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Electronic Supplementary Information

Ultra-rapid synthesis of 2D graphitic carbon nitride nanosheets via direct microwave heating for field emission

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1. Experimental section

1.1 Preparation of 2D g-C_3N_4 nanosheets

The synthesis of 2D g-C_3N_4 nanosheets was performed in a multimode microwave vacuum sintering furnace (Jiequan Microwave Co., China). By adjusting the irradiation power, which is in the range of 0~10 kW, the temperature of raw materials can be ultra-rapid heated to a few hundred degrees centigrade in tens of seconds. The temperatures were measured by an optical pyrometer (Reytek) on the top of insulation glass.

The 2D g-C_3N_4 nanosheets were successfully prepared via direct microwave heating of melamine and carbon fiber. In a typical synthesis, 10 g melamine and 0.5 g carbon fiber were taken into the ceramic crucible. The crucible together with the

specimens was placed at the center of a commercial microwave vacuum sintering furnace, the furnace cavity was then evacuated to below 1.5×10^4 Pa by a water circulating pump. After the microwave heating with irradiation power of 5.0 kW for 5 min**,** the light yellow 2D g-C_3N_4 nanosheets were obtained. During the microwave irradiation process, the highest temperature detected by optical pyrometer was approximately 574 ºC**.**

1.2 Characterizations

Powder X-ray diffraction (XRD) patterns were recorded with a X'TRA X-ray diffractometer with Cu Ka radiation (λ = 1.5418 Å). The accelerating voltage and the emission current were 40 kV and 35 mA, respectively. Data were collected over the 2θ range of 5-80°, with a step width of 0.02° and a scan rate of 10 °/min. Flourier transformed infrared (FT-IR) spectra were obtained by a Nicolet 870 FT-IR spectrometer. The sample discs were prepared by the KBr pellet technique (1% of sample in dried KBr). Field emission scanning electron microscopy (FE-SEM) with the type of XL FEGSFEG-SIRION (FEI Ltd., Netherlands) was applied to investigate the morphological features of the obtained g-C_3N_4. Before investigation, the samples were placed on a sample holder by using conductive adhesive and then coated with a thin layer of platinum. Transmission electron microscopy (TEM) investigation was performed with a FEI Tecnai T20 microscopy (FEI, Eindhoven, Netherlands) operated at 200 kV acceleration voltages using a LaB6 filament. The images were recorded using a Megaview III CCD camera. Atomic force microscopy (AFM) measurement was carried out with a type of Dimension ICON (Veeco, USA). The UV-vis spectra were collected with a Cary 5000 UV-vis diffuse reflection

spectrophotometer. Photoluminescence (PL) spectrum was measured using a Hitach F-7000 typed fluorescence spectrophotometer at room temperature, with the excitation light of 350 nm from xenon lamp. The specific surface area was measured by using an Autosorb-IQ2 adsorption instrument (Quantachrome Instruments, USA) and calculated by the Brunauer-Emmett-Teller (BET) equation. The field emission properties of product were measured in a vacuum chamber at a pressure of ~6.0×10^{-5} Pa. The current-voltage (I-V) curves were tested on a self-made measurement system. The distance between the cathode and the anode was fixed at 2 mm.

1.3 Field emission performance measurement

All the chemicals were used without any further purification. In brief, 0.2 g of Ethyl cellulose (AR) was gradually taken into 20 mL of terpilenol (AR) at 90 °C with magnetic stirring for 1 h to completely dissolve. Subsequently, 20 mg of 2D g-C_3N_4 nanosheets were added into the mixed solution, and the mixture was still magnetically stirred for 2 h to well disperse. After cool down, the mixture was coated onto a copper substrate as the cathode. The copper substrates were cleaned ultrasonically in ethanol and acetone consecutively before coating. The field emission characteristics of the 2D g-C_3N_4 nanosheets coating were carried out in a vacuum chamber at a pressure of ~6.0×10^{-5} Pa.

2. Results and discussion

2.1 Results of characterization

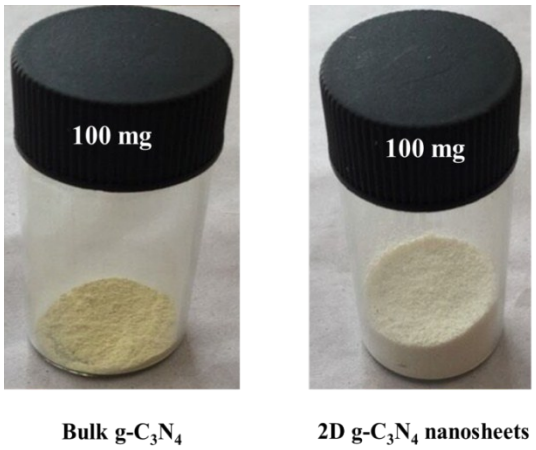


Fig. S1 The digital photographs of bulk g-C_3N_4 and 2D g-C_3N_4 nanosheets.

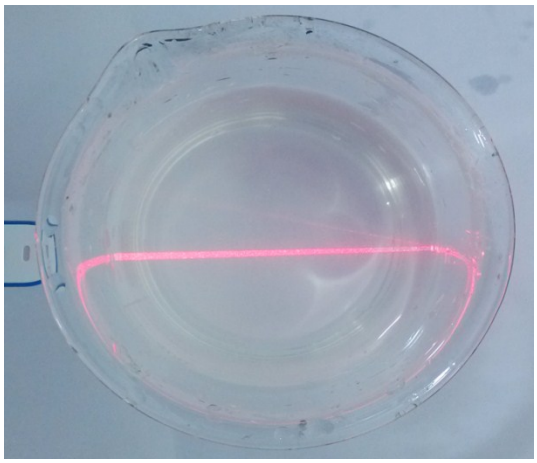


Fig. S2 Tyndall effect exhibition of 2D g-C_3N_4 nanosheets solution irradiated with red laser light.

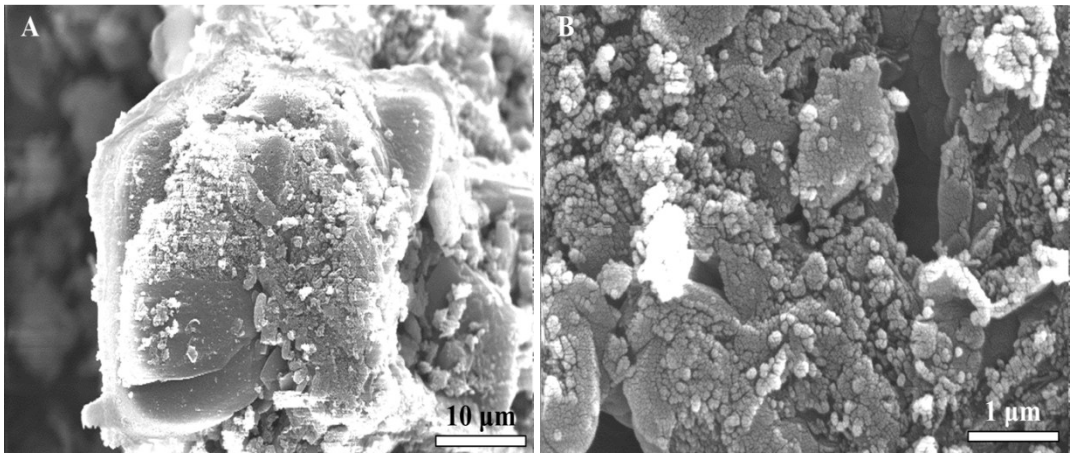


Fig. S3 SEM images of bulk g-C_3N_4 with Low- (A) and high- (B) magnification ratio.

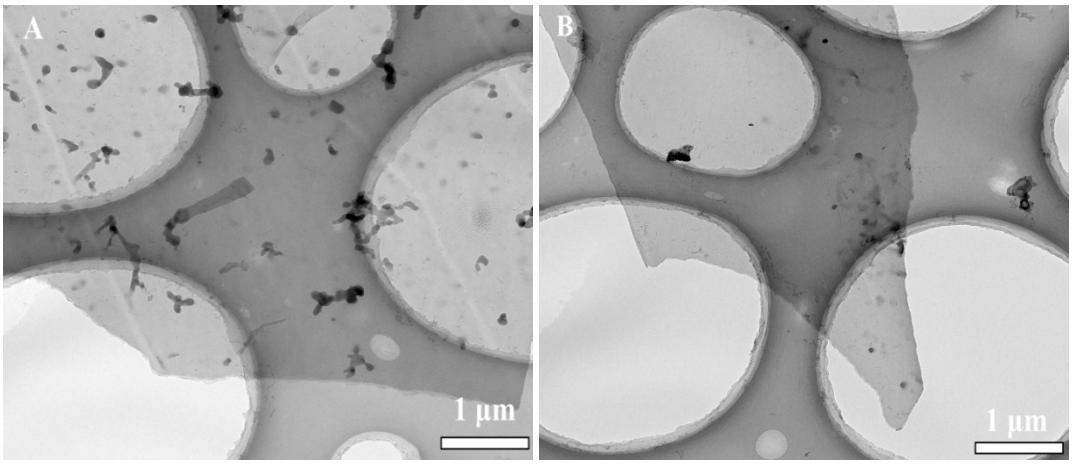


Fig. S4 TEM images of 2D g-C_3N_4 nanosheets.

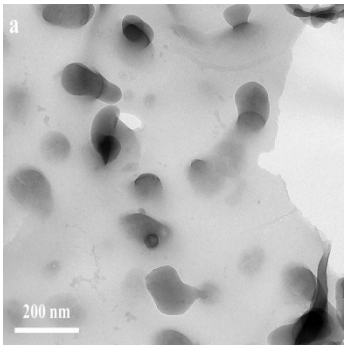
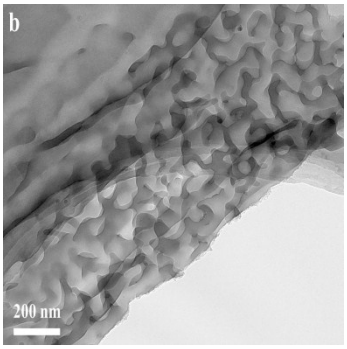


Fig. S5-a The initial structure of pyramid-like bumps (black spots shown in Fig. S4) on the surface of 2D g-C_3N_4 nanosheets synthesized with irradiation power of 5.0 kW for 5 min.



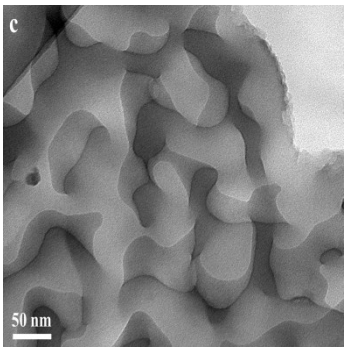


Fig. S5-b and **S5-c** The subsequent growth of these bumps on the surface of 2D g-C_3N_4 nanosheets synthesized with irradiation power of 5.0 kW for 7 min. and 9 min.

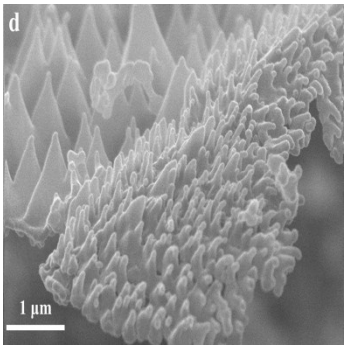


Fig. S5-d The array of the pyramid-like bumps on the surface of 2D g-C_3N_4 nanosheets synthesized with irradiation power of 5.0 kW for 7 min.

Fig. S5 The formation of the array of pyramid-like bumps on the 2D-g-C_3N_4 nanosheets in the different reaction time

2.2 The formation mechanism of 2D-g-C_3N_4 nanosheets

Under the high-energy microwave irradiation, plasma and electric arc might be produced, resulting in the formation of carbon atoms or carbon atom clusters. During the polycondensation reaction of melamine, these carbon atoms characterized with high reaction active also participate in the formation of g-C_3N_4 covering the formation of C–N bonds and the primary nucleation. By means of microwave heating, the temperature can rise to approximately 574 ºC in a few minutes. Results indicate that such temperature is a proper temperature for the synthesis of 2D g-C_3N_4 nanosheets via some special thermal polycondensation reactions.