Electronic Supplementary information

Highly crystalline anatase TiO_2 nanocuboids as an efficient photocatalyst for hydrogen generation

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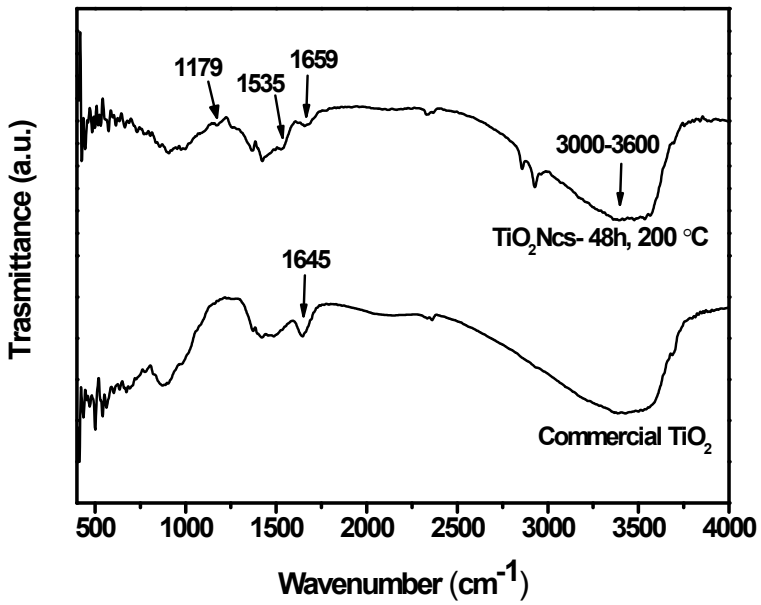

Figure S1. FTIR spectra of commercial TiO_2 and TiO_2 nanocuboids prepared at 200 °C for 48 h

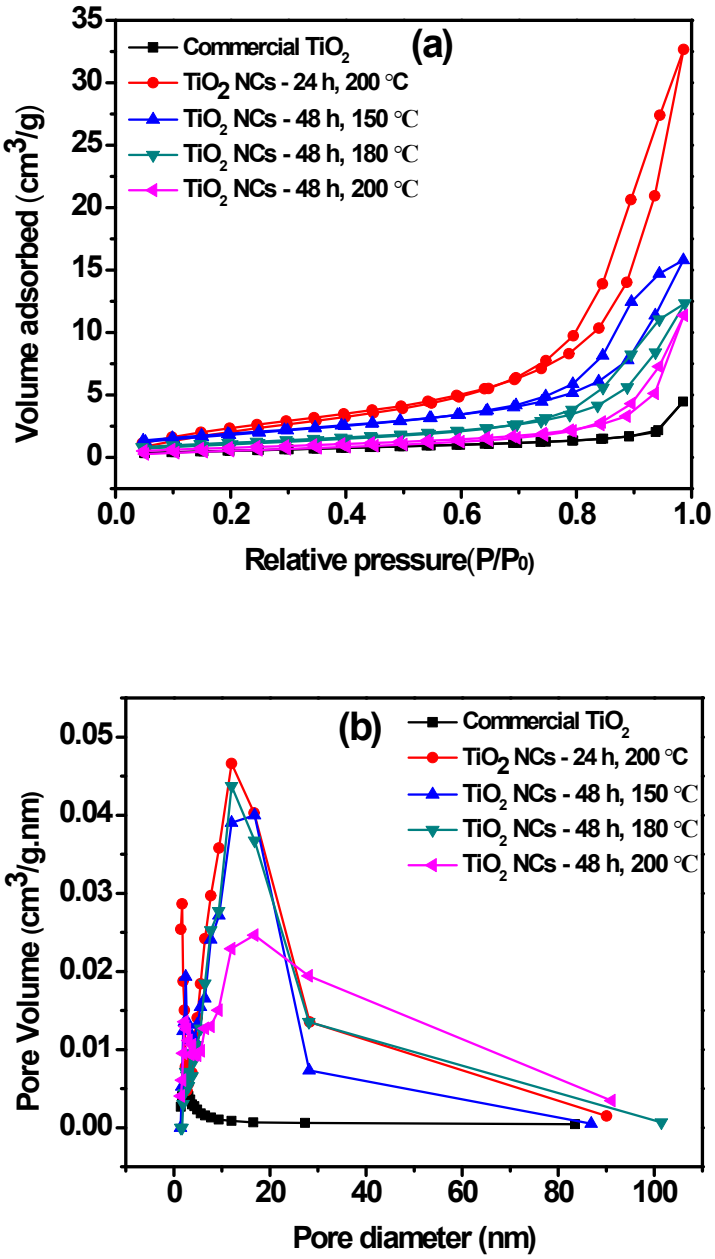

Figure S2. (a) N_2 adsorption-desorption isotherms and (b) corresponding pore size distribution curves of commercial TiO_2 and as prepared TiO_2 nanocuboids

Possible reactions involved in the formation of TiO_2 nanocuboids

$$TiO_2 + 6HF \longrightarrow H_2[TiF_6] + 2H_2O \quad \text{(S1)}$$

$$H_2[TiF_6] + 3H_2O + 6NH_3 \longrightarrow TiO_2(H_2O) + 6\,NH_4F \quad \text{(S2)}$$

$$TiO_2(H_2O) = H_2TiO_3$$

$$H_2TiO_3 + 3H_2O_2 + 2NH_3 \longrightarrow (NH_4)_2TiO_6 + 3H_2O \quad \text{(S3)}$$

$$(NH_4)_2TiO_6 \xrightarrow{EDTA} (NH_4)_2TiO_6\text{.EDTA} \quad \text{(S4)}$$

$$2(NH_4)_2TiO_6\text{.EDTA} + 2H_2O \xrightarrow[\text{condition}]{\text{Hydrothermal}} 2TiO_2 + 4NH_4OH + 3O_2 \quad \text{(S5)}$$

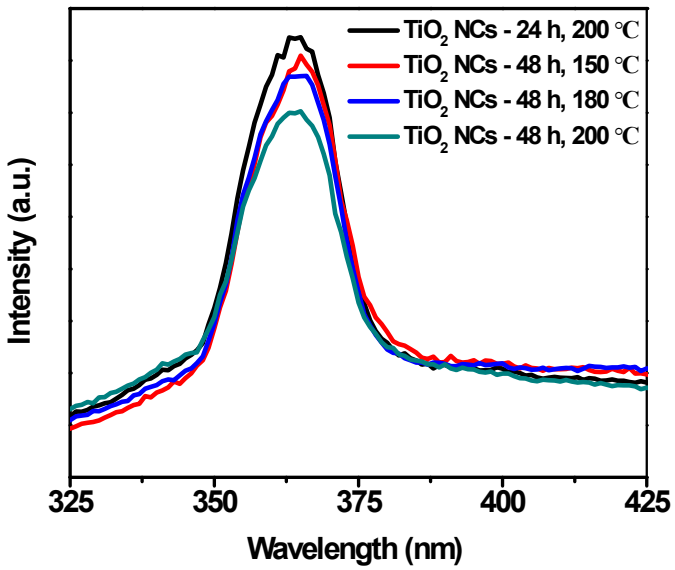

Figure S3. Photoluminescence spectra of TiO_2 nanocuboids prepared under different reaction time and temperature conditions

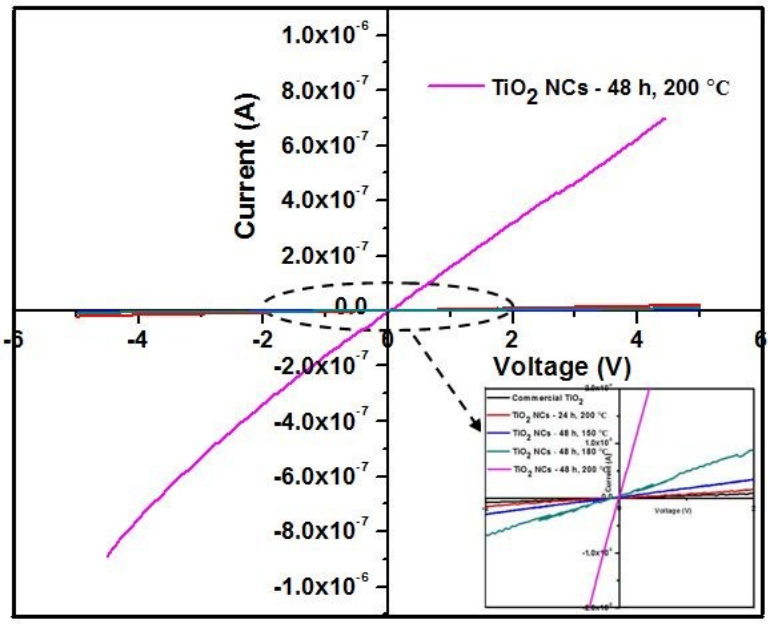

Figure S4. I-V plots of commercial TiO_2 and as prepared TiO_2 nanocuboids with illumination (1.5AM) of light.

Table S1. Specific surface area, pore volume and average pore size of commercial TiO_2 and as prepared TiO_2 nanocuboids

Sample	Specific surface area ($m^2 g^{-1}$)	Pore volume ($cm^3 g^{-1}$)	Average pore radius (nm)
Commercial TiO_2	29.3	0.038	6.64
TiO_2-NCs 24 h, 200 °C	122.9	0.459	9.91
TiO_2-NCs 48 h, 150 °C	111.6	0.346	6.99
TiO_2-NCs 48 h, 180 °C	103.2	0.402	8.85
TiO_2-NCs 48 h, 200 °C	85.7	0.495	12.04

Table S2. Comparison of the photocatalytic H_2 generation activity of anatase TiO_2 nanostructures reported by various researchers with as prepared anatase TiO_2 nanocuboids.

Sr. No.	Photocatalyst	Amount (g)	Light Source	H_2 generation ($\mu mol\ h^{-1}\ g^{-1}$)	Reference no.
1	TiO_2 nanocuboids	0.02	400W Hg lamp	3866.44	This work
2	TiO_2 (3D-flowerlike nanosheets)	1	450W Hg lamp	117.6	1
3	TiO_2 (nanosheets)	0.1	300W Xe lamp	2036	2
4	TiO_2 (cube-like morphology)	0.05	300W Xe lamp	104.54	3
5	TiO_2 (twinned nanocrystals)	0.05	300W Xe lamp	1272.66	3
6	TiO_2 (mesoporous nanofibers)	0.1	300W Xe lamp	231.7	4
7	TiO_2 (hollow spheres)	0.015	300W Xe lamp	21.2	5

References:

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3. G. Cheng, Y. Wei, Ji. Xiong, Y. Gan, J. Zhu and F. Xu, Same titanium glycolate precursor but different products: successful synthesis of twinned anatase TiO_2 nanocrystals with excellent solar photocatalytic hydrogen evolution capability, *Inorg. Chem.Front.,* 2017, **4,** 1319–1329.
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