Data Article

Dataset of emission and excitation spectra, UV–vis absorption spectra, and XPS spectra of graphitic C3N4

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In this data article, the normalized emission and excitation spectra, the ultraviolet-visible (UV–vis) absorption spectra, and the X-ray photoelectron spectroscopy (XPS) of bulk-powders and nano-structured graphitic C3N4 (g-C3N4) were presented, which are helpful to get insight into the crystal and electronic structures of g-C3N4, especially on determining the energy levels and the mechanisms of luminescence originating from electron transitions. This data article is related to our recent publication (He et al., in press) [1]. The absorption, excitation and emission spectra are vital to illustrate the optoelectronic performances in terms of photoluminescence, photocatalysis,

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The electron transitions and their corresponding energy levels could be identified from the normalized emission and excitation spectra.

- The fast relaxation of electrons from excited states to the ground state without the Stokes shift radiation could be discriminated by comparing absorption spectra with emission spectra together.
- The XPS data are helpful to reason out the way and reaction process of the thermal condensation of melamine to form g-C₃N₄ and thereby, to illustrate the performances from viewpoint of crystal structure.
- The emission, excitation and absorption spectra of g-C₃N₄ are helpful to recognize its electronic structure.
- The electronic and crystal structures are useful to interpret the intrinsic properties of g-C₃N₄ in terms of photoluminescence, electroluminescence, photocatalysis, etc.

1. Data

The normalized emission spectra of g-C₃N₄ powders synthesized at various temperatures in air and N₂ atmospheres are shown in Fig. 1a,b, respectively, which shows that the emission peaks red-shift with temperature increasing from 450 to 600 °C. The asymmetrical emission band mainly consists of the π*-Lp (lone pair electrons) and π*-π transitions. The normalized excitation spectra of g-C₃N₄ powders synthesized at various temperatures in air and N₂ atmospheres are displayed in as Fig. 1c,d, in which the excitation peaks at 339, 375, 399 and 431 nm were attributed to the LP–δ* (* indicates the antibond), LP–π*, π–π*, and traps absorption, respectively.
The normalized emission and excitation spectra of g-C₃N₄ quantum dots (QDs) are presented in Fig. 2. After being exfoliated into QDs, the emission and excitation peaks shift towards high energy direction (i.e., blueshift) evidently, as seen by the comparison of Fig. 2a–d with Fig. 1a–d. Besides, the δ*–LP transition peaked at 405 nm was observed in Fig. 2b for the sample synthesized at 450 °C under N₂ atmosphere, suggesting the electrons cannot relax from the high-energy δ* state to the low-energy π* state efficiently. Maybe, the π orbital is not well formed under the condition of 450 °C in N₂ atmosphere.

The absorption spectra of g-C₃N₄ bulk powders synthesized at various temperatures in air and N₂ atmospheres, respectively, are shown in Fig. 3. Besides the LP–δ*, LP–π*, and π–π* transitions as correspond to the excitation bands in Fig. 2a, b, one band peaked at about 266 nm was observed in Fig. 3a, b, which was attributed to the charge effect band (i.e., photocurrent, marked with CTB). The CTB was nearly not observed under 450 °C. However, the increase of CTB in intensity with an increase of temperature from 500 to 650 °C suggests that the exorbitant condensation of the g-C₃N₄ easily result in the photocurrent, which can explain the decrease of g-C₃N₄ luminescence upon increasing temperature from 500 to 650 °C. Moreover, the absorption within 440–600 nm increases with temperature increasing from 450 to 650 °C. The position of this absorption band is consistent with the
absorption wavelength of traps in Fig. 1c, d. So, it is naturally to assign the absorption band within 440–600 nm to crystal defects. Besides, the absorption within 440–600 nm overlaps with the emission band very well, as comparison of absorption spectra with emission spectrum displayed in Fig. 3a, b. Therefore, the absorption within 440–600 nm is mainly caused by the fast relaxation of electrons from the high-energy excited states to the ground band without the Stokes shift, overlaying with the absorption of crystal traps. After being exfoliated into QDs, the absorption bands of CTB and fast relaxation disappear, as seen from Fig. 3c, d.

The intensity of the LP-δ*, and π–π* absorptions of g-C₃N₄ QDs in Fig. 3c, d are far weaker over those of bulk powders in Fig. 3a, b, suggesting the π orbital was partially damaged or broken during the process of ultrasonic exfoliating and in turn resulting in electrons cannot relax from high-energy δ* to low-energy π* state efficiently. Accordingly, the LP-δ* transition dominates the absorption of g-C₃N₄ QDs in Fig. 3c, d.
The raw C 1s and N 1s XPS of g-C₃N₄ bulk powders and their fitted spectra by using the XPS PEAK 4.1 program are depicted in Fig. 4 and Fig. 5, respectively. The composition of C, N and O elements for the samples synthesized at various temperature in air and N₂ atmospheres was summarized in Table 1. The analyses on XPS spectra in Figs. 4 and 5 confirm that the structure of g-C₃N₄ consists of basic unit of tri-s-triazine ring, which is connected by the N atoms to form a π-conjugated polymeric network [2–8].

Moreover, the highest ratio of graphitic-to-triazine carbon was observed in the sample synthesized at 500 °C in N₂ atmosphere and the second highest ratio of graphitic-to-triazine carbon was observed in the sample synthesized at 450 °C in air ambient. These ratios are consistent with the strongest and the second strongest luminescence of g-C₃N₄ powders presented in Fig. 2 and Fig. 3, respectively, in Ref. [1], indicating the luminescence efficiency is related with the type of carbon existence closely.

2. Experimental design, materials and methods

The material and methods used to obtain the data of emission and excitation, absorption, and XPS spectra were described in [1]. The emission and excitation spectra of bulk-powders and nano-structured g-C₃N₄ were collected with Hitachi F4600 spectrometer, and the spectra in Figs. 1 and 2

![Fig. 3. The comparison of absorption spectra with emission spectra for the bulk powders and quantum dots: a,b, the bulk powders synthesized at variant temperatures in Air and N₂ atmospheres, respectively; c,d, the quantum dots obtained by ultrasonic exfoliating the bulk powders in a,b.](image-url)
Fig. 4. The survey, C 1s, N 1s, and O 1s XPS spectra of bulk g-C₃N₄ powders synthesized at variant temperatures in air and N₂, respectively.

the survey analysis on XPS spectra of bulk g-C₃N₄ synthesized in air (left) and N₂ (right).

the C 1s XPS spectra of bulk g-C₃N₄ synthesized in air (left) and N₂ (right).
the N 1s XPS spectra of bulk g-C₃N₄ synthesized in air (left) and N₂ (right).

the O 1s XPS spectra of bulk g-C₃N₄ synthesized in air (left) and N₂ (right).

Fig. 4. (continued)
were normalized to determine the energy levels. The absorption spectra in Fig. 3, recorded with UV-3600 spectra, were further in comparison with emission spectra to determine electron transition and the mechanisms thereof. The original XPS spectra, including the survey, C 1s, N 1s, and O 1s, of bulk g-C$_3$N$_4$ powders, measured with using the Thermo ESCALAB250Xi X-ray Photoelectron Spectrometer, were displayed in Fig. 4. The C 1s and N 1s XPS were fitted by using the XPS PEAK 4.1 program, as shown in Fig. 5, to reveal the way of thermal condensation and chemical bonding.

![Fig. 5. The fitted C 1s and N 1s XPS spectra, as corresponding to Fig. 4b,c, by using the XPS PEAK 4.1 program, of g-C$_3$N$_4$ powders synthesized at variant temperatures in air and N$_2$, respectively.](image-url)
Table 1

the percent of C, N and O atoms in bulk g-C₃N₄ powders obtained by fitting the XPS spectra in Fig. 4 by using the XPS PEAK 4.1 program, as corresponding to Fig. 5.

| Items | 450 °C | 500 °C | 550 °C | 600 °C | 650 °C | Conditions       |
|-------|--------|--------|--------|--------|--------|-----------------|
| C1s   | 42.04  | 42.91  | 42.14  | 42.12  | 41.42  | Synthesized in Air |
| N1s   | 51.8   | 52.62  | 54.01  | 53.63  | 54.58  |                 |
| O1s   | 6.16   | 4.47   | 3.85   | 4.26   | 4      |                 |
| C1s   | 39.44  | 43.55  | 41.81  | 41.91  | 41.83  | Synthesized in N₂ |
| N1s   | 55.95  | 52.78  | 54.68  | 54.66  | 54.45  |                 |
| O1s   | 4.61   | 3.68   | 3.51   | 3.43   | 3.72   |                 |
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Transparency document. Supplementary material

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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at https://doi.org/10.1016/j.dib.2018.09.123.

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