

## Supporting Information

# **An economic method to prepare vacuum activated photocatalysts with high photo-activities and photo-sensitivities**

**Mingyang Xing, Jinlong Zhang\*, Feng Chen and Baozhu Tian**

### **Experimental Details**

#### **Materials:**

The TiO<sub>2</sub> particles used in our experiment are Degussa P25 with the particle size of approximately 21 nm and BET surface area of 50 ± 15 m<sup>2</sup>/g. The type of vacuum drying oven was DZF-6020 and purchased from Shanghai Hualian Medical Devices Co., Ltd.

#### **Synthesis method:**

The TiO<sub>2</sub> powders (Degussa, P25) were spread homogeneously in a crucible. After the temperature of vacuum drying oven rose up to 470 K, the crucible was put into the oven and pumped vacuum immediately. The P25 was vacuum activated at a high vacuum degree for different time to obtain different vacuum activated samples.

### **Characterization**

X-ray diffraction (XRD) patterns of all samples were collected in the range 10-80° (2θ) using a Rigaku D/MAX 2550 diffractometer (Cu K radiation, λ = 1.5406 Å), operated at 40 kV and 100 mA. The surface morphologies were observed by scanning electron microscopy (JEOL.JSM-6360LV).

The instrument employed for XPS studies was a Perkin-Elmer PHI 5000C ESCA system with Al Kα radiation operated at 250 W. The shift of the binding energy due to relative surface charging was corrected using the C1s level at 284.6 eV as an internal standard.

The UV-vis absorbance spectra were obtained for the dry-pressed disk samples using a Scan UV-vis spectrophotometer (Varian, Cary 500) equipped with an integrating sphere assembly, using BaSO<sub>4</sub> as the reflectance sample. The spectra were recorded at room temperature in air within the range 200-800 nm.

The X-band EPR spectra were recorded at room temperature (Varian E-112).

Fourier transform infrared (FTIR) spectra were recorded with KBr disks containing the powder sample with the FTIR spectrometer (Nicolet Magna 550).

### **Photocatalytic Activities**

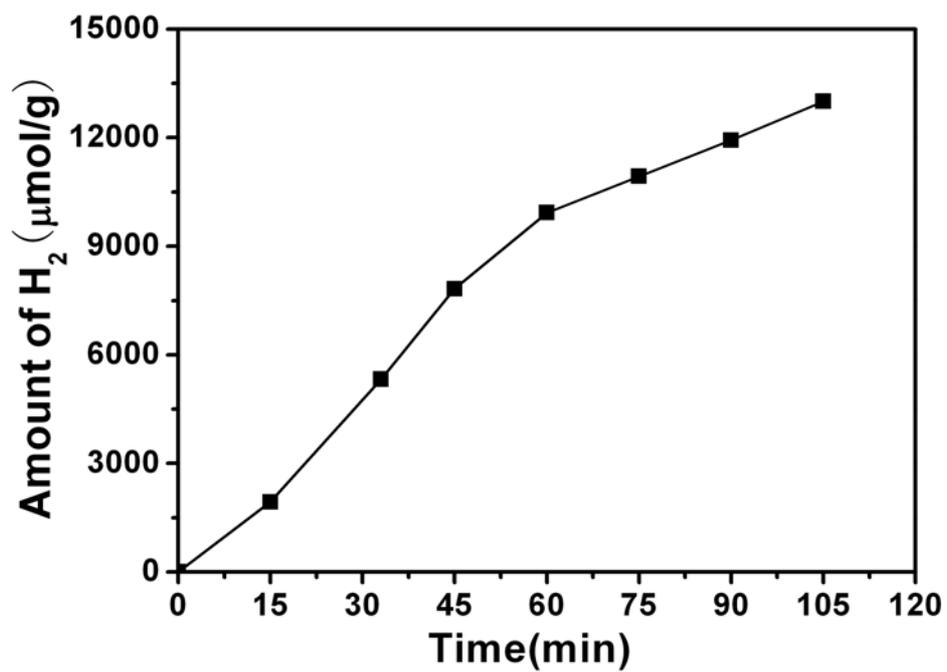
The photocatalytic activity of each sample was evaluated in terms of the degradation of methyl-orange (MO, 5 mg/L) and phenol (20 mg/L). The photocatalyst (0.07 g) was added into a 100 mL quartz photo-reactor containing 70 mL of a 5 mg/L MO or 20 mg/L phenol solution. The mixture was stirred for 30 min in the dark in order to reach the adsorption-desorption equilibrium. A 1000-W tungsten halogen lamp equipped with a UV cut-off filters (λ > 420 nm) was used as a visible light source and a 300-W high-pressure Hg lamp for which the strongest emission wavelength is 365 nm was used as a UV light source. The lamp was cooled with flowing water in a quartz cylindrical jacket around the lamp and ambient temperature is maintained during the photocatalytic reaction. At the given time intervals, the analytical samples were taken from the mixture and immediately centrifuged, then filtered through a 0.22 μm Millipore

filter to remove the photocatalysts. The filtrates were analyzed by recording variations in the absorption in UV-vis spectra of MO and phenol using a Cary 100 ultraviolet visible spectrometer.

Photocatalytic H<sub>2</sub> generation experiments were carried out in a sealed circulation system. 0.200 g of sample was suspended in 80 mL 25% methanol aqueous solution (containing 2 mL H<sub>2</sub>PtCl<sub>6</sub> (1g/L)) under magnetic stirring. Then the above solution was irradiated under a 300 W Xe lamp for 120 minutes, which resulted into the loading of Pt nanoparticles on the surface of catalyst. After degassing the system, a 400 nm cut-on filter (Newport Corp.) was used to cut off the UV light of Xe lamp and execute the photocatalytic reaction. The products were analyzed by gas chromatography (Techcomp GC-7890II) equipped with a thermal conductivity detector (TCD). The external quantum efficiency (EQE) was calculated based on the equation:

$$\text{EQE} = \frac{\text{The number of evolved H}_2 \text{ molecules} \times 2}{\text{The number of incident photons}} \times 100\% \quad (1)$$

To measure the EQE at 420 nm, a 420 nm band pass filter (Newport Corp.) was used. The number of the incident photons was determined using a radiant power energy meter (Newport Corp., Optical Meter Model 1918-C).



**Fig. S1** Time course of evolved H<sub>2</sub> under the 300 W Xe lamp (without 400 nm cut-on filter) light irradiation on the sample after vacuum activated for 180 minutes.

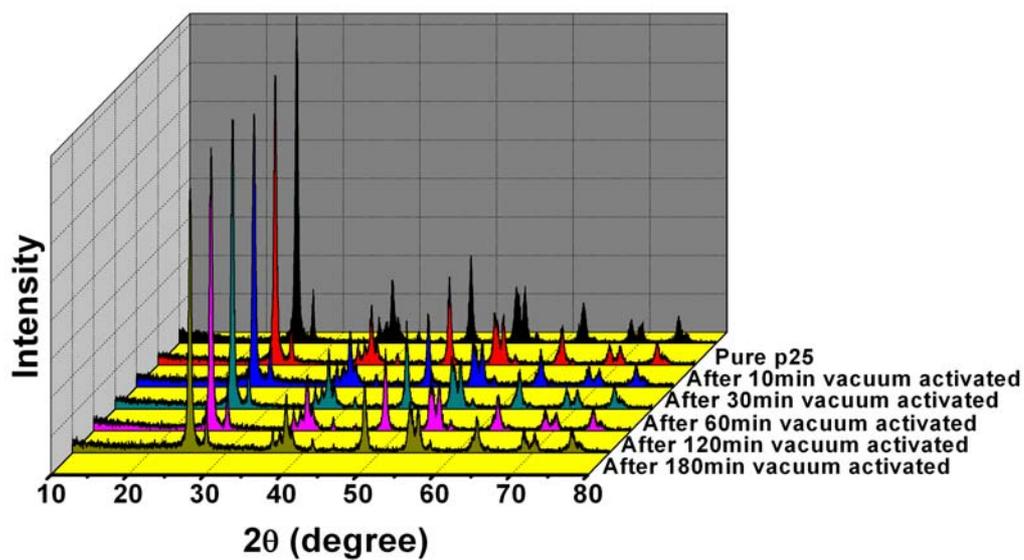
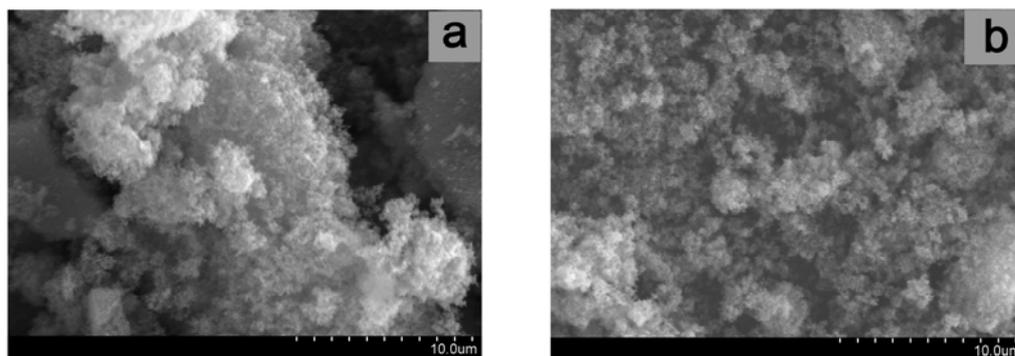
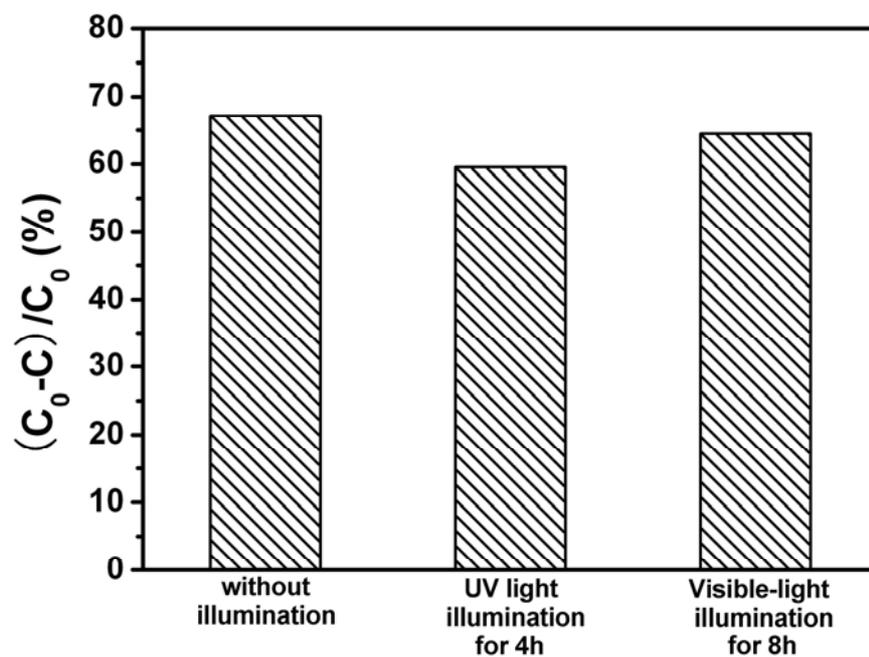


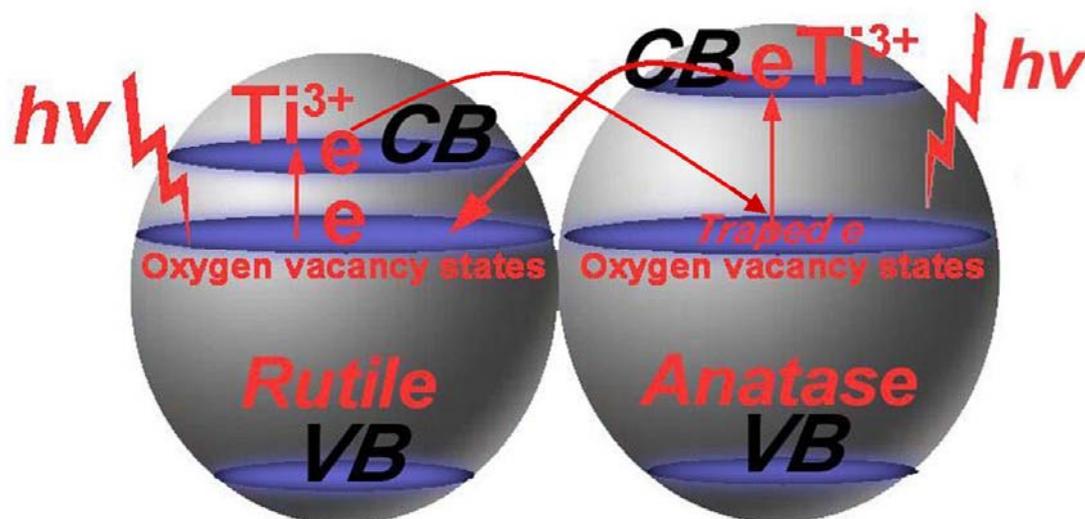
Fig. S2 XRD patterns for pure P25 and the vacuum activated samples.



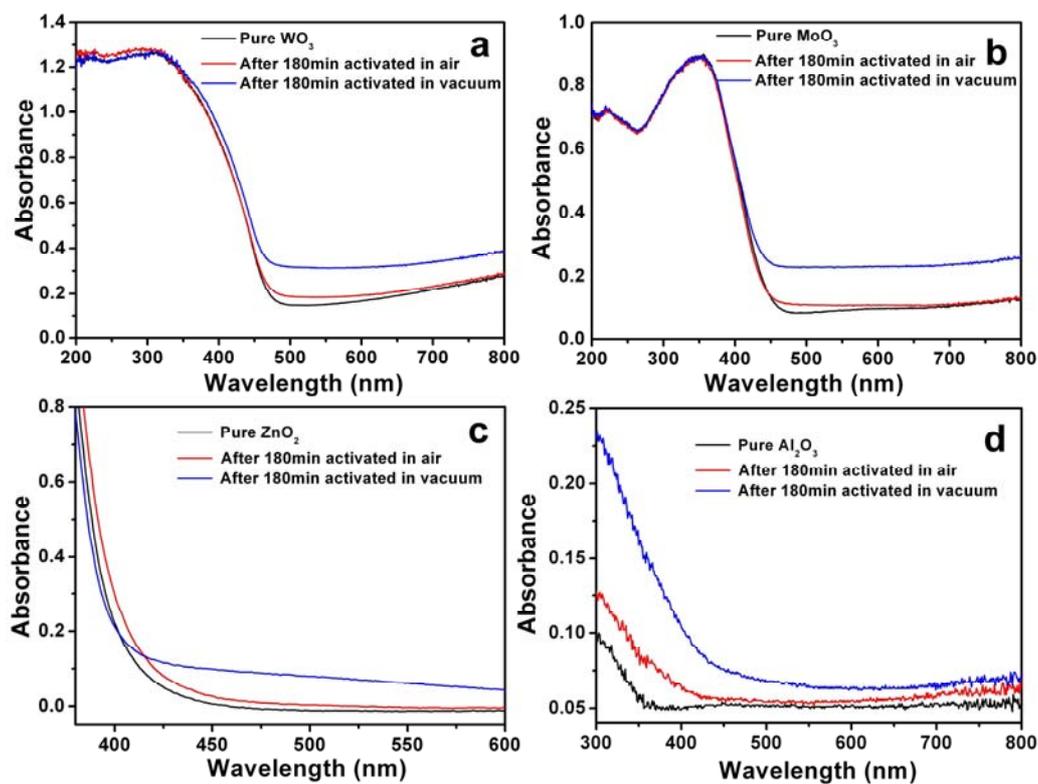
**Fig. S3** SEM images for different samples. a) pure P25. b) P25 after 120 minutes vacuum activated.



**Fig. S4** Photo-oxidation 20 mg/L phenol under the visible light irradiation (>420 nm) for 6 hours on the 180 minutes vacuum activated sample with the illumination treatment (without light illumination, under UV light illumination for 4 hours and under visible light illumination for 8 hours).



**Fig. S5** The photo-excited electrons-transfer from anatase to rutile under the light irradiation for the vacuum activated P25.



**Fig. S6** UV-vis diffuse reflectance spectra for different pure metal oxides and activated in different conditions for 180 minutes.