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Boosting charge separation and nitrogen vacancies in graphitic carbon nitride by implanted strontium vanadate for highly efficient photocatalytic reduction of hexavalent chromium

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Strontium vanadate nanoparticles embedded graphitic carbon nitride (g-C₃N₄/Sr₂V₂O₇) was facilely prepared *in situ* via a hydrothermal method. It was shown that the Sr₂V₂O₇ nanoparticles implanted into g-carbon nitride had a small size and high distribution. Importantly, compared with some other photocatalysts, the as-prepared g-C₃N₄/Sr₂V₂O₇ nano hybrid showed excellent photocatalytic activity for reduction of Cr(VI), and as high as 99% efficiency for Cr(VI) reduction (100 mg L⁻¹) was reached within 8 min. Moreover, its activity was hardly changed after five cycles, demonstrating that the developed g-C₃N₄/Sr₂V₂O₇ nano hybrid was highly stable and promising an efficacious disposal of Cr(VI) in water. It was confirmed that the improved charge separation owing to more nitrogen vacancies in the hybrid was the main reason for the improved performance of the g-C₃N₄-Sr₂V₂O₇ nano hybrid.

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Introduction

Nowadays, heavy metal ions in water have received extensive attention because of their serious harm to human and animal health. Among these ions, Cr(VI) is one of the most commonly found toxic ions in wastewater,¹ which seriously threatens ecological systems and could cause great damage to human health and induce many diseases such as cancer, dermatitis, liver damage, and inherited gene deficiency.² Therefore, eliminating Cr(VI) from aquatic environments has drawn much attention all around the world.

In those technologies for Cr(VI) removal,³ photocatalytic technology plays a dominant role in solving the problem of environmental pollution because of its simple operation that is clean, and has high repeatability.⁴ As a significant organic semiconductor photocatalyst, g-C₃N₄ has been widely used in H₂ evolution,⁵ pollutants degradation,⁶ CO₂ reduction,⁷ and other fields due to its facile preparation, nontoxicity, excellent electronic properties. Nevertheless, the photocatalytic activity of single g-C₃N₄ is generally undesirable as a result of fast e⁻/h⁺ recombination.⁸

Several strategies have been developed to facilitate separation and transfer of photoinduced charges, such as doping, coupling with other semiconductors.⁹ For instance, 95% of Cr(VI) can be removed by porous (P,Mo)-g-C₃N₄ prepared using a polycondensation strategy under sunlight irradiation for 120 min.¹⁰ A phosphorus-doped g-C₃N₄/SnS synthesized *via* hydrothermal method can completely remove 100 mg L⁻¹ Cr(VI) after irradiation for 60 min.¹¹ However, tedious synthetic

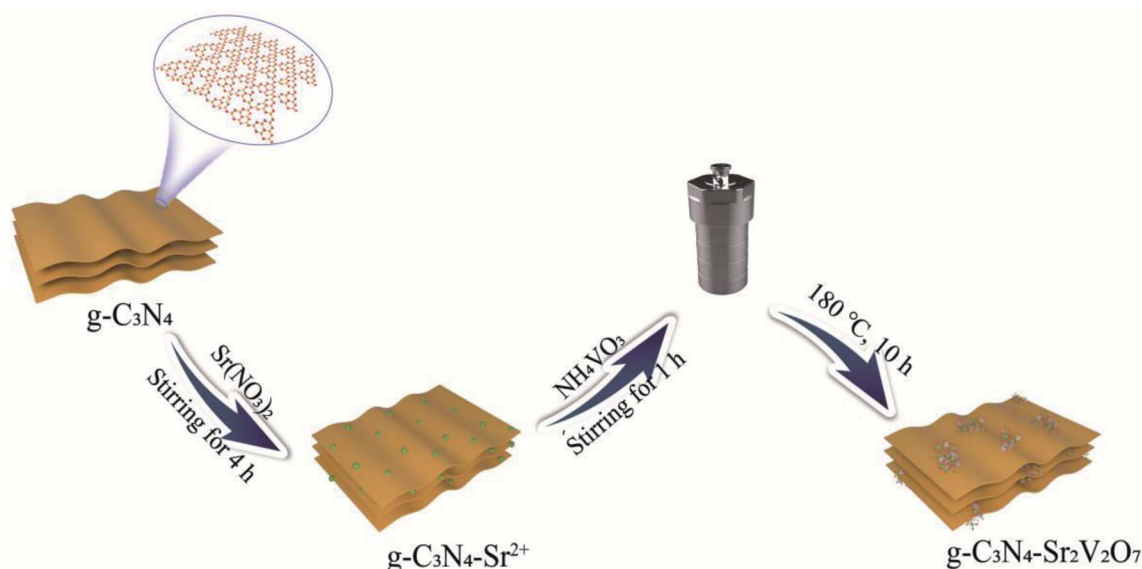
process¹² or quick photocorrosion of some metal sulfides restricts their application in Cr(VI) removal. Moreover, the stability of some common photocatalysts and the degradation rate towards Cr(VI) are still unsatisfying. Therefore, it is highly challenging to develop effective and reusable g-C₃N₄-based materials for photocatalytic reduction of Cr(VI).

Metal vanadates possessing high thermal stability have received significant interest due to their potential application in chemical sensors, transparent conductors and photocatalysts.¹³ As one of the important vanadates, strontium vanadate is widely used in the field of white-light-emitting devices and microwave-dielectrics.¹⁴ While there is few application in photocatalysis. Interestingly, the result of electronic structure calculation confirms that the valence band of Sr₂V₂O₇ is mostly supported by oxygen atoms, and the bottom of the conduction band is mostly composed of the d orbitals of vanadium atoms.¹⁴ And the 3d orbital of vanadium is generally situated below the analogous d orbitals of some transition metals, and the bottom of conduction band is decreased.¹⁵ Therefore, Sr₂V₂O₇ could be an excellent candidate for e⁻/h⁺ separation if a heterojunction system composed of g-C₃N₄ and Sr₂V₂O₇ was smartly constructed in that it would greatly boost the charge transmission.

Herein, by hydrothermal method, small sized Sr₂V₂O₇ is evenly *in situ* incorporated in g-C₃N₄. The as-prepared g-C₃N₄/Sr₂V₂O₇ nano hybrid is characterized, and its performance is evaluated by photocatalytic reduction of Cr(VI). Moreover, the durability and stability of the g-C₃N₄/Sr₂V₂O₇ hybrid are examined by consecutive cycling experiment. The research could pave a new way for fabricating highly active and reusable semiconductor photocatalysts for the removal of heavy metal ions in wastewater.

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Scheme 1 Preparation process for the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ nanohybrid.

Experimental

Synthesis of the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$

$g\text{-C}_3\text{N}_4$ (0.075 g) prepared by the reported method¹⁶ was dispersed into DI water (140 mL). $\text{Sr}(\text{NO}_3)_2$ (10 mL, 0.01 mol L^{-1}) was slowly dropped into the $g\text{-C}_3\text{N}_4$ dispersion, and stirred for 4 h. Then the separated solid ($g\text{-C}_3\text{N}_4\text{-Sr}^{2+}$) was mixed with 70 mL DI water, and NH_4VO_3 (5 mL, $0.0132 \text{ mol L}^{-1}$) was put in and continuously reacted for another 1 h. After that, the mixture was put into a stainless Teflon-lined autoclave and heated at $180 \text{ }^\circ\text{C}$ for 10 h. It was naturally cooled to $25 \text{ }^\circ\text{C}$. The solid ($g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ nanohybrid) was washed thoroughly with DI water, and dried (Scheme 1).

Characterizations

Crystal structure of the samples was performed using Bruker D8 Advance X-ray diffractometer (XRD) equipped with $\text{Cu K}\alpha$ irradiation (Germany). The interaction in materials were measured on a Thermo ESCALAB 250 X-ray photoelectron spectrometer (XPS) with a monochromatic X-ray source ($\text{Al K}\alpha h\nu = 1486.6 \text{ eV}$) (USA). The light adsorption property was measured with a UV-3900 spectrophotometer (Japan). The morphology was observed by transmission electron microscopy (TEM, JEM-1400F, JEOL, Japan). The element maps were analyzed by a scanning electron microscope (SEM, S-3400N, Hitachi, Japan). The electron paramagnetic resonance (EPR) measurements were carried out on a Bruker ESP A300 spectrometer (Germany). The photoluminescence (PL) spectra were measured with an FL-4600 fluorescence spectrophotometer (Japan).

Photocatalytic measurement

$\text{Cr}(\text{VI})$ (with $\text{K}_2\text{Cr}_2\text{O}_7$ as $\text{Cr}(\text{VI})$ resource) reduction with the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ nanohybrid as a photocatalyst was conducted in a quartz reactor under a 300 W xenon lamp. In order to reach

the adsorption/desorption equilibrium, in each photocatalytic test, before irradiation, the mixture containing $\text{K}_2\text{Cr}_2\text{O}_7$ (represented by $\text{Cr}(\text{VI})$, 40 mL , 100 mg L^{-1}) aqueous solution and tartaric acid (30 mg) as well as $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ nanohybrid (10 mg) was stirred continuously for 1 h. During photocatalytic reaction, the residual $\text{Cr}(\text{VI})$ in the solution was monitored by a UV-vis spectrophotometer. Its concentration was obtained according to the absorbance of $\text{Cr}(\text{VI})$ at 540 nm and Lambert Beer's law.

Result and discussion

The characterization of the nanohybrid

XRD result of the as-prepared nanohybrid is displayed in Fig. 1A, a peak at 27.4° corresponds to (002) crystal plane of $g\text{-C}_3\text{N}_4$.¹⁷ While the diffraction peaks appeared at $2\theta = 33.2^\circ$ and 45.3° are originated from the (215) and (1012) planes of $\text{Sr}_2\text{V}_2\text{O}_7$, respectively (JCPDS No. 32-1268),¹⁸ demonstrating that the $\text{Sr}_2\text{V}_2\text{O}_7$ is bound in the $g\text{-C}_3\text{N}_4$. In addition, the peaks of $\text{Sr}_2\text{V}_2\text{O}_7$ are weaker, which could be attributed to lower content (about 0.12 wt% measured by XPS), small size or high dispersion of $\text{Sr}_2\text{V}_2\text{O}_7$ in the nanohybrid. The morphology of the as-synthesized sample is studied by SEM and TEM. It shows some thicker sheet-like structures (Fig. 1B–D). The thickness of the blocky structure in SEM is about $0.92 \text{ }\mu\text{m}$ measured by Digital Micrograph 3.9, and the thinner sheet-like structure is about 15.40 nm as observed in TEM image. Besides the sheet-like structure, no other morphology or crystal lattices are displayed. It is suggested that a small amount of $\text{Sr}_2\text{V}_2\text{O}_7$ is evenly distributed in the $g\text{-C}_3\text{N}_4$, which has little influence on the morphology of $g\text{-C}_3\text{N}_4$. The result is consistent with that in XRD. The $\text{Sr}_2\text{V}_2\text{O}_7$ in the sample is further identified by SEM mapping and XPS followed.

To further confirm the elemental distribution in the nanohybrid, the energy-filtered SEM image and elemental mappings



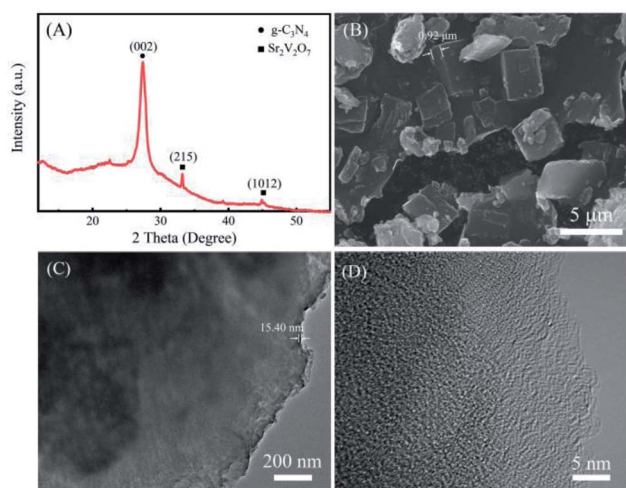


Fig. 1 The structure and morphology of the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ nano-hybrid. (A) XRD, (B) SEM, (C) TEM, (D) HRTEM.

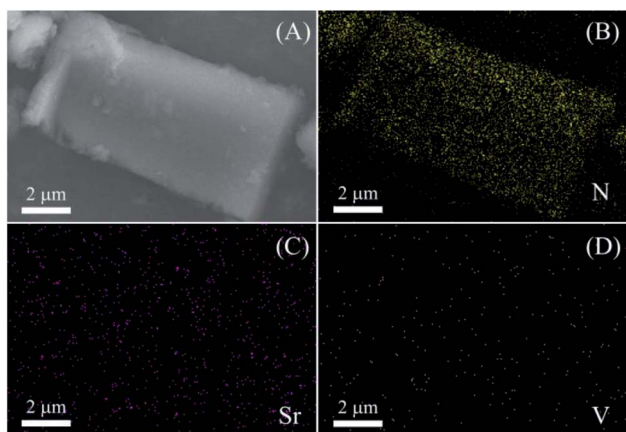


Fig. 2 (A) SEM image of the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$, (B) N map, (C) Sr map, and (D) V map.

for the nano-hybrid are taken. In Fig. 2, N, Sr and V can be observed in their mappings, which are matched with the theoretical composition of the obtained $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ nano-hybrid. N element comes from $g\text{-C}_3\text{N}_4$, Sr and V elements come from the embedded $\text{Sr}_2\text{V}_2\text{O}_7$. Moreover, as displayed in Fig. 2C and D, the density of Sr and V is obviously lower than N, and the distribution of Sr and V mostly conforms to that of the N, indicating that the $\text{Sr}_2\text{V}_2\text{O}_7$ is relatively evenly implanted in the $g\text{-C}_3\text{N}_4$ hybrid.

According to the XPS shift in binding energy, the interaction among materials can be investigated. To confirm the interaction between $\text{Sr}_2\text{V}_2\text{O}_7$ and graphitic carbon nitride, XPS analysis is performed. In Fig. 3A, C, N, O, Sr and V elements in the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ can be observed, which is consistent with those in Fig. 2. In C 1s spectrum of pristine $g\text{-C}_3\text{N}_4$ (Fig. 3B), the peaks at 287.68 and 284.38 eV are assigned to sp^2 hybridized carbon atom and the surface carbon, respectively.¹⁹ The peak corresponding to C–N in the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ is shifted toward lower binding energy relative to pristine $g\text{-C}_3\text{N}_4$. In Fig. 3C, the signals of N 1s in pristine $g\text{-C}_3\text{N}_4$

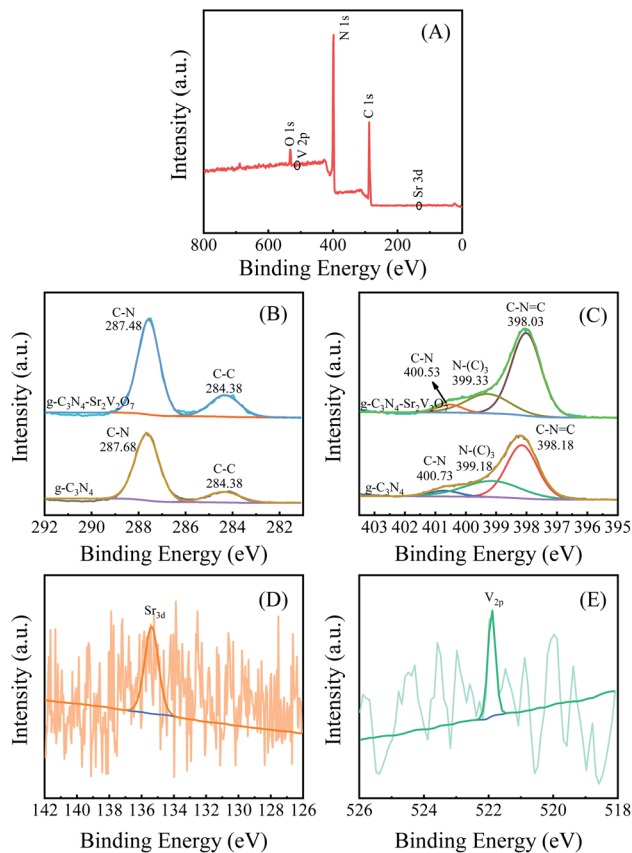


Fig. 3 (A) Survey XPS of the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$. The contrasting HRXPS of C 1s (B) and N 1s (C) in $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ and $g\text{-C}_3\text{N}_4$. The HRXPS of Sr 3d (D) and V 2p (E) in $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$.

can be fitted into three peaks at 400.73, 399.18, and 398.18 eV, which are assigned to N–H, N–C₃, and C–N=C, respectively.²⁰ When $\text{Sr}_2\text{V}_2\text{O}_7$ is embedded into the graphitic carbon nitride, the peaks of C–N=C and N–H slightly shift towards lower binding energy, while the peak of the N–C₃ shifts towards higher energy, which are attributed to strong interaction between strontium vanadate and graphitic carbon nitride in the nano-hybrid. However, the low signal-to-noise ratio of Sr and V spectra is observed in Fig. 3D and E, which could be attributed to the low content and lower sensitivity of Sr and V.

Photocatalytic Cr(vi) reduction

Performance of the prepared $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ nano-hybrid is investigated by photocatalytic reduction of Cr(vi). Due to low content (only 0.12 wt%) of $\text{Sr}_2\text{V}_2\text{O}_7$ in the $g\text{-C}_3\text{N}_4$, its role in the composite photocatalyst is similar to a co-catalyst. Therefore, in photocatalytic measurement, the photocatalytic activity of the $g\text{-C}_3\text{N}_4\text{-Sr}_2\text{V}_2\text{O}_7$ nano-hybrid was mainly compared with that of $g\text{-C}_3\text{N}_4$. Fig. 4A shows the time course of C/C_0 of Cr(vi) (C_0 and C are the concentrations of Cr(vi) at adsorption–desorption equilibrium and irradiated for t time, respectively). It is reported that tartaric acid can show photocatalytic activity for Cr(vi) reduction.²¹ In this experiment, without any photocatalysts, only with tartaric acid, it needs about 13 min for complete



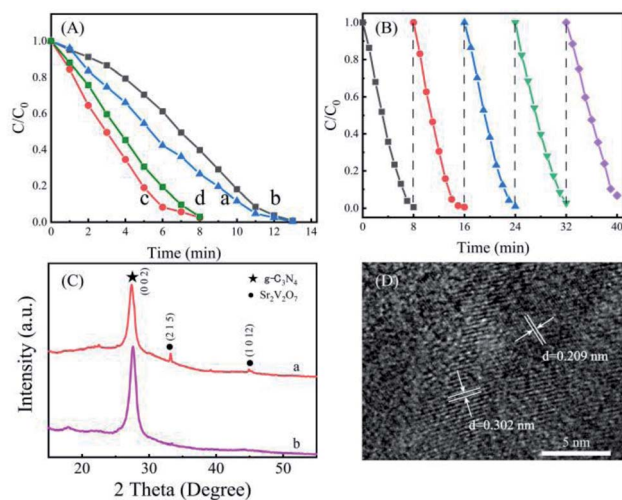


Fig. 4 (A) Time course of C/C_0 of Cr(VI) contained tartaric acid (a) without any photocatalysts, with (b) $g-C_3N_4$, with (c) $g-C_3N_4-Sr_2V_2O_7$ (0.12 wt%) nanohybrid, and with (d) $g-C_3N_4-Sr_2V_2O_7$ (0.06 wt%) nanohybrid; (B) photocatalytic recycling of the nanohybrid for Cr(VI) reduction; (C) the XRD patterns of the $g-C_3N_4-Sr_2V_2O_7$ nanohybrid before recycling experiment (a) and after recycling experiment (b); (D) HRTEM image of the $g-C_3N_4-Sr_2V_2O_7$ nanohybrid after recycling experiment.

reduction of Cr(VI). However, after pure $g-C_3N_4$ was introduced in the solution, its photocatalytic rate is lower than that only with tartaric acid, especially in the first ten minutes. Surprisingly, after the $g-C_3N_4-Sr_2V_2O_7$ is added, under same conditions, 100% Cr(VI) was quickly reduced within 8 min. The reduction time of Cr(VI) is decreased about 5 min compared with that of pure $g-C_3N_4$. When the content of $Sr_2V_2O_7$ is decreased from 0.12 wt% to 0.06 wt%, as shown in Fig. 4A(d), photocatalytic activity of nanohybrid is decreased. However, it is still higher than that of pure $g-C_3N_4$. It is indicated that the photocatalytic performance of graphitic carbon nitride can be effectively improved by embedded strontium vanadate.

A good photocatalyst, besides its activity, its stability is also an important parameter that needs to be considered in practical applications. Therefore, as an active photocatalyst for Cr(VI) removal, the reusability of the $g-C_3N_4-Sr_2V_2O_7$ hybrid is

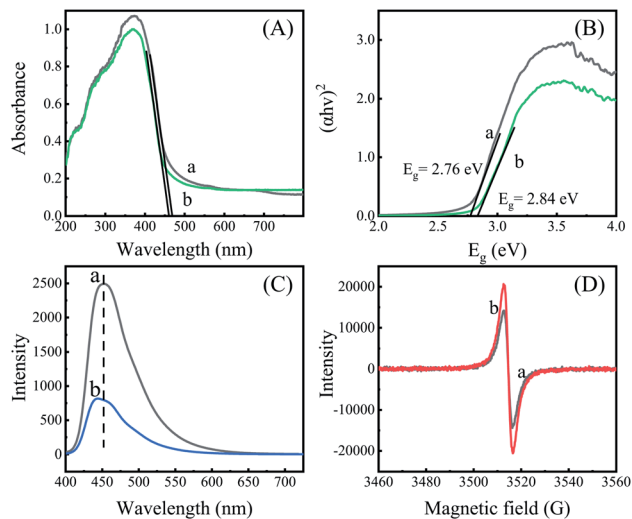


Fig. 5 (A) Solid diffuse reflectance spectra; (B) Tauc plots; (C) fluorescence spectra, $\lambda_{ex} = 380$ nm; (D) EPR spectra. (a) $g-C_3N_4$, (b) $g-C_3N_4-Sr_2V_2O_7$.

evaluated by five successive recycling tests. In the experiment, after the first photocatalytic reaction, the solution was removed by centrifugation, and the fresh Cr(VI) solution (100 mg L^{-1} , 40 mL) was added for the next cycle. As displayed in Fig. 4B, after 5 recycles, the photocatalytic performance of the nanohybrid is changed little, and almost 100% Cr(VI) is reduced in each recycle. TEM and powder XRD after recycling experiment were measured. As shown in Fig. 4C and D, after recycling experiment, the intensity of diffraction peaks of $Sr_2V_2O_7$ in the nanohybrid is decreased. It is deduced that some $Sr_2V_2O_7$ fall off the nanohybrid during recycling. In the TEM image of the $g-C_3N_4-Sr_2V_2O_7$ nanohybrid after recycling experiment, some crystal planes ((1012), (215)) corresponding to $Sr_2V_2O_7$ are shown, which cannot be observed in TEM image of the nanohybrid before recycling experiment (Fig. 1D). It is indicated that some $Sr_2V_2O_7$ particles grow up, and the crystallinity is improved during recycling. The two aspects are opposite to the photocatalytic activity. Subsequently, the photocatalytic activity of nanohybrid changes little after five recycling. The result demonstrates that the $g-C_3N_4-Sr_2V_2O_7$ nanohybrid *in situ*

Table 1 Performance of some common photocatalysts for Cr(VI) reduction

| References | Materials | Dosage of catalyst (mg mL^{-1}) | The concentration of Cr(VI) (mg L^{-1}) | Time (min) and efficiency (%) | Light source |
|---------------|--|--|--|-------------------------------|---------------|
| Ref. 22 | TiO ₂ /CN/rGO | 0.5 | 100 | 240 min, 97% | 300 W Xe lamp |
| Ref. 23 | Biochar- $g-C_3N_4$ microspheres | 40 | 50 | 240 min, 100% | 300 W Xe lamp |
| Ref. 24 | Br-doped $g-C_3N_4$ | 1 | 20 | 120 min, 62% | 300 W Xe lamp |
| Ref. 10 | (P,Mo)-CN | 1.25 | 100 | 120 min, 95% | 300 W Xe lamp |
| Ref. 25 | CaFe ₂ O ₄ / $g-C_3N_4$ /CNT | 1 | 10 | 120 min, 97% | 300 W Xe lamp |
| Ref. 26 | CNT@MoS ₂ /SnS ₂ | 1 | 50 | 90 min, 100% | 300 W Xe lamp |
| Ref. 11 | P-CN/SnS | 0.5 | 100 | 60 min, 100% | 300 W Xe lamp |
| Ref. 27 | γ -CD/MoS ₂ / $g-C_3N_4$ | 0.4 | 10 | 50 min, 81% | 300 W Xe lamp |
| Ref. 28 | $g-C_3N_4$ @bio-Fe(III)-hydroxysulfate | 0.2 | 20 | 20 min, 100% | 300 W Xe lamp |
| In this paper | $g-C_3N_4-Sr_2V_2O_7$ | 0.25 | 100 | 8 min, 99% | 300 W Xe lamp |



prepared by hydrothermal method is with relatively higher stability, and is an effective and potential material for photocatalytic Cr(VI) removal.

For photocatalytic reduction of Cr(VI), many materials can be used as photocatalysts. As shown in Table 1, using the g-C₃N₄-Sr₂V₂O₇ hybrid as the photocatalyst, in a shorter time (8 min), Cr(VI) reduction can be completed. Moreover, the Cr(VI) with higher concentration can be reduced by less dosage of the g-C₃N₄-Sr₂V₂O₇ nanohybrid. It further demonstrates that the hybrid is a more active photocatalyst for Cr(VI) removal.

To explore the reason for the improved photocatalytic performance in the g-C₃N₄-Sr₂V₂O₇, the light adsorption property of the samples is studied. Clearly, in Fig. 5A, the g-C₃N₄ shows an absorption edge at ca. 468 nm. The corresponding band gap is 2.76 eV (Fig. 5B), which is in agreement with the reported result.²⁹ And the nanohybrid displays comparable light-absorption, absorption edge is at 459 nm, and the corresponding band gap is 2.84 eV, showing that light adsorption and band gap of graphitic carbon nitride is changed little by the incorporation of Sr₂V₂O₇.

In addition, fluorescence spectra are measured in order to explore charge separation in the nanohybrid. Generally, a higher emission indicates more e⁻/h⁺ recombination and poorer photocatalytic performance.³⁰ It can be seen in Fig. 5C, g-C₃N₄ shows a high emission at 453 nm, while the g-C₃N₄-Sr₂V₂O₇ exhibits a weaker emission. Besides, an about 9 nm blue-shift is seen in the emission spectrum of the nanohybrid, suggesting that the embedding of strontium vanadate in the graphitic carbon nitride plays an important role in inhibiting recombination of e⁻/h⁺ pairs due to strong interaction between strontium vanadate and graphitic carbon nitride. It is very advantageous to increase photocatalytic performance of the g-C₃N₄-Sr₂V₂O₇ nanohybrid.

To better substantiate and comprehend the charge separation in the nanohybrid, the EPR measurements are performed. In Fig. 5D, only one single Lorentzian line at $g = 2.003$ is shown for the two samples. It is ascribed to nitrogen vacancies.³¹ In the nanohybrid, the EPR signal is increased compared with that of the graphitic carbon nitride, suggesting more nitrogen vacancies generated in the nanohybrid. According to the results in Fig. 5, it is concluded that more nitrogen vacancies generated in the g-C₃N₄-Sr₂V₂O₇ could be the intrinsic reason for increased charge separation and photocatalytic performance in the nanohybrid.

Conclusions

g-C₃N₄-Sr₂V₂O₇ nanohybrid was smartly *in situ* synthesized by hydrothermal method. The nanohybrid with intimate contact showed fast and highly efficient removal of Cr(VI). Moreover, its photocatalytic performance was hardly changed after five successive recycling tests. The lower fluorescence and increased EPR signals achieved in the nanohybrid evidenced that highly efficient charge separation originated from more nitrogen vacancies in the nanohybrid was the main reason for the increased photocatalytic performance in the g-C₃N₄-Sr₂V₂O₇. The work will provide an idea for designing a greatly preferable

and recyclable photocatalyst for the removal of other heavy metal ions in wastewater.

Conflicts of interest

There are no conflicts to declare.

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