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Preparation of Water-Dispersible Porous g-C\textsubscript{3}N\textsubscript{4} with Improved Photocatalytic Activity by Chemical Oxidation

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Hydrophilic treatment of the bulk graphene-like carbon nitride (g-C\textsubscript{3}N\textsubscript{4}) for future applications has aroused extensive interest, due to their enhanced specific surface area and unusual electronic property. Herein, water-dispersible g-C\textsubscript{3}N\textsubscript{4} with a porous structure can be obtained by chemical oxidation of the bulk g-C\textsubscript{3}N\textsubscript{4} with K\textsubscript{2}Cr\textsubscript{2}O\textsubscript{7}/H\textsubscript{2}SO\textsubscript{4}. Acid oxidation results in the production of hydroxyl and carboxyl groups to its basal plane, and the formation of porous structure of the g-C\textsubscript{3}N\textsubscript{4} at the same time. The porous g-C\textsubscript{3}N\textsubscript{4} appears as networks with tens of micrometers in width and possesses a high specific surface area of 235.2 m\textsuperscript{2} g\textsuperscript{-1}. The final concentration of the porous g-C\textsubscript{3}N\textsubscript{4} can be up to 3 mg mL\textsuperscript{-1}. Compared with the bulk g-C\textsubscript{3}N\textsubscript{4}, the as-obtained porous g-C\textsubscript{3}N\textsubscript{4} exhibits excellent water dispersion stability and shows great superiority in photoinduced charge carrier separation and transfer. The photocatalytic activities towards organic pollutants degradation of the porous g-C\textsubscript{3}N\textsubscript{4} are much higher than those of the bulk due to the larger band gap (by 0.2 eV) and specific surface areas.

1. Introduction

Global researches on visible-light-active photocatalysts for environmental protection and solar fuels have attracted lots of attention recently. Among the various catalytic materials, graphitic carbon nitride (g-C\textsubscript{3}N\textsubscript{4}) is a metal-free photocatalyst with appropriate band edges and a large band gap of 2.70 eV, which can catalyze a number of reactions, such as photodegradation of organic pollutants or photochemical splitting of water. However, the bulk g-C\textsubscript{3}N\textsubscript{4} materials prepared by direct polycondensation from suitable precursors, including melamine, thiourea and guanidine hydrochloride (GndCl), generally have very low surface areas (less than 10 m\textsuperscript{2} g\textsuperscript{-1}) and poor photocatalytic activity. Despite the new modification methods, for instance, morphology control, coupling with other materials or element doping, the insoluble property of carbon nitrides in common solvents (water, ethanol, acetone and so on) also makes further functionalization inconvenient and limits their applications.

The studies on the structure of g-C\textsubscript{3}N\textsubscript{4} have indicated that there are stronger covalent C-N bonds instead of C-C bonds in each layer. The planar cohesion between strands of polymeric melon units is mainly attributed to hydrogen bonding, which is less strong compared with the covalent bonding cohesion in graphite. Motivated by the liquid-phase exfoliation methods to prepare graphene oxide, researchers have successfully exfoliated the bulk g-C\textsubscript{3}N\textsubscript{4} into water-dispersible C\textsubscript{3}N\textsubscript{4} nanosheets of a high quality. The exfoliation techniques involves intercalation and surface passivation by solvent molecules (H\textsubscript{2}O, isopropanol, 1,3-butylene glycol, and H\textsubscript{2}SO\textsubscript{4}). Layer materials could adsorb solvent molecules into the spacing between layers. The intercalation of solvents between the layers increases the layer spacing and weakens the interlayer attraction. Subsequent treatment such as sonication or thermal shock can result in exfoliation. If the surface energy of the solvent matches with that of bulk g-C\textsubscript{3}N\textsubscript{4}, the energy disparity between aggregated and exfoliated states will be very small. The as-obtained nanosheets are then stabilized as exfoliated dispersion. However, these strategies require a rather long sonication time (10-16 h), and the yield is relatively low (0.15-0.35 mg mL\textsuperscript{-1}). Recently encouraged by the protonation method used to disperse CNTs, Wang et al. firstly reported the reversible protonation of carbon nitride by stirring it with strong mineral acids (37% HCl) for 3 h. The protonation method provides better dispersion in water and a higher surface area. The BET surface area of the pronated g-C\textsubscript{3}N\textsubscript{4} has increased from 8 to 30 m\textsuperscript{2} g\textsuperscript{-1}. Then Qiao et al. have prepared proton-functionalized g-C\textsubscript{3}N\textsubscript{4} nanosheets, which exhibit large specific surface area of 305 m\textsuperscript{2} g\textsuperscript{-1}.

Although different methods have been developed to produce water-dispersible g-C\textsubscript{3}N\textsubscript{4}, the chemical oxidation of bulk g-C\textsubscript{3}N\textsubscript{4} has rarely been reported in literature. As we know, various oxidants have been used to react with carbon nanotubes or graphene in the liquid-phase oxidation methods, including H\textsubscript{2}O\textsubscript{2}, HNO\textsubscript{3}, KMnO\textsubscript{4}, K\textsubscript{2}Cr\textsubscript{2}O\textsubscript{7}, K\textsubscript{2}SO\textsubscript{4} and so on. Treatment with oxidizers results in the production of hydroxyl and carboxyl groups on the basal plane, and contributes to water intercalation and its hydrophilicity. The nature of the reactants and oxidants would have a great impact on the degree of oxidation. For example, the widely used Hummers method is not suitable for the exfoliation of g-C\textsubscript{3}N\textsubscript{4}. 

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The strong oxidizing acid, KMnO$_4$/H$_2$SO$_4$, is more capable of making ortho hydroxyls and would seriously destroy the planar atomic structure of g-$C_3N_4$, only leading to the formation of carbon nitride based particles. Considering the special structure of g-$C_3N_4$, it is worth anticipating that oxidants with a lower redox potential (K$_2$Cr$_2$O$_7$/H$_2$SO$_4$) could be used to allow the hydrophilic modification and avoid heavy structure destruction.

Herein, we report a facile method to prepare dispersible g-$C_3N_4$ by chemical oxidation of bulk g-$C_3N_4$ at room temperature. The obtained g-$C_3N_4$ possesses a porous structure with a large surface area (235.2 m$^2$ g$^{-1}$) and appears as networks with tens of micrometers in width. Due to the oxygen-containing functional groups on its surface, the final concentration of the g-$C_3N_4$ solution can even be up to 3 mg mL$^{-1}$. The crystal structure, chemical states and electronic structure of the resultant porous g-$C_3N_4$ have been thoroughly studied. Moreover, the porous g-$C_3N_4$ has an efficient photocatalytic activity towards organic pollutants degradation.

2. Experimental section

2.1 Synthesis of porous g-$C_3N_4$

g-$C_3N_4$ was prepared by heating dicyandiamide in a muffle furnace at 550 °C for 4 h with a ramp rate of 2.3 °C/min. The porous g-$C_3N_4$ was synthesized via the chemical oxidation of the bulk g-$C_3N_4$ with K$_2$Cr$_2$O$_7$/H$_2$SO$_4$. 10 g of K$_2$Cr$_2$O$_7$ was mixed with 50 mL of H$_2$SO$_4$ (98 wt%) in a 100-mL flask and stirred till the solution became brown. 0.5 g of the bulk g-$C_3N_4$ was added into the solution and stirred thoroughly for 2 h at room temperature. Then the mixture was slowly poured into 400 mL of deionized water and cooled to room temperature. After centrifugation at 6000 rpm, the obtained light yellow solid was dialedized in a dialysis bag to remove all residual acids, and dispersed in water for sonication (1–2 h). Finally, the obtained milk-white suspension was centrifuged at 3000 rpm to remove undispersed g-$C_3N_4$. The supernatant liquid could be supercritical dried for future use.

2.2 Characterization of the samples

The X-ray powder diffraction (XRD) patterns were recorded using a D8 advance X-ray diffractometer (Bruker Company, Germany) in transition mode and Cu K radiation ($\lambda = 1.54056$ Å). The X-Ray photoelectron spectroscopy (XPS) was performed on a Kratos Axis Ultra-DLD X-ray photoelectron spectrometer (Shimadzu, Kyoto, Japan). The UV-Vis absorption spectroscopy and room-temperature UV-Vis diffuse reflectance spectroscopy (DRS) were taken at room temperature on a UV-3150 spectrophotometer (Shimadzu, Kyoto, Japan). The photoluminescence (PL) spectroscopy was performed by a RF-5301pc fluorescence spectrometer (Shimadzu, Kyoto, Japan). The Fourier transform infrared (FT-IR) experiment was carried out on a Nicolet Nexus 470 FT-IR spectrometer in the wavenumber range of 400 to 4,000 cm$^{-1}$. The samples were prepared in the form of pellets together with KBr. Raman spectra were recorded on a SPEX403 laser Raman spectrometer (JY, France) at room temperature using the 785 nm line of an argon ion laser as the excitation source.

The transmission electron microscope (TEM) images were acquired on a FEI Tecnai G20 field emission electron microscope operating at an accelerating voltage of 200 kV. The field emission scanning electron microscopy (FESEM) images were taken on an Ultra 55 scanning electron microscope. N$_2$ and CO$_2$ adsorption and desorption isotherms were collected on an Autosorb-IQ$_2$ Quantachrome Instrument at 77 K and 273 K, respectively. Ultra-high purity N$_2$ and CO$_2$ (99.995 %) were purchased and used as received. The total pore volumes were estimated from the adsorbed amount at a relative pressure P/P$_0$ of 0.995. The Mott-Schottky experiments were taken on an electrochemical workstation (CHI660D, Shanghai Chenhua, China). The perturbation signal was 20 mV with the frequency at 0.5 kHz.

2.3 Photocatalytic activity

The photocatalytic activities were evaluated by the degradation of RhB in a 100-mL quartz reactor with a circulating water system to maintain a constant temperature. A 300 W Xe lamp (BL-GHX-Xe-300k, Shanghai Bilon) with a 420 nm cutoff filter was used as the light source. In each run, 25 mg of the sample was mixed with 50 mL of RhB solution (10 mg mL$^{-1}$). Prior to irradiation, the suspension was magnetically stirred in the dark for 100 min to reach the absorption-desorption equilibrium of RhB on the catalyst. The suspension was collected at an interval of 20 min and detected.

3. Results and discussions

3.1 Mechanism of the Formation of the Porous g-$C_3N_4$

The chemical oxidation method has resulted in the formation of porous structure and hydrophillicity of g-$C_3N_4$ at the same time. The synthetic strategy is illustrated in Fig. 1. Typically, after acid treatment, various hydrophilic functional groups (e.g. –COOH, –OH, –C=O) can be generated at the open ends or defect sites of the bulk g-$C_3N_4$ structure (Fig. 1A). The intercalation and exfoliation of the pristine g-$C_3N_4$ by these oxygen-containing groups could improve the hydrophillicity of g-$C_3N_4$. Then the mixture was slowly poured into water, and the producing heat made the g-$C_3N_4$ immediately expand into large floc floatings, which is poorly dispersible in highly acidic solutions.
The mechanism of oxidation is based on previous work about the oxidation of carbon nanotubes by K₂Cr₂O₇/H₂SO₄. The redox potential of K₂Cr₂O₇ is 1.679. Due to the appropriate oxidizing ability of K₂Cr₂O₇/H₂SO₄, the slow oxidative etching process could improve the formation of the porous structure, while the two-dimensional (2D) plate-like structure of g-C₃N₄ is still well-preserved.

3.2 Crystal Structure and Chemical States of the Porous g-C₃N₄

The crystal and chemical structure of the porous g-C₃N₄ and the bulk g-C₃N₄ are studied by XRD, XPS, FTIR and Raman spectra. In the XRD patterns presented in Fig. 2A, the low-angle reflection peak at 13.1° stems from the interplanar structural packing motif, such as the hole-to-hole distance. The strong XRD peak (002) of the bulk g-C₃N₄ at around 27.56° originates from the characteristic interlayer stacking reflections like in graphite. With respect to the bulk g-C₃N₄, the (002) peak of the porous g-C₃N₄ is slightly broadened and shifted from 27.56° to 27.91° which indicated a decreased gallery distance between the layers. In our case, the oxidized layers can be planarized by the π-π stacking and hydrogen bonding interactions, which would lead to a denser packing and thus shorten the gallery distance. Moreover, a decrease in the overall intensity can be observed in the porous g-C₃N₄. This change of XRD patterns are similar with that of mesoporous g-C₃N₄ prepared by using SiO₂ nanoparticles or mesoporous SiO₂ as hard templates, which reflects the effect of porous microstructure on the geometric confinement.

Strong evidence of oxygen species in the porous g-C₃N₄ was firstly confirmed by XPS. As shown in Fig. 2B, the profile of bulk g-C₃N₄ does not need to be applied with the Stokes deconvolution method due to its symmetrical nature. The O 1s core level at 532.6 eV is ascribed to the absorbed water and no signal related to the O=C or C=O bond is observed. After acid treatment, line a in Fig. 2B shows a higher density of oxygen-containing species on the porous g-C₃N₄ surface. Also, a slope peak of higher values appears, indicating oxidation-induced acid-bonding characteristics. This main peak could be deconvoluted into four Gaussian-Lorentzian peaks. Three new core levels at 530.7 eV, 531.8 eV and 533.1 eV have been detected, contributed from O=C=O, C=O and O-C=O groups respectively. These signals suggest that the oxygen species are presented not simply as absorbed water but various functional groups on the surface, as a result of the acid treatment of bulk g-C₃N₄. The calculated atomic percentage in the fitting data of O=C=O at 533.1 eV is 28.9 %, C=O at 531.8 eV is 36.2% and O=C=O at 530.7 eV is 16.8%.

The C 1s spectra of the bulk g-C₃N₄ show three peaks located at about 288.0 eV, 286.1 eV and 284.6 eV. The main contribution peak centered at 288.0 eV could be further deconvoluted into two Gaussian-Lorentzian peaks which are originated from sp² hybridized carbon bonded to N in C-N-C coordination. The peak at 284.6 eV is typically assigned to graphitic sp² C=C bonds, while the lowest peak at 286.1 eV is attributed to the C-NH₂ species. In addition, a new-generated peak at 289.1 eV has been shown in the porous g-C₃N₄, which could be assigned to the COOH species. Meanwhile, the peak in the N 1s spectra can also be deconvoluted into three different peaks at around 398.5 eV, 399.8 eV and 401.1 eV. The peaks are ascribed to sp³ N atoms in triazine rings (C-N=C), bridging N atoms (N-(C)_3) and amino groups carrying hydrogen (C-NH) separately. Another peak at 404.3 eV is due to the charging effect.
the electrophilic effect of the oxygen atoms in new C-O bonds adjacent to C-N.

Raman spectroscopy is also used to confirm the presence of oxygen species in the porous samples. A comparison between the bulk g-C3N4 and porous g-C3N4 has been shown in Fig. 3B. Obviously, the characteristic bands can be observed on both materials5, 16. while a new broad band at 1257 cm\(^{-1}\) has replaced the old peak. According to Lebrun et al.’s work,45, 46, this replacement might be ascribed to the OH in-plane bending mode in C-OH, which further supported our assumptions about the FTIR results.

To further confirm the oxidation process of the resultant porous g-C3N4, several confirmatory experiments have been done as shown in Fig. 3C and 3D. Adding Fe\(^{3+}\) or dendrimer aqueous solution directly into the as-prepared porous g-C3N4 dispersion has caused obvious floccules. Either coordination of Fe\(^{3+}\) with the oxygen-containing functional groups on porous g-C3N4, or electrostatic interaction between the protonated amino groups on dendrimer and the oxygen-containing functional groups is assumed to be the driving force for the above phenomenon.47 Moreover, no floccules appeared when adding Fe\(^{3+}\) solution into the water outside the dialysis bag, which excluded the interaction between metal ions and the very few residual acids. Furthermore, the floccules would disappear after injecting an appropriate amount of NaOH solution into the dendrimer-g-C3N4 solutions, which should be owing to the fact that alkali can destroy the electrostatic interaction by releasing these oxygen-containing functional groups. All the results mentioned above could support the oxidation process of the porous g-C3N4 with oxidizer.

3.3 Morphology of the Porous g-C3N4

The morphology of the porous g-C3N4 was investigated with TEM as shown in Fig. 4. The bulk material displays a typical dense and stacked lamellar structure of several micrometers (Fig. 5A, Fig. S2), while the porous g-C3N4 was found to have agglomerated into non-uniform networks with tens of micrometers. The formation of networks might be driven by the π-π stacking effect between the layers and hydrogen bonding interactions mediated by the oxygen-containing groups (–COOH, -OH, -C=O).38 The TEM images with higher magnification have shown that the structure is still graphene-like and a slightly less dense distributed with disordered pores, which is in agreement with the less intense XRD peaks shown in Fig. 2A. The high resolution transmission electron microscopy (HRTEM) has indicated that the etching edges are rather rough. The pores might be caused by the oxidative etching of the melon units on the edge or defect sites of the bulk g-C3N4 layers, which is similar to the disordered pores in samples prepared by template methods.31 Chen et al.11 have also demonstrated that the oxygen-doped g-C3N4 appears with hierarchical edges due to the H2O2 hydrothermal treatment.

The N2 adsorption isotherms have shown that the measured surface area of the porous g-C3N4 is 235.2 m\(^2\) g\(^{-1}\), which is almost 26 times larger than that of bulk g-C3N4 (9.2 m\(^2\) g\(^{-1}\)) (Fig. 4E). Their corresponding pore diameter distributions were calculated by BJH method. A centred peak at 10 nm of the porous g-C3N4 is observed on the pore-size distribution curve, which should be attributed to the random pores formed by chemical etching. The extended wide peak at 25~100 nm might owe to the pores in the networks formed by stacking effect. The BET surface area is comparable compared with those mesoporous g-C3N4 materials synthesized via the nanocasting methods or g-C3N4 nanosheets prepared by exfoliation methods.5, 15, 16, 31 Besides, the CO2 adsorption performance at 0 °C was also investigated shown in Fig. 4F. Compared with the bulk samples, the porous g-C3N4 presents a higher CO2 capture capacity of 1.20 mmol g\(^{-1}\) at 0 °C. It is reported that the nitrogen-containing groups in mesoporous carbon nitride materials can provide lewis base active sites for absorbing CO2.49 In our work, the porous structure in the g-C3N4 is beneficial to CO2 capture capacity due to the capillary

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Fig. 3 FTIR spectra (A) and Raman spectra (B) of the bulk g-C3N4 and the porous g-C3N4. The photograph of identification tests by Fe\(^{3+}\) solution (C) and dendrimer solution (D).

Fig. 4 (A-D) Typical TEM images with different magnification of the as-prepared porous g-C3N4 reacting for 2 h. (E) The N\(_2\) and (F) CO\(_2\) adsorption isotherm recorded for the bulk g-C3N4 and the porous g-C3N4. Inset of (E) is the pore size distribution for the bulk and porous g-C3N4.
condensation effect. Besides, owing to the high surface area, more basic sites could be provided, which would improve more CO$_2$ binding. As a result, the adsorption performance of the porous materials is higher than that of the bulk g-C$_3$N$_4$. In addition, the reaction time is also very important in the formation of porous structures. When the oxidation time of the bulk g-C$_3$N$_4$ is extended to 12 h, the obtained products are presented as small patches hanging over the walls of lacy support films shown in Fig. 5B. The planar structure has been seriously destroyed which might be resulted from the defect-consuming step. In this step, the parental graphite-like structure of the carbon nitride was continuously consumed by the oxidation of the already generated defects. Thus, large plates of the carbon nitride was continuously consumed by the oxidation of the already generated defects.

The electronic and optical properties have been investigated by UV-Vis diffuse reflectance spectroscopy (DRS), Mott-Schottky plots and photoluminescence (PL) (Fig. 6). As can be seen in Fig. 6A, the absorption edge of the porous g-C$_3$N$_4$ shows a remarkable blue shift in comparison with the bulk g-C$_3$N$_4$. The corresponding bandgaps of the bulk g-C$_3$N$_4$ and porous g-C$_3$N$_4$ are about 2.54 eV and 2.74 eV, from the inset in Fig. 6A. The blue shift of the absorption edge is in agreement with the color change from yellow of bulk g-C$_3$N$_4$ to light yellow of porous g-C$_3$N$_4$ (see Fig. S3), since the color of a sample is determined by the absorption spectrum. Aside from the bandgaps, electrochemical Mott-Schottky experiment was performed to determine the relative positions of CB and VB edges. The plots disclose that the porous g-C$_3$N$_4$ is a typical n-type semiconductor owing to the positive slope (Fig. 6B). More importantly, the flat band potentials of the bulk and porous g-C$_3$N$_4$ were determined to be -1.23 V and -0.88 V versus Hg/Hg$_2$Cl$_2$. Combined with the bandgaps obtained from UV-vis DRS curves, the VB level of the porous sample is calculated to be 1.86 V, which is much lowered with a downshift from 1.31 V of the pristine g-C$_3$N$_4$ (Fig. 6C). Such optical properties are important for the improvement of photocatalysis. Furthermore, under photoexcitation at 330 nm, the PL spectra of the porous g-C$_3$N$_4$ demonstrate that the position of the emission peak is blue shifted by about 33 nm to 435 nm compared with the bulk g-C$_3$N$_4$ (Fig. 6D). As shown in the inset of Fig. 6D, the porous g-C$_3$N$_4$ emitted blue PL under UV light. The larger band gap and blue shift of PL spectra can be attributed to the strong quantum confinement effect, which are caused by the conduction and valence band shifting in opposite directions and the decrease of conjugation length. The above results clearly demonstrate that the porous g-C$_3$N$_4$ own significantly enhanced photocatalytic activities compared to the bulk g-C$_3$N$_4$. As reported before, three steps exist in the semiconductor photocatalysis process, including the photoexcitation, the separation and transportation of photoexcited charge carriers, and the photocatalytic reaction. Studies have shown that the synergistic effects of surface area, light absorption and electron transport ability can greatly influence the photocatalytic activity of g-C$_3$N$_4$. In our case, more adsorption sites and photocatalytic reaction sites could be provided by the larger Brunauer-Emmett-Teller (BET) specific surface area, during the photocatalysis reaction of the porous g-C$_3$N$_4$. On the other hand, the porous structures might contribute to the improvement of photoinduced charge carrier separation and transportation efficiency. More importantly, the larger band gap (0.2 eV) could enhance the conduction.

Fig. 5 (A) TEM image of the bulk g-C$_3$N$_4$. (B-D) TEM images of different magnification of the as-prepared g-C$_3$N$_4$ patches reacting for 12 h.

Fig. 6 (A) UV-Visible absorption spectra, (B) Mott-Schottky plot, (C) band structure, and (D) PL spectra of the bulk g-C$_3$N$_4$ and the porous g-C$_3$N$_4$. Inset of (A) is estimated band gaps of the bulk g-C$_3$N$_4$ and the porous g-C$_3$N$_4$. Inset of (D) is the color change of the porous g-C$_3$N$_4$ solution before and under UV light illumination.
ability of photogenerated electrons due to the quantum confinement effect.13, 15 As is known, the photoreduction and photooxidation abilities of semiconductors are determined by the potentials of their conduction and valence bands. The positive shift of VB in the porous g-C₃N₄ would make the hole more oxidative and increase its photooxidation ability.38, 59 Moreover, the effect of possible residual K+ on the degradation of RhB rather than the traces of residual acids.  

Theoretical concentration of Cr³⁺ in the 25 mg porous g-C₃N₄ for 50 mL RhB degradation is 0.0023 mg/L. Based on this calculation, we have prepared 50 mL RhB solution containing 0.01 mg/L and 0.005 mg/L Cr³⁺ by using K₂Cr₂O₇/H₂SO₄. The degradation result is not good, compared to the porous g-C₃N₄. Thus, we believe that it is the porous g-C₃N₄ that has a major effect on the degradation of RhB rather than the traces of residual acids.

Conclusions

In summary, water-dispersible porous g-C₃N₄ with a high concentration (3 mg mL⁻¹) has been successfully prepared via a chemical oxidation method of the bulk g-C₃N₄. The porous g-C₃N₄ exhibits a large surface area and a wide band gap, which greatly improve its photocatalytic activities under visible light. Further efforts will be made towards fabrication of self-assembled composites of g-C₃N₄ with other materials or applications in the fields of electrocatalysts or solar cells.

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Notes and references


† Electronic Supplementary Information (ESI) available: FTIR spectra, SEM and sample photos, photocatalytic degradation of RhB by Cr³⁺. See DOI: 10.1039/b000000x/