Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information for

Regulating TiO_2/MXenes Catalysts to Promote Photocatalytic Performance of Highly Selective Oxidation of D-Xylose

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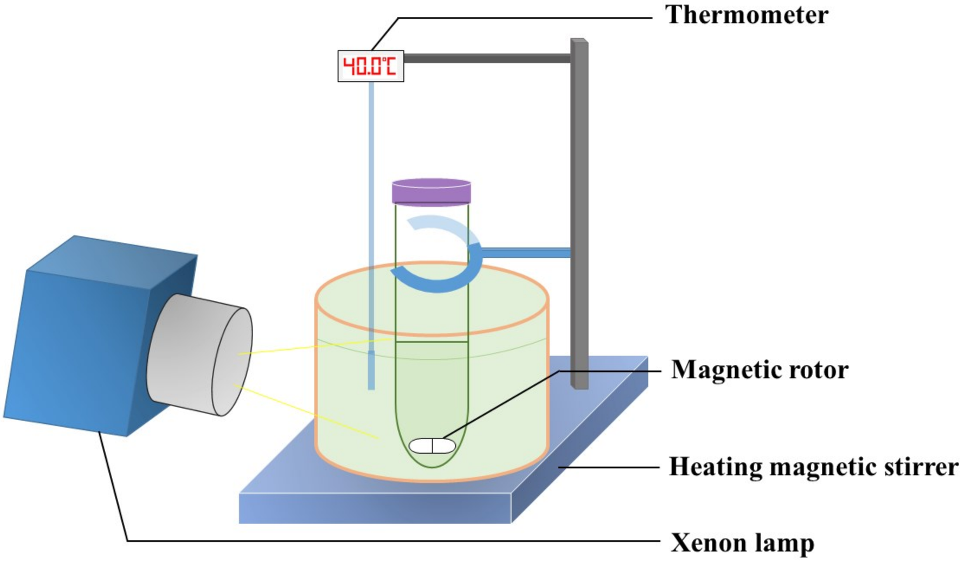
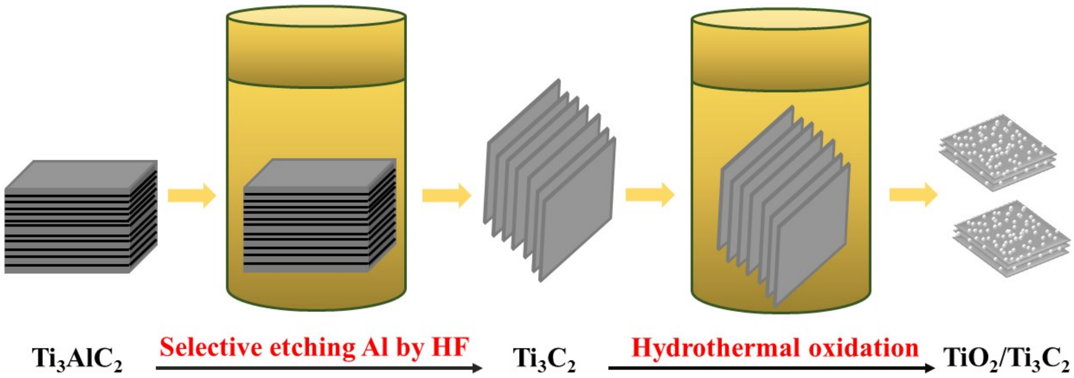
Reaction device diagram



Schematic diagram of catalyst preparation process



Cycle Experiment

The photocatalyst TT-12 after the reaction was recycled by washing, filtration, drying, grind. The recycled TT-12 was then used to repeat the previous reaction under the optimal conditions. The above operations were repeated four times to test the yield of xylonic acid and the conversion of xylose.

Product detection

All the samples were immediately syringed out, filtered and analyzed by high-performance liquid chromatography (HPLC, Agilent 1260 series) with a UV detector at 210 nm and a Bio-Rad Aminex HPX-87H column (300 mm × 7.8 mm × 9 μm). 5 mM H_2SO_4 was used as the mobile phase at 55 °C with a flow rate of 0.6 mL min^{-1}. Xylose was quantified using a High Performance Ion Chromatography (HPIC, Dionex ICS-3000) system with an integrated amperometric detector and a CarboPac PA1 column at 30 ^{o}C using pure water as eluent. The concentrations of the products were determined by comparing the calibration curve established with the external standards. The conversion of xylose and the yields of the products ($_{D}$-xylonic acid, lactic acid, acetic acid and formic acid) were calculated by the following equations:

$$Conversion\ (\%) = \frac{Moles\ of\ carbon\ in\ feedstock\ consumed}{Moles\ of\ carbon\ in\ feedstock\ input} \times 100\%$$

$$Product\ yield\ (\%) = \frac{Moles\ of\ carbon\ in\ organic\ acid}{Moles\ of\ carbon\ in\ feedstock\ input} \times 100\%$$

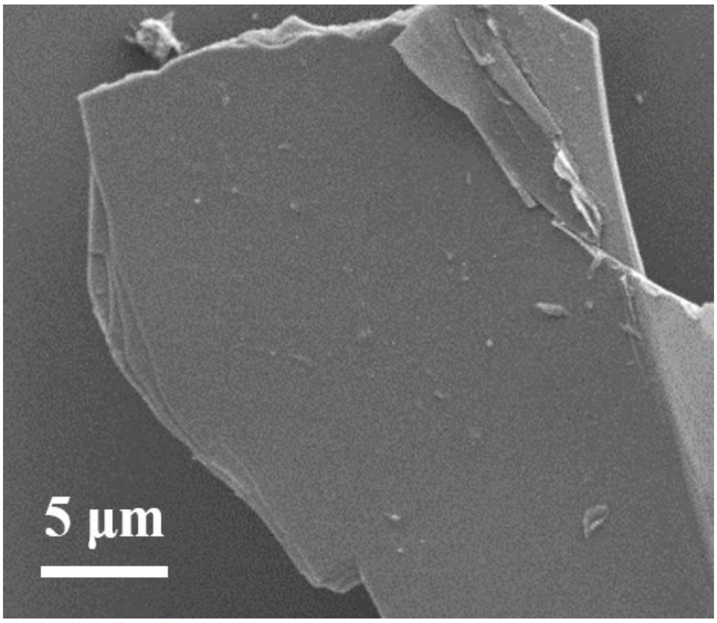


Fig. S1 The SEM image of Ti_3C_2

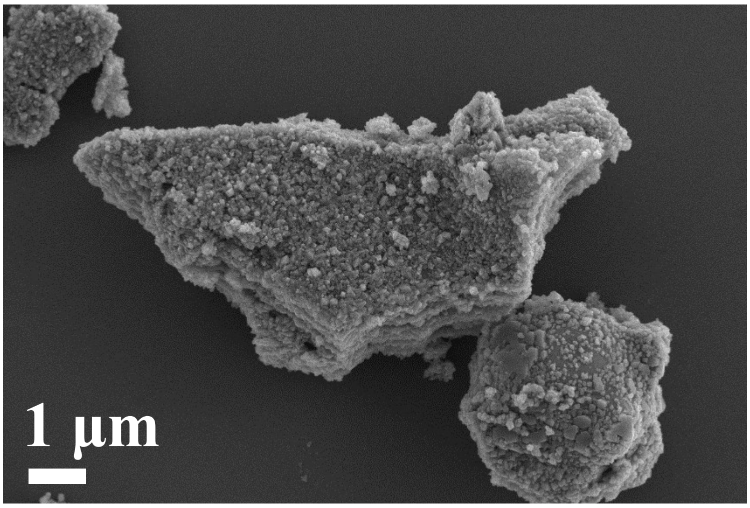


Fig. S2 The SEM image of TiO_2/Ti_3C_2

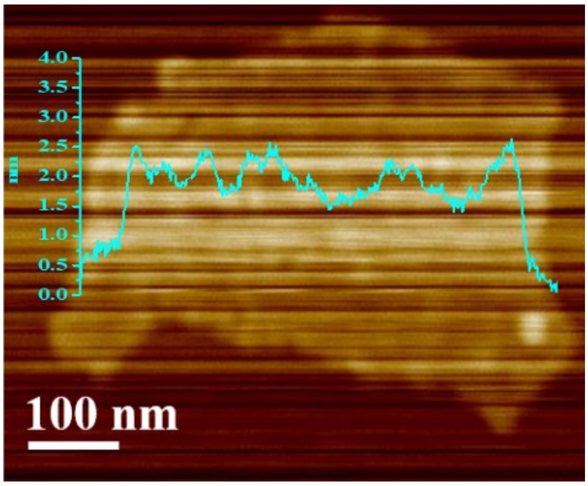


Fig. S3 AFM image of composite photocatalysts TiO_2/Ti_3C_2

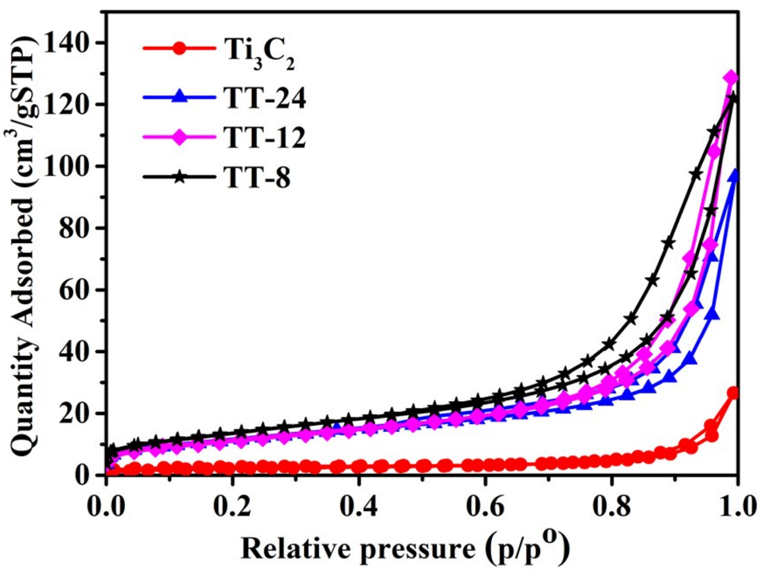


Fig. S4 N_2 adsorption-desorption isotherms of Ti_3C_2, TT-8, TT-12 and TT-24

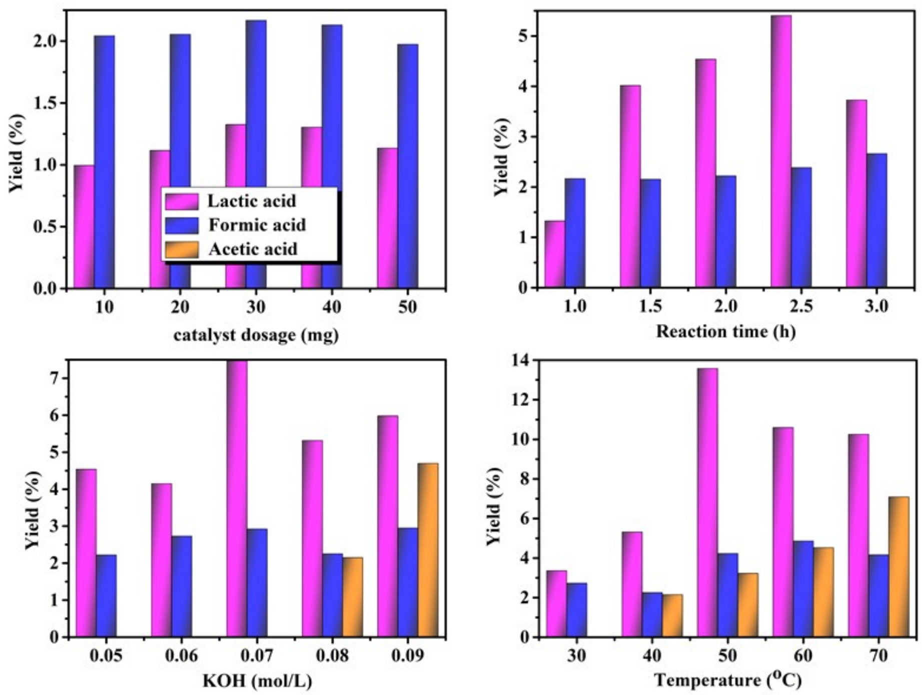


Fig. S5 The yield of byproducts under different reaction conditions

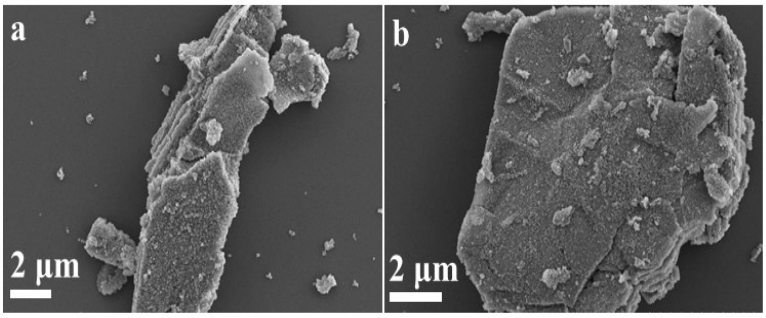


Fig. S6 The SEM images of recycled TT-12

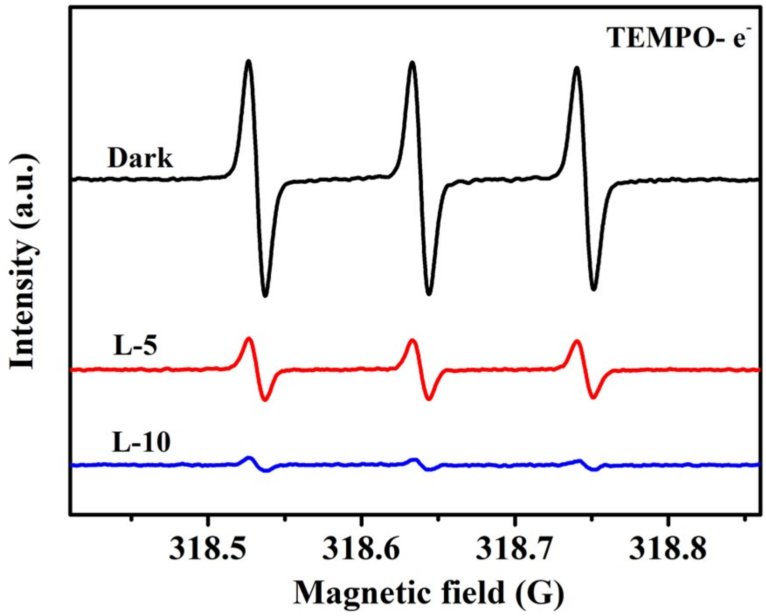


Fig. S7 TEMPO ESR spin-labelling for e^-

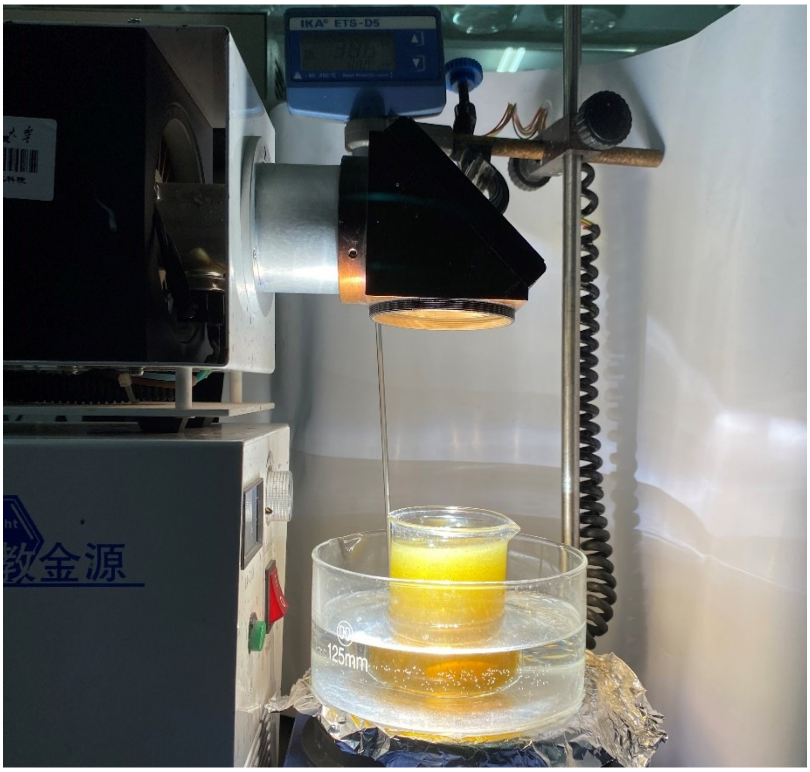


Fig. S8 A hundred-fold scale-up experiment

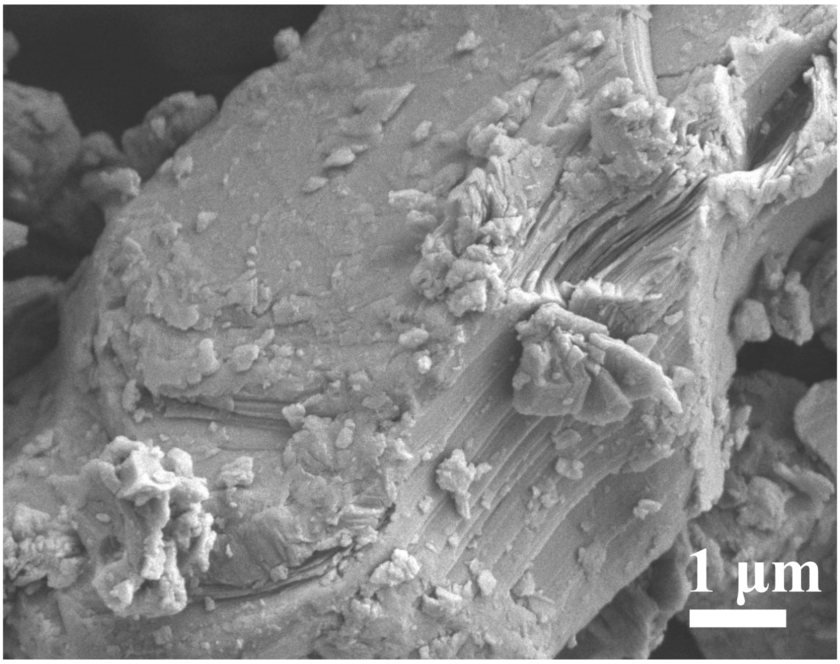


Fig. S9 The SEM image of Ti_3AlC_2

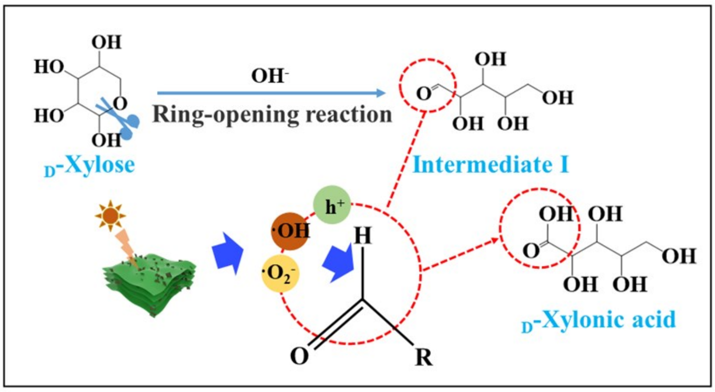


Fig. S10 Schematic diagram of primary reaction mechanism

Table S1 BET Surface Area of different samples.

Sample Number	Sample Name	BET Surface Area (m^2/g)
1	Ti_3C_2	8.7370
2	TT-8	50.3050
3	TT-12	47.5700
4	TT-24	42.0429

Table S2 Photocatalytic properties of different samples.

Sample Number	Sample Name	Xylonic acid Yield
1	Ti_3C_2	trace
2	TiO_2	13.2%
3	TT-8	33.3%
4	TT-12	35.1%
5	TT-24	29.7%

Notes: Reaction conditions: sample, 10 mg; xylose, 100mg; 0.05 mol/L KOH, 10 mL; reaction temperature, 40 ^{o}C; reaction time, 1 h; light source, xenon lamp.

Table S3 Elemental content of different samples measured by EPMA (Electron Probe Micro-analyzer).

Samples	Element (at%)				
	Ti	C	O	F	Al
Ti_3C_2	47.61	29.63	13.98	8.15	0.64
TT-8	43.33	29.54	24.53	2.26	0.34
TT-12	43.12	25.46	29.69	2.23	0.51
TT-24	42.55	20.88	34.74	1.40	0.43

Table S4 Elemental content of different samples measured by EPMA.

Samples	Element (wt%)				
	Ti	C	O	F	Al
Ti_3C_2	75.22	11.74	7.38	5.10	0.57
TT-8	72.19	12.34	13.65	1.49	0.32
TT-12	71.28	9.42	17.47	1.38	0.45
TT-24	71.09	7.44	20.14	0.93	0.40

Table S5 The mole fraction of each compound (TiO_2, Ti_3C_2O and C).

Samples	Component content (%)		
	TiO_2	Ti_3C_2O	C
Ti_3C_2	-	100	-
TT-8	26.09	53.91	20.00
TT-12	39.37	48.46	12.17
TT-24	52.79	40.74	6.47

The content of TiO_2 in samples with different hydrothermal time are determined by EPMA analysis, as shown in Table S2, S3 and S4. The Ti: C: O atomic ratio of the Ti_3C_2 are 47.61: 29.63: 13.98. Hence, The O content in Mxenes is determined, and the chemical formula is Ti_3C_2O. During the hydrothermal process, the Ti_3C_2O is consumed and transformed to TiO2 and C. Based on the balances of Ti, O and C atoms, the contents of Ti_3C_2O, TiO_2 and C can be calculated according to EPMA results (Table S4).

Table S6 Photocatalytic properties of different experiments.

Number	Xylonic acid Yield
1	trace
2	trace
3	trace
4	55.43%

Notes: 1: No catalyst in the best reaction condition; 2: No light in the best reaction condition; 3: No O_2 in the best reaction condition; 4: A hundred-fold scale-up experiment in the best reaction condition.