Supporting Information

One-Step Hydrothermal Synthesis of N-TiO_2/C Nanocomposites with Highly Visible-Light Photocatalytic Activity

*Dong-Hong Wang, Li Jia, Xi-Lin Wu, Li-Qiang Lu, An-Wu Xu**

Division of Nanomaterials and Chemistry, Hefei National Laboratory for Physical Sciences at Microscale, Department of Chemistry, University of Science and Technology of China, Hefei 230026, China

Correspondence author: E-mail: anwuxu@ustc.edu.cn (A. W. Xu)

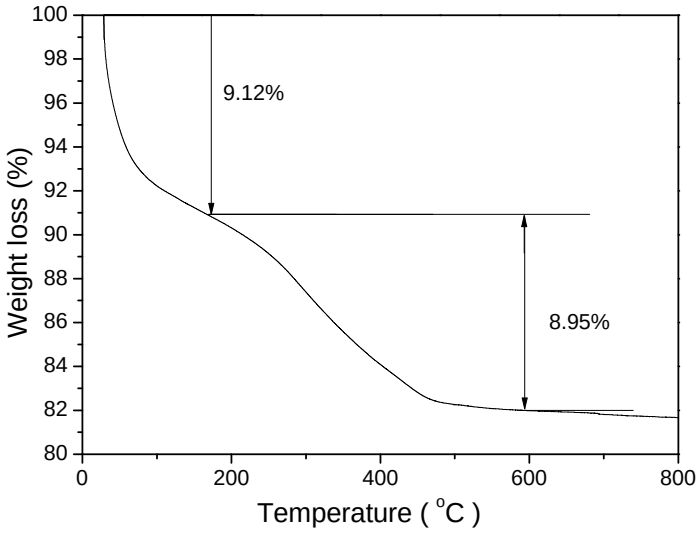

Fig. S1 Thermogravimetric **(**TGA) trace of N-TiO_2/C nanocomposites under a stream of air. The TGA curve exhibits a total mass loss of 18.07 % for the sample. The curve

can be divided into two stages. Below 200 ^{o}C, a mass loss of up to 9.12 % is observed, which is caused by dehydration from the powders. The second stage is from 200 to 600 ^{o}C, where the mass loss is about 8.95 %. This can be assigned to the combustion of carbon species and doped nitrogen in the N-TiO_2/C. There is no mass loss above 600 ^{o}C, indicating that all the impurities were burned out from the nanocomposites. Doped nitrogen in the N-TiO_2/C sample was found to be 2.26 atom % estimated from the XPS data, and the weight content is about 1.0 wt %. Therefore, we can conclude that the total content of the carbon species in the final N-TiO_2/C sample is about 7.95 wt%, in agreement with XPS analysis.

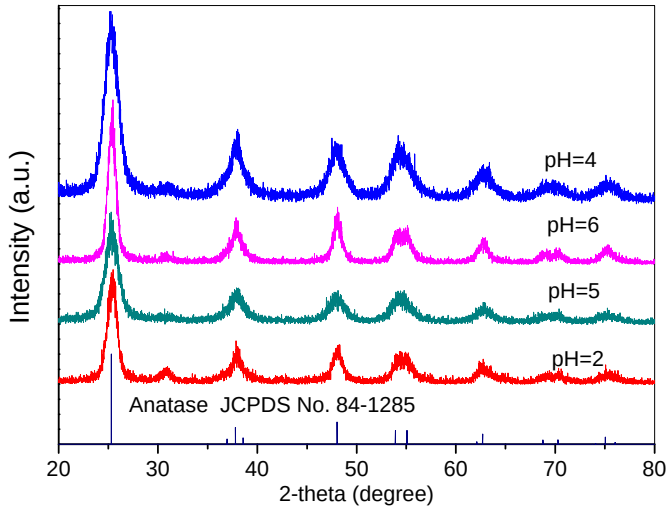

Fig. S2 XRD patterns of N-TiO_2/C samples prepared at different pH. The change of the pH value ranging from 2 to 6 has little effect on the crystal phase of N-TiO_2/C. All samples were crystallized into anatase phase by hydrothermal treatment at different

pH.

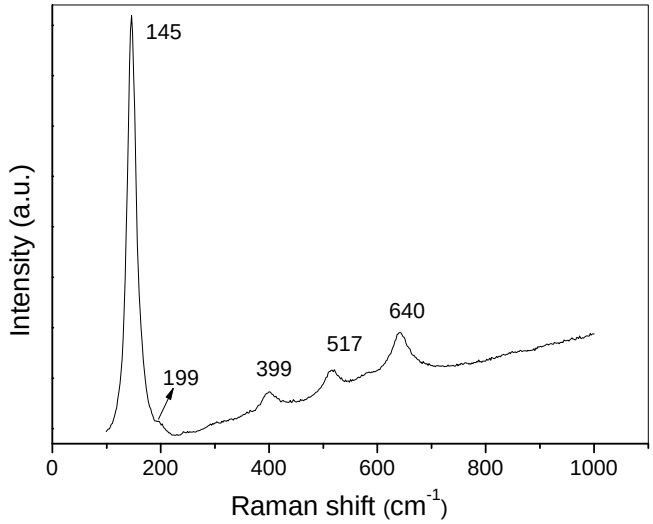

Fig. S3 Raman spectrum of N-TiO_2/C sample under hydrothermal treatment at 180 ºC for 12 h (L-lysine/$TiCl_4$ molar ratio = 8/1 and pH = 4).

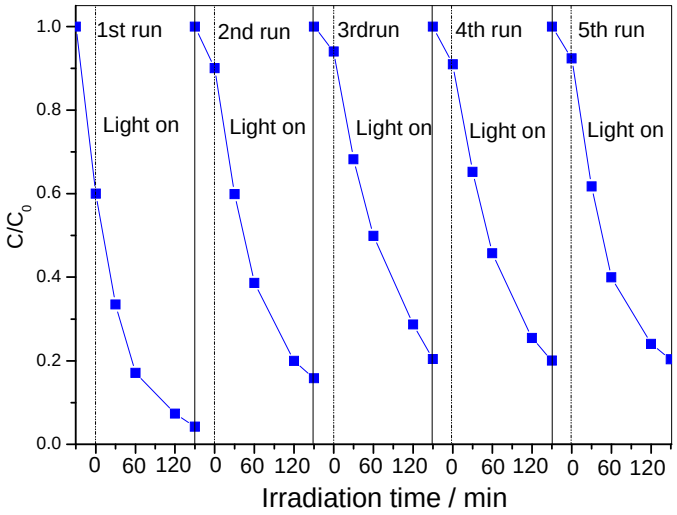

Fig. S4. Reusable stability of N-TiO_2/C nanocomposites under the same reaction conditions. Additional experiments for five cycles show that the degradation rate keeps almost constant after five cycles, confirming that the N-TiO_2/C photocatalyst is stable during prolonged photocatalytic reaction. After the first cycle, the adsorption capability decreases a little, which could be attributed to the surface active sites of the sample were covered by some groups decomposed from adsorbed-dyes.

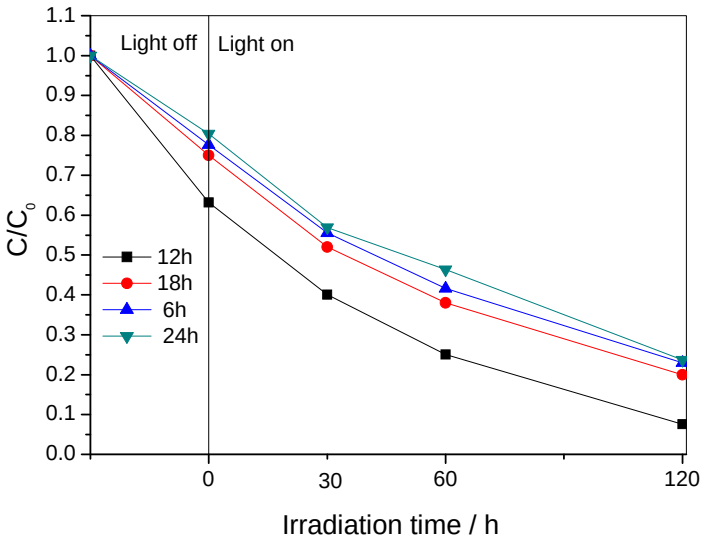

Fig. S5. The effect of hydrothermal treatment time on the photodegradation of MO with the N-TiO_2/C powders at 180 ºC.

The effect of hydrothermal treatment time on the photodegradation of MO for the N-TiO_2/C powders at 180 ºC is shown in Fig. S5. The optimal reaction time is 12 h, an increase or decrease in the reaction time all lead to the decrease in photocatalytic activity. From TGA and XPS analysis, the carbon and the nitrogen content decrease as the hydrothermal treatment time increases. The nitrogen and carbon species are responsible for the light harvesting, electron transferring, and dyes adsorbing. However, higher N content means higher concentration of oxygen vacancies and Ti^{3+}

species, which can become the recombination center of photo-produced holes and electrons as the N content increases,[27] leading to the decrease in the photocatalytic activity. In addition, with the increase of the hydrothermal time, the particle size of N-TiO_2/C powders increases, leading to a decrease in the specific surface area.[6] The decrease of the reaction time leads to a poor crystallinity. A high crystallinity is often required to reduce the formation of electron traps, which enhances the photocatalytic efficiency. A large specific surface area can supply more active sites and number of substrates adsorbed. The N-TiO_2/C sample prepared at 12 h by hydrothermal treatment has a good compatibility with the wonderful absorption in the visible light region, the small size, high surface area and the efficient electron transfer. Therefore, the optimal sample prepared at 12 h shows the highest photocatalytic degradation efficiency.