Electronic Supplementary Information

TiO₂ mesocrystals built of nanocrystals with exposed {001} facets: Facile synthesis and superior photocatalytic ability

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Experiment Section:

Preparation of NH_4TiOF_3 **:** Typical reaction conditions for the preparation of NH₄TiOF₃ precursors are as follows: 2 cm³ of NH₄OH aqueous solution with concentrations varying from 0.5 to 2.0 mol dm⁻³ are mixed with 8 cm³ of (NH₄)₂TiF₆ aqueous solution with concentrations varying from 0.125 to 0.5 mol dm⁻³. The concentration of NH₄TiOF₃ or NH₄OH in the final solution varied from 0.1 to 0.4 mol dm⁻³. The mixture solutions were kept at 25 °C for 16 h. Precipitates were collected, separated by centrifugation and washed (×3) sequentially with DI water and ethanol.

*Preparation of TiO*₂ *mesocrystals:* The NH₄TiOF₃ precursors are sintered at a high temperature between 700 °C and 1000 °C for 15 mins in air.

Characterization of materials: Scanning electron micrographs were obtained using a Philips XL30 or FEI Quanta 200 SEM. Transmission electron micrographs were obtained using a Philips CM 200 TEM. Powder X-ray diffraction studies were performed using a PANalytical X'Pert PRO diffractometer.

Photodegradation experiments: Photodegradation experiments were carried out at 25 °C in a 300ml glass container containing 100 mg catalyst and 50 cm³ of a methylene blue aqueous solution (10 mg dm⁻³). The suspension solution was stirred in the dark for 1 h first in order to reach adsorption equilibrium, and then exposed to 365 nm UV light (provided by a 6 W UV lamp) for 2 h.

Recycling use of mesocrystals: Recycling use was separately studied. All parameters are as same as the above general photodegradation experiments except changing the UV irradiation time to 40 mins. Each time the suspension solution was centrifuged. The TiO_2 residue was washed carefully using DI water and used again for the next photodegradation batch without further treatment. After three batches, the decoloring ratio of methylene blue are 90.1%, 93.4% and 93.3% respectively. There is not obvious decrease for the photocatalytic ability, which means a good stability.

Figures:

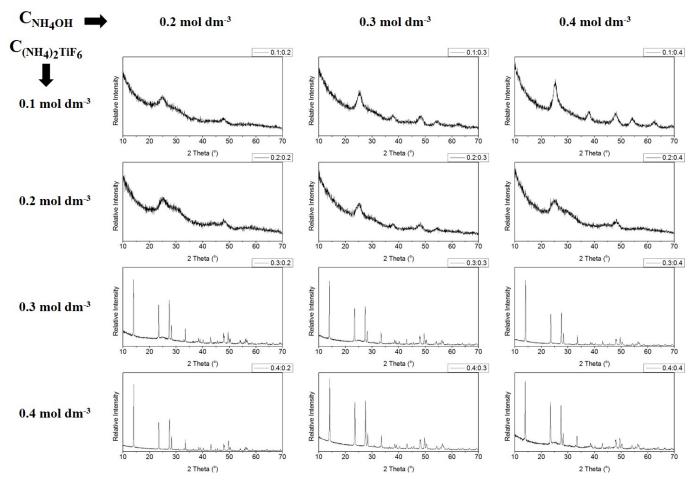


Figure S1. XRD patterns of the samples prepared in the solutions with different concentrations of $(NH_4)_2 TiF_6$ and $NH_4 OH$.

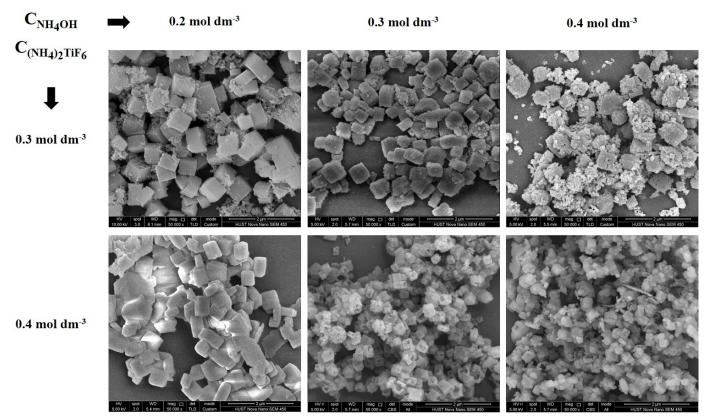


Figure S2. SEM images of the samples prepared in the solutions with different concentrations of (NH₄)₂TiF₆ and NH₄OH.

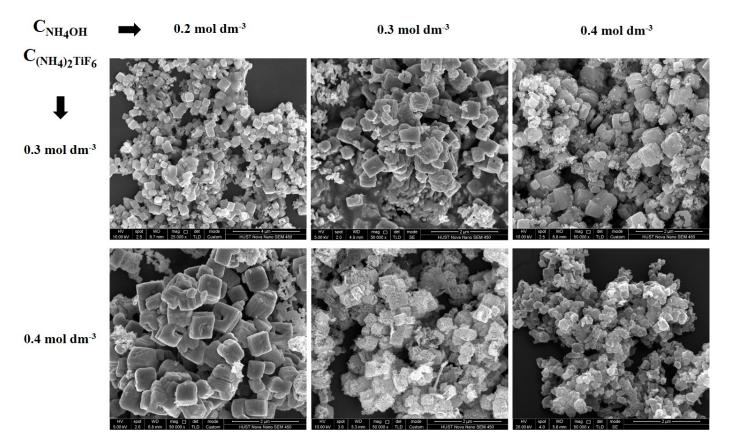


Figure S3. SEM images of the samples prepared by sintering the as-prepared samples.

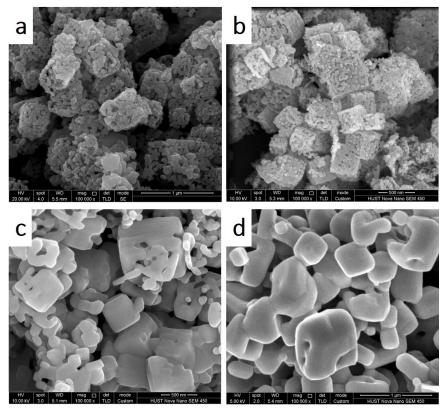


Figure S4. SEM images of the samples prepared by sintering the sample S_{43} at different temperatures. a) 700 °C, b) 800 °C, c) 900 °C and d) 1000 °C.

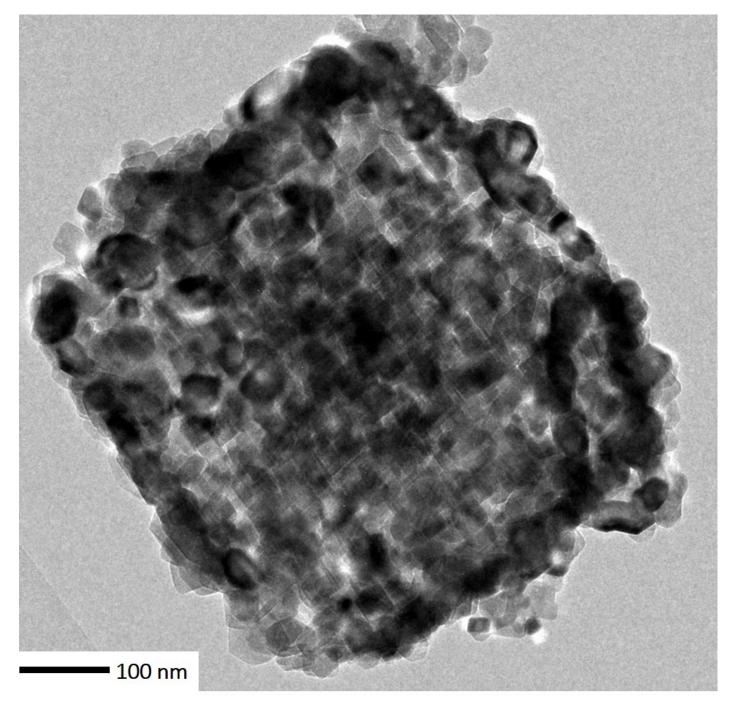


Figure S5. Magnified TEM image of Fig. 4a.

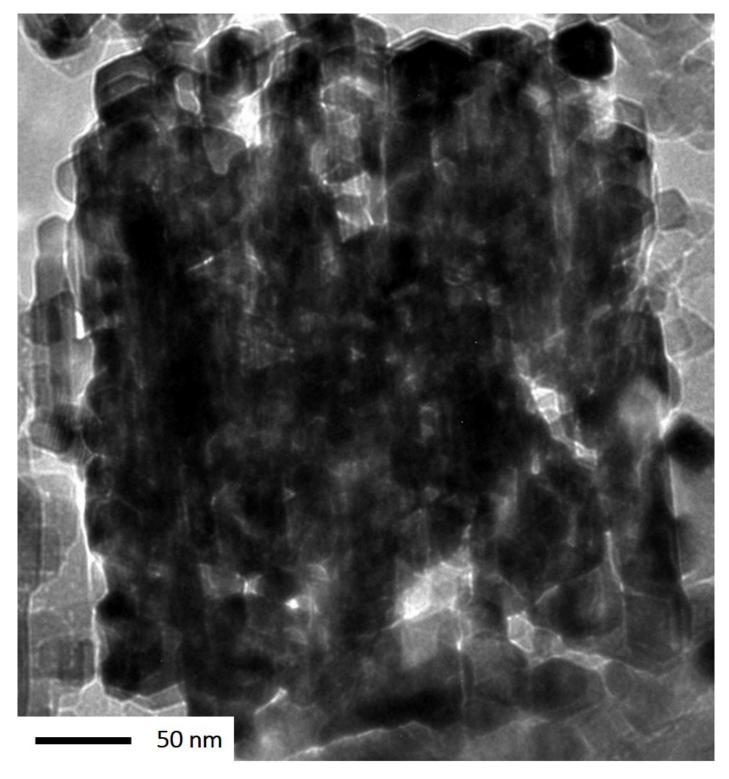


Figure S6. Magnified TEM image of Fig. 4d.

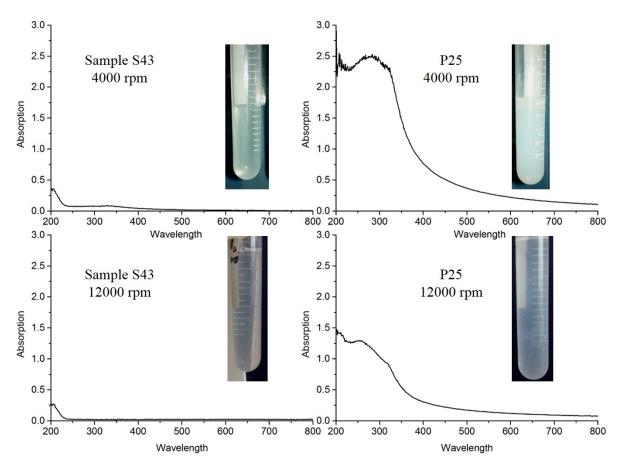


Figure S7. UV/Visible spectrums and photographs of the sample S_{43} and P25 after centrifugation at different rotation speed. *Experimental Details and discussion:* Two suspension solutions were prepared, which have the same amount of the sample S43 and P25. The suspension solutions were centrifugated at a speed of 4000 rpm / 12000 rpm. The UV spectrums of the supernate were examined. The results indicate even after centrifugation at a speed of 12000 rpm for 10 mins, P25 particles still obviously exist in the supernate (causing a broaden adsorption peak at 200~400 nm). For the sample S43, only 4000 rpm is enough to remove almost all the suspended particles.

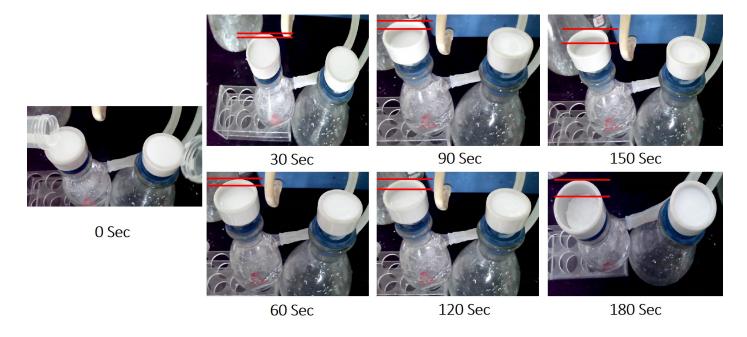


Figure S8. Screenshots at different time from a video showing the filtration process of the two suspension solutions. *Experimental Details and discussion:* The suspension solutions that contain the same concentration of the sample S43 and P25 were added into two funnels separately at the same time. The two funnels are under a same vacuum level. After 3 mins, the suspension containing the sample S43 completely passed through the funnel, however only a very small part of the P25 suspension passed through the funnel, suggesting that the sample S₄₃ is much easier to be removed from the reaction system.