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journal or publication title	RSC Advances
volume	5
number	6
page range	4026-4029
year	2015
URL	http://hdl.handle.net/10228/00006480

doi: <https://doi.org/10.1039/C4RA13355B>

Synthesis high specific surface area nanotube g-C₃N₄ with two-step condensation treatment of melamine to enhance photocatalysis properties

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Experimental

Preparation of g-C₃N₄ and Hx-C₃N₄

First-step condensation was hydrothermal treatment of melamine in water as follows. Melamine (5 g) was added to 50 mL deionized water and dispersed for 30 min, and then it was heated in a Teflon-lined autoclave at 200 °C for 12 hour and 24 hour (denoted as HM-12 and HM-24, respectively). After filtration and evaporation, a white powder was obtained. Then the white powder was ground in an agate mortar.

Second-step condensation was heating treatment of the white powder as follows. Three grams of powder (HM-x) was packed in an alumina crucible with a cover and was heated in air at a rate of ca. 10 °C min⁻¹ to reach a temperature of 550 °C. After being heated at 550 °C for 4 h, the g-C₃N₄ obtained was ground in an agate mortar. The sample obtained by two-step condensation was denoted as Hx-C₃N₄, where x is time of hydrothermal treatment. For comparison, bulk g-C₃N₄ was also obtained by melamine treatment as second-step condensation process.

Characterization

Crystal structure was performed by using an X-ray diffractometer (Rigaku, MiniFlex II) with CuK α radiation ($\lambda = 1.5405 \text{ \AA}$). Morphology of the prepared particles was observed by a field emission scanning electron microscope (FE-SEM; JEOL, JSM-6701FONO) and a transmission electron microscopy (TEM; Hitachi H-9000NAR). Diffuse reflectance spectra (DRS) were measured using a UV-vis spectrophotometer (Shimadzu, UV-2600) equipped with an integrating sphere unit (Shimadzu, ISR-2600 Plus). Specific surface area (S_{BET}) was determined with a surface area analyzer (Quantachrome, Nova 4200e) by the Brunauer-Emmett-Teller method. Functional group vibrations were confirmed by using a Fourier transform infrared spectrometer (FTIR; JASCO, FT / IR 4200) with a diffuse reflectance accessory (JASCO, DR-81). Fluorescence spectra were obtained using a photoluminescence spectrometer (JASCO, FP-8500).

Photocatalytic measurement

Before evaluation of the photocatalytic activity, each sample was irradiated with UV light using a black light (UVP, XX-15BLB) in order to remove organic contaminants on the sample. The photocatalytic activity of the sample was evaluated by decomposition of Rhodamine B (Rh B). Photocatalytic reaction was performed in a reactor vessel (SIBATA, SPC 15). Five milligrams of powder, which has complete extinction of incident radiation, was dispersed in Rh B aqueous solution (100 mL, 4 mg L⁻¹, pH=6.84). Before the photocatalytic reaction, Rh B solution should be magnetically stirred with the catalyst in dark for 1 hour to establish an adsorption and desorption balance. The light irradiation system contains a 500 W Xe lamp (USHIO Optical Modulex SX-U1501XQ) with cutoff filter L42 for visible light ($\lambda_{\text{max}} \geq 420\text{nm}$) and a heat absorbing glass to removing heat effects. The intensity of the corresponding incident light is 50 mW/cm². The degradation efficiencies of Rh B were evaluated using the UV-vis absorption spectra to measure the peak value of a maximum absorption of Rh B solution ($\lambda_{\text{max}} = 554\text{nm}$). During the irradiation, about 3

mL of suspension was continually taken from the reaction cell at given time intervals for subsequent target dye concentration analysis after centrifuging.

Photodegradation of Rh B over the bulk $g\text{-C}_3\text{N}_4$ and $\text{Hx-C}_3\text{N}_4$

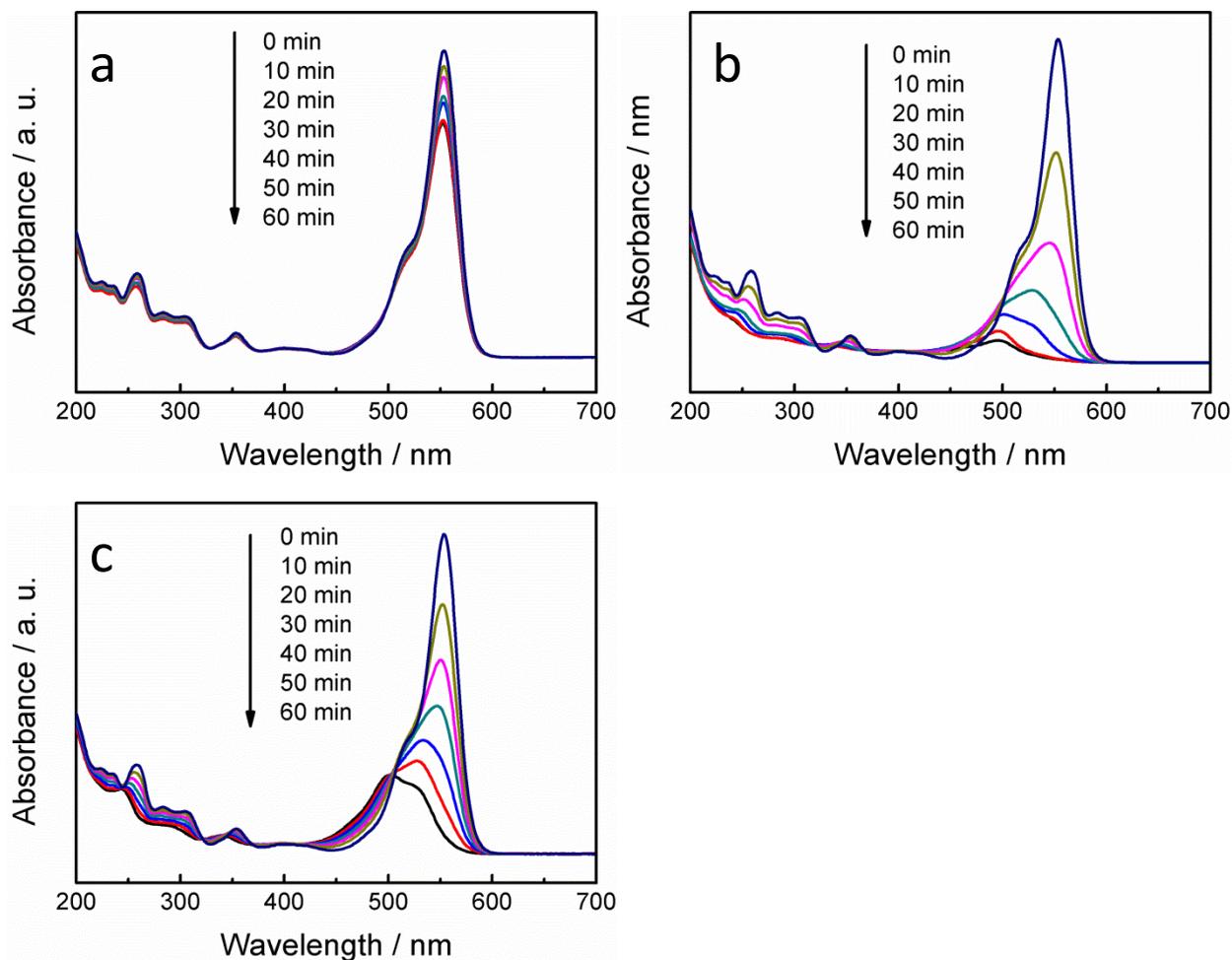


Figure S1 UV-vis spectroscopic changes of photodegrading Rh B over (a) bulk $g\text{-C}_3\text{N}_4$, (b) $\text{H12-C}_3\text{N}_4$ and (c) $\text{H24-C}_3\text{N}_4$ samples under visible light irradiation ($\lambda \geq 420$ nm).

Tests of stability

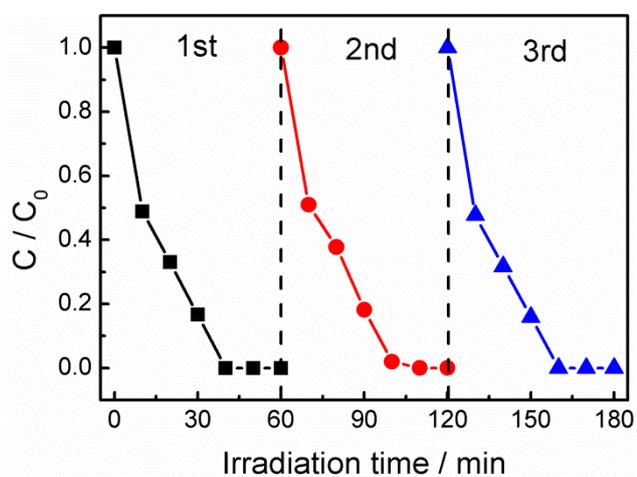


Figure S2 Cycling runs of Rh B degradation over $\text{H12-C}_3\text{N}_4$ under visible light irradiation. C_0 : initial

concentration.

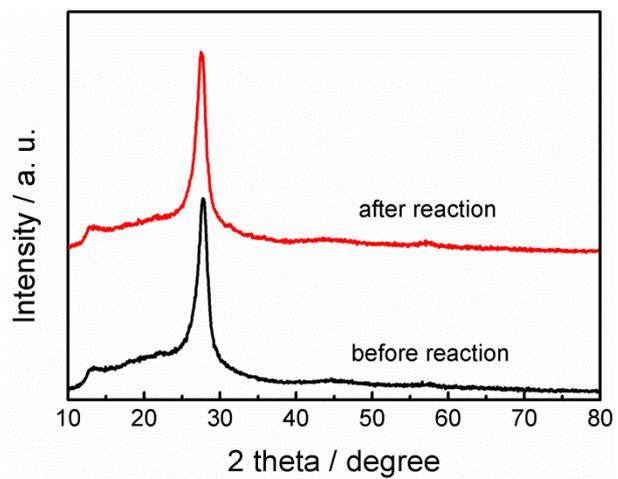


Figure S3 XRD patterns of H12-C₃N₄ before and after reaction.