

Recent Advances in Carbon Nitride-based Nanomaterials for the Removal of Heavy Metal Ions from Aqueous Solution

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Abstract: Graphitic-like carbon nitride ($g\text{-C}_3\text{N}_4$), one of the most significant two-dimensional layered materials, has attracted worldwide attention in multidisciplinary areas such as photocatalysis, energy conversion and environmental pollution management. Its derivative compounds have also attracted multifarious attention owing to the intrinsic characters of their stable physicochemical properties, low cost and environmentally friendly features. This review focus on the design of high-performance $g\text{-C}_3\text{N}_4$ -based nanomaterials and their potential for pollutant elimination in environmental pollution cleanup. Over the past few years, significant advances have been achieved to synthesize $g\text{-C}_3\text{N}_4$ and $g\text{-C}_3\text{N}_4$ -based nanomaterials, and their properties have been enhanced and characterized in detail. In this review, recent developments in the synthesis and modification of $g\text{-C}_3\text{N}_4$ -based nanomaterials are summarized. The applications in heavy metal ions adsorption from wastewaters are gathered and their underlying reaction mechanisms are discussed. Finally, a summary and outlook are also briefly illustrated.

Key words: carbon nitride; adsorption; heavy metal ions; interaction mechanism; review

With the rapid progress of modern industries and human activities, human health and environmental ecosystems have been extremely threatened by varied pollutants such as organic and inorganic contaminants^[1-4]. Especially, the toxic heavy metal ions released from metal-finishing plants and electroplating manufactory have serious impacts on human health and environmental ecosystem due to their physicochemical properties of nondegradable, highly virulent and widely dispersed in water solutions^[5-8]. Thereby, it is urgent to exploit eco-friendly and highly efficient materials for the decontamination and solidification of heavy metal ions from aqueous environment^[9-11].

During the past few decades, various modern and conventional technologies^[12-13], such as membrane separation^[14], reverse osmosis^[15], ion-exchange^[16], chemical precipitation^[7] and adsorption^[17] have been applied extensively to separate and preconcentrate heavy metal ions from contaminated water^[18-20]. All of these methods have been scientifically investigated intensively by theoretical

calculations and experimental techniques. Nevertheless, most of these strategies still suffer from various drawbacks such as secondary pollution, complex operation process and expensive installation costs^[21]. Considering the physicochemical properties of heavy metal ions, adsorption strategy has achieved rising attention for its advantages as an easy-to-operation, versatile and highly efficient technique for removing pollutants in large scale.

To the best of our knowledge, many researchers have focused on layered double hydroxides (LDHs)^[22-24], nanosized metal oxides (NMOs)^[25-27], clay minerals^[28-30], ordered mesoporous carbon (OMC)^[31-32], carbon nanotubes^[21,33-34], activated carbon^[35-36], and graphene oxides^[4,22,26] as efficient materials for the adsorption of heavy metal ions from aqueous solutions^[26,37]. Among these adsorbents, graphitic-like carbon nitride ($g\text{-C}_3\text{N}_4$) based materials represent a class of two-dimension materials which were widely used for the removal of heavy metal ions. $g\text{-C}_3\text{N}_4$ is very stable in extreme strong acid and base conditions^[32]. In addition, it possesses excellent

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thermal stability above 500 °C because of the high temperature of synthesis condition^[38-39]. It is well known that the structural property of g-C₃N₄ exhibits outstanding characteristics, for example: (1) the existence of amino functional groups and the triazine ring on the surface of C₃N₄ makes it possible to form strong surface complexes with metal ions; (2) the stability and excellent physico-chemical properties are beneficial to the composite to form with other functional groups, and for application under different conditions^[40-43].

Herein, the object of this review is to provide a systematic overview of g-C₃N₄-based nanomaterials and their applications in heavy metal ions removal from aqueous solutions. Firstly, we focus on their preparation and modification strategies to present an intuitive comprehending of g-C₃N₄. Secondly, the research status and removal mechanisms by theoretical calculations and experimental methods in the elimination of heavy metal ions are summarized and discussed in detail. Finally, the outlook and challenges of the g-C₃N₄-based adsorbents in real applications are prospected.

1 Synthesis of g-C₃N₄ and g-C₃N₄-based nanomaterials

1.1 Synthesis of g-C₃N₄ by traditional methods

In the past decades, g-C₃N₄ has attracted a matter of great concern as a favorable adsorbent for elimination of various heavy metal ions, radionuclides (*e.g.*, Pb(II), Cu(II), Cd(II), U(VI), Am(III) *etc.*) and as catalyst for the photocatalytic degradation of organic pollutants (*i.e.*, biphenyl compounds and organic dyes) from wastewater. One of the traditional methods for the synthesis of g-C₃N₄ is thermal treatment. The synthesis of pristine g-C₃N₄ by thermal treatment of nitrogen-rich precursors (*e.g.*, melamine, dicyandiamide, thiourea, urea and cyanamide) is shown in Fig.1. The host layers in g-C₃N₄ coexist interaction, which are stacked each other *via* weak intermolecular electrostatic force or van der Waals force with interlayer^[40]. Chemical or physical strategies can be applied to destroy the interlayer interaction, such as thermal oxidation exfoliation, ultrasonic exfoliation and chemical exfoliation, achieving the alleged nanosheets. g-C₃N₄ was fabricated through thermal condensation way at ~550 °C and the inert atmosphere, while g-C₃N₄ nanosheets were manufactured by heating bulk-g-C₃N₄ in the air at 500 °C^[44]. Ultrasound-assisted method is one of the extensively strategies applied to liquid exfoliation of layered materials lately. Liao *et al.*^[45] prepared g-C₃N₄ nanosheets by ultrasonic exfoliation strategy for the elimination of serial contaminants. Briefly, the powder of pristine g-C₃N₄ was dispersed in

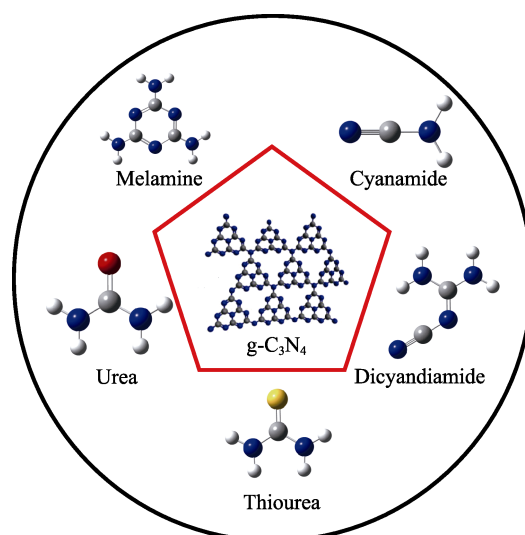


Fig. 1 Schematic illustration of the synthesis process of g-C₃N₄ by thermal polymerization of different precursors

deionized water and exfoliated by ultrasonic process for 10 h. After the modification and exfoliation of g-C₃N₄ into 2D nanosheets, the water solubility and the specific surface area (up to 418.28 m²/g) were greatly improved. These results showed that the traditional techniques, such as thermal method and ultrasonic exfoliation method, to modified g-C₃N₄ nanosheets were facile and promising for wastewater treatment.

1.2 Synthesis of g-C₃N₄ by hard template method

The synthesis of porous g-C₃N₄ for a variety of species and structural diversities as well as tunable cavities has been studied recently. The advantage of high surface area could provide abundant active sites for the adsorption of metal ions. g-C₃N₄ was recombined with porous solid template, such as mesoporous silica hard template KIT-6, a salt melt precursor, commercial nanosized silica nanosphere template and mesoporous biomass carbon, to remove contaminants from wastewater. In 2015, Shen *et al.*^[46] reported a simple and environmental-friendly saltmelting method for the synthesis of g-C₃N₄ particles with a size of approximate 100 nm, which had an effective removal capacity for remediation of metal polluted water. Tan *et al.*^[31] fabricated the coral reef-like C₃N₄ by refluxing nitrogen source through the growing mechanisms of Ostwald ripening and oriented attachment. In the process of particle formation, two phases (β - and α -phase) transformation occurred in 1.5 h upon refluxing. However, sphere-shaped particles converted to coral reef-like particles at the reflux time of 2.0 h. The high surface area (24.5 m²/g) and the incorporated of -COOH sites were the dominant reasons for superior adsorption abilities for heavy metal ions.

Among the various template strategies, well-ordered

mesoporous silica family precursors loaded g-C₃N₄ have been widely researched because of their excellent physicochemical properties, such as SBA-15, MCM-41 and KIT-6^[47]. Either random structures or highly ordered structures of template strategy-modified g-C₃N₄ showed potential to preconcentrate metal ions from wastewater. The versatility and simplicity of both methods to enrich functional groups and to increase the specific surface area provided the basis for further researches. For ordered templates, mainly regulating the morphology and size seems to be a very promising field. Random templating, with its diversified functional groups, has already been applied in wastewater purification through adsorption or photocatalytic degradation methods.

1.3 Surface Modification of g-C₃N₄

Two significant limitations, low specific surface area and single active functional group, restricted the adsorption performance of bulk g-C₃N₄. The unmodified C₃N₄ tends to aggregate because of van der Waals interactions, resulting in the decrease of the available functional groups and active sites to bind metal ions. Thereby, various surface modified strategies have been exploited by introducing the stabilizer to improve the dispersion in solution and to prevent the aggregation of bulk g-C₃N₄.

Many investigators have concentrated on coating g-C₃N₄ nanomaterials with polymer attachment. Various polymer coatings have been applied successfully, such as β -cyclodextrin (β -CD) modified multifunctional layer-layer nanosheet^[48], decorating with polydopamine (PDA) and grafting with polyethyleneimine (PEI) brush^[49], EDTA-immobilized method^[50], and melamine-based dendrimer amine^[51]. These layered coating materials can attach onto the surface of g-C₃N₄ to provide electrostatic repulsion and electric steric stabilization, which can prevent the layer-by-layer aggregation and improve the adsorption ability to heavy metal ions. As a result of modification and functional decoration, the modified C₃N₄, with high specific surface area, high porosity and diverse functional groups, showed high removal capacity for heavy metal ions from large volumes of aqueous solutions.

Zou *et al.*^[48] synthesized g-C₃N₄-based materials by loading β -CD onto g-C₃N₄ for Pb(II) removal. On the one hand, the modification method kept the graphite-like layer and irregular wrinkles morphology of g-C₃N₄. On the other hand, diverse oxygen-containing functional groups (*i.e.*, C-C/O, C-O-C and C=O groups) were introduced owing to the abundant hydroxyl groups of β -CD. Wang *et al.*^[49] successfully synthesized the effective and environmental-friendly laminated C₃N₄, decorated with PDA and grafted with PEI brush, named *l*-C₃N₄/PDA/PEI₃. Then the obtained material was ap-

plied to heavy metal ions' remediation from water system under various environmental conditions. From scanning electron microscopy (SEM) images, the morphology of *l*-C₃N₄ changed attributed to PDA and PEI grafting on the surface of *l*-C₃N₄. The adsorption ability reduced with the increase of PEI content. The results could be explained by two reasons: (1) amino-groups on the surface of *l*-C₃N₄/PDA/PEI_x were the active sites for metal ion removal. By increasing PEI content, the active amino-groups in loading PEI became less, which resulted in the lower adsorption capacity; (2) with the increase of PEI content, the diffusion resistance increased, which unfavorably affected metal ions' elimination^[49]. Teng *et al.*^[50] fabricated a voltammetry sensor for selective determination of ultra-trace Pb(II) which was prepared based on EDTA-immobilized g-C₃N₄ nanosheets (EDTA-CN-NS). The result indicated that Pb(II) could be extensively accumulated onto the surface of the adsorbent due to the chelating effectiveness of EDTA groups. The specificity between functional groups and a specific kind of heavy metal ion was the inspiration of modified strategy. Similarly, Anbia *et al.*^[51] attached amino groups on the surface of C₃N₄ with post-grafting process. The synthesized dendrimer amine-grafted mesoporous C₃N₄ (MDA-MCN-1) was employed to the adsorption of Cu(II) and Pb(II) from wastewater.

Surface modification to bulk g-C₃N₄ could improve the specific surface area, water solubility and the oxygen- and nitrogen-containing functional groups (*e.g.*, carboxyl, hydroxyl, epoxide and surface amino). In general, these functional groups could form strong surface complexes with heavy metal ions. Based on the above discussion, researchers could modify C₃N₄ according to the characteristics of the target pollutant for selective elimination and preconcentration target toxic metal ion. The beneficial conditions for *in-situ* elimination and solidification of metal ions under complicated conditions could be applied attributed to specific surface modification.

1.4 Element doping of g-C₃N₄

Doping of g-C₃N₄ could be regarded as the development of introducing specific impurities and additional elements into pristine g-C₃N₄ to distinctly tune the solubility, outer electron density, specific surface area and other physicochemical properties of bulk g-C₃N₄. The elemental doping strategies have been divided into two main types: metal doping and nonmetal doping. As for adsorption of toxic heavy metal ions, the effective combination between active sites and target ions is the most fundamental purpose. Hitherto, there are several studies have been reported about the element doping of metals and nonmetals such as Li, Na, K, Mg, Ca, Sr, Ba, B and S.

Li *et al.*^[52] reported the synthesis of sulfur-doped $g\text{-C}_3\text{N}_4$ ($S\text{-}g\text{-C}_3\text{N}_4$) by pyrolysis for S- and N-rich supramolecular polymer precursor, which induced the new active sites for adsorption. The increased specific surface area was attributed to the introduction of trithiocyanuric acid (TCA), the new precursor. The N_2 adsorption-desorption isotherms showed that the specific surface area of $S_{3.9\%}\text{-}g\text{-C}_3\text{N}_4$ ($16.3\text{ m}^2/\text{g}$) was much higher than that of $g\text{-C}_3\text{N}_4$ ($8.6\text{ m}^2/\text{g}$), indicating that the $S_{3.9\%}\text{-}g\text{-C}_3\text{N}_4$ sample would expose more nitrogen- and sulfur-containing groups for the adsorption of heavy metal ions than $g\text{-C}_3\text{N}_4$. The fine structure of S replaced sites in the triazine ring was evidenced by the DFT calculations. Furthermore, the total densities of state (TDOS) patterns of the above structures were calculated. Combining with the experimental results, $\text{C}_3\text{N}_4\text{-S-N}^3$ might be the most stable species in the substituted $S_x\text{-}g\text{-C}_3\text{N}_4$ structures. Peng *et al.*^[53] reported porous boron C_3N_4 nanosheets (BCN NS) modified by the pyrolysis of the mixture of melamine and boric acid. BCN NS showed excellent adsorption capacities for Hg(II) and Pb(II) . Besides, the as-prepared material exhibited excellent chemical stability, recycling and renewable abilities.

Different methods for the modification of $g\text{-C}_3\text{N}_4$ with different functional groups are illustrated in Fig. 2. The surface modification is an efficient method to change the surface functional groups, the structures, the band gaps and the active sites of pure $g\text{-C}_3\text{N}_4$. Therefore, the sorption capacity and improve the sorption selectivity of $g\text{-C}_3\text{N}_4$ could be improved and applied under complicated environmental conditions.

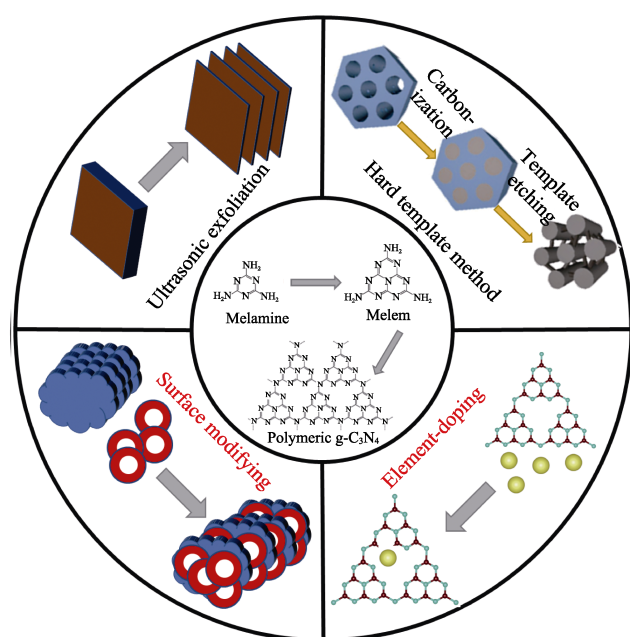


Fig. 2 Different modification strategies of $g\text{-C}_3\text{N}_4$ based materials

2 Removal of heavy metal ions by $g\text{-C}_3\text{N}_4$ -based materials

Heavy metal ions (such as Pb(II) , Cd(II) , Cu(II) , Zn(II) , *etc.*) and radioactive elements (such as U(VI) , Eu(III) , Am(III) and Th(IV) , *etc.*) have great potential risks to the environment and human health because of the severe harmfulness and permanency. $g\text{-C}_3\text{N}_4$ -based materials have been received much attention in recent years due to their unique properties and high sorption capacities. Furthermore, their unique structure, such as heptazine (C_6N_7) units or triazine ring (C_3N_3) units and $\text{sp}^2\text{ C-N}$ active sites, make them excellent candidates for heavy metal ions and radionuclides' elimination. Inchoative researches about heavy metal ions and radionuclides adsorption were reported using the bulk $g\text{-C}_3\text{N}_4$ nanomaterials. Following the basis material, the further progress for pollutant decontamination by $g\text{-C}_3\text{N}_4$ and its derivatives has become the hot spot in this field. The objective of this section is to review a classified and systemic summary of these meaningful progress. The adsorption researches about several representative heavy metal ions and radionuclides by the $g\text{-C}_3\text{N}_4$ and its derivatives are listed in Table 1. Besides, two probable interactions existed in the adsorption process. One is that heavy metal ions can be coordinated with $\text{C}\equiv\text{N}$ or N-H at the edge of $g\text{-C}_3\text{N}_4$ by forming complexes. The other is that the conjugated π -electron pairs of C_6N_7 units or C_3N_3 units and $\text{sp}^2\text{ C-N}$ can be regarded as Lewis base, meanwhile, the pollutant cations can be considered to be Lewis acid. From the point of Lewis acid-base theory, the reaction to form powerful complexes was very easy to take place between $g\text{-C}_3\text{N}_4$ and metal ions in the Lewis acid-base reactions. Besides, according to comparing the peak area, conjugation π -electron pairs of triazine units' interaction acted as the major contributor in metal ions' removal onto $g\text{-C}_3\text{N}_4$. In the other words, the metal cations accepted electrons, forming the coordination, were not constrained by the atomic, but around the triazine units.

2.1 Removal of Pb(II)

Pb(II) is an essential element in many industries. However, its leakage, discharge and pollution will arouse widespread worries because of its harmfulness to the aqueous environment and humans. Consequently, it is crucial to prevent Pb(II) from dispersing into the ecological system and scientific dispose of Pb(II) -wastewater. In 2015, Shen *et al.*^[46] reported a salt-melting method for the synthesis of $g\text{-C}_3\text{N}_4$ particles, which had an effective adsorption capability for Pb(II) (281.8 mg/g). Besides, the toxic metal ions could be eliminated quickly in a wide pH range. The interaction between the carbon-/

Table 1 The adsorption of heavy metal ions on g-C₃N₄-based materials

g-C ₃ N ₄ -based material	Adsorbate	(<i>m/V</i>)/(g·L ⁻¹)	pH	C ₀ /(mg·L ⁻¹)	Time/h	<i>T</i> /K	Q _{max} /(mg·g ⁻¹)	Interaction mechanism	Ref.
g-C ₃ N ₄	Pb(II)						282		
	Cu(II)	3.0	3.5	20	1	298	134	Inner-sphere surface complexation	[46]
	Cd(II)						112		
	Ni(II)						38		
Pb(II)		5				720			
Coral reef-like g-C ₃ N ₄	Cd(II)	0.2	5	10	2		480	Surface complexation	[31]
	As(V)		3				220		
g-C ₃ N ₄ /β-CD	Pb(II)	0.3	5.5	10	20	298	101	Complexation and electrostatic interaction	[48]
S-g-C ₃ N ₄	Pb(II)	0.2	4.5	10	2	298	53	Inner-sphere complexation	[52]
BCN NS	Pb(II)	0.4	7	108.0	0.7	298	211	Electrostatic interaction and molecular interaction	[53]
	Hg(II)			307.8			625		
Fe ₃ O ₄ &g-C ₃ N ₄	Pb(II)	1	6	200	1	298	424	Conjugation	[54]
GNS	Pb(II)	1	5.1		0.7		407	Ion exchange	[56]
	Cd(II)		6.2			73			
2D-g-C ₃ N ₄ nanosheets	Cd(II)	0.3	7	200	5	318.5	94	π-π conjugate interaction and electrostatic attraction	[57]
BCN	Cd(II)	2	5	11	0.2		159	Complexation	[59]
OM g-C ₃ N ₄	U(VI)	0.2	4	10	1	298	150	Chemisorption	[32]
l-C ₃ N ₄ /PDA/PEI ₃	U(VI)	0.5	5	40	10	298	101	Inner-sphere complexation and surface co-precipitation	[49]

nitrogen-containing active sites and heavy metal ions could be explained by inner-sphere surface complexation. Tan *et al.*^[31] synthesized the coral reef-like C₃N₄ particles with high surface area (24.5 m²/g) and the abundant of carboxyl functional groups, which possessed a superior adsorption ability for Pb(II) (720 mg/g) (Fig. 3(a)). Zou *et al.*^[48] used β-CD to fabricate the g-C₃N₄/β-CD adsorbent for the elimination of organic dye and Pb(II) ions under various environmental conditions (*e.g.*, temperature, pH, contact time and solid content). The as-prepared g-C₃N₄/β-CD could adsorb Pb(II) ions from solution in short time. Li *et al.*^[52] prepared C₃N₄-S-N³ for the elimination of Pb(II). According to hard-soft acid-base (HSAB) reactions, named the Pearson Theory, soft bases with donor atoms such as S prefer to coordinate to soft acid (*i.e.*, Pb(II)). The experimental results and theoretical calculation (Fig. 4) revealed that the structure of C₃N₄-S-N³ was the most stable, and the adsorption energy (*E*_{ad}) values of S-g-C₃N₄ for Pb(II) removal were higher than that of g-C₃N₄. According to the relevant result, the sample with lower sulfur content possessed higher adsorption capacity. The monosubstituted model, C₃N₄-S-N³, presented outstanding wastewater treatment performance with adsorption capacity of 53 mg/g for Pb(II) calculated from the Langmuir model under a relatively low pH (~4.5). Based on the study of Peng *et al.*^[53], the as-prepared BCN NS exhibited excellent adsorption performance toward Hg(II) and Pb(II) with the maximum adsorption capacity of

~625 and ~211 mg/g, respectively. Furthermore, BCN NS showed outstanding chemical stability and the adsorption capacities after 6 adsorption/desorption cycles (persist over 95%). Relying on the facile manufacture and cost performance approach, the BCN NS with the outstanding adsorption capacity showed great potential for practical application in the field of wastewater cleanup. Due to the relatively high cost of preparation, the application of advanced nanomaterials faces many obstacles. Thereby, the promotion of recycling capacity for adsorbents becomes the research attention.

Compositing adsorbent with magnetic property is a simple and operable technique to improve the rapid magnetic response. Guo *et al.*^[54] developed a novel lamellar structure magnetic Fe₃O₄@g-C₃N₄ adsorbent with high adsorption capacity for Pb(II). Fe₃O₄@g-C₃N₄ showed excellent removal performance under different conditions (*i.e.*, pH, initial concentration, contact time and ionic strength). The adsorption kinetics data were well described by the pseudo-second-order model. The adsorption isotherms were well simulated by Langmuir model with the adsorption capacity of 424 mg/g for Pb(II). From the XPS analysis, the binding energy value of C1s and N1s of material shifted after Pb(II) adsorption, which revealed the change of local bonding environments. The results proved that the lone pair of electrons was supplied by both C and N element in adsorption process. Actually, the shielding effect on the nucleus was

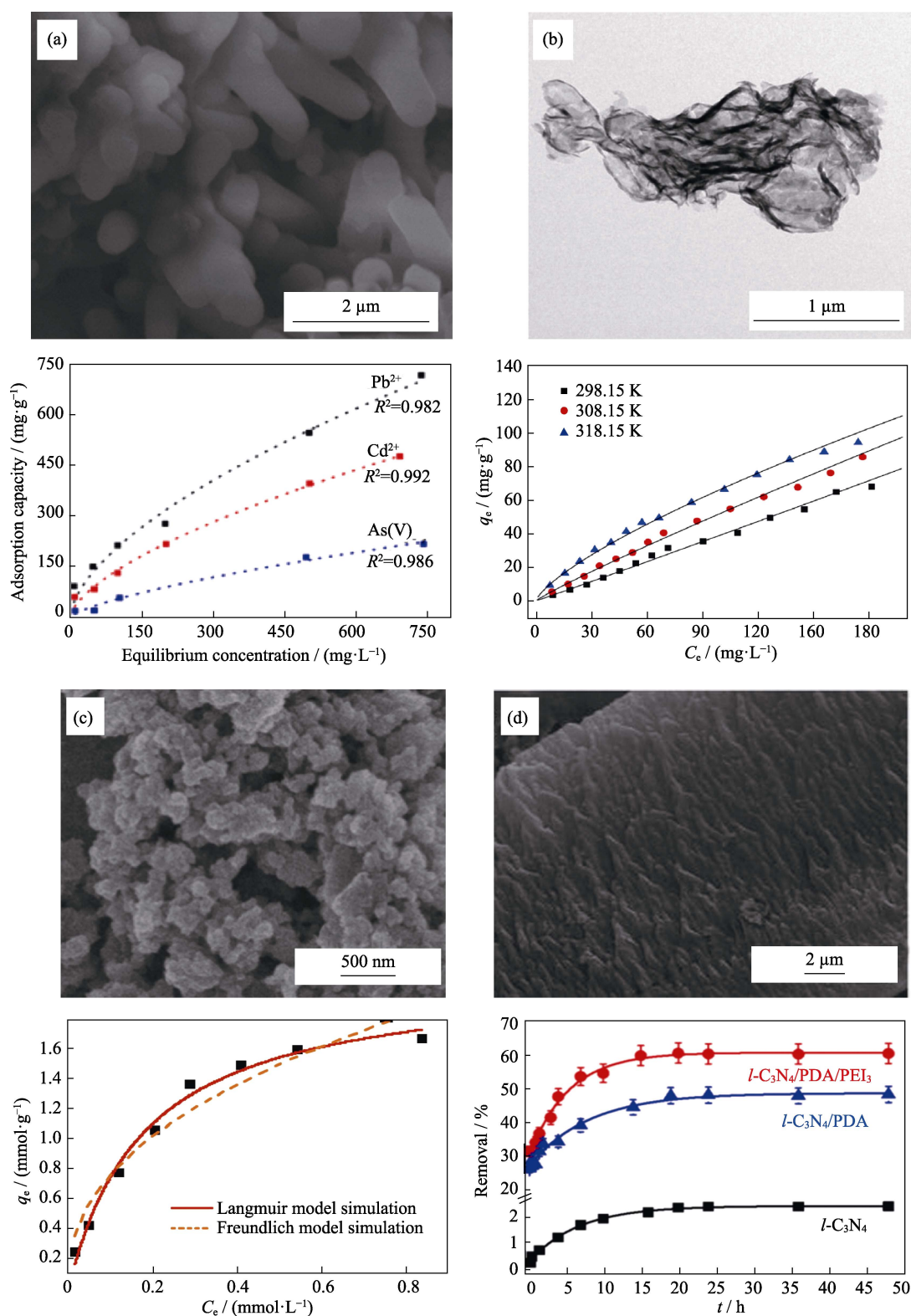


Fig. 3 SEM/TEM images and adsorption isotherm results for heavy metal ions (a) Pb(II), Cd(II) and As(V)^[31]; (b) Cd(II)^[57]; (c) Cu(II)^[46]; (d) U(VI)^[49]

weakened and the attractive force exerted by the nitrogen and carbon nucleus on the Pb2p electron. The conjugated π -electron pairs of C_6N_7 units or C_3N_3 units played a crucial role in the complexation reaction. Moreover, the Pb(II) adsorbed on $Fe_3O_4@g-C_3N_4$ could be regenerated by EDTA and the adsorption capacity of regenerative

adsorbent maintained 88.9% after 5 cycles^[55]. Typically, combining $g-C_3N_4$ with nanomaterials, like magnetic materials, possessing a high level of active sites is an effective strategy considered frequently. It was believed that this kind of excellent materials would contribute to efficient emerging adsorbents for real applications in

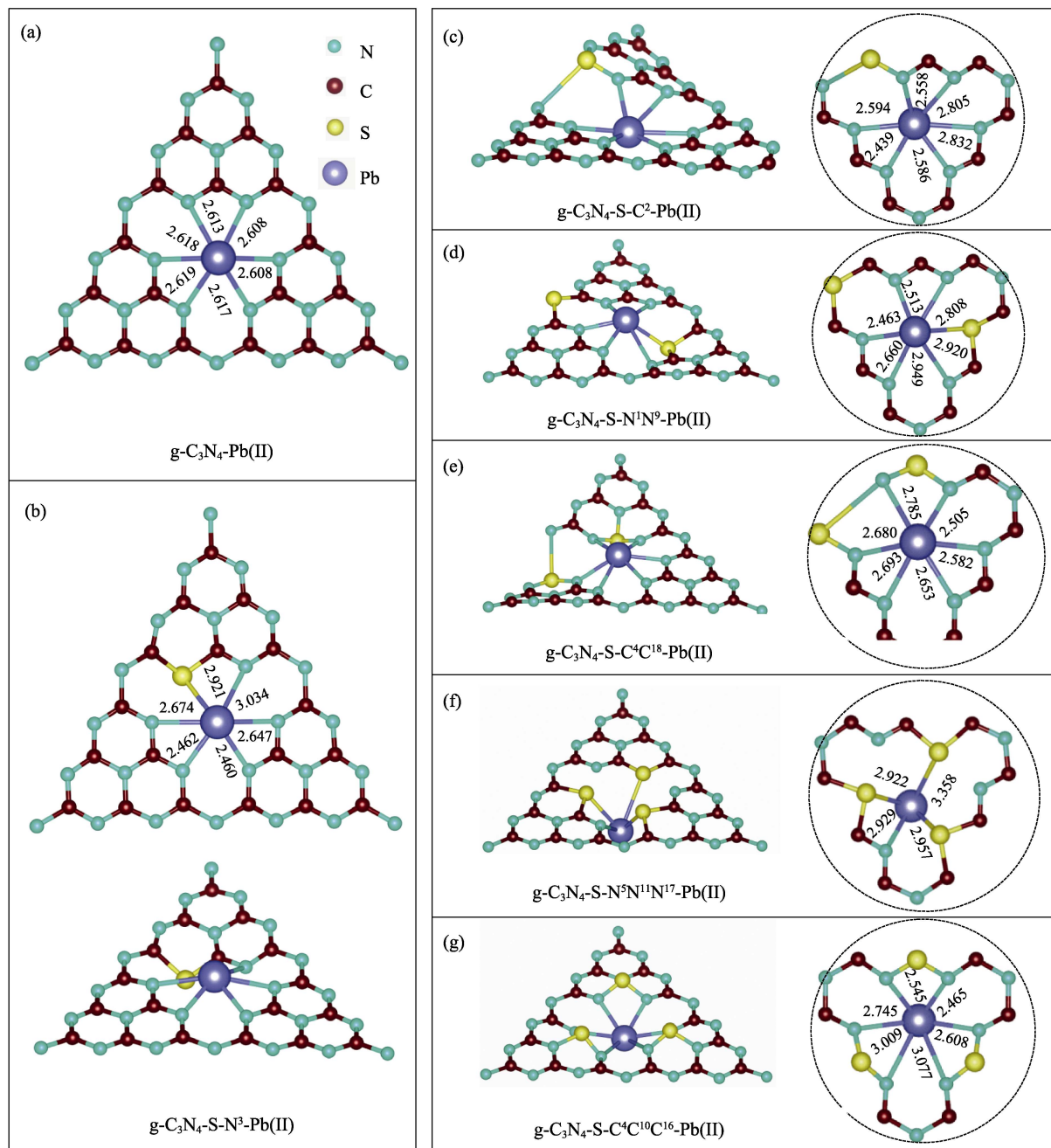


Fig. 4 Optimized structures and the corresponding bond lengths of (a) pure (g-C₃N₄)-Pb(II) and (b-g) (S-g-C₃N₄)-Pb(II) complexes^[52]

polluted water treatment.

2.2 Elimination of Cd(II)

Heavy metals such as cadmium (Cd) in water have been an issue in recent years because it could lead to accumulative poisoning and nervous system damages^[56]. The ordered C₆N₇ or C₃N₃ units contain abundant “nitrogen pots” in which six nitrogen lone-pair electrons possess negatively charged functionalities. The functional groups could be quite suitable for adsorption of Cd(II). Cai *et al.*^[57] reported a series of g-C₃N₄ synthesized at different temperatures (450, 500, 550, 600, and 650 °C), which were named as CN-450, CN-500,

CN-550, CN-600 and CN-650, respectively. Meanwhile, a series of materials fabricated with different pyrolysis time (0, 60, 120, 180, and 240 min) at 550 °C were labelled as CN-0, CN-60, CN-120, CN-180, and CN-240. From the experimental results, CN-500 possessed a high adsorption capacity of ~ 34.6 mg/g for Cd(II) at the initial concentration of 100 mg/L. The maximum adsorption capacity of g-C₃N₄ at the initial concentration of 200 mg/L was 94.4 mg/g (Fig. 3(b)). The desorption and reusability results indicated that g-C₃N₄ nanosheets had decent reusability and chemical stability, which were favorable for the recurring adsorption of Cd(II). The available adsorp-

tion sites on g-C₃N₄ were modulated by the surface properties, the conjugate π region and the aqueous dispersion of g-C₃N₄ colloids. There are many functional groups on the g-C₃N₄ sp³-hybridized edges. In addition to the planar π - π surface, the -NH₂ functional groups also serve as potential adsorption sites. XPS analyses demonstrated that the lone-pair electrons between the C and N atoms of C₆N₇ units in g-C₃N₄ coordinated with the unoccupied 5s orbital of Cd(II)^[58].

Magnet-based nanocomposites are also applied to decontaminate Cd(II). A large amount of magnetic adsorbents with high adsorption capacity and short magnetic response time have been developed by researchers. The adsorption of Cd(II) on magnetic Fe₃O₄@g-C₃N₄ showed that the pseudo-second-order model fitted better than pseudo-first-order model, indicating that the decisive process was the chemical process when adsorbing metal ions. The adsorption process could be explained by intraparticle diffusion model. The first stage was owing to metal ions adsorbed on surface of material. In this section, over 80% Cd(II) cations were adsorbed within a short time. When the exterior surface of Fe₃O₄@g-C₃N₄ was saturated, Cd(II) ions gradually diffused into the pores of adsorbent. With the pore diameter got smaller, the diffusion resistance of Cd(II) ions became larger, generating the diffusion rates to decline. The third stage was equilibrium process, *i.e.*, the adsorption and desorption reached equilibrium. The adsorption isotherms indicated that the Langmuir model was a little more appropriate than Freundlich model, suggesting that monolayer and homogeneous adsorption of Fe₃O₄@g-C₃N₄ were advocated in adsorption process. The maximum adsorption amount for Cd(II) was calculated to be 169 mg/g. The mechanism of adsorption process could be explained by the extreme difference (ED) ($ED=Q_{\max}-Q_{\min}$). The ED of Cd(II) was calculated to be 14 mg/g, indicating electrostatic force was not the major interaction mechanism. Herein, it could be summarized that the chemical adsorption was the rate controlling step. Besides, the N-H or C \equiv N of Fe₃O₄@g-C₃N₄ could combine with Cd(II) by forming strong complexes. The coordinate bonds are the main form between metal ions and adsorbent^[55]. Tan *et al.*^[59] fabricated the B-doped g-C₃N₄ for Cd(II) adsorption. The introduction of B, using boric acid as boron resource, also introduced oxygen atom into g-C₃N₄, which existed in the form of O-H groups. The batch experimental results illustrated that the sorption capacity of un-doped g-C₃N₄ was much lower than that of B-doped g-C₃N₄, indicating that the incorporation of B atom improved the Cd(II) adsorption capacity of g-C₃N₄. Based on FT-IR and XPS analysis, O-H groups were main active sites in the process of Cd(II) removal.

2.3 Uptake of Cu(II)

Copper (Cu) is an essential microelement in the process of plant and animal growth. However, it is toxic to environment at higher level. Excess Cu(II) could lead to biological enrichment effect, especially in the liver. Meanwhile, it could be toxic to fish at lower pH. It is essential to remove Cu(II) from natural water environment.

Normally, it was indisputable that the inner-sphere surface complexation was appropriate to clarify the interaction between the nitrogen- and carbon-containing functional groups of g-C₃N₄ and heavy metal ions. Thus, a series of studies were investigated in regard to the adsorption of Cu(II). Shen *et al.*^[46] fabricated g-C₃N₄ by a simple and environmental friendly salt melt method. The batch experimental results showed that the adsorption kinetics of Cu(II) followed the pseudo-second-order model and the as-prepared adsorbent exhibited much higher adsorption capacity toward Cu(II) ions (133.7 mg/g) (Fig. 3(c)). They also investigated the effect of ionic strength on the uptake of Cu(II). The influence of aqueous ionic strength could be related to a deduction between the outer-sphere surface complexation and the inner-sphere one. As for inner-sphere surface complexation, the formation of chemical bond was the main reason of adsorption interaction between metal ions and surface functional groups. On the contrary, in the case of outer-sphere one, the chemical force could not form during the interaction. Herein, with the increase of ionic strength, the decrease of adsorption capacity was always observed in outer-sphere surface complexation. The removal ability of inner-sphere surface complexation reaction showed no change or an increase with the increasing ionic strength.

2.4 Decontamination of actinides

The nuclear power, an excellent new energy source, commonly referred to as "solving the energy crisis", has received attention due to its capacity to relieve energy pressure and meet basic energy requirements^[60]. However, the wide application and exploiting of nuclear energy would undoubtedly lead to radionuclides pollution. Especially, actinides are the major and most important environmental contaminants associated with anthropogenic activities such as mining and milling of uranium ores, and generation of nuclear energy resulting in the production of spent fuels^[61]. For the sake of human health and ecological security, many advanced nanomaterials were fabricated and applied for the effective purification of radioactive wastewater. For instance, Zhang *et al.*^[32] synthesized the bio-template encapsulating urea to form ordered mesoporous g-C₃N₄ (OM-g-C₃N₄) with a high adsorption capacity for U(VI) ions (up to 150 mg/g). The hybrid

nanostructure was successfully introduced porous structure with high surface area, excellent thermal stability and multi-active adsorption sites. DFT calculations showed that the strong interaction between U(VI) and g-C₃N₄ with high E_{ad} (655.55 kJ/mol) was owing to the effective adsorption sites inside the holes of g-C₃N₄. Wang *et al.*^[49] manufactured l-C₃N₄/PDA/PEI₃ adsorbent for the removal of U(VI) (Fig. 3(d)). In general, the main driving force between U(VI) and l-C₃N₄/PDA/PEI₃ was surface complexation with various functional groups (C=C, C=O, N=C=C, and C-C or C-H) and electrostatic attraction. Besides, the surface polymer could prevent the aggregation between layers, improved the dispersion of the composites in solution, and increased the multi-functional groups of bulk g-C₃N₄. The compounds were also used to eliminate Pb(II), Cu(II) and Am(III) ions under various conditions. Through facile synthesis process, the efficient adsorption capacity demonstrated that the as-prepared l-C₃N₄/PDA/PEI₃ adsorbent could be a promising material for efficient purification of toxic metal ions and radionuclides from natural environment^[49].

3 Summary and outlook

In this review, we systematically summarized the development of g-C₃N₄-based materials for the adsorption of heavy metal ions over the past few years. Indeed, owing to the diversified structures of g-C₃N₄, progresses on the innovation of materials and clarification of mechanisms have so far accumulated gradually: (1) the advance of surface area, especially synthesized *via* hard template method, could provide more active sites for adsorption and unique property of g-C₃N₄; (2) as a result of modification and functional decoration, with high specific surface area, high porosity and diverse functional groups, the modified g-C₃N₄ kept high removal capacity; (3) doping of g-C₃N₄ could be regarded as the development of introducing useful impurities and additional elements into bulk g-C₃N₄ structure to distinctly tune the solubility, outer electron density, specific surface area and other physical properties of bulk g-C₃N₄.

Despite the tremendous achievements established, there are still some issues and challenges to further explore: (1) compared with other kinds of man-made nanomaterials, the surface properties of modified C₃N₄ is still required to promote; (2) the interaction mechanisms between g-C₃N₄-based materials and pollutants under complex natural environment are still unclear; (3) as excellent semiconductor materials, the modified C₃N₄ materials can be used for catalysis-adsorption integration, which is helpful for *in-situ* elimination and solidification

of high valent metal ions and radionuclides such as Cr(VI), U(VI), *etc.*

Although there are still many challenges in the field of environmental contamination management, we still believe that there would be a bright prospect for the practical application and large-scale construction of g-C₃N₄-based materials under the efforts of scientific researchers. The practical and engineering applications of g-C₃N₄-based materials will be available in the foreseeable future.

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氮化碳基纳米复合材料在重金属去除方面研究进展

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摘要: 石墨相氮化碳材料作为一种重要的二维层状材料, 在光催化、能源存储和环境污染治理等领域引起了广泛关注。氮化碳基复合材料以其稳定的物理化学性质、低成本和环境友好等特点成为不同领域的研究热点。在过去几年中, 氮化碳及其氮化碳基复合材料的制备、性质表征和不同领域应用取得了重要进展。本文总结了近几年氮化碳基复合材料的制备及掺杂和功能化研究, 及其在重金属离子废水中的去除应用, 以及不同研究方法对吸附机理的分析。最后还总结了氮化碳基材料在未来研究和应用中面临的主要问题、挑战和机遇。

关键词: 氮化碳; 吸附; 重金属离子; 作用机理; 综述

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