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# Photocatalytic $CO_2$ reduction over MXene/$TiO_2$ under visible light: process conditions and stability aspects

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## Synthesis

The synthesis of $Ti_3C_2$ MXenes and 5 wt% $Ti_3C_2$(F)/$TiO_2$ has been recently described in detail.[S1]

### $Ti_3C_2X$ Mxene ink preparation

The $Ti_3C_2$ Mxene ink was prepared from the $Ti_3AlC_2$ MAX phase by the etching with LiF/HCl mixture. Specifically, 2.00 g of LiF were dissolved in 40 ml of 7.0 M hydrochloric acid and 2.00 g of $Ti_3AlC_2$ powder was added in several portions with a constant stirring. The resulting mixture was heated at 45 °C for 48 h under constant stirring. The resulting suspension was centrifuged and the obtained precipitate was washed several times by centrifugation with water (400 ml totally) to bring the pH value of the supernatant to about 6–7. The washed precipitate was redispersed in 50 ml of water and resulting suspension was ultrasonicated for 1 h in an ice bath. The as-obtained suspension was centrifuged at 4000 rpm for 5 min and the supernatant was collected and designated as $Ti_3C_2$ ink.

### 5 wt% $Ti_3C_2$(F)/$TiO_2$ sample preparation

425 mg of $TiO_2$ (Evonik P25) was suspended in 7 ml of water and under constant stirring 6.10 ml of the $Ti_3C_2$ ink was added. The resulting suspension was stirred for 24 h at room temperature and then the solid material was separated by filtration on 0.22 μm PTFE porous membrane. The collected solid material was washed with water, acetone and dried in an air stream. Finally, the prepared material was dried at 60 °C in a vacuum and designated as 5 wt% $Ti_3C_2$(F)/$TiO_2$.

## Characterization

The prepared photocatalyst **5 wt% $Ti_3C_2$(F)/$TiO_2$** underwent characterization using high-resolution transmission electron microscopy (HR TEM) and X-ray photoelectron spectroscopy (XPS). HR TEM images were obtained for the samples **5 wt% $Ti_3C_2$(F)/$TiO_2$** before and after photocatalytic reaction at room temperature and 50 °C. XPS analysis was performed for the samples **5 wt% $Ti_3C_2$(F)/$TiO_2$** after photocatalytic reaction at room temperature and 50 °C; XPS analysis of fresh sample **5** wt% $Ti_3C_2$(F)/$TiO_2$ was described recently.[S1]

**XPS** was performed using an X-ray photoelectron spectrometer (SPECS Surface Nano Analysis GmbH, Germany) equipped with an XR-50 X-ray source with a dual Al/Mg anode and a PHOIBOS-150 hemispherical electron energy analyzer. Core-level spectra were recorded with Al Kα radiation ($h\nu$ = 1486.6 eV). The binding energy calibration was performed by setting C1*s* peak corresponded to titanium carbide at 281.9 eV. Curve fitting was performed with CasaXPS software.[S2]

**HR TEM** images were obtained with a Themis electron microscope (Thermo Fisher Scientific, USA), which operates at a 200 kV accelerating voltage. The microscope boasts a spherical aberration corrector affording a maximum lattice resolution of 0.06 nm and a SuperX energy-dispersive spectrometer (Thermo Fisher Scientific). Ceta 16 CCD sensor (Thermo Fisher Scientific) was employed to capture images.

**Table S1** Properties of synthesized 5 wt% $Ti_3C_2$(F)/$TiO_2$ sample.[S1]

| Sample | Phase composition | $E_g$/eV | $S_{BET}$/m$^2$ g$^{-1}$ | $V$/cm$^3$ g$^{-1}$ |
|---|---|---|---|---|
| 5 wt% $Ti_3C_2$(F)/$TiO_2$ | Anatase<br>Rutile<br>$Ti_3C_2$ MXene | 2.87 | 47 | 0.42 |

**Photocatalytic tests**

Photocatalytic tests were carried out in two types of the photocatalytic reactors. Ultrapure water ('NuZar Q' water system set) was used for photocatalyst deposition on the glass support and to generate saturated vapor pressure in the reactor in both cases. The gas samples were analyzed by means of gas chromatography over the flame ionization detector and thermal conductivity detector to identify the products of $CO_2$ reduction and $H_2$. Experiments with variable reaction temperature were carried out in the 140 ml batch reactor.[S3] Experiments were carried out at room temperature, as well as at temperatures of 35, 50 and 70 °C; each experiment lasted 4 h. The LED with maximum intensity at 405 nm (Figure S1) and power density of 60 mW $cm^{-2}$ was used as the light source. Cyclic experiments for $CO_2$ reduction were carried out in the 170 ml batch reactor.[S1] Four photocatalytic cycles of 5 hours each were carried out at a room temperature. The LED with maximum intensity at 397 nm (Figure S1) and power density of 60 mW $cm^{-2}$ was used as the light source.

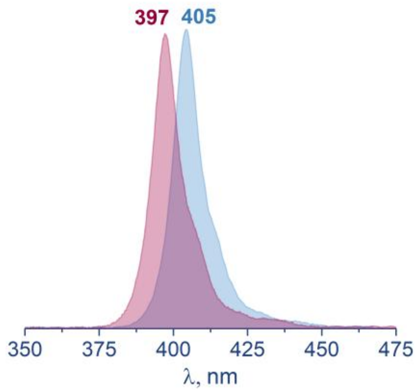


**Figure S1 Emission spectra of 397 nm and 405 nm LEDs.**

The total rate of photocatalytic $CO_2$ reduction (Table 1) was calculated according to the equation:

$$CO_2 \text{ RR } [\mu mol\ g^{-1}\ h^{-1}] = [(8n(CH_4) + 2n(CO)]/t\ m \qquad (I)$$

where $n(CH_4)$ and $n(CO)$ are the amounts of $CH_4$ and CO (µmol), 2 and 8 are the coefficients for electron balance, $t$ is the time of reaction (h), and $m$ is the weight of photocatalyst (g).

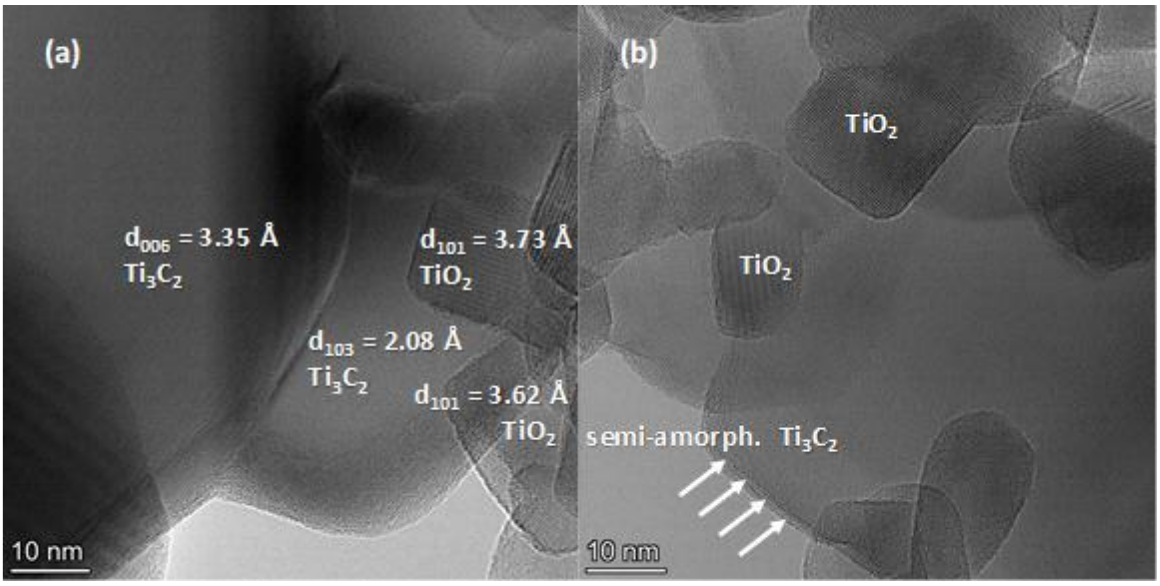


**Figure S2 HRTEM images of the samples 5 wt% $Ti_3C_2(F)/TiO_2$ after photocatalytic reaction at 50 °C and room temperature.**

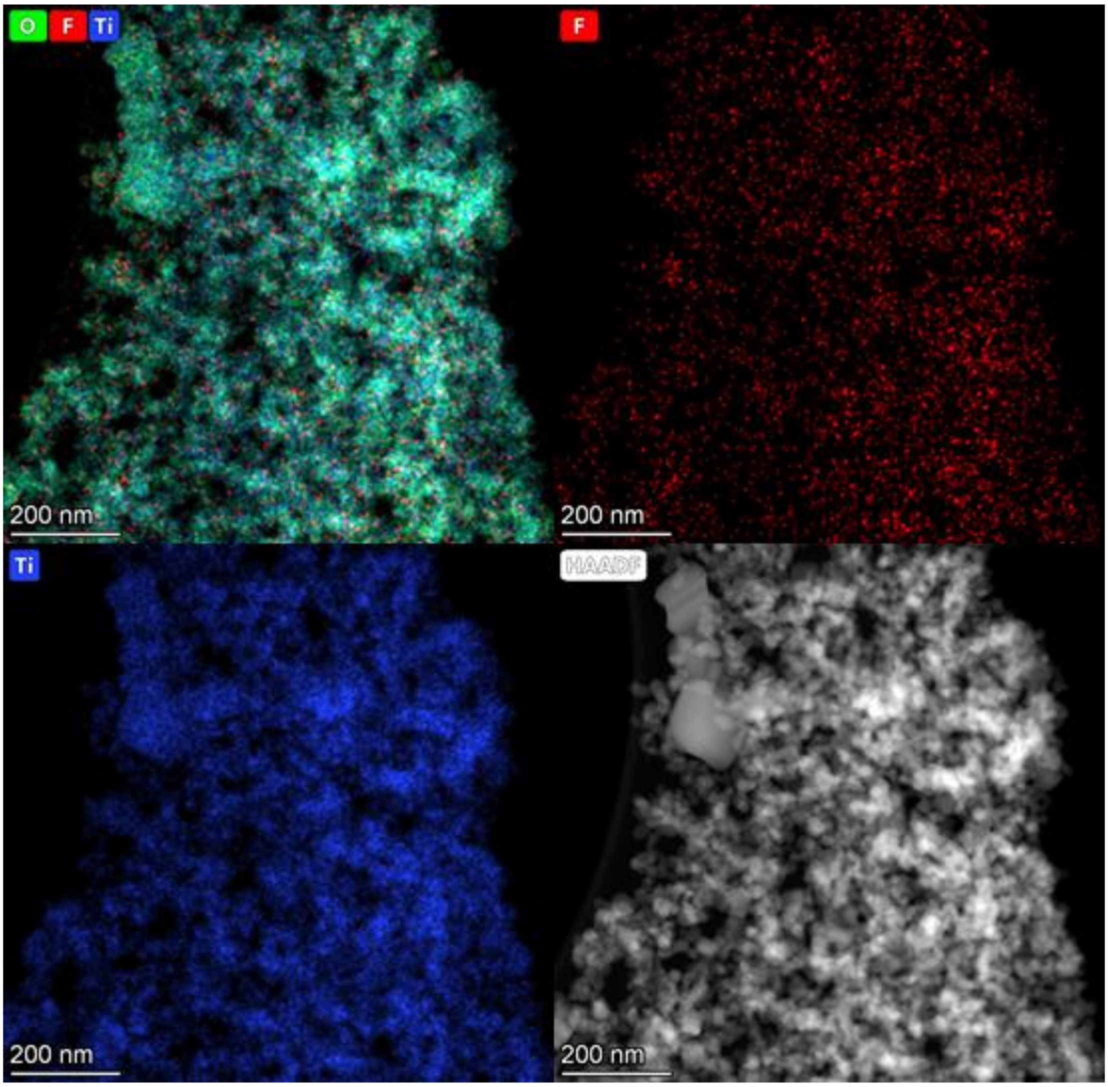


**Figure S3 Element mapping and HAADF-STEM images of the sample 5 wt% $Ti_3C_2$(F)/$TiO_2$ after photocatalytic reaction at a room temperature.**

Analysis of the HR TEM images of was carried out using JCPDS No. 21-1272 for $TiO_2$ and data[S4] for $Ti_3C_2$, respectively.

## References


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