Phase transition and Morphological evolution of titania/titanate nanomaterials under alkalescent hydrothermal treatment

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Fig. S1. XRD patterns of the products obtained in 0.00-1.50 M NaCl at 20-200 °C in the current work, indicating the phase transitions

from hydrogen titanate H₂Ti₂O₅·H₂O to brookite TiO₂ to anatase TiO₂.

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Fig. S2. Raman spectras for the samples of anatase TiO_2 obtained in 0.00 M NaCl at 180 °C (a), hydrogen titanate $H_2Ti_2O_5$ · H_2O obtained in 1.50 M NaCl at 180 °C (b) and pure brookite TiO_2 obtained in 0.25 M NaCl at 180 °C (c).

Raman spectra analysis was conducted on the samples using a Renishaw inVia+Reflex spectrometer and an excitation wavelength of 785 nm with incident power of 30 mW. The laser power was kept low enough to avoid heating of the samples by optical filtering and/or defocusing the laser beam at the sample surface. Spectra were collected in the range of 1000-100 cm⁻¹ with a resolution of 1 cm⁻¹.



Fig. S3. Photocatalytic degradation curves of MO under UV irradiation. Catalysts were labelled by the concentration of NaCl in the synthetic media and hydrothermal treated temperature.



Fig. S4. Photocatalytic degradation curves of 20 mg/L Phenol (a) and 20 mg/L Salicylic acid (b) under UV irradiation. Catalysts were labelled by the concentration of NaCl in the synthetic media and hydrothermal treated temperature.

Three photocatalysts with high photocatalytic activities (0.12 M NaCl-200 °C, 0.12 M NaCl-180 °C, 0.00 M NaCl-180 °C) have been also tested by two other model compounds (phenol and salicylic acid) and compared with the standard photocatalyst of Degussa P25 under identical conditions in order to further investigate the photocatalytic activities of brookite-anatase composites. Fig. S4 shows the photocatalytic degradation of phenol (a) and salicylic acid (b) under UV irradiation. All three samples exhibit high activities for photodegradation of phenol and salicylic acid. Whether in the phenol degradation or salicylic acid degradation, the photocatalytic activity of the best photocatalyst (200 °C, 0.12 M NaCl) is greater than that of standard photocatalyst of Degussa P25.

Sample ^a	0.00 M	0.12 M	0.25 M	0.50 M	0.75 M	1.00 M	1.50 M
Phases ^b	А	A + B	В	B + HTiO	B + HTiO	HTiO + B	HTiO
Specific surface area (S _{BET} , m ² ·g ⁻¹)	90.376	85.620	81.659	85.387	156.628	163.743	175.198

Table S1. Specific surface areas of classical samples obtained at 180 °C

a: Catalysts were denoted by the concentration of NaCl in the synthetic media.

b: Anatase, Brookite and layered hydrogen titanate (H2Ti2O5 H2O) were denoted as A, B and HTiO, respectively.

Specific surface areas were measured by Nitrogen adsorption-desorption method using ASAP 2020 Micromeritics apparatus following the BET analysis. Adsorption and desorption of N_2 were performed at -196 °C. Samples had been previously outgassed by heating at 100 °C under vacuum (3 mm Hg).