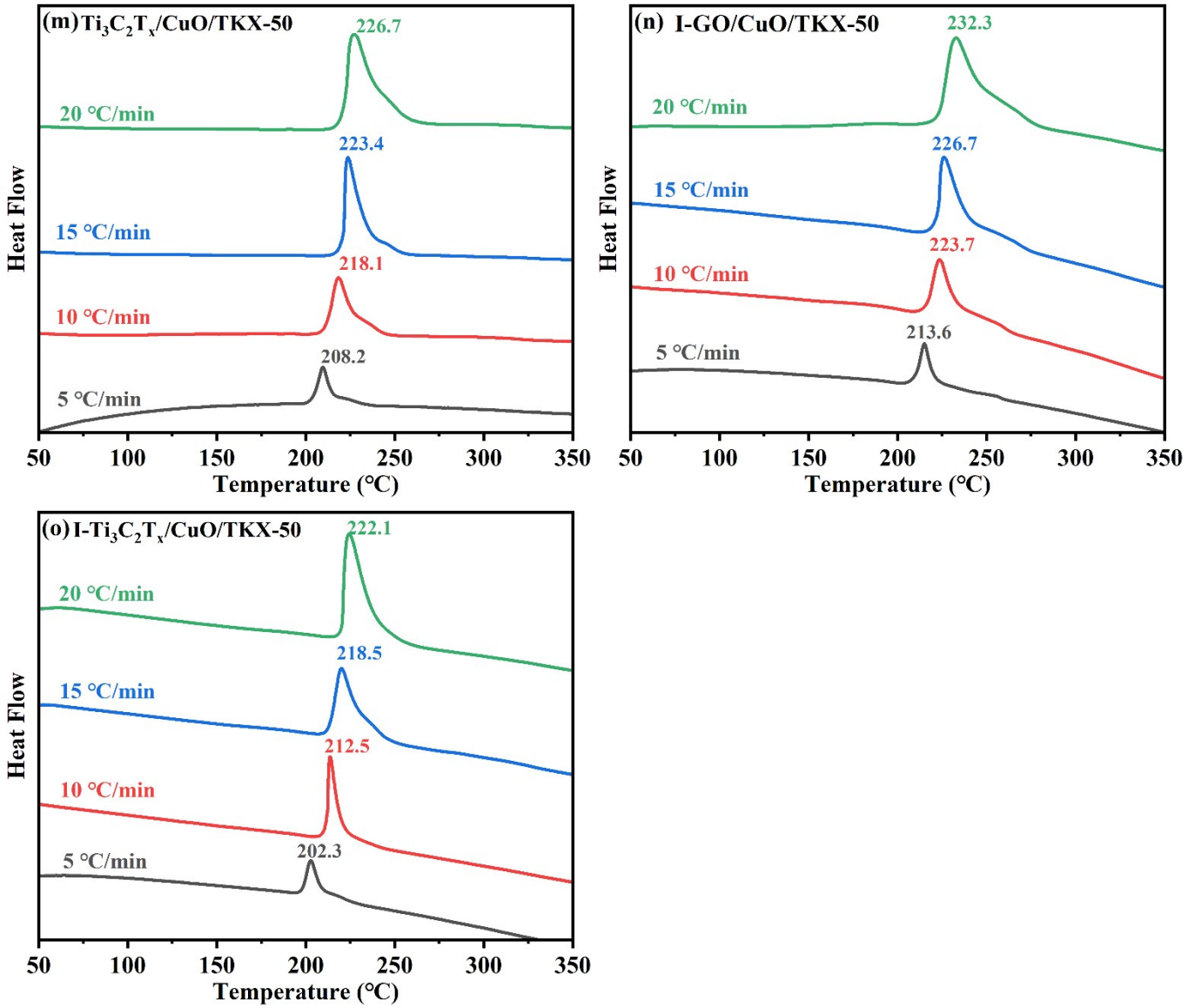



**Fig. S8** DSC curves of (a) TKX-50, (b) Pre GO/TKX-50, (c) GO/TKX-50, (d) $Ti_3AlC_2$/TKX-50, (e) 10%HF-$Ti_3C_2T_x$/TKX-50, (f) 20%HF-$Ti_3C_2T_x$/TKX-50, (g) 30%HF-$Ti_3C_2T_x$/TKX-50, (h) 40%HF-$Ti_3C_2T_x$/TKX-50. (i) CuO/TKX-50 (j)Pre GO/CuO/TKX-50 (k) GO/CuO/TKX-50 (l) $Ti_3AlC_2$/CuO/TKX-50 (m) $Ti_3C_2T_x$/ CuO/TKX-50 (n) I-GO/CuO/TKX-50 (o) I-$Ti_3C_2T_x$/ CuO/TKX-50

Hating rate K/min=℃/min, $T_p$(K)=273.15+$T_p$(℃)

**Table S1.** Thermal decomposition temperature established using DSC and Kissinger $E_a$ calculations of energetic compounds with the MXene catalysts

| Sample | $\beta$ (K/min) | $T_p$ (K) | 1000/$T_p$ | ln($\beta/T_p^2$) | $E_a$ (kJ/mol) | $R^2$ |
|---|---|---|---|---|---|---|
| TKX-50 | 5 | 512.25 | 1.9521717 | -10.8681876 | 228.3 | 0.9913 |
| | 10 | 519.55 | 1.9247425 | -10.2033410 | | |
| | 15 | 522.85 | 1.9125944 | -9.81053903 | | |
| | 20 | 525.25 | 1.9038553 | -9.53201640 | | |
| Pre GO/TKX-50 | 5 | 508.35 | 1.9671486 | -10.8529024 | 170.6 | 0.9828 |
| | 10 | 515.45 | 1.9400523 | -10.1874955 | | |
| | 15 | 522.25 | 1.9147917 | -9.8082426 | | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | 20 | 524.85 | 1.9053062 | -9.5304927 | | |
| GO/TKX-50 | 5 | 503.25 | 1.9870839 | -10.8327362 | 163.4 | 0.9751 |
| | 10 | 509.55 | 1.9625159 | -10.1644708 | | |
| | 15 | 515.65 | 1.9392999 | -9.7828062 | | |
| | 20 | 520.65 | 1.9206760 | -9.5144237 | | |
| $Ti_3AlC_2$/TKX-50 | 5 | 508.55 | 1.9663749 | -10.8536891 | 160.1 | 0.9722 |
| | 10 | 515.25 | 1.9408054 | -10.1867193 | | |
| | 15 | 522.45 | 1.9140587 | -9.8090083 | | |
| | 20 | 526.45 | 1.8995156 | -9.53658044 | | |
| 10%HF-$Ti_3C_2T_x$/TKX-50 | 5 | 491.95 | 2.0327269 | -10.78731626 | 150.8 | 0.9833 |
| | 10 | 501.95 | 1.9922303 | -10.13441593 | | |
| | 15 | 507.25 | 1.9714144 | -9.749957756 | | |
| | 20 | 509.55 | 1.9625159 | -9.471323693 | | |
| 20%HF-$Ti_3C_2T_x$/TKX-50 | 5 | 491.35 | 2.0352091 | -10.7848755 | 148.6 | 0.9803 |
| | 10 | 501.45 | 1.9942167 | -10.13242271 | | |
| | 15 | 506.95 | 1.9725811 | -9.7487745 | | |
| | 20 | 509.05 | 1.9644435 | -9.4693602 | | |
| 30%HF-$Ti_3C_2T_x$/TKX-50 | 5 | 490.05 | 2.0406081 | -10.7795769 | 141.1 | 0.9887 |
| | 10 | 500.25 | 1.9990005 | -10.1276308 | | |
| | 15 | 505.75 | 1.9772614 | -9.7440347 | | |
| | 20 | 508.45 | 1.9667617 | -9.4670014 | | |
| 40%HF-$Ti_3C_2T_x$/TKX-50 | 5 | 489.95 | 2.0410245 | -10.7791687 | 146.1 | 0.9924 |
| | 10 | 499.95 | 2.0002000 | -10.1264310 | | |
| | 15 | 505.25 | 1.9792182 | -9.7420565 | | |
| | 20 | 508.25 | 1.9675356 | -9.4662146 | | |
| CuO/TKX-50 | 5 | 495.55 | 2.0179598 | -10.8018986 | 169.6 | 0.9913 |
| | 10 | 505.45 | 1.9784350 | -10.1483131 | | |
| | 15 | 508.45 | 1.9667617 | -9.7546835 | | |
| | 20 | 511.85 | 1.9536973 | -9.4803309 | | |
| Pre GO/CuO/TKX-50 | 5 | 491.95 | 2.0327269 | -10.7873162 | 150.7 | 0.9833 |
| | 10 | 501.95 | 1.9922303 | -10.1344159 | | |
| | 15 | 507.25 | 1.9714144 | -9.7499577 | | |
| | 20 | 509.55 | 1.9625159 | -9.4713236 | | |
| GO/CuO/TKX-50 | 5 | 491.05 | 2.0364525 | -10.7836544 | 149.8 | 0.9896 |
| | 10 | 500.95 | 1.9962072 | -10.1304275 | | |
| | 15 | 505.25 | 1.9753086 | -9.7460110 | | |
| | 20 | 507.95 | 1.9648295 | -9.4689672 | | |
| $Ti_3AlC_2$/CuO/TKX-50 | 5 | 498.85 | 2.0046106 | -10.8151729 | 158.1 | 0.9878 |
| | 10 | 508.95 | 1.9648295 | -10.1621144 | | |
| | 15 | 512.85 | 1.9498878 | -9.7719166 | | |
| | 20 | 516.75 | 1.9351717 | -9.4993861 | | |
| $Ti_3C_2T_x$/CuO/TKX-50 | 5 | 481.35 | 2.0774903 | -10.7437514 | 141.7 | 0.9929 |
| | 10 | 491.25 | 2.0356234 | -10.0913212 | | |
| | 15 | 496.55 | 2.0138958 | -9.7073181 | | |
| | 20 | 499.55 | 2.0018016 | -9.4316831 | | |
| I-GO/CuO/TKX-50 | 5 | 486.75 | 2.0544427 | -10.7660633 | 146.9 | 0.9736 |
| | 10 | 496.85 | 2.0126798 | -10.1139912 | | |
| | 15 | 499.85 | 2.0006001 | -9.7205659 | | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | 20 | 505.45 | 1.9784350 | -9.4551659 | | |
| I-$Ti_3C_2T_x$/CuO/TKX-50 | 5 | 475.45 | 2.103270586 | -10.71908554 | 127.6 | 0.9978 |
| | 10 | 485.65 | 2.059096057 | -10.06839131 | | |
| | 15 | 491.65 | 2.033967253 | -9.687483961 | | |
| | 20 | 495.25 | 2.019182231 | -9.414393098 | | |

1. Cao, Y.; Deng, Q.; Liu, Z.; Shen, D.; Wang, T.; Huang, Q.; Du, S.; Jiang, N.; Lin, C.-T.; Yu, J., Enhanced thermal properties of poly(vinylidene fluoride) composites with ultrathin nanosheets of MXene. *RSC Advances* **2017,** *7* (33), 20494-20501.