## SUPPORTING INFORMATION

## Aerosol-Spraying Synthesis of SiO<sub>2</sub>/TiO<sub>2</sub> Nanocomposites and Their Alternative Conversion to Porous TiO<sub>2</sub> and Single-Crystal TiOF<sub>2</sub>

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Summary: 11 Pages; 9 Figures

## Experimental procedures of photocatalysis

The photocatalytic reaction was carried out at 30 °C in a 100 mL quartz reactor containing 0.050 g catalyst and 50 mL of  $5.0 \times 10^{-5}$  M 4-chlorophenol aqueous solution at pH = 6~7. The reaction system was stirred vigorously (800 rpm) to eliminate diffusion effect on the reaction kinetics. The mixture was allowed to stir for enough time (>1 h) for reaching adsorption equilibrium. Then, the photocatalytic reaction was initiated by irradiating with three 500 W Xenon lamp (CHF-XM500, light intensity =  $600 \text{ mW/cm}^2$  located at 10 cm away from the solution. All the UV lights with wavelength lower than 420 nm were removed by a glass filter (JB-420). Each reaction was lasted for 4 h and the 4-chlorophenol concentration was analyzed by UV spectrophotometer (XINMAO UV-7504PC) at its characteristic wavelength ( $\lambda = 224$  nm). Besides CO<sub>2</sub>, no other organic products were identified by HPLC-MS, indicating the complete decomposition of 4-chlorophenol under the present conditions. Preliminary tests showed that there was a good linear relationship between the light absorbance and the concentration of organic compounds. Meanwhile, experimental results also confirmed that only less than 1.0 % 4-chlorophenol was decomposed after reaction for 6 h in the absence of photocatalyst (see Fig. S8) and thus, could be neglected in comparison with the degradation yield resulting from real photocatalysis. The reproducibility of the results was checked by repeating the experiments at least three times and was found to be within acceptable limits ( $\pm 5\%$ ).

In order to determine catalyst durability, the photocatalyst was allowed to settle down after each reaction and the photocatalyst was separated from aqueous solution by centrifugation, washed with  $H_2O$  for 3 times, followed by drying at 100 °C for 12 h under vacuum. Then, the subsequent reaction was conducted under the identical conditions for 6 h and the 4-chlorophenol degradation yield was determined to show the change in activity.



Fig. S1 SEM and TEM (inset) images of the  $TiO_2$  particles obtained via aerosol-spray before (a) and after (b) being calcined at 450 °C for 2 h.



Fig. S2 FTIR spectra of (a) the undoped  $TiO_2$  and (b) the  $TiO_2/SiO_2$  samples after being calcined at 450 °C for 2 h.



**Fig. S3** N<sub>2</sub> adsorption-desorption isotherms of the  $TiO_2/SiO_2$  sample calcined at 450 °C for 2 h and the  $TiO_2$  obtained by exposing the  $TiO_2/SiO_2$  in NaOH solution.

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Fig. S4 EXD patterns of (a) the  $TiO_2/SiO_2$  calcined at 450 °C and (b) the  $TiOF_2$  samples.



Fig. S5 XPS spectra of (a)  $TiOF_2$  and (b) F-doped  $TiO_2$  samples.



**Fig. S6** The EDX pattern (a) and HRTEM image (b) of the  $TiOF_2$  sample obtained by HF etching  $TiO_2/SiO_2$  composite particles without calcinations. The inset is the SAED pattern.



**Fig. S7** XRD patterns of the  $TiO_2/SiO_2$  sample after being etched for 40 h with (a) 40 ml, (b) 50 ml, and (c) 100 ml HF solution with volume ratio of HF (aq. 40%) : ethanol : water = 1 : 1 : 2.



**Fig. S8** Photocatalytic degradation of 4-chlorophenol under UV-light irradiation. Reaction conditions: 0.050 g catalyst, 100.0 mL  $1.0 \times 10^{-4}$  M 4-chlorophenol, three 8 W UV lamp (wavelength = 365 nm), reaction temperature = 30 °C, stirring rate = 800 rpm.



Fig. S9 SEM image of the single crystal  $TiOF_2$  after being used repetitively for 8 times in photocatalysis.