Electrospinning preparation of g-C_3N_4/Nb_2O_5 nanofibers heterojunction for enhanced photocatalytic degradation of organic pollutants in water

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In this study, graphitic carbon nitride (g-C_3N_4) and niobium pentoxide nanofibers (Nb_2O_5 NFs) heterojunction was prepared by means of a direct electrospinning approach combined with calcination process. The characterizations confirmed a well-defined morphology of the g-C_3N_4/Nb_2O_5 heterojunction in which Nb_2O_5 NFs were tightly attached onto g-C_3N_4 nanosheets. Compared to pure g-C_3N_4 and Nb_2O_5 NFs, the as-prepared g-C_3N_4/Nb_2O_5 heterojunction exhibited remarkably enhanced photocatalytic activity for degradation of rhodamine B and phenol under visible light irradiation. The enhanced catalytic activity was attributed predominantly to the synergistic effect between g-C_3N_4 sheets and Nb_2O_5 NFs, which promoted the transferring of carriers and prohibited their recombination, confirmed by the measurement of transient photocurrent responses and photoluminescence spectra. In addition, the active species trapping experiments indicated that superoxide radical anion (·O_2⁻) and hole (h⁺) were the major active species contributing to the photocatalytic process. With its high efficacy and ease of preparation, g-C_3N_4/Nb_2O_5 heterojunction has great potentials for applications in treatment of organic pollutants and conversion of solar energy.

In recent years, water pollution caused by textile dyes and other organic pollutants has made serious damage to the ecosystem and human health as they are toxic, mutagenic, and mostly non-biodegradable¹². For the sustainable development of human being, there is urgent demand to remove water contamination. Traditionally, physical, chemical, and biological wastewater treatment processes are used but generally have several disadvantage such as high cost, low degradation efficiency, etc.¹. Recently, as an ideal “green strategy” to deal with increasing environmental issues, metal oxide semiconductor based photocatalysis has drawn great attention because of their versatile properties⁴. In photocatalysis process, when the semiconductors are illuminated by photons with energy higher than their band gap, active charges are generated to cause photocatalytic reactions toward pollutant degradation⁶. Nowadays, semiconductor materials such as TiO₂, ZnO, Nb₂O₅, CeO₂, BiOCl, graphene, g-C_3N_4 and their heterojunction composites like Nb₂O₅/TiO₂, BiOCl/TiO₂, g-C_3N_4/TiO₂, Nb₂O₅/ZnO and CeO₂/Nb₂O₅ have been used to overcome the water pollution issues⁷–²⁰. Among these semiconductor materials, Nb₂O₅, a promising traditional semiconductor material with a band gap of ca. 3.2 eV, has been widely used in a variety of fields, for instance, electrode materials, catalysis, photodecomposition of water and especially photodegradation of harmful organic pollutants in water, due to its outstanding advantages of thermodynamic stability, nontoxicity and relatively high photocatalytic activity²¹. However, the rapid recombination of photogenerated charges hindered the practical application of the pure Nb₂O₅, similar to other traditional semiconductor photocatalysts²²,²³. With the purpose of facilitating the separation of photoinduced charge, novel Nb₂O₅-based composites which are suitable for catalysis of pollutant degradation should be constructed. Consequently, researches found that Nb₂O₅-based heterojunctions with other materials, such as metal (Ag, Au, Pt, etc.), metal oxide (TiO₂, NiO, Ag₂O, Fe₂O₃, etc.) and graphene is a prominent method²⁴–³⁰. Particularly, Nb₂O₅ heterojunctions coupled with visible-light-responsive semiconductor are recognized as the most effective photocatalysts for wastewater treatment because of the internal electric field, which can suppress the recombination of photogenerated charge and

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effectively improve mutual transfer of photogenerated charge in the heterojunctions, thus ultimately enhance the photocatalytic activity. In addition, visible-light-responsive semiconductor coupling Nb$_2$O$_5$ can also improve the light absorption capacity to a significant extent.$^{31}$

Nb$_2$O$_5$, a two-dimensional polymeric semiconductor, has attracted much attention in recent years due to its unique delocalized conjugated $\pi$ structure formed by sp$^2$ hybridization of C and N atoms which offers a rapid photoinduced charge separation in the electron transfer process.$^{32}$ In particular, g-C$_3$N$_4$ can activate molecular oxygen and produce superoxide radicals, which effectively enhances the activities for photocatalytic reactions. Furthermore, g-C$_3$N$_4$ shows massive prospects for application of photocatalytic degradation due to its advantages of narrow band gap, low cost, eco-friendliness and excellent optical and thermal properties.$^{33,34}$ Unfortunately, the photocatalytic activity of pure g-C$_3$N$_4$ is also usually restricted because of the fast recombination of photogenerated electron/hole pairs. In order to improve its efficiency in photocatalytic processes, strategies such as coupling g-C$_3$N$_4$ with TiO$_2$, CeO$_2$, BiVO$_4$ have been proposed, which can not only effectively reduce the photoinduced electron–hole recombination rate, but also form intermediate energy levels in the forbidden band of metal oxide owing to the matching band structure of g-C$_3$N$_4$ and metal oxide.$^{35–37}$ So far, many g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction photocatalysts have been successfully prepared to enhance photocatalytic activity for pollutants degradation due to the well match of band gap edges between Nb$_2$O$_5$ and g-C$_3$N$_4$, which facilitates the charge carrier separation and thus improves the photocatalytic performance. Carvalho et al. reported that g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction photocatalysts, assembled as Nb$_2$O$_5$ nanoparticles decorating the g-C$_3$N$_4$ surface via hydrothermal process, exhibited remarkable enhanced photocatalytic activity in the degradation of methylene blue and rhodamine B dyes.$^{38}$ Silva et al. reported that g-C$_3$N$_4$/Nb$_2$O$_5$ heterostructures, assembled as Nb$_2$O$_5$ nanospheres decorating the g-C$_3$N$_4$ surface by a sonochemical method, showed high activity for dye and drug pollutants degradation under visible light irradiation.$^{39}$ Hong et al. reported Nb$_2$O$_5$/g-C$_3$N$_4$ heterojunctions prepared by a simple heating method showed significantly enhanced photocatalytic activity in the degradation of tetracycline hydrochloride.$^{40}$ As shown in these reports, the g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction had excellent photocatalytic performance and remarkable optoelectronic characteristics for degrading organic pollutants in wastewater compared with individual g-C$_3$N$_4$ and pure Nb$_2$O$_5$. Nevertheless, most of these studies were based on powder-form Nb$_2$O$_5$, which was prepared by complicated means such as solvothermal method, chemical precipitation method, et al. These conventional preparation methods may lead to agglomeration of Nb$_2$O$_5$ nanoparticles and then reduce the photocatalytic activity. Hence, it is of great interest to develop a facile and practical method for effective preparation of the evenly nanostructured g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction with large specific surface area and improved photocatalytic activity.

Thus, to date, one-dimensional semiconductor metal oxide nanofibers fabricated through electrospinning has been greatly attractive due to the advantages of high surface areas and large surface-to-volume ratio, which can provide quick charge transfer channels and more active sites.$^{41–44}$ More significantly, the electrospun nanofibers with a rather high surface area will be optimal carriers for fabricating heterojunction photocatalysts and then provide more active reaction sites for interaction with pollutants.$^{45}$ Meanwhile, the electrospun nanofibers with nonwoven web structure results in an easy separation and recovery from fluid in photocatalytic process.$^{46–47}$ On the basis of above, constructing heterojunctions of electrospun Nb$_2$O$_5$ nanofibers coupled with g-C$_3$N$_4$ would be expected as promising composite photocatalyst for practical applications.

In the present work, we developed a g-C$_3$N$_4$/Nb$_2$O$_5$ nanofibers heterojunction via a simple electrospinning technique, which exhibited a photocatalytic activity superior to the pure g-C$_3$N$_4$, and electrospun Nb$_2$O$_5$ NFs. Meanwhile, the as-prepared heterojunction could be recovered easily by filtration without reducing the photocatalytic activity. Moreover, a possible degradation mechanism is also proposed based on the detailed structural analysis of the heterojunction.

**Experimental Synthesis of g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction.** In a typical procedure, g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction was synthesized by electrospinning. First, 10 g of CH$_6$ClN$_3$ was heated in an open crucible in static air at a heating rate of 10 °C/min to 600 °C and kept at that temperature for 4 h. The product was collected and ground into powder in an agate mortar to obtain the g-C$_3$N$_4$. Subsequently, 0.25 g of NbCl$_5$ and 0.025 g of g-C$_3$N$_4$ were added into 2.65 mL of N,N-dimethylformamide (DMF) and ultrasonicated for 1 h. Then 0.35 g of polyvinylpyrrolidone (PVP) was dissolved in the mixture. After magnetic stirring for 8 h, the precursor solution of g-C$_3$N$_4$/NbCl$_5$/PVP composite was afforded and transferred into a 5 mL plastic syringe with a 25 gauge stainless steel needle for electrospinning. In this electrospinning experiment, the collector was positioned 20 cm away from the tip of needle and the applied direct voltage between the collector and the needle tip was ~18 kV, the precursor solution flow rate was 0.25 mL/h. Then, the collected precursor nanofibers were calcined in muffle furnace at 600 °C for 1 h in air with a heating rate of 10 °C/min to obtain g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction. For comparison, pure Nb$_2$O$_5$ nanofibers was prepared under the same condition without adding g-C$_3$N$_4$.

**Characterizations and photocatalytic experiment.** Supporting Materials showed their details.

**Results and discussion Characterization.** The Fourier Transform infrared (FTIR) spectra of as prepared samples were shown in Fig. 1. In the FTIR spectrum of Nb$_2$O$_5$ NFs, the peaks at 827 cm$^{-1}$ and 665 cm$^{-1}$ were assigned to Nb–O–Nb vibration modes, respectively. The peaks around at 1411 cm$^{-1}$, 1317 cm$^{-1}$ and 1238 cm$^{-1}$ were attributed to aromatic C–N stretching. The unique absorption peak at approximately 806 cm$^{-1}$ was related to the s-triazine ring vibrations of narrow band gap, low cost, eco-friendliness and excellent optical and thermal properties.$^{33,34}$ Unfortu-
modes. As expected, all of the major characteristic absorption peaks of g-C₃N₄ and Nb₂O₅ were present in the spectrum of g-C₃N₄/Nb₂O₅ heterojunction, suggesting that composite samples contained g-C₃N₄.

Figure 2 depicted the X-ray diffraction (XRD) patterns of g-C₃N₄, Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction. As shown in Fig. 2a, the quintessential characteristic diffraction peaks of pure g-C₃N₄ at 13.4° and 27.6° associated to the (100) and (002) planes of the graphite-like structure of C₃N₄, respectively. The XRD pattern revealed that the diffraction peaks of Nb₂O₅ NFs correspond to the orthorhombic phase (standard JCPDS card 30-0873). It is noticeable that the synthesized g-C₃N₄/Nb₂O₅ heterojunction exhibited similar pattern to Nb₂O₅ NFs. The characteristic peak of g-C₃N₄ (002) closed to orthorhombic (180) peak was not extrusive in the pattern of g-C₃N₄/Nb₂O₅ heterojunction owing to the comparatively low dosage of g-C₃N₄. Moreover, compared with Nb₂O₅ NFs, the diffraction peaks of Nb₂O₅ in the heterojunction became weaker and shifted slightly to a smaller value diffraction angle (Fig. 2b) with the addition of g-C₃N₄, which may be caused by the interaction between g-C₃N₄ and Nb₂O₅ in the heterojunction. Similar phenomenon was also reported in an earlier literature.

Fig. S1 showed the thermogravimetric analysis (TGA) curves of g-C₃N₄ and g-C₃N₄/Nb₂O₅ heterojunction. The thermal profile of g-C₃N₄ indicated that the as-prepared material was stable in air flow below 600 °C, and heating to 700 °C resulted in no residue of the material being observable. The weight of the g-C₃N₄/Nb₂O₅ heterojunction decreased rapidly in the temperature range 600–700 °C, owing to the demoposition of g-C₃N₄ occurred in this temperature range. Hence, the g-C₃N₄ content for the g-C₃N₄/Nb₂O₅ heterostructure could be estimated to be 9.2 wt% neglecting the amount of surface-bound water.

The morphology of g-C₃N₄, Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction were observed by scanning electron microscopy (SEM) and transmission electron microscope (TEM). It was found that the Nb₂O₅ NFs represent 1D nanofiber morphology, which had an average diameter of 150 nm with random orientation (Fig. S2a). The
g-C₃N₄ sample displayed an irregular sheet like structure (Fig. S2b). It was perceptible on the SEM image of g-C₃N₄/Nb₂O₅ heterojunction (Fig. 3a) that two semiconductors, Nb₂O₅ nanofibers and g-C₃N₄, directly and sufficiently contacted through mixed electrospinning process and calcination. Such line/area contact increased porosity-related characteristics of the heterojunction and resulted in less agglomeration. In particular, such heterojunction formed by nanofiber and nanosheet can enhance the transport of charges and reduce the recombination probability of photoexcited charge carriers, ultimately further improve the photodegradation efficiency. TEM image (Fig. 3b) further revealed that the g-C₃N₄/Nb₂O₅ heterojunction was composed of homogeneous, long and narrow Nb₂O₅ NFs that were in direct contact with g-C₃N₄ nanosheets. In addition, the selected area electron diffraction (SAED) pattern (inset of Fig. 3b) depicted broad and strong spots that was assigned to the essentially 1D characteristic of the long nanofibers, indicating the highly single crystallinity of the orthorhombic phase Nb₂O₅, which was in accordance with the XRD analysis results of Nb₂O₅. No diffraction spots/rings of g-C₃N₄ were detected in the SAED spectrum, indicating that g-C₃N₄ was amorphous in the g-C₃N₄/Nb₂O₅ heterojunction. Figure 3c exhibited that Nb₂O₅ NFs with a distribution of diameters ~150 nm were attached onto the surfaces of g-C₃N₄ nanosheets, forming g-C₃N₄/Nb₂O₅ heterojunction structure. Moreover, the high resolution TEM image (Fig. 3d) revealed the lattice fringe with d-spacing of approximately 0.245 nm, which was in agreement with the (181) spacing of orthorhombic Nb₂O₅. With these, a heterojunction formation between Nb₂O₅ and g-C₃N₄ was confirmed. This heterojunction would promote charge transfer between Nb₂O₅ and g-C₃N₄ and separation of photogenerated electron–hole pairs, both of which would enhance the photocatalytic activity.

The X-ray photoelectron spectroscopy (XPS) survey spectrum and high resolution spectrum of g-C₃N₄/Nb₂O₅ heterojunction were performed to illuminate the surface composition and the chemical environment. As shown in Fig. 4a, the survey XPS spectrum exhibited that the g-C₃N₄/Nb₂O₅ heterojunction not only contained Nb, O elements related to the Nb₂O₅ phase, but also contained C, N elements related to the g-C₃N₄ phase. Correspondingly, the C 1s high-resolution spectra (Fig. 4b) was divided into three fitted peaks at 284.78 eV, 286.28 eV and 288.43 eV. The peak at 284.78 eV corresponded to sp²-hybridized carbon atoms of the carbon standard used to calibrate the binding energies. The peaks at 286.28 eV and 288.43 eV was attributed to the C–N–C and N–C=N backbones coordination in the triazine rings of g-C₃N₄, respectively. The N 1s high-resolution spectra (Fig. 4c) was deconvoluted into two fitted peaks at 398.93 eV and 401.13 eV, which could be assigned to N sp²-bonded to C (N–sp²C) and tertiary nitrogen groups (N–(C)₃) of g-C₃N₄, respectively. The Nb 3d spectrum (Fig. 4d) exhibited two significant peaks at around 207.43 and 210.13 eV, corresponding to 3dₓ₂₋ᵧ₂ and 3dᵧ₂ states of Nb, respectively. The O 1s XPS spectrum (Fig. 4e) could be fitted to two peaks centered at around 530.28 and
Figure 4. (a) XPS survey spectrum and high resolution XPS spectra of (b) C 1s, (c) N 1s, (d) Nb 3d and (e) O 1s in g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction.
531.63 eV, which were assigned to the Nb–O bond and adsorbed oxygen, respectively. Notably, compared with those of pure Nb₂O₅ NFs, the Nb 3d and O 1s peaks of g-C₃N₄/Nb₂O₅ heterojunction shifted slightly to higher energies, evidencing the intense interaction between the Nb₂O₅ and g-C₃N₄ that resulted from the formation of effective heterojunction. These phenomena are in good agreement with previous report53.

Figure 5 showed N₂ absorption–desorption isotherms and pore size distribution curves of Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction. As illustrated in Fig. 5a, both the isotherms belonged to type IV isotherm possessed obvious type H₃ hysteresis loop at relative higher p/p₀, suggesting the existence of slit-shaped pores due to the aggregation of nanoparticles61. The BET specific surface areas of Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction are 30.09 m²/g and 36.18 m²/g, respectively. In addition, Fig. 5b displays the pore-size distributions of Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction estimated with the BJH method. As seen from the spectra, two characteristic diameters of mesopores (2–50 nm) are located primarily at 3.7 and 10.6 nm for pure Nb₂O₅ NFs, 3.7 and 7.1 nm for g-C₃N₄/Nb₂O₅ heterojunction, respectively. The increased BET specific surface area and decreased pore size were likely caused by the incorporation of g-C₃N₄, which was fundamental in enhancing the photodegradation activity of electrospun g-C₃N₄/Nb₂O₅ heterojunction.

The UV–Vis absorption spectra of as-prepared samples were depicted in Fig. 6a. It is obvious that the g-C₃N₄ has absorption in the visible region, which is in good agreement with the narrow band gap. However, the Nb₂O₅ NFs only absorbs UV light, consistent with wide band gap. Compared to Nb₂O₅ NFs, the absorption edge of g-C₃N₄/Nb₂O₅ heterojunction shifts to the visible light region with the absorbance edge between those of Nb₂O₅ NFs and g-C₃N₄. It can be concluded that the combination of g-C₃N₄ and Nb₂O₅ may enhance the visible light absorption of the sample and thus improve their catalytic activity in the visible region35. In addition, the bandgap

Figure 5. (a) N₂ sorption isotherm and (b) pore size distribution of Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction.

Figure 6. (a) UV–vis diffused reflectance spectra and (b) Tauc plot graph of g-C₃N₄, Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction.
energy of samples were obtained from Tauc plot by extrapolation to the photon energy-axis. As illustrated in Fig. 6b, the band gap values of the g-C$_3$N$_4$ and Nb$_2$O$_5$ NFs were estimated to be approximately 2.7 and 3.2 eV, respectively, consistent with those described for these phases in the literature$^{31,62}$. In addition, the band gap of g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction was found to be 2.84 eV. This suggested that the synthesized g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction had smaller band gap than that of pure Nb$_2$O$_5$ NFs, which was the precondition of effective photocatalytic activity in visible region. Besides, Mott–Schottky plots (Fig. S3) of the Nb$_2$O$_5$ NFs and g-C$_3$N$_4$ were collected to define their conduction band potential (E$_{CB}$). The derived E$_{CB}$ of Nb$_2$O$_5$ NFs and g-C$_3$N$_4$ were estimated to be −0.59 and −0.98 eV, respectively$^{63}$. Thus, the valence band potential (E$_{VB}$) of them were calculated to be 2.61 and 1.72 eV, respectively.

**Photocatalysis.** The photocatalytic activities of the synthesized samples were investigated by decomposing rhodamine B (RhB) under visible light irradiation. As shown in Fig. 7a, compared to Nb$_2$O$_5$ NFs and g-C$_3$N$_4$, g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction possessed the highest photocatalytic degradation rate owing to the existence of synergistic effect between g-C$_3$N$_4$ and Nb$_2$O$_5$. After 120 min irradiation, the photodegradation efficiency of RhB for g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction was 98.1%. The superior photocatalytic activity of g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction over RhB could also be confirmed from kinetics experiment. On the base of the rate equation of $-\ln(C/C_0) = kt$, $k$ representing the degradation rate constant, the kinetic curves for the RhB photodegradation over three photocatalysts were obtained, as shown in Fig. 7b. It was obviously that the $k$ value of g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction is larger than that of pure Nb$_2$O$_5$ NFs and g-C$_3$N$_4$. In addition, phenol, a colorless organic pollutant model, was selected to further evaluate the photocatalytic activity of g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction. It can be found that the g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction also displayed the highest photocatalytic activity for phenol degradation. It was observed that essentially complete degradation of phenol occurred over 120 min for g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction (Fig. 7c). The degradation rate constant of phenol over different catalysts were also calculated by the above rate equation. It is found that g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction still achieved the highest apparent rate constant among all these samples (Fig. 7d). Meanwhile, the total organic carbon (TOC) removal rate of g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction sample for degradation of RhB and phenol were shown in Fig. 6. It can be seen that

Figure 7. The degradation efficiency of (a) RhB and (b) phenol, kinetics curves for (c) RhB and (d) phenol over g-C$_3$N$_4$, Nb$_2$O$_5$ NFs and g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction under visible light irradiation.
the TOC of both solutions decreased as the reaction time increased and reached almost to zero indicating that mineralization of the pollutants occur during the photocatalytic reaction. Table 1 presented the photocatalytic degradation rate of various g-C₃N₄/Nb₂O₅ composites, which are comparable to as-synthesized g-C₃N₄/Nb₂O₅ heterojunction, is exhibited remarkable degradation rate.

**Photocatalysis mechanism.** Investigating the lifetime of photoexcited charge carriers is fundamental to understand the photocatalysis mechanism. The photocurrent responses of g-C₃N₄, Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction were undertaken to manifest the separation efficiency of the electron–hole pairs. As shown in Fig. 8a, g-C₃N₄/Nb₂O₅ heterojunction exhibited the strongest photocurrent density in comparison with that of the single component, which reflected the reduced interface resistance and the enhanced separation and migration efficiency of the photoinduced electron–hole pairs in g-C₃N₄/Nb₂O₅ heterojunction. This was also consistent with the photoluminescence (PL) spectroscopy results. Based on Fig. 8b, it can be seen that the peak intensities gradually decrease in the order of g-C₃N₄, Nb₂O₅ NFs and g-C₃N₄/Nb₂O₅ heterojunction, indicating the g-C₃N₄/Nb₂O₅ heterojunction possessing the lowest electron–hole recombination efficiency. It can be concluded that the recombination of photogenerated charge carriers were inhibited effectively due to the heterojunction construction of g-C₃N₄ and Nb₂O₅.

On the other side, hydroxyl radical (·OH), hole (h⁺) and superoxide radical anion (·O₂⁻) as major active species play key roles in photocatalytic reaction. To elucidate the mechanism of g-C₃N₄/Nb₂O₅ heterojunction, free radicals trapping experiments were performed to confirm the active species contributing to RhB and phenol photodegradation. In these experiments, isopropanol (IPA), triethanolamine (TEA) and p-benzoquinone (BZQ) acted as the scavenger for ·OH, h⁺ and ·O₂⁻, respectively. Figure 9 revealed the photocatalytic efficiencies of RhB and phenol with g-C₃N₄/Nb₂O₅ heterojunction after adding various scavengers. The reaction rate constant showed almost unchanged in the presence of IPA, while the addition of TEA and BZQ led to an obviously decrease of the reaction rate constant. In view of the above results, it can be proposed that ·O₂⁻ is the main active species and h⁺ also play important role in g-C₃N₄/Nb₂O₅ heterojunction for RhB and phenol degradation.

Based on the above analysis and experiments, the mechanism was proposed and schematically illustrated in the Fig. 10. It was well understood that the distinction of CB edge potentials between semiconductors strongly
promoted the electrons transfer at the heterojunction interface and reduced the recombination of carriers, ultimately improved the photodegradation activity of heterojunction. When g-C₃N₄/Nb₂O₅ heterojunction was exposed to irradiation, g-C₃N₄ component was easy to be excited by visible light due to its narrow band gap. And the photogenerated electrons were easy to transfer from CB of g-C₃N₄ to that of Nb₂O₅ because the CB edge potential of g-C₃N₄ (−0.98 eV) was more negative than that of Nb₂O₅ (−0.59 eV). The photogenerated electrons could react with dissolved O₂ near the Nb₂O₅ NFs to form the main active specie of ·O₂⁻, which was mostly responsible for degrading pollutants. Similarly, the VB edge potential of g-C₃N₄ (1.72 eV) was also more negative than that of Nb₂O₅ (2.61 eV), and the photogenerated h⁺ transfer to VB of g-C₃N₄ from VB of Nb₂O₅ through the tight heterojunction interface. The residual h⁺ on VB of Nb₂O₅ could oxidize organic molecules to photodegradation products directly. Moreover, the multi-point connected Nb₂O₅ nanofiber and porous g-C₃N₄ were also helpful to promote the electron migration.

Reusability of g-C₃N₄/Nb₂O₅ heterojunction. To investigate the stability and reusability of the g-C₃N₄/Nb₂O₅ heterojunction, recycling test of the photodegradation were performed. After each photodegradation cycle, the catalyst was filtered from the solution and dried at 80 °C for further use. As depicted in Fig. 11a, the g-C₃N₄/Nb₂O₅ heterojunction showed no obvious reduction of photocatalytic activity after four cycles for both RhB and phenol, demonstrating its excellent stability and reusability for the multiple times for organic pollutants degradation. And the XRD patterns of the g-C₃N₄/Nb₂O₅ heterojunction before and after the reaction were similar (Fig. 11b). From a typical SEM image of g-C₃N₄/Nb₂O₅ heterojunction (inset of Fig. 11b) after photocatalytic reaction, it can be clearly seen that the Nb₂O₅ NFs is still tightly attached with g-C₃N₄ nanosheet, which indicates that the morphology of g-C₃N₄/Nb₂O₅ heterojunction remains unchanged after photocatalytic reaction.
Conclusions
In summary, the g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction was successfully synthesized using a simple and facile electrospinning-calcination process and displayed excellent photocatalytic activity for RhB and phenol degradation. Particularly, the as-prepared g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction exhibited higher photocatalytic activities towards the photodegradation of RhB and phenol under visible irradiation, compared to the pure g-C$_3$N$_4$ and Nb$_2$O$_5$ phases. The low bandgap energy (2.84 eV) as well as the synergistic effect between Nb$_2$O$_5$ NFs and g-C$_3$N$_4$ in the g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction enhanced the photocatalytic activity, which were beneficial to a rapid photoinduced charge separation in the electron transfer process and a slow charge recombination. In addition, both superoxide radical anion (·O$_2$–) and hole (h$^+$) were the major oxidative species for RhB and phenol degradation over the g-C$_3$N$_4$/Nb$_2$O$_5$ heterojunction photocatalyst. This work may provide a promising future of applying photocatalyst to solving dye pollutant problems and solar energy conversion.

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Author contributions
L.W. prepared photocatalysts and wrote the main manuscript text. Y.L. designed catalyst and measured the chemical structure of catalyst. P.H. performed manuscript revision and editing.

Competing interests
The authors declare no competing interests.

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