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REVIEW

Review on Enhancing Separation of Heavy Metal Ions by Cyclodextrin Adsorbent Materials

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Abstract: The traditional techniques for treating wastewater contaminated by heavy metals mostly involve chemical precipitation, solvent extraction and adsorption, ion-exchange, chemical precipitation, and membrane separation. The main shortcomings of traditional procedures are low economic efficiency, lack of environmental friendliness, and poor selectivity. Cyclodextrins are artificial compounds that resemble cages. Through host-guest interaction, pollutants can be adsorbed by its stable inner hydrophobic chamber and exterior hydrophilic surface. It is not only inexpensive and environmentally friendly, but also quite selective. The synthesis and application of materials were reviewed, as well as the primary influencing factors, and the reaction principle of cyclodextrin adsorbent materials for better separation of heavy metal ions. And the future trend of discovery was described.

Key words: β-cyclodextrin; adsorption; heavy metal; composite material; influencing factor

1 Introduction

With the rapid development of economy and technology, smelting, leather production, metals, battery manufacturing, and other industries have developed rapidly. A large amount of industrial wastewater containing metal ions is generated. If it is released into the environment without treatment, it will seriously pollute the ecosystem and waste resources[1]. Generally, the ions of Cd^{2+}, Pb^{2+}, Cr^{6+}, Cu^{2+}, Hg^{2+}, and other precious metal ions are poisonous and difficult to biodegrade. Once they enter and remain in the environment, they will cause cancer and endanger health of people. At present, chemical precipitation, ion exchange, solvent extraction, and membrane separation are the primary techniques used to separate heavy metal ions from wastewater. However, these techniques have some drawbacks, such as low selectivity, the convoluted procedure, high costs, and a large potential for secondary contamination[2].

The adsorption method usually uses porous solid adsorbent to adsorb one or more components in water. In order to well fulfill the aims of enrichment and separation, activated carbon (AC), silica gel, activated alumina, resin, molecular sieves, and other materials are frequently utilized as adsorbents. These adsorbents can effectively absorb heavy metal ions from wastewater without secondary pollution of environment. If magnetic materials are prepared on this basis, the problem of material recycling can also be solved[3–4]. However, the traditional adsorption method still has a low selectivity. For the majority of metal ions, its adsorption capacity is high, but there is a high concentration of impurity ions in the eluted solution. So the products are useless and turn into new hazardous wastes. Therefore, it is urgent to research and develop a kind of new adsorption material with high selectivity, so it can efficiently remove metal ions from wastewater and realize the recycling of metal resources.

β-cyclodextrin (β-CD) is a white crystal formed by hydrolyzing cyclodextrin translocase on starch. In a cyclodextrin molecule, a truncated cone-like plane with a secondary hydroxyl group is called the secondary plane. And a truncated cone-like plane with a primary hydroxyl group is called the primary plane. Six primary hydroxyl groups located on the main plane are the most active. The active order of the three hydroxyl groups in the β-CD molecule is 6-OH>2-OH>3-OH. The most acidic groups are two secondary hydroxyl groups

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on the secondary surface. The acidic order of three groups is 2-OH>3-OH>6-OH. During the chemical modification process, the main modification objects are hydroxyl groups at 6 and 2 positions[5–6], as shown in Fig.1. This structure makes the cyclodextrin cavity hydrophobic, and more hydroxyl groups are distributed on the surface of the cyclodextrin ring, forming the hydrophilic periphery. The cavity size is 0.50–0.95 nm, as shown in Fig.2[6]. The cavity structure of cyclodextrins enables them to form inclusions with guest molecules, which can greatly improve the pollutant removal ability and selectivity.

Nevertheless, β-CD is easily soluble in water, which leads to environmental pollution and the loss of β-CD. In order to resolve the above problems, researchers grafted β-CD onto a solid substrate. For example, β-CD cross-linking and electrospinning PA66 were used to produce nanofiber membranes that were used to absorb heavy metal ions[7]. Pectin β-CD composite was successfully prepared and used for Zn^{2+} and Cu^{2+} adsorption for the first time[8]. By incorporating a crosslinked hydrogel network based on acrylic acid and N, N′-methylenebisacrylamide monomers into a β-CD-based metal-organic framework (MOF) structure, the β-CD-MOF-based porous hydrogel was developed. The synthesized porous hydrogel, denoted as A/M-CDMOF-gel, not only showed enhanced structural stability but also exhibited excellent properties for simultaneous adsorption of Au^{3+}, Ag^{+}, and Pb^{2+} ions[9]. Similarly, β-CD cross-linking and a highly soluble pea starch derivative were used to produce a kind of environmentally friendly nanosponges that had a high carboxyl group content and were used to absorb Cu^{2+}[10]. MoS_2@PEI@β-CD synthesized by crosslinking β-CD and nanosheet MoS_2 is a kind of polymer adsorption material, and usually used to absorb Cr^{5+}[11]. A novel hybrid material was prepared by grafting β-CD onto the surface of oxidized AC for the rapid adsorption of naphthalene[12]. When the AC/β-CD grafting ratio value was 1∶1 and the reaction time was just 10 s, the adsorption equilibrium capacity could reach 178.73 mg/g. It had a faster adsorption rate than many reported adsorbents. The synthesis of aminopyrimidine-modified cyclodextrin MOF (AP-β-CD-MOF) was performed at room temperature with pentanal as a cross-linking agent[13]. This process introduced additional adsorption sites and highly delocalized conjugated systems, consequently enhancing the adsorption performance of material. The synthesis of a hybrid organic/inorganic nanomaterial based on the chemical modification of Ht by grafting modified β-CD units was reported[14]. The synthesized nanomaterial was tested as a sorbent for the removal of antibiotics, organic dyes, and heavy metal ions. From the above, it can be found that the solubility of β-CD in water has been resolved, and the adsorption and selection performance also has been improved. However, it is difficult to recycle β-CD nanoparticles dispersed in aqueous solution, which brings out secondary pollution and wasting of resources.

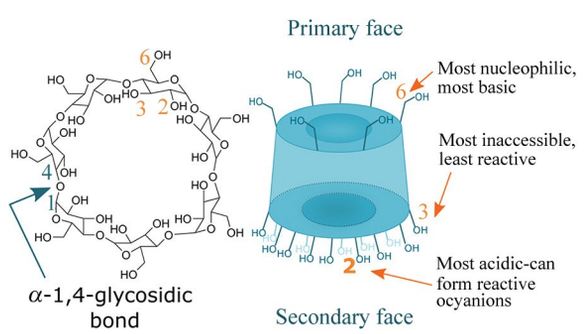

Fig.1 Structure of β-CD[6]

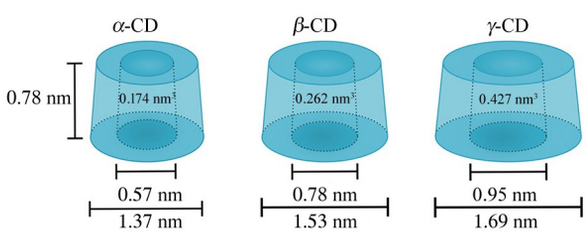

Fig.2 Geometry of α, β, and γ-CDs[6]

In order to solve the recycling problems, the researchers attempted to combine β-CD nanoparticles with magnetic technique, such as magnetic graphene[15–17], magnetic hydrocarbon[18], and magnetic alginate[19]. Kumar et al[20] reported a method to synthesize a synthetic magnetic composite material β-CDs-GO@Fe_3O_4NPs. The graphene oxide (GO) was functionalized by magnetic nanoparticles (MNPs, Fe_3O_4 NPs) decorated with β-CD. The hydroxyl and carboxyl groups of the new adsorbent have excellent water dispersibility and superior magnetic properties. Therefore, it can be used as an effective adsorbent to enhance the adsorption efficiency of As^{3+} and As^{5+}. The Fe_3O_4/NiO magnetic nanomaterials were prepared by microwave-assisted and hydrothermal synthesis in β-CD solution, which could be employed as absorbents and photocatalysts for the degradation of Congo red[21]. Badruddoza et al[22] synthesized Fe_3O_4 nanoparticles modified with carboxymethyl-β-CD (CM-β-CD) polymer for selective removal of Pb^{2+}, Cd^{2+}, and Ni^{2+} ions from water. After the graft of MNPs, many hydroxyl and carboxyl groups in the polymer skeleton were complexed with metal ions, which enhanced the adsorption capacity of the polymer.

In recent years, with the rapid development of science and technology, β-CD has received great attention of researchers due to its unique advantages. It is combined with porous solid matrix materials to form new adsorbent materials. The efficient recovery of valuable metal ions from wastewater using the high selectivity of β-CD has become an industry hotspot. Therefore, the reaction principle of heavy metal ions separation, material synthesis and application, the key influencing factors of the cyclodextrin adsorption material, and the future development trend were reviewed.

2 Reaction Principle

The unique cavity structure of β-CD provides a host-guest interaction center, and the abundance of hydroxyl groups provides an acidic environment. So, β-CD can be covalently bonded with hydrophilic metal ions. Researchers have carried

out a lot of studies on the reaction principle of β-CD adsorption and separation of metal ions.

Chen et al[23] prepared a multifunctional magnetic nanosorbent of β-CD and polyvinylimide (Fe_3O_4-PEI/β-CD), which can simultaneously capture methyl orange (MO) and Pb^{2+} from complex wastewater. Fe_3O_4-PEI/β-CD removes MO and Pb^{2+} from aqueous solutions by electrostatic attraction, host-guest inclusion, chelation, and other mechanisms. Huang et al[24] used citric acid (CA) to cross-link β-CD for preparing a polymer adsorbent (CA-β-CD). CA-β-CD was used to remove bisphenol A (BPA), methylene blue (MB), and Cu^{2+}. Meanwhile, the mechanism of adsorption was proposed. One is that the carboxyl group on CA is not only a crosslinking agent, but also the adsorption site of Cu^{2+} and MB. And the other is that the hydrophobic cavity of CA-β-CD can effectively trap non-polar BPA molecules through the host-guest inclusion. The adsorption mechanism is shown in Fig.3. Wu et al[25] used ethylenediaminetetraacetic acid (EDTA) modified β-CD/chitosan (CTS) to rapidly remove Pb^{2+} and acid red (AR) from aqueous solution. And they proposed that the adsorption mechanism of Pb^{2+} was chelation by EDTA as well as electrostatic attraction of hydroxyl and amino groups. The adsorption mechanism of AR was mainly subject-object interaction and electrostatic attraction. The adsorption mechanism is shown in Fig.4. Li et al[26] prepared a sulfhydryl rich β-CD polymer (SH-CDP) to adsorb Pb^{2+} and BPA, and studied its mechanism. The result shows that SH-CDP adsorbed Pb^{2+} because of the chelation of Pb^{2+} with sulfhydryl group.

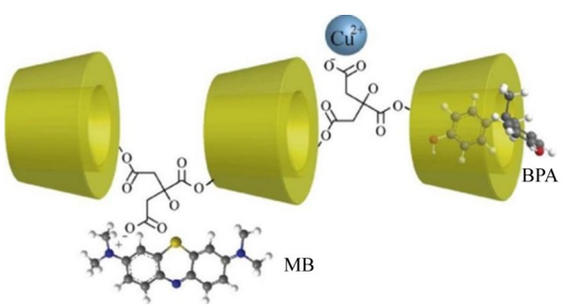

Fig.3 Schematic diagram of CA-β-CD adsorption of BPA, MB, and Cu^{2+}[24]

To sum up, the adsorption mechanism of cyclodextrin is mainly electrostatic attraction, chelation reaction, ion exchange, and surface complexation. This makes β-CD not only have adsorption selectivity but also have excellent adsorption capacity[27].

3 Synthesis and Application of β-CD Adsorption Materials

β-CD is easy to dissolve into water phase and has low mechanical strength, so it is generally grafted on solid materials to improve adsorption and separation performance. Depending on whether the β-CD synthetic material has magnetic properties, it can be divided into non-magnetic and magnetic β-CD adsorption materials.

3.1 Non-magnetic β-CD adsorption material

At present, the commonly used solid carrier materials are mainly CTS, cellulose, graphene, silicon dioxide, etc. They can be polymerized with crosslinking agents to form polymers, such as CA, epichlorohydrin, which use their pores and functional groups to adsorb contaminants.

3.1.1 β-CD and CTS polymerized adsorption material

CTS is an environmentally friendly adsorption material. It has active groups such as amino and hydroxyl groups, and it is easy to form complexes with metal ions. Therefore, it is widely used in the adsorption and removal of heavy metals in water solution. Researchers polymerized CTS and β-CD to prepare β-CD-CTS polymeric adsorbent materials. It has been applied to wastewater treatment and good results have been obtained.

Sikder et al[28] used functionalized CTS impregnation β-CD as a novel adsorbent to adsorb Cd^{2+} in aqueous solution. When the pH value is 6.5, the maximum removal rate of Cd^{2+} was 378 mg/g. Usman et al[29] synthesized a novel nitrotriaceticacid-β-CD-CTS adsorbent (NTA-β-CD-CS) that can effectively remove dyes and metal ions simultaneously in water. The cavity of β-CD trapped MB through host-guest contact. The remaining functional groups on β-CD were able to serve as adsorption sites for metal ions and MO. In a single process, the maximum adsorbed amount of Hg^{2+}, MB, and

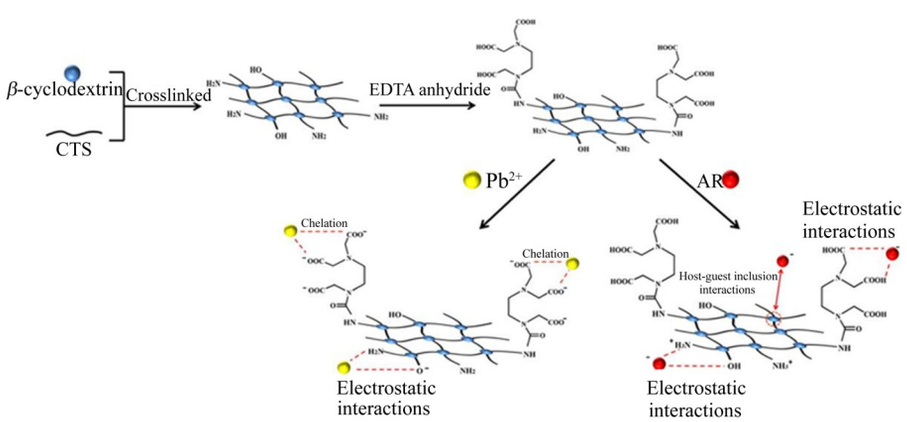

Fig.4 Schematic diagram of EDTA-modified β-CD/CTS adsorption of Pb^{2+} and AR[25]

MO by NTA-β-CD-CS was 178.3, 162.6, and 132.5 mg/g, respectively. After 4 cycles, the performance efficiency of the adsorbent remained unchanged, with excellent regeneration performance and other advantages. Xiong et al[30] used CTS and sodium alginate (SA) as the matrix, crosslinked β-CD and zeolite, and prepared a microsphere adsorbent (SCDO) with high efficiency for adsorbing Cu^{2+} by the microdroplet condensation method. The results showed that when the temperature was 30 °C, the dosage of SCDO was 12 g/L, the initial concentration of Cu^{2+} was 100 mg/L, the solution pH was 6.0, and the adsorption time was 120 min, the Cu^{2+} removal rate could reach 97.08%, and the maximum adsorption capacity was 24.32 mg/g. The adsorption process of Cu^{2+} by SCDO conformed to the quasi-second-order kinetic model and Langmuir adsorption isotherm. It can be seen that β-CD and CTS polymer adsorption material can effectively adsorb precious metal ions, and improve the stability and effectiveness of β-CD.

3.1.2 β-CD and cellulose polymerized adsorption material

Cellulose has the advantages of large specific surface area and high adsorption rate, which is widely used for the removal of precious metals in wastewater. Related research shows that cellulosic materials can adsorb precious metal ions from aqueous solutions, but the effect and selectivity of using cellulose alone are not ideal. In order to improve the adsorption properties and selectivity of cellulose, researchers loaded cellulose and β-CD together to create a new composite adsorption material to adsorb precious metal ions from water.

Qu et al[31] prepared β-CD functionalized rice husk-based cellulose (β-CD@RH-C) and applied it to remove Pb^{2+} from water. The synthesis process is shown in Fig. 5. β-CD@RH-C removed Pb^{2+} from water through complexation and electrostatic interaction, and β-CD@RH-C maintained a high removal rate of Pb^{2+} after 4 cycles. The results indicated that β-CD@RH-C was an efficient adsorbent for removing heavy metal ions from water. Kundu et al[32] used ethylene glycol diglyceride ether as a crosslinking agent, and crosslinked carboxymethyl cellulose (CMC), microcrystalline cellulose (MCC), and xylan with β-CD to prepare hydrogels in alkaline medium with a molar ratio of 1:1 to obtain β-CD-CM, β-CD-CMC, β-CD-xylan, respectively. These three hydrogels were used to adsorb Cd^{2+} and Ni^{2+} in aqueous solution. The results showed that, among all hydrogels, β-CD-CMC hydrogels had the best selective adsorption on Cd^{2+} and Ni^{2+}. When pH=6, the adsorption capacity of Cd^{2+} and Ni^{2+} can reach 24.66 and 15.93 mg/g, respectively. Xia et al[33] used β-CD modified cellulose fiber to prepare a new type of cellulose fiber that could adsorb Cu^{2+} and other precious metal ions. The modified cellulose fiber had a high adsorption capacity of Cu^{2+}, and the adsorption saturation capacity of Cu^{2+} can reach 6.24 mg/g at 293 °C, while the unmodified cellulose had no adsorption. Therefore, cyclodextrin-modified cellulose fibers have good selective adsorption of Cu^{2+}.

3.1.3 Other polymerized adsorption materials

In addition to polymerization with CTS, cellulose, and other materials to form composite materials, β-CD also can be combined with MOFs, graphene, and rigid materials, or directly combined with its own crosslinker to form polymers.

Usman et al[34] prepared β-CD-based nanocomposite adsorbent (β-CD/ZrO_2) for simultaneous adsorption of Pb^{2+} and BPA in wastewater. The adsorption mechanism is shown in Fig. 6. The oxygen-containing group is bound to Pb^{2+}, while the β-CD cavity adsorbs BPA through host-guest interaction. The results show that, in the single pollutant system, the nanocomposites show good adsorption capacity for Pb^{2+} and BPA. The maximum adsorption capacities of Pb^{2+} and BPA at 298 K are 274.4 and 174.9 mg/g, respectively. Due to different adsorption mechanisms, competitive behavior is avoided, and Pb^{2+} and BPA can be effectively removed at the same time. Shahabi et al[35] synthesized a novel insoluble polymer β-CD-

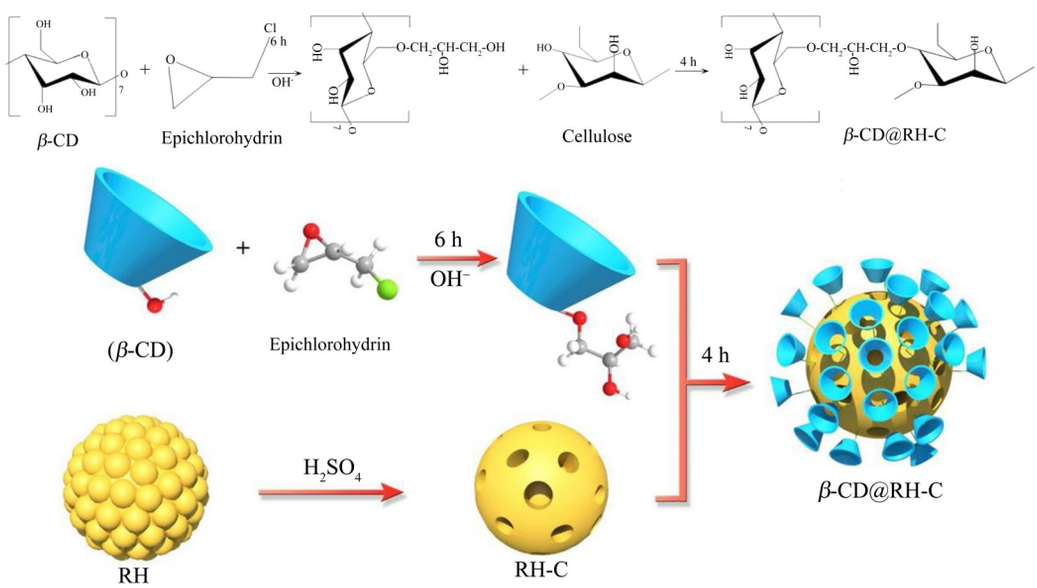

Fig.5 Schematic diagram for synthesis of β-CD functionalized cellulose[31]

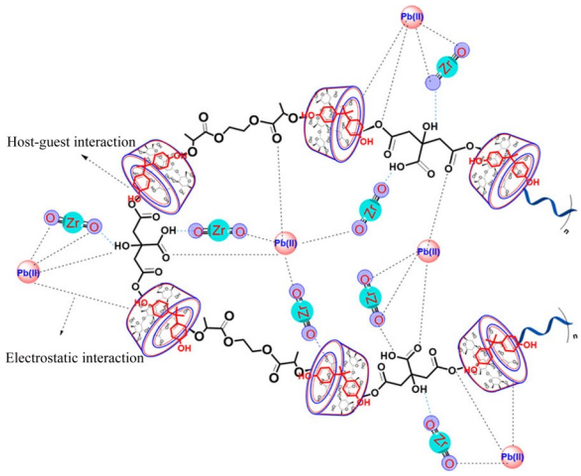

Fig.6 Adsorption mechanism of Pb^{2+} and BPA by β-CD/ZrO_2 nanocomposites[34]

ECH-TU with thiourea (TU) and epichlorohydrin (ECH), which is used to remove Pb^{2+}, Cd^{2+}, and other heavy metal ions from wastewater. The results show that the maximum adsorption capacities of Pb^{2+} and Cd^{2+} are 285.126 and 126.580 mg/g, respectively. The adsorbent is preferred to adsorb Pb^{2+}. The adsorption efficiency of the β-CD-ECH-TU polymer remains unchanged after the regeneration cycle. Yan et al[36] prepared a novel β-CD-CTS-graphene oxide (β-CD-CS-GO) composite for adsorption of Mn^{2+} in aqueous solutions. The results show that the adsorption system has good correlation with Freundlich isotherm model, and the maximum adsorption capacity of Mn^{2+} reaches 177.94 mg/g. The thermodynamic parameters show that the adsorption process is exothermic and spontaneous. The proposed method shows that the prepared β-CD-CS-GO can be applied to adsorb Mn^{2+} in wastewater treatment.

Loading β-CD on CTS, graphene, cellulose, and other materials not only improves the adsorption properties of the materials, but also improves the selectivity of the materials. However, in the actual production process, there are the following problems. (1) If adsorption material is installed in the reaction column, the use of the fixed bed reaction principle can solve the problem of adsorption material recovery. Whereas the processing capacity will be greatly reduced, and the operation process is complex. (2) If it is dispersed in the reactor, the processing capacity is improved, but the adsorbed particles are difficult to recover. This is also the bottleneck problem, which has limited the large-scale industrial application of non-magnetic β-CD adsorption materials.

3.2 Magnetic β-CD adsorption material

In order to solve the problem that non-magnetic β-CD adsorption materials are difficult to recover, researchers polymerized them with magnetic CTS, magnetic silicon dioxide, magnetic graphene, magnetic cellulose, etc, to form magnetic nanosorbents. Magnetic nanosorbents have the advantages of both magnetic separation technique and nanomaterials, and can achieve rapid separation and recovery by external magnetic field while adsorbing metal ions quickly.

3.2.1 β-CD and magnetic silica composite material

Silica has a relative pore size and large specific surface area. Coating silica on the surface of Fe_3O_4 nanoparticles can enhance the adsorption performance and stability of magnetic particles. If the novel magnetic silica combined with β-CD, the adsorption capacity and selectivity of the new material can be improved.

Long et al[37] introduced β-CD/ethylene imide polymer (EIP) into magnetic mesoporous silica, and successfully prepared a new magnetic composite material (EIP-β-CD) to adsorb Hg^{2+}. The results showed that EIP-β-CD had excellent adsorption properties, high selectivity, and fast adsorption rate. Under the optimized experimental conditions, the maximum adsorption capacity of Hg^{2+} was 248.72 mg/g, and the best removal rate was 99.49%. After four times of adsorption and desorption, the removal rate and the adsorption capacity of Hg^{2+} were still as high as 97.48% and 243.69 mg/g, respectively.

Jahanbakhsh et al[38] used novel functionalized magnetic Fe_3O_4@SiO_2 core-shell nanoparticles with CM-β-CD to adsorb Pb^{2+} from aqueous solutions. The grafting reaction was performed via the carbodiimide method using 1-ethyl-(3-dimethylaminopropyl) carbodiimide (EDC) and N-ydroxysuccinimide. Due to the strong adsorption capacity for Pb^{2+} of -OH and -COOH functional groups in CM-β-CD, CM-β-CD was grafted on Fe_3O_4@SiO_2 core-shell nanoparticles to improve the ion adsorption capacity. The adsorption mechanism is shown in Fig. 7. The results showed that the nanoadsorbents had a good adsorption capacity, with a maximum value of 170 mg/g at 25 °C and pH=6.0. All the equilibrium adsorption kinetics of Pb^{2+} was well fitted to the pseudo-second-order model. The adsorption data were also found to follow the Langmuir adsorption isotherm model.

Nkinahamira et al[39] synthesized functional composites PCDP-M-SHM by β-CD hybrid (PCDP@Fe_3O_4) and 2-ethylhexyl phosphonic acid mono-2-ethylhexyl (PC88A) doped silica. The simulated adsorption of rare earth elements (REEs) was studied. It was found that PCDP-M-SHM could selectively purify REEs from synthetic solutions and

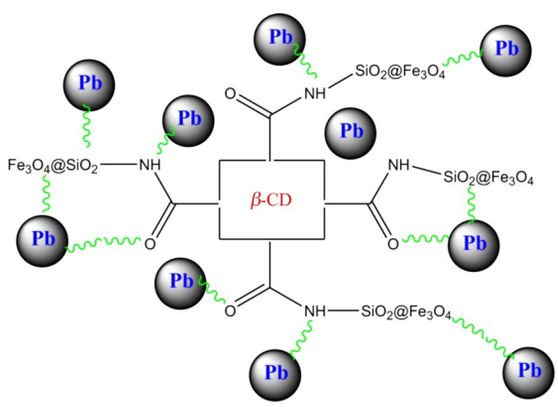

Fig.7 Adsorption mechanism diagram of Fe_3O_4@SiO_2@CM-β-CD magnetic nanocomposite adsorbents on Pb^{2+}[38]

industrial wastewater during the continuous adsorption process, and the enrichment coefficient was between 10 and 30. The separation factor confirmed the effectiveness of PCDP-M-SHM to recover REEs from digested sludge, and its distribution coefficient was much higher than that of coexisting elements. The adsorption and desorption of the adsorbent were carried out five times with a dilute acidic solution, and the adsorption performance of the composite did not decrease.

3.2.2 β-CD and magnetic CTS composites

The product of CTS and β-CD polymerization can adsorb metal ions either by the functional groups of CTS or by the cavity structure of β-CD, but it is difficult to recover. If a magnetic nucleus is added inside the composite adsorbent to make it magnetic, it can be used to recover the adsorption material.

Li et al[40] reported a simple chemical bonding method to synthesize magnetic cyclodextrin-CTS/graphene oxide (CCGO). The adsorption behavior of Cr^{6+} in aqueous solution on CCGO was systematically investigated. The results showed that Cr^{6+} could be quickly extracted from water by CCGO because of its high specific surface area and abundant hydroxyl and amino groups. Due to the magnetic properties of Fe_3O_4, separation can be easily accomplished by magnetic adsorption. The adsorption equilibrium of Cr^{6+} by CCGO was consistent with Langmuir isotherm, and the removal effect of Cr^{6+} by CCGO was better at low pH. The principle is shown in Fig.8.

Liu et al[41] crosslinked CTS with β-CDP to synthesize a biomass-based composite membrane CTS/β-CDP and simulated the adsorption of Zn^{2+} from wastewater using the CTS/β-CDP. The results showed that the maximum adsorption of CTS/β-CDP was 123.7 μg/g and the adsorption rate of Zn^{2+} was 94.14%. Comparing the adsorption effects of CTS, β-CD, β-CDP membranes, and CTS/β-CDP composite membrane on Zn^{2+}, the CTS/β-CDP composite membrane showed the highest adsorption efficiency to Zn^{2+}. The surface of CTS/β-CDP composite membrane was rougher than that of porous CTS membrane, which increased the number of adsorption sites and adsorption efficiency (Fig.9).

3.2.3 β-CD and magnetic graphene composites

Graphene material is an excellent adsorption material with strong mechanical properties, a large specific surface area, and strong electrical conductivity. Magnetically modified GO can achieve efficient separation and recovery of adsorbed materials through external magnetic fields, improving resource utilization and reducing costs. In order to further improve the selectivity of magnetic graphene adsorption materials, researchers grafted β-CD molecules onto magnetic GO to form supramolecular materials. It not only prevented the self-aggregation of graphene, but also avoided the uneven particle size distribution caused by agglomeration. The chemical stability of magnetic nanoparticles becomes stronger, and they have the characteristics of molecular recognition, which makes the synthesized adsorbent material more selective when adsorbing the target substance.

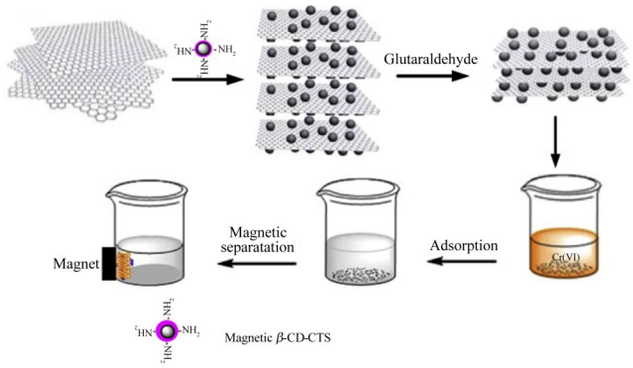

Fig.8 Schematic diagram of synthesizing CCGO and its application in removal of Cr^{6+} in an external magnetic field[40]

Cui et al[42] successfully synthesized resin-loaded magnetic β-CD and GO sheet (MCD-GO-R), and found that it was a good adsorbent of Hg^{2+}. The synthesis and adsorption separation processes are shown in Fig. 10. The results showed that the maximum adsorption capacity was 88.43 mg/g at 323 K and pH=7.1. The prepared adsorbents had good adsorption properties in a wide pH range of 4.0–10.0. Static adsorption experimental data showed good correlation with pseudo-second-order models and Freundlich isotherm models. It was found that the adsorption was accomplished mainly via chelation or ion exchange, and the adsorption reached equilibrium within 30 min. MCD-GO-R had good magnetic properties and could be recovered from aqueous solutions

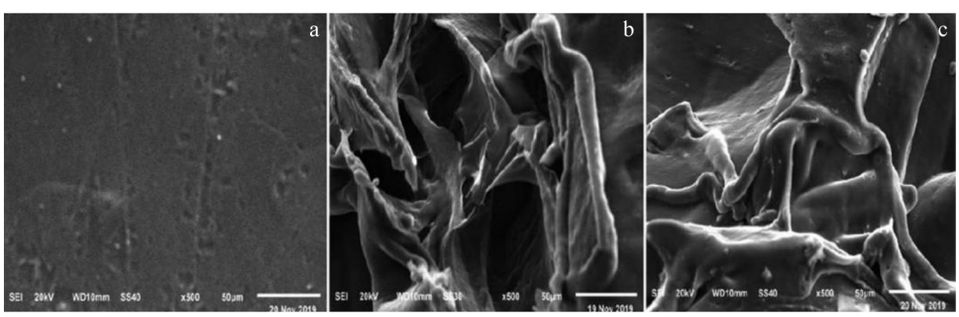

Fig. 9 SEM images of CTS membrane (a), CTS/β-CDP composite membrane (b), and CTS/β-CDP composite membrane after Zn^{2+} adsorption (c)[41]

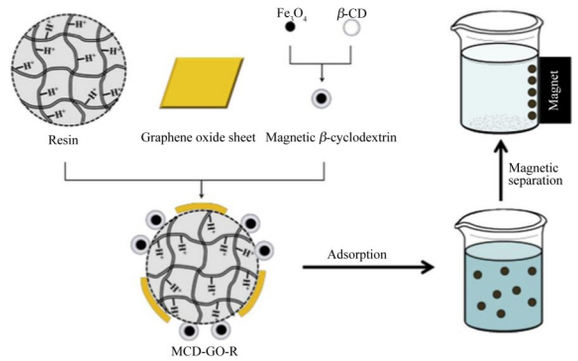

Fig.10 Schematic diagram of synthesis, adsorption, and magnetic separation process of MCD-GO-R[42]

under low magnetic field conditions within 30 s.

Ma et al[43] prepared β-CD modified magnetic graphene oxide (β-CD/MGO) nanohybrids. The adsorption properties of β-CD/MGO for Pb^{2+}, Cu^{2+}, and MB were investigated by batch experiments. The results showed that β-CD/MGO had good adsorption properties for Pb^{2+}, Cu^{2+}, and MB. At 298 K, the maximum adsorption capacities of β-CD/MGO nanohybrid for Pb^{2+}, Cu^{2+}, and MB were 279.21, 51.29, and 93.97 mg/g, respectively.

Einafshar et al[44] developed a novel and facile route for the synthesis of cyclodextrin-conjugated graphene oxide (CDs-GO) nanocomposites by esterification reaction with EDC/4-dimethylaminopyridine (DMAP) as catalyst, as shown in Fig. 11. Then, competitive adsorption capacity of cadmium ion by CDs-GO composites was studied. The results showed that β-CD-GO was suitable for removing Cd^{2+} with 90% removal efficiency at pH=7. The γ-CD-GO and α-CD-GO showed high adsorption capacity of 222.22 mg/g toward Cd^{2+}, which was pointedly more than that of β-CD-GO (208.33 mg/g).

β-CD attached to magnetic materials not only improves the adsorption performance and adsorption selectivity of adsorption materials, but also solves the problems of difficult recovery and a low recycling rate. It improves the utilization rate of resources and has a good research prospect. However,

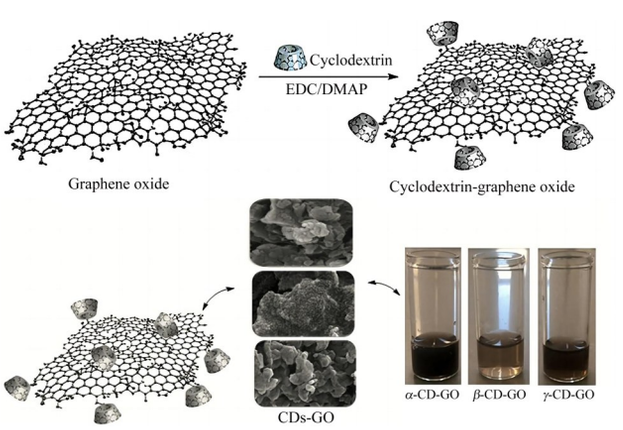

Fig.11 Schematic diagram of synthesizing α, β, and γ-CD-GO[44]

in the actual production process, there are following problems. Agglomeration of magnetic nucleus causes the non-uniform particle size distribution and fails to reach the ideal adsorption effect. In the process of industrial production, it is difficult to realize mass production, which makes practical application difficult. The presence of magnetism, pH values, and other factors have a great influence on synthesis of β-CD magnetic composites, which are the bottleneck problems that limit the large-scale industrial application.

4 Influencing Factor

When β-CD adsorption material is used to remove metal ions from water, it is often affected by pH value, initial metal ion concentration, temperature, coexisting ions, and other factors. In order to optimize the process conditions, researchers have carried out a lot of studies about the influence of the above factors on the removal effect of metal adsorption.

4.1 Effect of pH

In the process of metal ion removal from water by β-CD adsorption material, pH value has an important influence on the removal effect. With the change in pH value in the system, the existence of metal ions and the surface charge distribution of the material will also change. It can directly affect the adsorption mechanism, including electrostatic attraction, electrostatic repulsion, anion exchange, cation exchange, inner surface complexation, and outer surface complexation, and it can affect the interaction and the reaction rate between ions and functional groups of adsorbents.

Ansari et al[45] synthesized a novel Fe_3O_4/hydroxy-apatite/β-CD (Fe_3O_4@HA-CD) nanocomposite adsorbent for the removal of heavy metals from water. The effect of Fe_3O_4@HA-CD on the adsorption of Cd^{2+} and Cu^{2+} was studied when the pH of the aqueous solution was in the range of 2 – 6. The results showed that the adsorption capacity of the adsorbent could be improved by increasing the pH of the solution. At pH =6, the adsorption rates of Cd^{2+} and Cu^{2+} were the highest. There may be the following reasons. (1) When pH<6, copper ions exist in the form of Cu^{2+}, and cadmium ions exist in the form of Cd^{2+} and $Cd(OH)^{+}$. Therefore, in the solution, with the decrease in H^{+}, the competitive adsorption will weaken, and the adsorption amount of metal ions will increase. (2) When the pH value increases, cationic metal ions and adsorbents will generate electrostatic attraction, so the adsorption capacity will increase.

Chen et al[46] modified β-CD with polydopamine and CA. The effect of pH values on the adsorption of Cu^{2+} was studied. The results showed that with the change in pH value, the adsorption effect of the polymer on Cu^{2+} was different. (1) When pH=1.0, the adsorption effect of Cu^{2+} was the most unsatisfactory. This may be due to electrostatic effects. (2) When the pH value was increased to 6.0, the Cu^{2+} removal rate finally reached 73.97%.

Dong et al[47] used β-CD-MOF materials to adsorb Hg^{2+} from wastewater. Results showed that, in a low acid

environment, the adsorption capacity of β-CD-MOF materials for Hg^{2+} was low. Thus, the adsorption amount was increased with the increase in pH. When pH=6.0, the adsorption capacity of β-CD-MOF reached its maximum value of 1392 μg/g.

4.2 Effect of initial metal ion concentration

It is found that the initial concentration of metal ions in an aqueous solution has an important effect on the adsorption removal effect when β-CD adsorption material is used to adsorb metal ions from water. With an increase in the initial concentration of metal ions, the adsorption capacity of adsorbed materials increases first and then reaches equilibrium. In particular, at low concentration, the adsorption of metal ions does not reach saturation, so the adsorption capacity increases with the increase in the initial concentration of metal ions in the aqueous solution. When the adsorption reaches saturation and the initial metal ion concentration is increased, the adsorption tends to be balanced.

Wang et al[48] prepared poly-β-CD conjugated MGO, which was expressed as MGO@poly(β-CD). The adsorption behavior of MGO@poly(β-CD) for Cd^{2+} and sulfamethazine (SMT) in single and binary systems has been investigated. Due to the presence of various functional groups, Cd^{2+} and SMT could be removed through different mechanisms of action. Therefore, at the initial stage of adsorption, the adsorption capacity of Cd^{2+} and SMT increased rapidly with the increase in Cd^{2+} and SMT concentration. In the later stage, all empty binding sites were filled by Cd^{2+} or SMT, and adsorption reached equilibrium.

Nasiri et al[49] prepared hydroxypropyl-β-CD-polyurethane magnetic nanoconjugated/reduced graphene oxide (HPMNPU/GO) supramolecules. The removal effect of the adsorbent on Cr^{6+} and Pb^{2+} in aqueous solution was studied by batch adsorption experiments. The effect of initial metal concentration on the adsorption of Cr^{6+} and Pb^{2+} was investigated, as shown in Fig. 12. The results showed that when the adsorbent content was 10 mg, the temperature was 43 °C and the pH was 2, the maximum removal rate of Cr^{6+} was 97.8%. The initial concentration of Cr^{6+} was inversely proportional to the adsorption efficiency, and the percentage of adsorption efficiency decreased with the increase in initial value of Cr^{6+}. When the adsorbent content was 20 mg, the temperature was 45 °C, and the pH was 7.5, the maximum removal rate of Pb^{2+} was 98.4%.

Yadav et al[50] used functionalized iron oxide (Fe_3O_4), AC particles, β-CD, and SA as raw materials to synthesize Fe_3O_4/CD/AC/SA new polymer gel beads, and then tested their adsorption properties. The results showed that the removal rate of metal ions decreased from 94.11% to 89.70% with the increase in Cu^{2+} concentration in the solution. In the initial stage, the number of molecules adsorbed on the surface of nanocomposite materials was the largest, so the removal rate of metal ions was higher in the initial stage. At higher concentrations of adsorbents, when all the active sites of adsorbents on the surface were saturated, the adsorption sites were reduced, resulting in a decrease in adsorption.

4.3 Effect of temperature

Temperature also has an important effect on the behavior of β-CD adsorption material to separate precious metal ions in water. If the adsorption capacity of the adsorbent is proportional to the temperature, it indicates that the process is endothermic. The main reason is that the number of active adsorption sites increases with the increase in temperature, and the molecular motion is enhanced. Instead, in the adsorption amount decreases with the increase in temperature, it indicates that the adsorption process is exothermic. Because as the temperature increases, the force between the pollutant and the adsorption site decreases.

Yan et al[51] prepared a functionalized magnetic recyclable polyamphoteric hydrogel adsorbent using β-CD and GO, which was used for treatment for cationic and anionic dyes and heavy metal ion wastewater. Adsorption thermodynamic analysis showed that $\Delta H_0 > 0$, indicating that the adsorption process is endothermic. It is also showed that with the increase in temperature, adsorbent's metal ions adsorption capacity increased.

Yadav et al[50] used functionalized iron oxide (Fe_3O_4), AC particles, β-CD, and SA as raw materials to synthesize Fe_3O_4/CD/AC/SA new polymer gel beads to adsorb Cu^{2+}. The effect of temperature on the adsorption behavior of Cu^{2+} was studied.

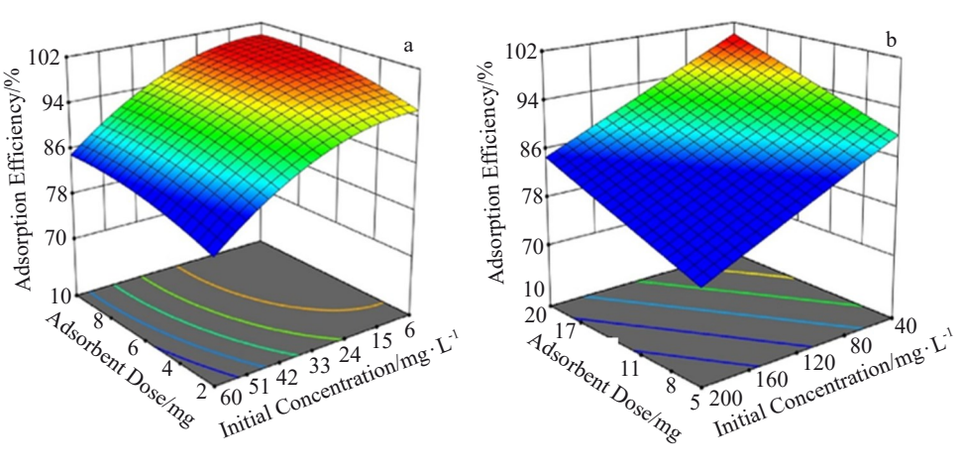

Fig.12 3D surface and contour plots of adsorption efficiency response for Cr^{6+} (a) and Pb^{2+} (b) initial concentration[49]

The results showed that the removal rate increased with the increase in temperature. At 45 °C, the maximum removal rate of Cu^{2+} was 96.8%. It showed that the adsorption process was endothermic.

Yan et al[52] used TiO_2/β-CD modified AC to prepare composite adsorption materials. The simulated adsorption of Pb^{2+}, Cd^{2+}, Cu^{2+}, and Zn^{2+} was carried out in the wastewater environment. The effect of temperature on adsorption was discussed. The results showed that the optimum adsorption temperature was 25–30 °C, and the adsorption efficiency was about 90%.

4.4 Influence of coexisting ions

In general, coexisting ions in water will produce competitive adsorption with target pollutants, which will have a negative impact on the adsorption performance of target pollutants. Therefore, the influence of coexisting ions on the adsorption process of target pollutants has received more attention.

Yadav et al[50] prepared the Fe_3O_4/CD/AC/SA composite polymer and used it to adsorb metals. The effect of coexisting ions on adsorption was investigated. The results showed that cations (Na^+, Ca^{2+}) produced competitive adsorption at the active adsorption sites. The coexisting ions weakened the adsorption of metal ions by the adsorbent.

Verma et al[53] prepared the functionalized β-CD chitosan adsorbent of EDTA (β-CD-CS-EDTA). The adsorbent was used to remove heavy metals and ciprofloxacin micropollutants from wastewater. The effects of coexisting ions (Na^+, K^+, Ca^{2+}, and Mg^{2+}) on the properties of the adsorbent were investigated. The results showed that high concentration of interfering ions had little effect on the adsorption of Ni^{2+}. The adsorption properties of β-CD-CS-EDTA for Ni^{2+} were almost unaffected by coexisting ions.

Wang et al[54] crosslinked β-CD, trihydroxymelamine, and epichlorohydrin in a PVA hydrogel to remove heavy metal ions such as Pb^{2+} and Ni^{2+}. In the experiment, in order to study the selective adsorption of Pb^{2+} and Ni^{2+}, mixed ionic solutions of lead nitrate, nickel nitrate, zinc nitrate, and copper nitrate were added to the adsorption solution (C_0=100 mg/L). The results are shown in Fig.13, and the removal rate of Pb^{2+} was 98.7%, which was much higher than that of other heavy metals. However, the removal rates of Cu^{2+}, Ni^{2+}, and Zn^{2+} also reached 79.52%, 35.5%, and 50.5%, respectively. Therefore, the hydrogel prepared in this study could preferentially adsorb Pb^{2+}. And it also had the ability to remove some other heavy metal ions from the mixed solution.

4.5 Regeneration effect

In practical application, whether the adsorbent can be regenerated and its regeneration effect is of great significance to its application prospects. Abdolmaleki et al[55] prepared a new type of nanoadsorbent, by conducting nucleophilic substitution reactions of monochlorotriazinyl-β-cyclodextrin (T-β-CD) with MNPs and further reacting with Fe_3O_4 magnetic nanoparticles. The adsorbent was used to remove heavy metal ions in an aqueous solution (Pb^{2+}, Cu^{2+}, Zn^{2+}, and Co^{2+}). In this study, in order to investigate the reusability of T-β-CD-MNPs, the adsorption and desorption of T-β-CD-MNPs were carried out over six cycles. In the later cycle, the Pb^{2+} removal rate was still above 92%. These results indicated that T-β-CD-MNP can be used repeatedly for the removal of heavy metal ions in wastewater treatment.

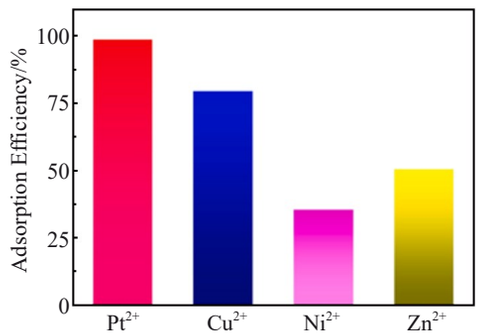

Fig.13 Adsorption efficiency of hydrogel for mixed ions when pH=5, T=298 K, C_0=100 mg/L[54]

Jiahe et al[51] prepared a functionalized magnetic recyclable polyamphoteric hydrogel adsorbent using β-CD and GO to study the stability and reusability of hydrogels. The results showed that, after 8 adsorption-desorption cycles, the adsorption capacity of the prepared adsorbent did not lose significantly. Therefore, the adsorbent had good recyclability and stability.

Hu et al[56] used plasma-induced β-CD grafting to multiwalled carbon nanotubes to synthesize adsorbent. The recycling of β-CD grafted multiwall carbon nanotube adsorbent in the removal of Pb^{2+} was studied. The results showed that with the increase in reuse times, the adsorption capacity of Pb^{2+} decreased slightly from 11.94 mg/g to 10.56 mg/g. After repeated use for 5 times, the efficiency reduction was less than 5%. The results indicated that the β-CD grafted multiwall carbon nanotube adsorbent had a good reusability.

It can be seen from the above parts that in the process of adsorption and separation of heavy metals using β-CD based adsorption materials, the pH value, temperature, initial metal ion concentration, and coexisting ions all have certain effects on the adsorption behavior. The change in pH value will affect the existence of metal ions in the solution and also affect the adsorption mechanism. The adsorption amount increases with the increase in temperature, indicating that the adsorption is an endothermic process. The results show that the adsorption of heavy metals by the β-CD composite adsorbent is generally endothermic. With the increase in the initial concentration of metal ions, the adsorption capacity of the adsorbent for metal ions generally increases first and then reaches equilibrium. The adsorption site on the surface of the adsorbent has the same affinity for the adsorbent. When the initial concentration of metal ions is low, a large number of empty adsorption sites adsorb metal ions quickly. When the adsorption site is filled and the initial ion concentration is increased, the adsorption efficiency will not continue to increase. The specific

Table 1 Effects of various factors on adsorption

Influence factor	pH	Initial metal concentration	Temperature	Coexisting ions	Regeneration effect
Effect	Optimum pH range of 4–7	Increase before balance	Endothermy, optimum temperature of 25–45 °C	Have little effect	Cycle 5–8 times
Ref.	[50,54–55,57]	[45,49,57]	[26,37,58]	[26,33,37]	[46,58–60]

functional groups of the β-CD composite adsorbent can be selectively bound to metal ions. And the pore size of β-CD can also selectively adsorb metal ions, so the coexistence of Na^+, K^+, Ca^{2+}, and Mg^{2+} ions in the solution has little effect on the adsorption of heavy metals. However, to selectively adsorb precious metals with small ionic radius differences, it may be necessary to further explore the functional groups or ligands that can bind them. In order to realize the adsorption and recovery of these precious metals, the effects of various factors on adsorption are shown in Table 1[26,33,37,45,49,50,54–55,57–60].

5 Conclusions and Prospects

β-CD is an environmentally friendly functional material with high selectivity and low cost. In order to obtain the β-CD composite material with larger surface area, stronger stability, and higher selectivity, researchers use pre-synthesis and post-modification methods to introduce specific functional groups into β-CD materials or load the β-CD material with other porous matrix materials. Although great progress has been made in adsorption separation and other fields, the relevant research is not deep enough and needs further exploration. Regarding the research on cyclodextrin materials in adsorption separation, the following aspects are worthy of attention.

5.1 Focus on improving stability of β-CD based adsorption materials

Although a large number of β-CD composites have been synthesized, β-CD materials have the problem of instability after grafting with other materials. Moreover, MNPs are also prone to problems such as uneven particle size distribution. Therefore, it is necessary to further modify β-CD based materials to improve their stability and to broaden the practicability as an adsorption material, so it can be better applied in various fields.

5.2 Developing economical and green synthesis methods

At present, the main problems in the synthesis of β-CD materials are high cost and low yield. This is also one of the bottlenecks that makes it difficult for β-CD materials to achieve industrial applications. Therefore, to develop a simple and easy synthesis method with high yield and low cost is important. This not only facilitates laboratory research, but also lays the foundation for large-scale industrial applications of β-CD materials.

To sum up, combining β-CD with a variety of magnetic and non-magnetic materials not only improves the adsorption performance and selectivity of adsorption materials for heavy metal ions, but also solves the problem of adsorption material recycling. This is an effective method for the recovery of precious metal resources and the adsorption of heavy metal pollutants in wastewater. It has good economic, social, and environmental benefits, in line with the concept of sustainable development. Further research on the above technological challenges is expected to continue, leading to wider applications in environmental, metallurgical, and other fields for the benefit of human society.

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环糊精吸附材料强化分离重金属离子的研究进展

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摘　要：重金属污染废水治理的传统方法主要有化学沉淀法、溶剂萃取法、离子交换法、电化学法和膜分离法等。传统方法存在的问题在于选择性差，经济效益低且环境不友好。环糊精是一种人工合成的笼状分子，具有稳定的内疏水腔和外亲水表面，可以通过主客体作用将污染物封装在其内部，具有选择性强、绿色环保且成本较低的优势。主要综述了环糊精吸附材料强化分离重金属离子的反应原理、材料的合成与应用及其主要影响因素，并对未来发展趋势进行了展望。

关键词：β-环糊精；吸附；重金属；复合材料；影响因素

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