Supplementary Materials: Fast and Large-Scale Anodizing Synthesis of Pine-Cone TiO_2 for Solar-Driven Photocatalysis

Yan Liu, Yanzong Zhang, Lilin Wang, Gang Yang, Fei Shen, Shihuai Deng, Xiaohong Zhang, Yan He, Yaodong Hu, and Xiaobo Chen

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Methods

Synthesis of graphene

Graphene was synthesized by an electrochemical exfoliation method. Two graphite rods (99.99%, Shanghai Zichen Co., China) were inserted into an electrolyte (0.5g NaCl and 20 mL isopropanol)serving as anode and cathode, and a bias of 15 V was applied between the two electrodes using a direct current power supply. After a continuous reaction for 10 h, a dark solution was formed in the reaction beaker. The dark solution was then centrifuged at 956 ×*g* (E-5480R, Germany) to remove the large agglomerates. The graphene powder was collected by centrifuging at 27,530 ×*g* (Allegra 64R Centrifuge, Beckman Coulter), and washed several times with absolute ethanol and deionized water until no Cl^- was detectable. The obtained graphene was then dried at 333 K.

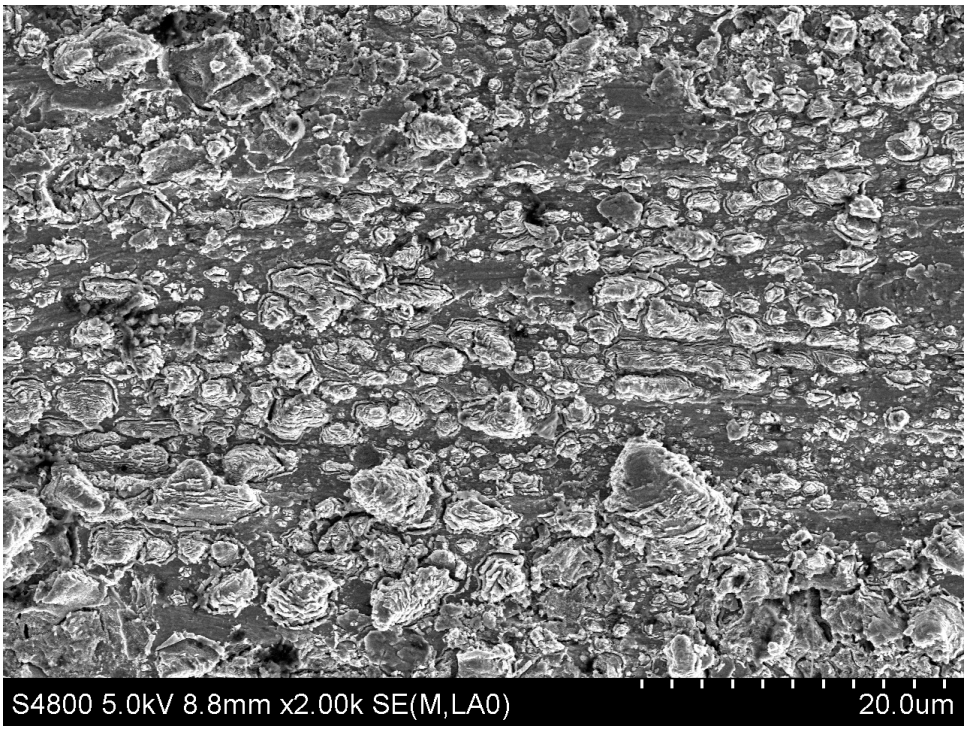

Figure S1. FE-SEM image of overall PCT film.

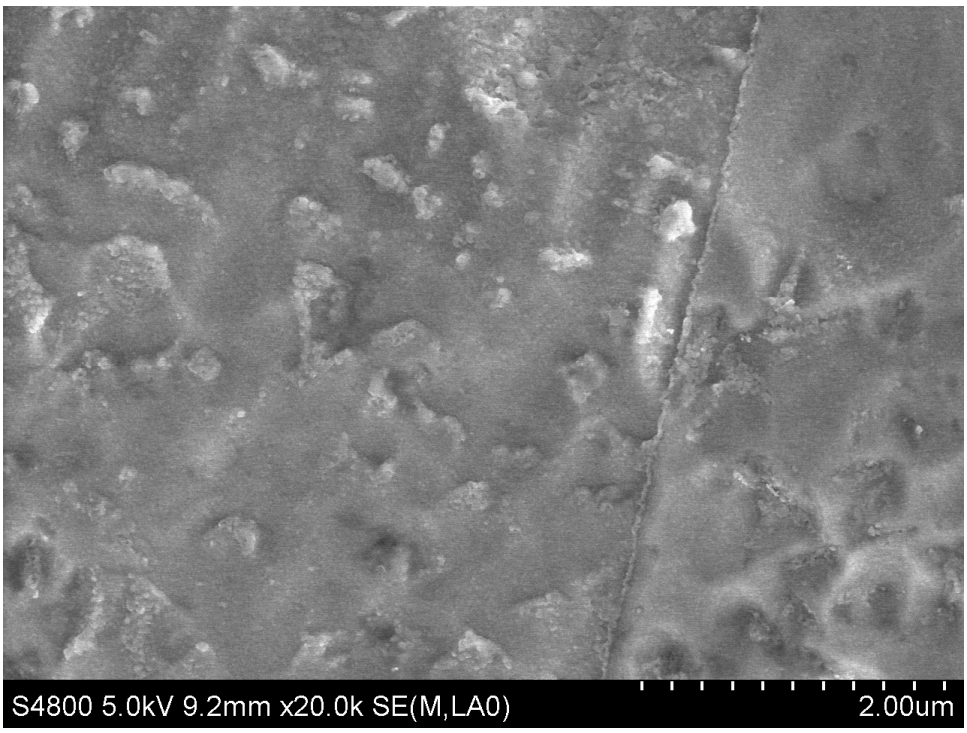

Figure S2. FE-SEM images of the TiO_2 film anodized in the electrolyte of magnesium nitrate solution (100 mg L^{-1}, 50 mL) at 60 V for 2 min.

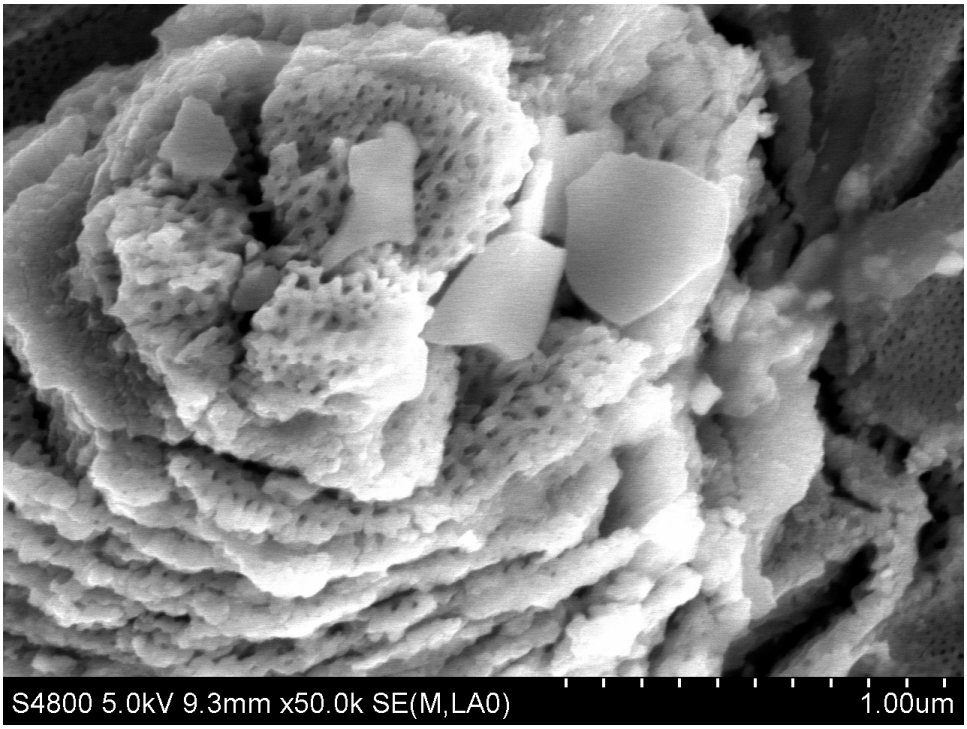

Figure S3. FE-SEM image of top view of the PCT film.

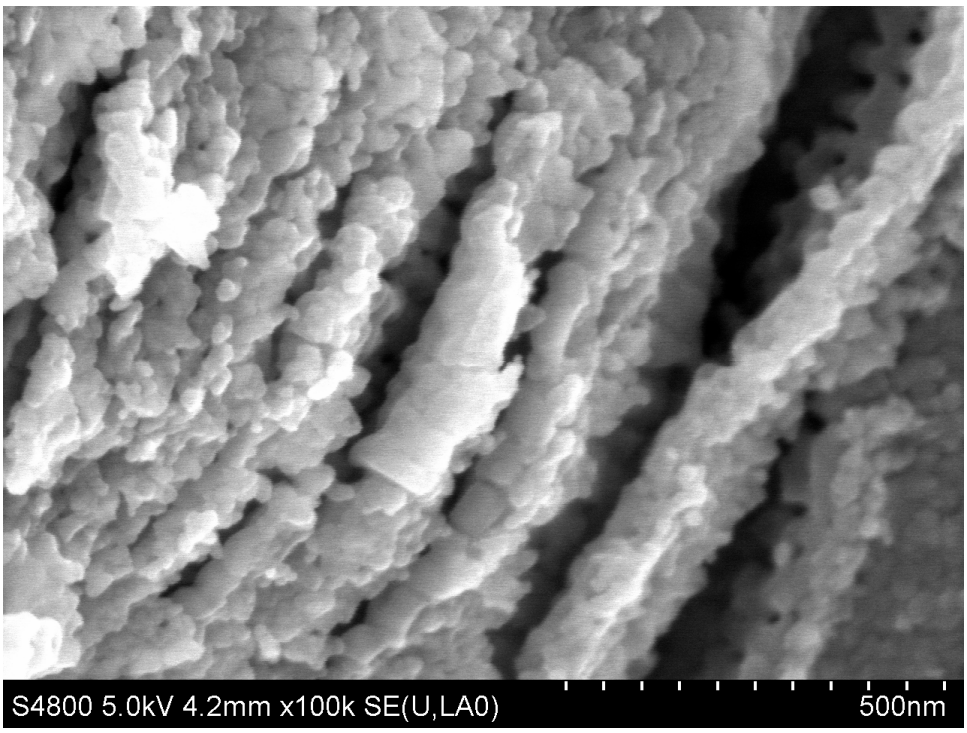

Figure S4. FE-SEM image of layer structure of the PCT film.

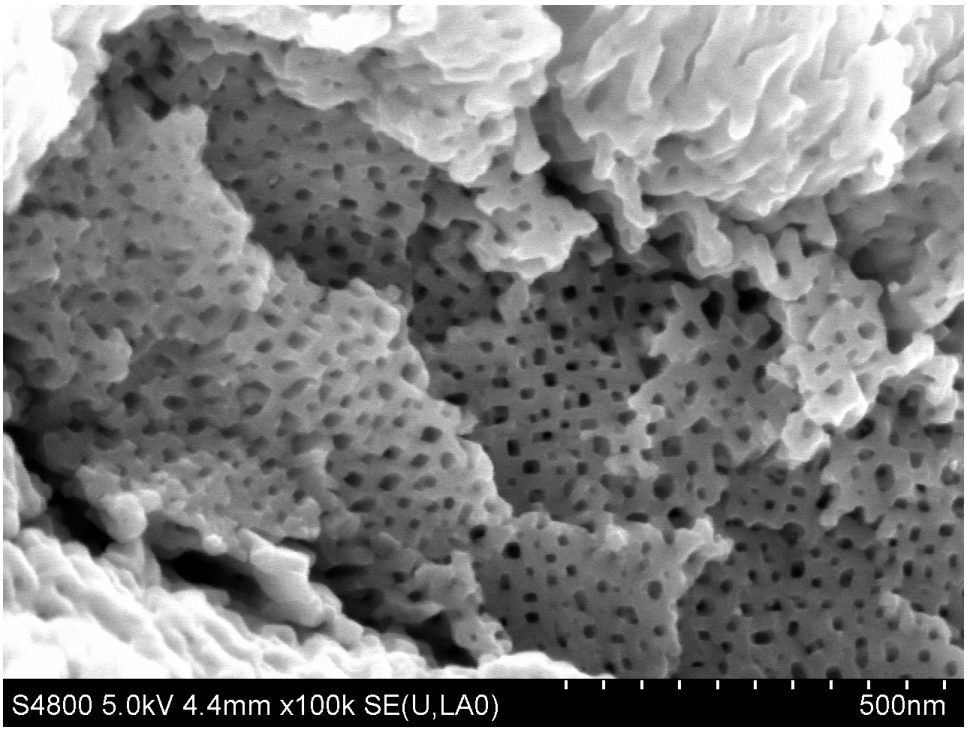

Figure S5. FE-SEM image of cracks on the oxide layer.

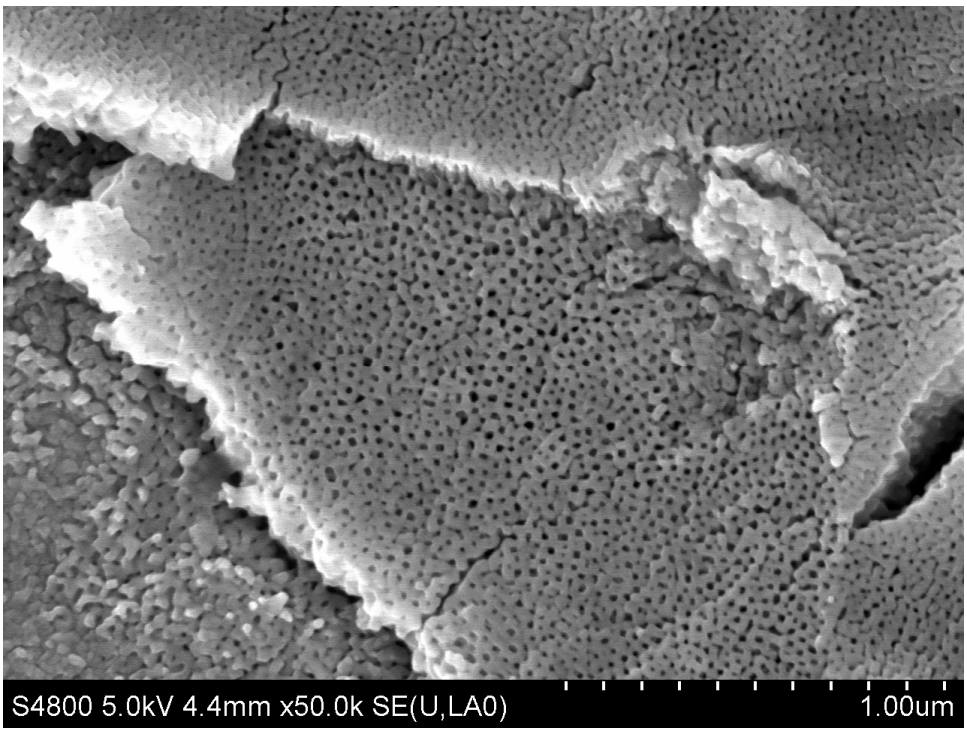

Figure S6. FE-SEM image of the nanoporous oxide layer.

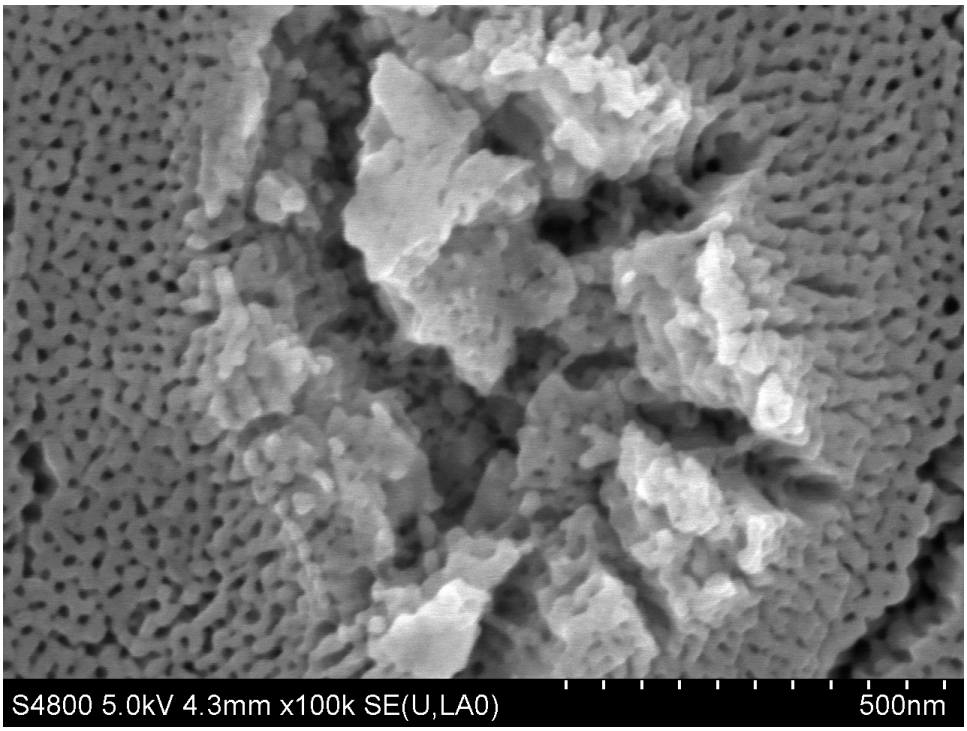

Figure S7. FE-SEM image of small cracks in the Figure 1A.

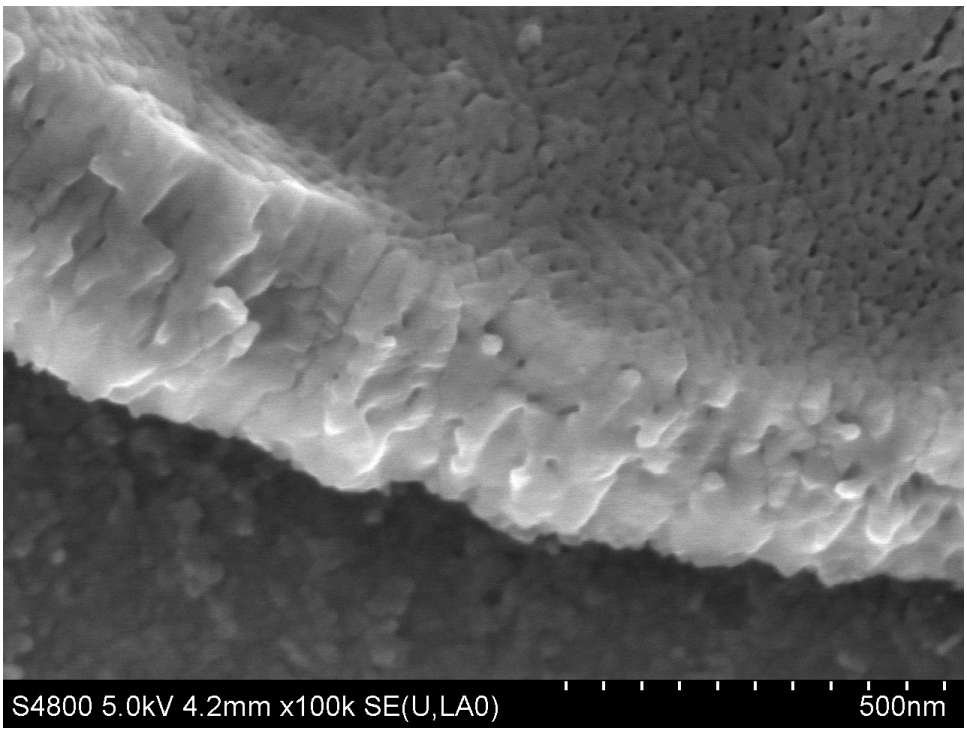

Figure S8. FE-SEM image of TiO_2 fragment washed up by O_2 gas.

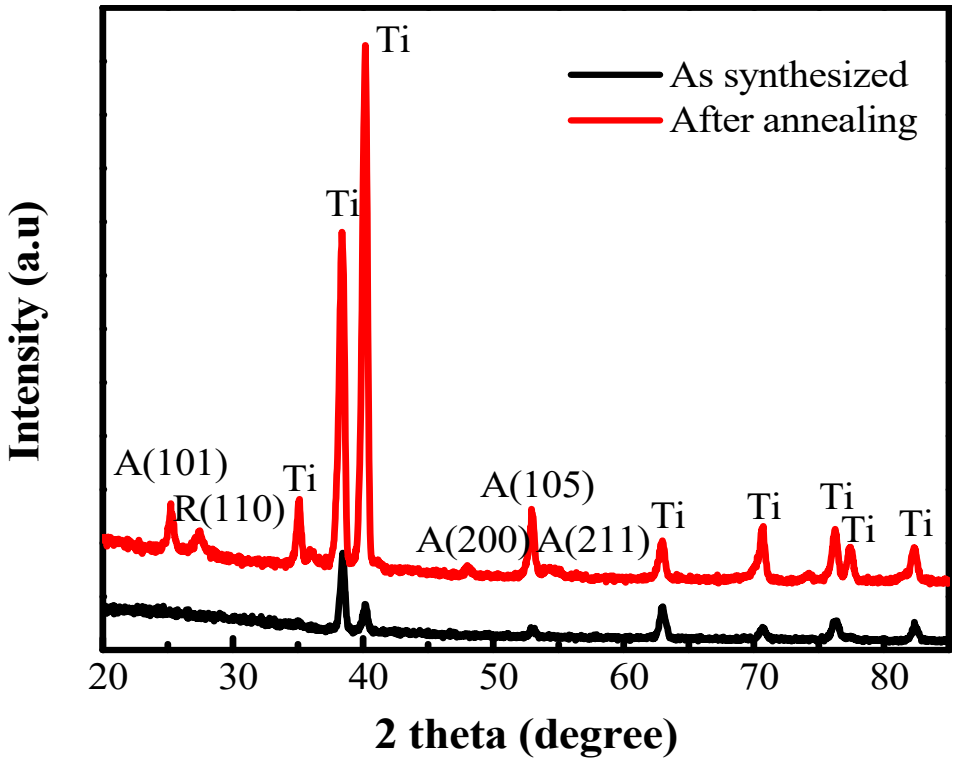

Figure S9. XRD patterns of the PCT films. Specific diffraction peaks for anatase (JCPDS#21−1272) and rutile (JCPDS#21−1276) are labeled according to A(hkl) and R(hkl), respectively.

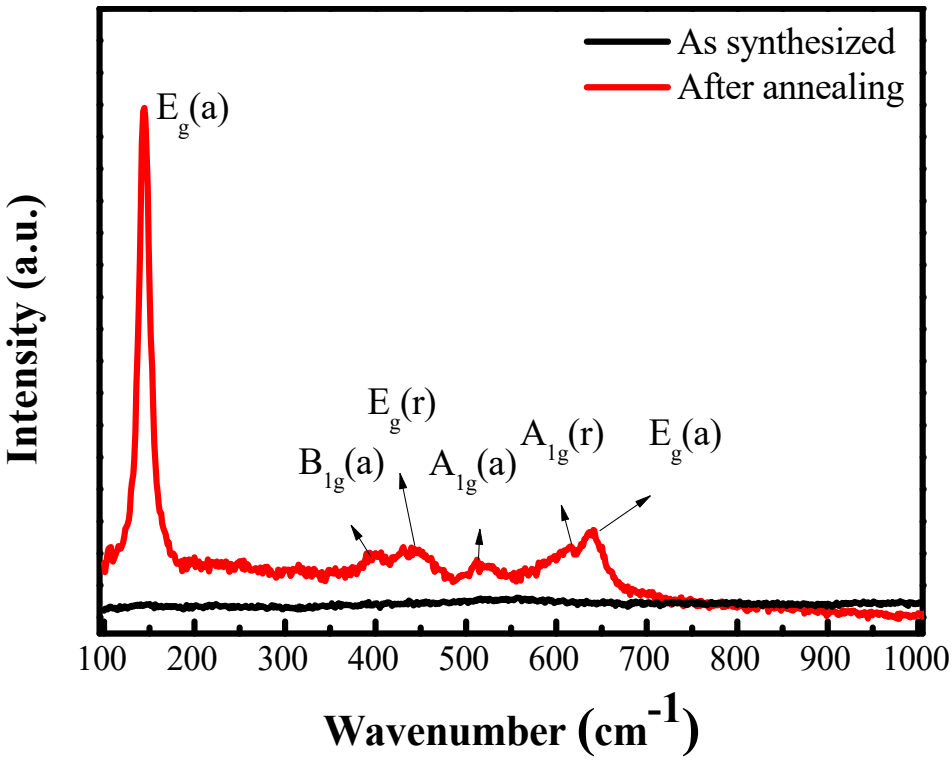

Figure S10. Raman scattering patterns of the PCT films, where the anatase and rutile vibration modes are labeled according to mode (a) and mode (r), respectively.

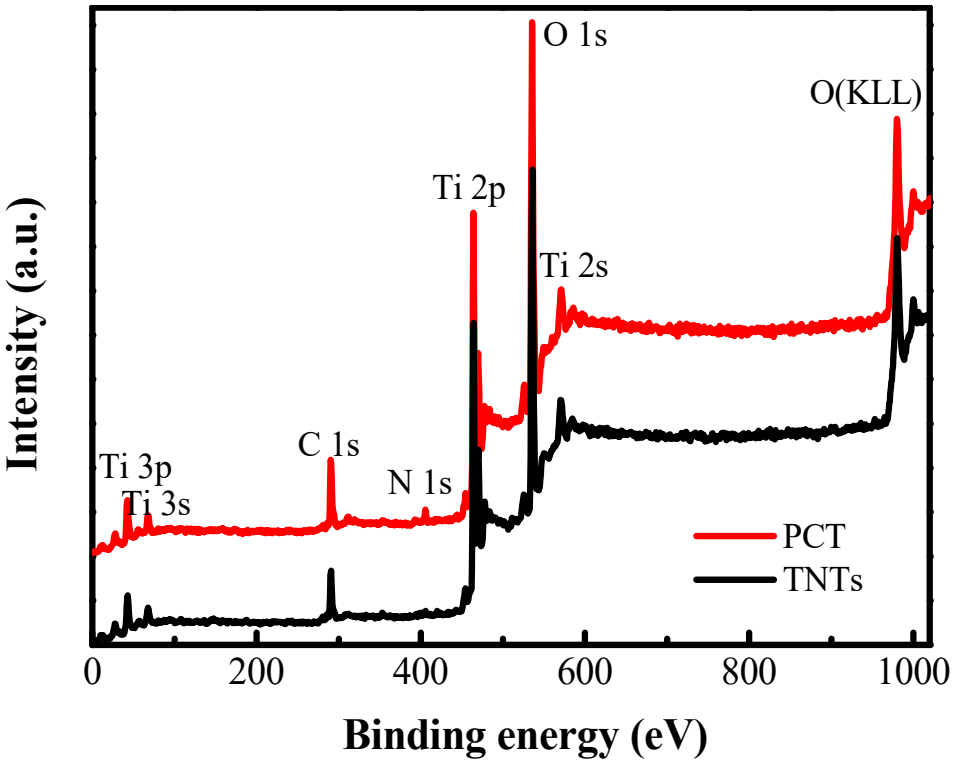

Figure S11. Full XPS spectra of PCT film and TNTs.

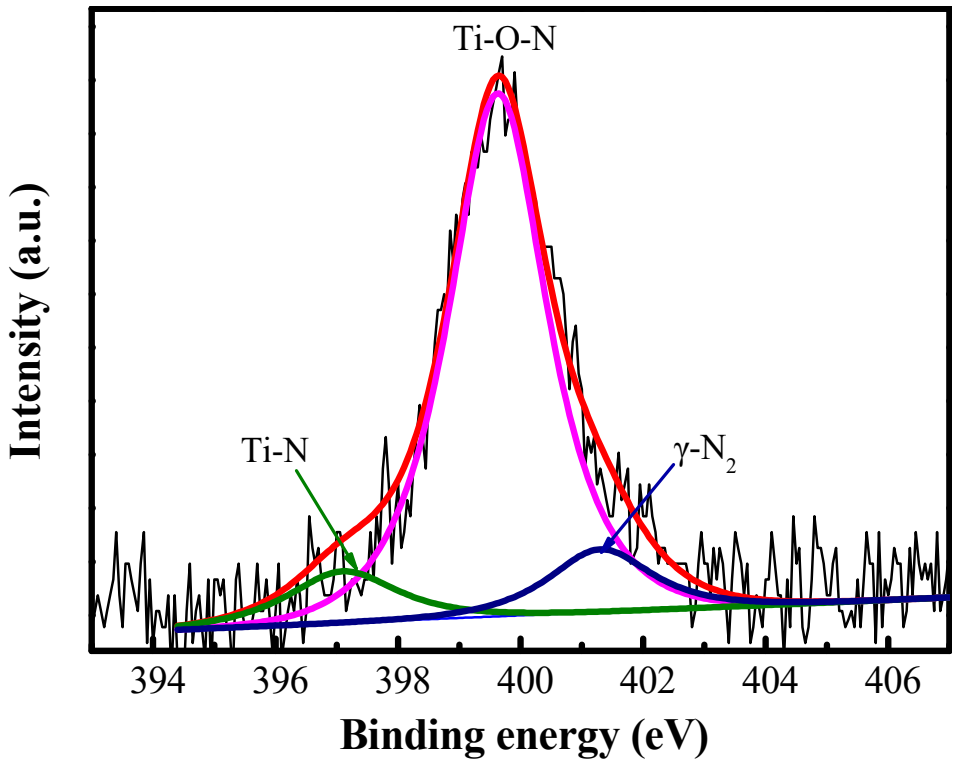

Figure S12. N 1s XPS spectrum of the PCT film.

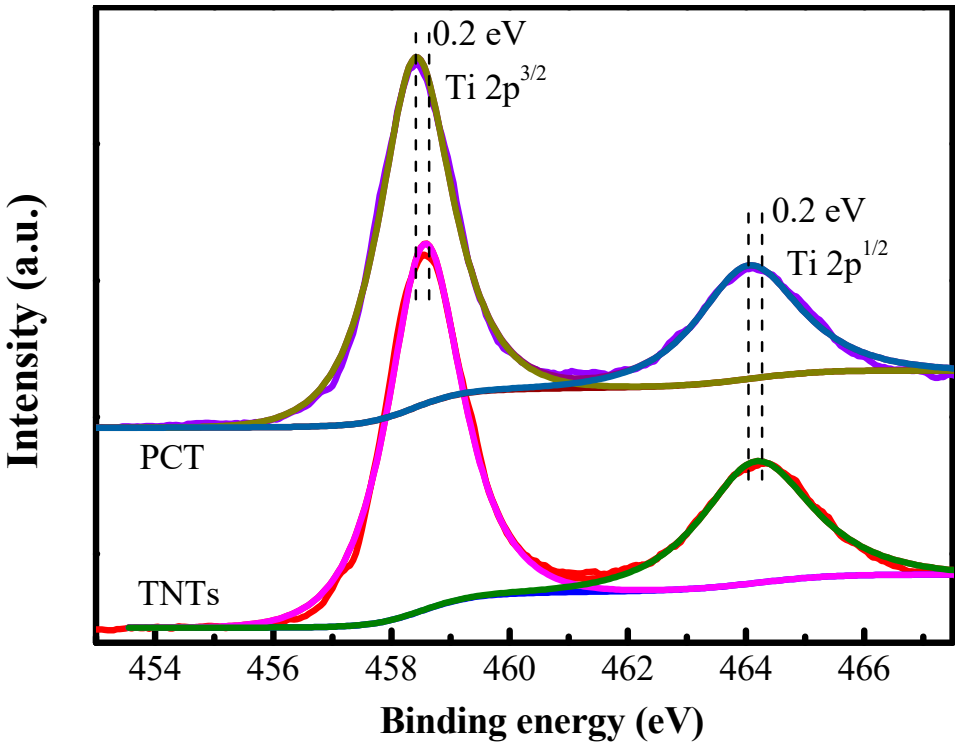

Figure S13. Ti 2p XPS spectra of the PCT film and TNTs.

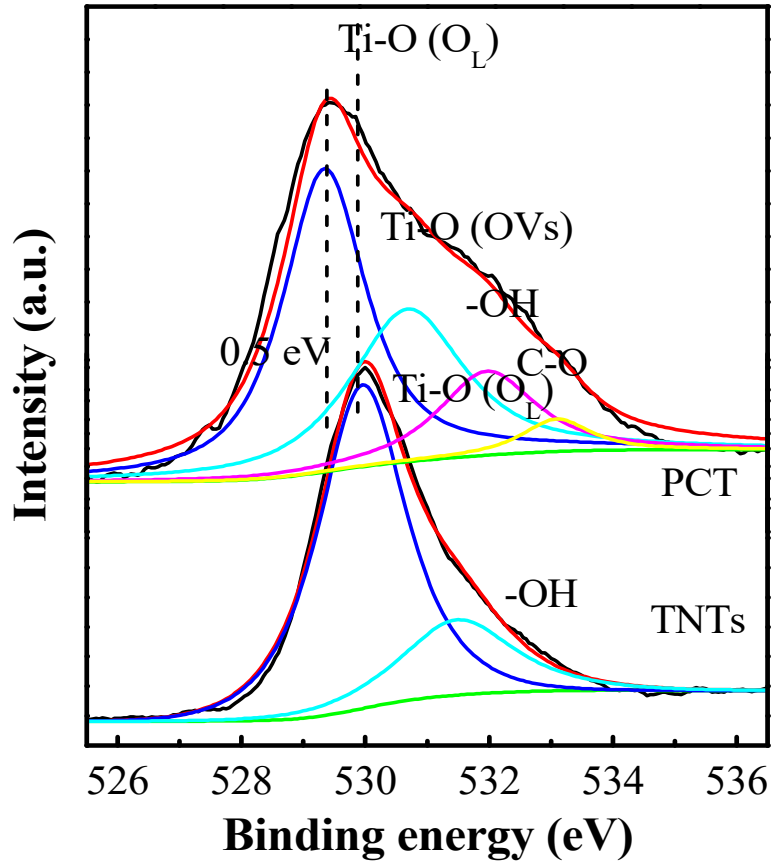

Figure S14. O 1s XPS spectra of the PCT film and TNTs.

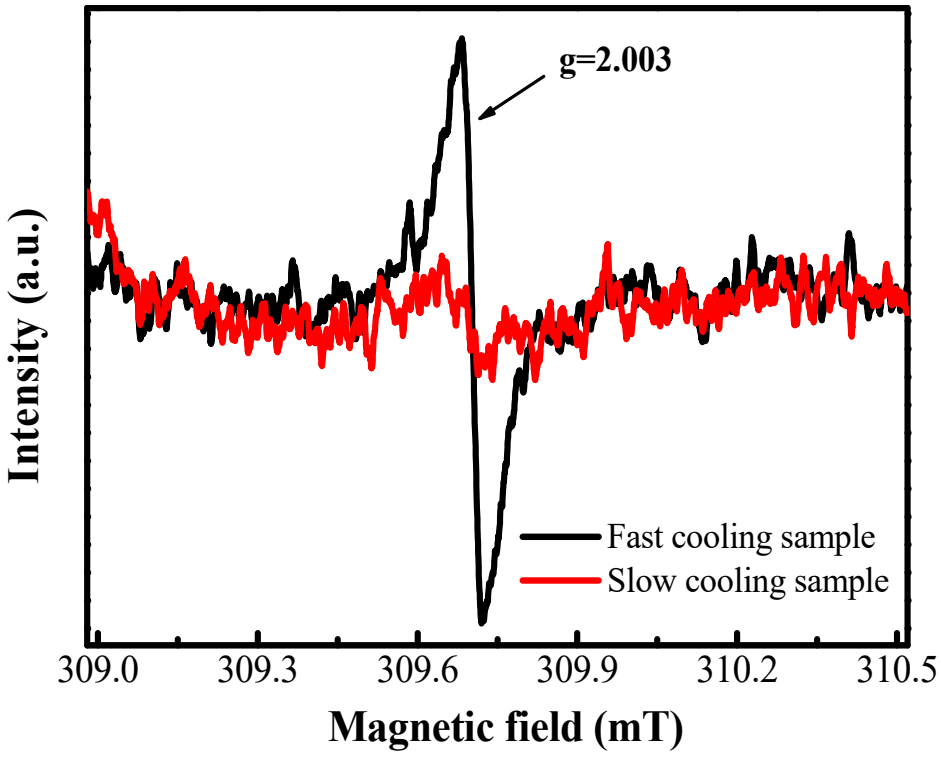

Figure S15. ESR spectra of PCT film with different cooling styles after calcination at 723 K for 2 h.

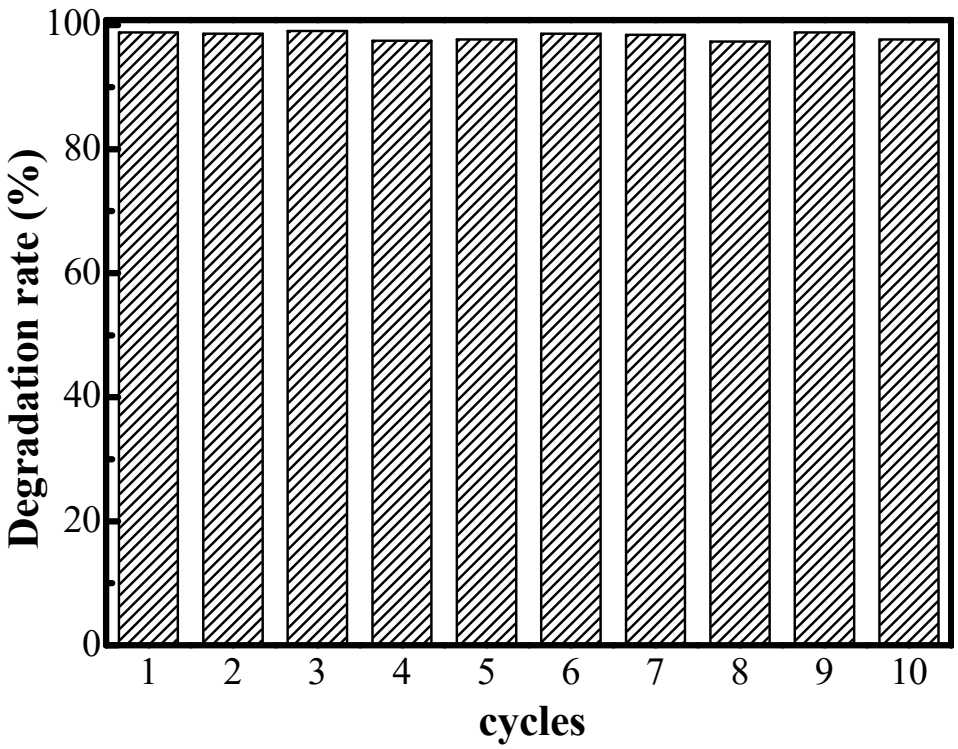

Figure S16. Stability test of methyl orange degradation for ten cycles under natural sunlight (C_{MO} = 20 mg L^{-1}, V_{MO} = 40 mL).

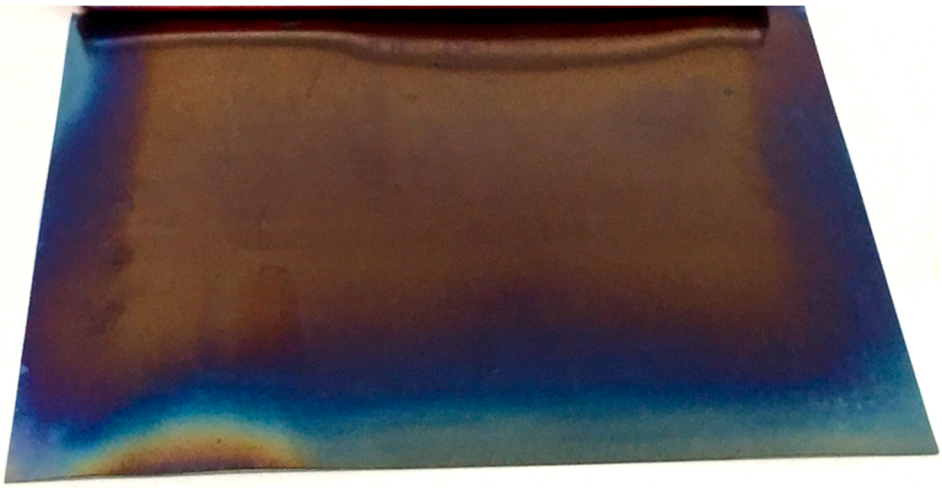

Figure S17. Pictures for the large-scale PCT films prepared by different anodization conditions. A laptop was used as a reference.

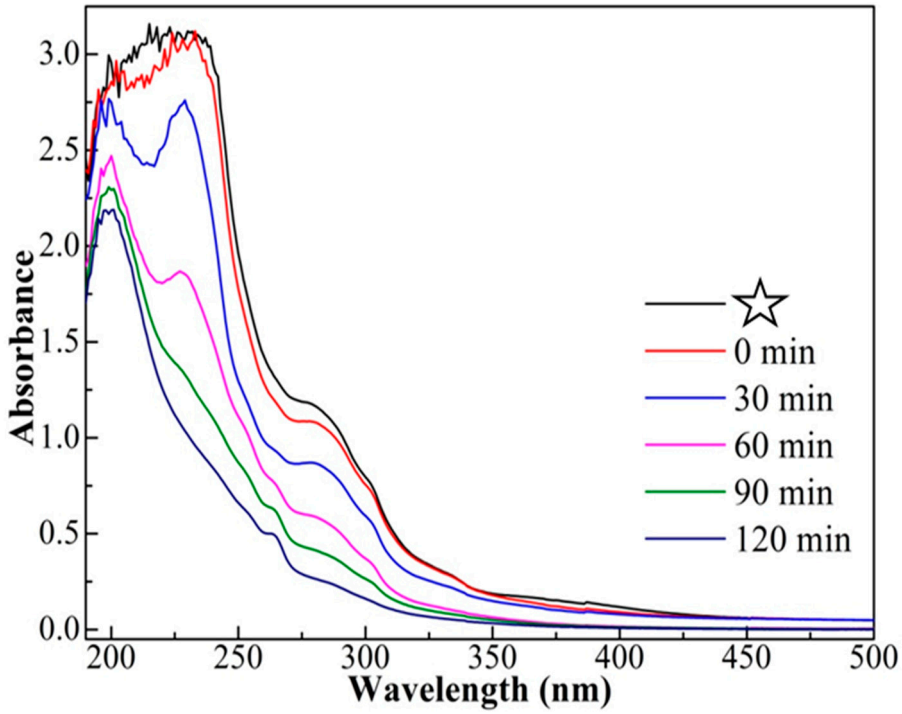

Figure S18. UV-Vis absorbance of the printing and dyeing wastewater during the photoelectrochemical degradation process. ☆ represents the absorbance of the waste water after flocculation and precipitation.

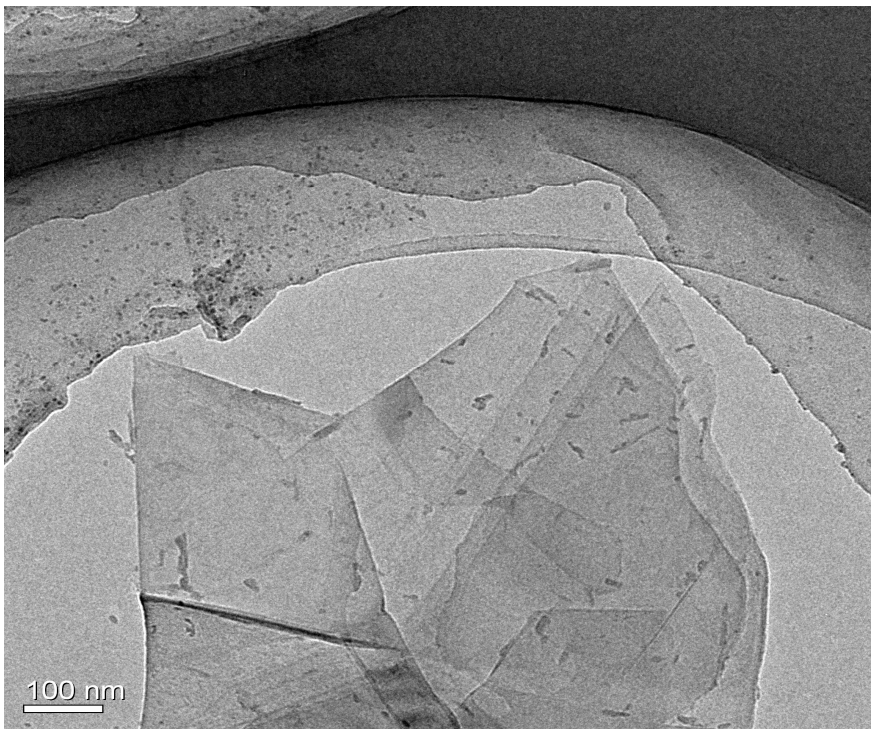

Figure S19. TEM images of prepared graphene. The as-prepared graphene was almost completely transparent and had an average length and width of about 500 and 300 nm.

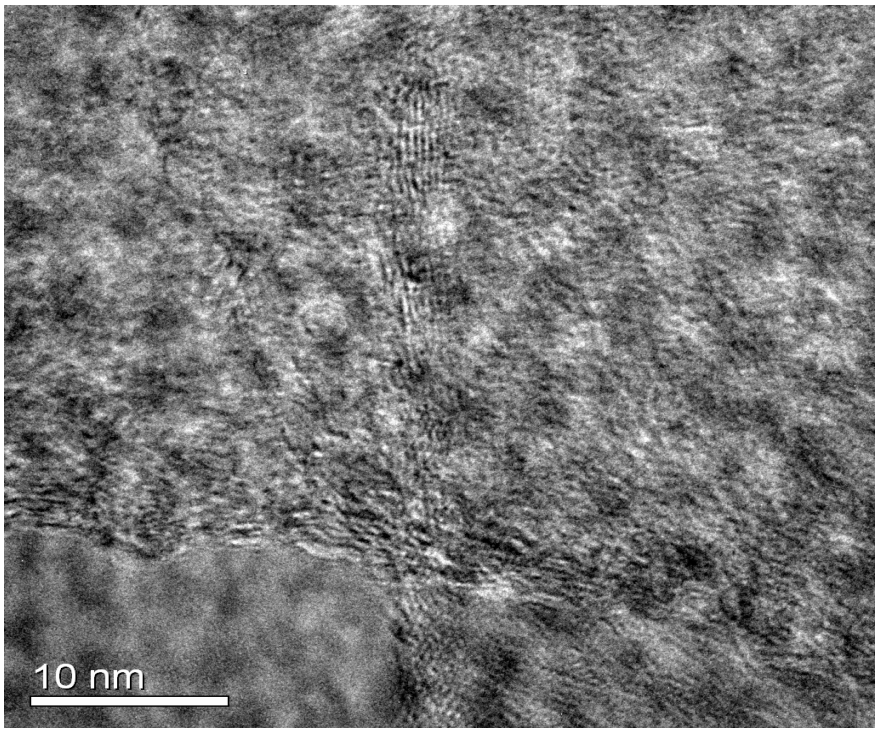

Figure S20. HRTEM images of prepared graphene. The graphene was no more than 7 layers thick.

Tables S1-S2

Table S1. Lattice parameters and particle sizes of the PCT film

Crystal phase	*d*-spacing (Å)		FWHM (Å)	Crystallite size (nm)	Phase ratio
	theoretical	experimental			
Anatase	3.5200	3.5270	0.410	19.6	0.78
Rutile	3.2470	3.2425	0.445	18.1	0.22

Table S2. Average illumination intensity of sunlight and outside temperature during the MO photodegradation.

Weather	Photodegradation time (min)	30	60	90	120
Sunny Day	Illumination intensity (lx)	87,020	88,220	89,800	90,080
	Temperature (K)	306	306	307	307
Cloudy day	Illumination intensity (lx)	51,030	50,850	36,220	41,890
	Temperature (K)	301	301	301	302