Synthesis of reusable cyclodextrin polymers for removal of naphthol and naphthylamine from water

Weifeng Xu1 · Xiang Liu1 · Jianzhe Cai1 · Tiemeng Xue1 · Kewen Tang1

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Abstract
As one group of important naphthalene derivatives, naphthol and naphthylamine are diffusely employed as dye intermediates. The presence of naphthol and naphthylamine in water systems may pose risks to the environment and public health due to their carcinogenicity. In this study, four mesoporous polymers prepared by β-cyclodextrin derivatives and tetrafluoroterephthalonitrile were obtained and applied to adsorbing 1-naphthylamine, 2-naphthylamine, 1-naphthol, and 2-naphthol from water. The impact of adsorption time, initial concentration of naphthol and naphthylamine, and temperature on the adsorption efficiency of the four polymers were explored separately. The four polymers present fast adsorption kinetics toward naphthol and naphthylamine, attaining 93~100% of adsorption equilibrium uptake for 1-naphthol, 1-naphthylamine, 2-naphthylamine in 15 min, and 87~90% of equilibrium uptake for 2-naphthol in 15 min. The kinetics could be depicted well by the pseudo-second-order kinetic model. The adsorption isotherms of the four polymers toward naphthol and naphthylamine accord with the Redlich-Peterson or Sips model. The maximum adsorption capacities of 1-naphthylamine, 2-naphthylamine, 1-naphthol, and 2-naphthol are 189.9 mg/g, 82.8 mg/g, 137.7 mg/g, and 88.7 mg/g, respectively. The adsorption ratio increases fast with reducing the initial concentration of naphthol and naphthylamine, and the adsorption ratio of naphthol and naphthylamine in 5 mg/L can achieve over 95% in 25 °C. In addition, the four polymers can be effortlessly regenerated by a gentle and simple washing procedure with little reduction in performance. The adsorption performance of the four polymers toward the four naphthalene derivatives can be improved by increasing the adsorption temperature. In conclusion, the prepared β-cyclodextrin polymers exhibit rapid water treatment in removing the four low-concentration naphthalene derivatives with convenient regeneration and good reusability.

Keywords 1-Naphthylamine · 2-Naphthylamine · 1-Naphthol · 2-Naphthol · Cyclodextrin polymers · Adsorption

Introduction
Polycyclic aromatic hydrocarbons (PAHs), released into the environment through natural and anthropogenic activities, are known to have immunotoxicity, genotoxicity, carcinogenicity, and mutagenicity (Haritash and Kaushik. 2009). Naphthalene and its derivatives, a type of PAHs, are frequently present in wastewater produced from dye intermediates (Zhang et al. 2020). Naphthylamine and naphthol are one group of important naphthalene derivatives and diffusely employed as dye intermediates. For example, 1-naphthol is a raw material for synthetic dyes, while it is highly toxic and difficult to be degraded (Zhang et al. 2020). 1-Naphthol and 1-naphthylamine, among top priority contaminants, are regulated as carcinogens and extensively adopted as industrial intermediates. They are ubiquitous in 1-naphthol production wastewater (Yang et al. 2021; Zhang et al. 2008). 2-Naphthylamine, a strong carcinogen, has been banned in the textile industry due to its susceptibility to bladder cancer (Czubacka and Czerczak 2020). Therefore, there is an urgent need to explore an economical, efficient, and green treatment technology to treat naphthol and naphthylamine in water. Compared to other treatment processes, adsorption is generally used as the preferred separation method for removing inorganic and organic pollutants from
water due to its simpleness and effectivity (Hamed 2014; Hamed et al. 2016; Hu et al. 2011; Rebekah et al. 2020; Rizk and Hamed 2015). The feasibility and efficiency of the adsorption process are generally determined by the nature of the adsorbent. The designs of new high-performance adsorbents with longer lifetime are still the research interests and technical challenges (Hu et al. 2020; Yang et al. 2020).

Naphthol and naphthylamine are aromatic pollutants with nonpolar portion (benzene ring) and polar portion (amidogen or oxhydryl). The existence of polar functional groups makes them have certain solubility in water, resulting in their difficult removal from water. The presence of nonpolar portion makes them be efficiently adsorbed by non-polar sorbents. For example, the presence of hydrophobic poly-aromatic groups of un-oxidized benzene rings in graphene makes graphene-based composites be usually used to remove aromatic pollutants from aqueous media, due to the formation of hydrophobic interaction, π-π stacking between graphene and organic molecules (Rebekah et al. 2020). The strong hydrophobic nature and unique graphitic structures of carbon nanotubes (CNTs) allow them interact strongly with organic contaminants through specific π-electron–related interactions (Hou et al. 2013; Hu et al. 2011; Lin and Xing 2008). The presence of benzene ring groups in polyaniline (PANI) polymers facilitates the development of PANI-based materials to adsorb organic compounds with π-π interaction (Zhou et al. 2016).

Cyclodextrin (CD) has a well-defined cylindrical cavity—essentially a “cage” structure. Both ends of the nanoscale cavity have multiple hydroxyl groups. The central cavity of the cyclodextrin cage is hydrophobic, providing ideal coordination for small organic molecules to form the “host–guest” complex compound (Connors 1997). The inclusion process of cyclodextrin involves a variety of forces (such as hydrophobic force, van der Waals force, intermolecular hydrogen bond, and electrostatic attraction), making it much stronger than simple physical adsorption. In an aqueous solution, the driving force for cyclodextrin and its derivatives to form inclusion compounds with guest molecules is the removal of water molecules with high energy from the hydrophobic cavity of cyclodextrin and its derivatives (Crini and Morcellet 2002). High-efficiency cross-linking agents can transform molecular nanocavities into 3D nanoporous polymers. By adjusting the degree of cross-linking, a hydrophobic polymer with a “molecular host” can be obtained, which can capture the target organic compound (Morin-Crini and Grégorio 2013; Zhao et al. 2017). The non-covalent mutual attraction between the guest molecules and the host cavity can effectively remove organic matter even at very low concentrations (Alsbaee et al. 2016; Alzate-Sánchez et al. 2019; Cova et al. 2021; Hu et al. 2020; Li and Ma 1999; Mhlanga et al. 2007; Tian and Liu 2020). Using small polyfunctional molecule as the cross-linking agent, such as epichlorohydrin, would result in a weak mechanical strength of the polymer with a gel-like appearance. The presence of pore structure may be beneficial to the adsorption of polymers toward organic pollutant with low concentration (Alsbaee et al. 2016; Cova et al. 2021; Yang et al. 2020). To synthesize CD polymers with permanent microporosity and high CD content, a multifunctional cross-linking agent with small molecular size and strong structure is needed (Alsbaee et al. 2016; Gu et al. 2006; Mhlanga et al. 2007). As a new cross-linking agent, rigid tetrafluoroterephthalonitrile is proposed by Alsbaee et al. (2016) and Ling et al. (2017). Using this new cross-linking agent, an insolubly porous β-cyclodextrin polymer was obtained and applied to removing aromatic organic micropollutants effectively from water.

Herein, we explore the cyclodextrin derivative polymers with tetrafluoroterephthalonitrile as cross-linking agent to adsorb the naphthol and naphthylamine from water. The cyclodextrin derivatives include β-cyclodextrin, methylated-β-cyclodextrin, hydroxypropyl-β-cyclodextrin, and hydroxyethyl-β-cyclodextrin. The structures of the synthesized polymers and their adsorption performance for naphthol and naphthylamine removal from water were studied comprehensively.

### Materials and methods

#### Materials

- β-Cyclodextrin (β-CD, 98%), hydroxyethyl-β-cyclodextrin (HE-β-CD, 98%), hydroxypropyl-β-cyclodextrin (HP-β-CD, 98%) and methylated-β-cyclodextrin (Me-β-CD, 98%) were purchased from Shandong Binzhou Zhiyuan Biotechnology Co., Ltd. (Shandong, China). Tetrafluoroterephthalonitrile (TFPPN) (98%) and potassium carbonate (K₂CO₃, 99%) were supplied by Saen Chemical Technology Co., Ltd. (Shanghai, China). Tetrahydrofuran (THF, 99.0%) and N,N-diemthylformamide (DMF, 99.5%) were provided by Shanghai Titan Scientific Co., Ltd. (Shanghai, China). Other reagents used were purchased from different vendors and were of analytical grade. HPLC grade acetonitrile (CH₃CN, 99.9%) was purchased from Sigma-Aldrich Co., Ltd. (Shanghai, China) as the solvents for chromatography.

#### Synthesis of polymers

Cyclodextrin and its derivative polymers were synthesized according to the reference (Alsbaee et al. 2016). First, 0.88 mmol of β-CD, HE-β-CD, HP-β-CD, and Me-β-CD were separately added into different 500 mL round-bottomed flasks, and then 2.5 mmol TFPPN and 10.93 mmol K₂CO₃ were added into the flasks. Then, 300 mL of mixed solvent containing THF and DMF with volume ratio of 9:1 was...
added to the flasks under N₂ condition. The mixtures were stirred at 85 °C. After 48 h, the reactions were cooled to room temperature and filtered. The solids in the filter cake were washed with 1 mol/L dilute hydrochloric acid until no bubbles escape. Then, the solids were washed for three times with water for 15 min and soaked in THF for 30 min and in dichloromethane for 15 min. Finally, the yellow solids for β-CD polymer and Me-β-CD polymer, the light-yellow solid for HP-β-CD polymer, and the earthy yellow solid for HE-β-CD polymer were obtained by filtration and freeze-drying for 24 h. The solid materials were ground into powder for the next step of analysis and adsorption experiments. The above four polymers were called poly(TFPNN-β-CD), poly(TFPNN-HE-β-CD), poly(TFPNN-HP-β-CD), and poly(TFPNN-Me-β-CD), respectively.

Characterization of CD polymers

Thermogravimetric analysis (TGA) was conducted on a NETZSCH STA 449F3 thermal gravimetric analyzer under nitrogen atmosphere ranging from room temperature to 600 °C at a ramp rate of 5 K·min⁻¹. The Brunauer–Emmett–Teller (BET) surface and pore size distribution of polymers was determined by applying nitrogen adsorption–desorption isotherm method using an ASAP 2020 instrument (Micromeritics, USA). Scanning electron microscopy (SEM) images were obtained by a FEI Tecnai G² 20 Twin (FEI, USA). The infrared spectrum was conducted using a Fourier transform infrared spectrometer (FT-IR, Avatar 370, Thermo Nicolet, USA) in the range of 400–4000 cm⁻¹. The particle size distributions of polymers were measured using a highly sensitive zeta potential and particle size analyzer (Brookhaven 90Plus PALS, USA) and were determined using a dynamic light scattering (DLS) at 25 °C. Samples were prepared by dispersing 3 mg of each sample in 3 mL of methanol under ultrasonic for 5 min.

Batch adsorption experiment

Synthetic contaminant solutions were obtained by dissolving certain amounts of naphthalene derivatives into deionized water. Adsorption tests were carried out as the following procedures. A total of 10 mg of the synthetic polymer was added into a 50 mL centrifuge tube, and then 10 mL of contaminant solution with a certain concentration was transferred into the centrifuge tube. The centrifuge tube was fully oscillated by cyclotron rotation in a stable temperature bath. The rotation speed is fixed at 250 rpm. After a certain period of oscillation, the centrifuge tube was taken out. The solution of each centrifuge tube was filtered using a 0.22-μm organic nylon filter, and the contaminant concentration of the sample was detected by high-performance liquid chromatography (HPLC). The initial concentration of contaminant solution was 50 mg/L except for isothermal adsorption experiments. Except for kinetic adsorption experiments, the contact time was 120 min. The adsorption temperature was set at 25 °C except for specified description. The pH for all experiments is the original pH value of the contaminant solution without any adjustment.

HPLC analyses were performed on a Waters 2998 HPLC system including a quaternary pump (Waters e2695) and a wavelength-variable UV detector. An InertSil ODS-3 C18 column (250 mm × 4.6 mm, 5 μm) was used for all HPLC analyses. The column temperature was kept at 303.15 K, and injection volume was 10 μL. For all analyses, acetonitrile and water (volume ratio, 8:2) were used as the mobile phase at a flow rate of 0.8 mL·min⁻¹. The wavelength of 1-naphthol, 2-naphthol, 1-naphthylamine, and 2-naphthylamine were monitored at 232 nm, 230 nm, 241.4 nm, and 236.7 nm, respectively. The adsorption capacity (Q, mg·g⁻¹) and adsorption ratio (R%) of CD polymer for naphthol and naphthylamine were used to analyze the adsorption efficiency and calculated by the following equations:

\[
Q_t = \frac{(C_0 - C_t) \times V}{m_1} \quad (1)
\]

\[
R\% = \frac{C_0 - C_t}{C_0} \times 100\% \quad (2)
\]

where \(C_0\) (mg·L⁻¹) and \(C_t\) (mg·L⁻¹) represent the initial concentration and the concentration at \(t\) time; \(V\) (mL) is the volume of the pollutant solution; and \(m_1\) (mg) is the mass of polymer.

Recycling experiment

For regeneration, methanol was used as the desorption agent. For the adsorption experiment, 100 mg of β-CDP was placed in contact with 10 mL of pollutant solution (50 mg/L) for 120 min to reach equilibrium at 25 °C. The suspension was filtered through a filter and the concentration of pollutant in the filtrate was measured. For the desorption experiments, polymer loaded with pollutant was stirred in 10 mL of methanol solution at 25 °C for 120 min. Then, the suspension was filtered, and the desorbed polymer was dried overnight in vacuum freeze-drying agent for the next cycle.

Results and discussion

Characterization

The characterization of the as-synthesized polymers was investigated by thermal gravimetric analyzer, scanning electron microscopy, Fourier transform
infrared spectroscopy, and N₂ adsorption–desorption method. Firstly, the structures of β-CD, poly(TFPPN-β-CD), HE-β-CD, poly(TFPPN-HE-β-CD), HP-β-CD, poly(TFPPN-HP-β-CD), Me-β-CD, poly(TFPPN-Me-β-CD), and TFPPN were characterized by FT-IR. As presented in Fig. 1, the characteristic absorption peaks of C≡N, C–F, and C–C aromatic stretches are obviously observed at 2252 cm⁻¹, 1260 cm⁻¹, and 1250 cm⁻¹ in the FT-IR spectrum of TFPPN, respectively (Lu et al. 2019). The absorption peaks of hydroxyl group at 3386 cm⁻¹, aliphatic C–H stretch at 2929 cm⁻¹, and strong C–O stretch at 1033 cm⁻¹ are observed in the FT-IR spectrum of β-CD, HE-β-CD, HP-β-CD, and Me-β-CD (Duan et al. 2020; Lu et al. 2019). All of the above characteristic absorption peaks of C≡N vibration, C–F vibration, C–C aromatic stretches, O–H vibration, aliphatic C–H stretch, and strong C–O stretch are obviously observed in the FT-IR spectra of poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD), which indicate that the four polymers of β-CD, HE-β-CD, HP-β-CD, and Me-β-CD cross-linked with TFPPN have been successfully prepared.

The surface morphologies of the four prepared cyclodextrin polymers were observed by scanning electron microscope. As revealed in Fig. 2, the surface shapes of the four polymers are irregular, and it can be clearly seen that the four cyclodextrin polymers have rough surface morphology and uneven distribution of pores. The pores in β-CDP were attributed to the introduction of rigid groups in its structure (Hu et al. 2020). The N₂ adsorption–desorption isotherms at 77 K were obtained as shown in Fig. 3, which are type IV isotherms indicative of mesoporosity (Dias and Ciminelli 2000; Wilson et al. 2011). It can be obviously seen from Fig. 3 that a sharp rise in the adsorbed volume for \( p/p^* > 0.9 \) is emerged. This feature is ascribed to the agglomeration between particles, and this type of particle domain represents the large amount of nitrogen adsorption by such low surface area mesoporous materials (Wilson et al. 2011). The
specific surface areas and porosity of poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) calculated by Brunauer-Emmett-Tell (BET) method according to N2 adsorption–desorption isotherms are 20.09 m²·g⁻¹, 19.02 m²·g⁻¹, 21.37 m²·g⁻¹, and 23.51 m²·g⁻¹, respectively. The pore volume and pore diameter from the adsorption isotherm were estimated by the Barrett-Joyner-Halenda (BJH) method. The inset figures in Fig. 3 illustrate the pore size of the four CD polymers ranges from 2 to 5 nm, indicating poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) have the characteristics of mesoporous materials. The average particle sizes of the four CD polymers determined by highly sensitive zeta potential and particle size analyzer (Brookhaven 90Plus PALS) are 0.9 μm, 1.5 μm, 0.4 μm, and 2.0 μm, respectively.

The four cyclodextrin polymers were also characterized by thermogravimetry, exploring the stability of materials at different temperatures. Figure 4 shows that the thermal stability of the four cyclodextrin polymers is similar. The mass loss of water adsorbed on poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) are 6.72%, 6.66%, 6.73%, and 7.31%, respectively, in the temperature range of 25 to 100 °C (Wang and Harrison 2018; Xie et al. 2020). From 100 to 245 °C, the thermogravimetric analysis curves remain stable, suggesting that the synthetic cyclodextrin polymers are stable in this range of temperature studied. The mass loss of poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) were 70.39%, 73.43%, 65.95%, and 67.6%, respectively, in the temperature ranging from 245 to 325 °C, due to the decomposition of materials. When the temperature is higher than 325 °C, the carbon chains start to break down, making the decomposition rates slow down. The above results show that the prepared polymers have good thermal stability.
Kinetics study

To study the adsorption equilibrium time of the four CD polymers for naphthol and naphthylamine, and the kinetics of the adsorption processes, the adsorption time is set from 0.5 to 180 min. As depicted in Fig. 5, the uptake capacities of the four cyclodextrin polymers for 1-naphthylamine, 2-naphthylamine, 1-naphthol, and 2-naphthol increase rapidly at first and then increase slowly until to be constant with the increasing of the adsorption time. The adsorption equilibrium can be achieved within 60 min. In addition, the four CD polymers present fast adsorption kinetics toward naphthol and naphthylamine, attaining 93~100% of adsorption equilibrium uptake for 1-naphthylamine, 2-naphthylamine, and 1-naphthol and 87~90% of equilibrium uptake for 2-naphthol in 15 min, as presented in Table 1.

The kinetics of the adsorption processes were analyzed by the pseudo-first-order (PFO) kinetic model and pseudo-second-order (PSO) kinetic model (Simonin 2016; Ho 2006). The PFO and PSO kinetic models are described as Eq. 3 and Eq. 4, respectively.

\[
\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303}
\]  

\[
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}
\]

where \( k_1 \) (min\(^{-1}\)), \( k_2 \) (g·mg\(^{-1}\)·min\(^{-1}\)), and \( k_i \) are the pseudo-first-order model and pseudo-second-order model rate constants, respectively. The fitting parameters of the PFO and PSO kinetic models are presented in Table 2. As displayed in Fig. 6 and Table 2, the adsorption process of the four cyclodextrin polymers toward four naphthalene derivatives can be depicted well by PSO kinetic model, suggesting chemical adsorption may be involved in the above adsorption processes (Simonin 2016; Ho 2006). The adsorption rate constant of HP-β-CDP for 1-naphthol (Table 2) is larger than that of the other three cyclodextrin polymers, while there

![Fig. 3 The N2 adsorption–desorption isotherms and pore size distribution maps of CD polymers: a poly(TFPPN-β-CD), b poly(TFPPN-HE-β-CD), c poly(TFPPN-HP-β-CD), d poly(TFPPN-Me-β-CD)]](image)

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is no significant difference in the adsorption rate constant ($K_2$) of the four cyclodextrin polymers for 1-naphthylamine, 2-naphthylamine, and 2-naphthol. Additionally, Fig. 5 and Table 2 also show that the equilibrium uptake capacity ($Q_e$) of the four cyclodextrin polymers for the four substances has certain regularity. For 1-naphthylamine and 2-naphthylamine, the order of adsorption effect of the four cyclodextrin polymers is as follows: poly(TFPPN-β-CD) > poly(TFPPN-Me-β-CD) > poly(TFPPN-HE-β-CD) > poly(TFPPN-HP-β-CD). For 1-naphthol and 2-naphthol, the order of adsorption effect of the four cyclodextrin polymers is as follows: poly(TFPPN-Me-β-CD) > poly(TFPPN-HP-β-CD) > poly(TFPPN-β-CD) > poly(TFPPN-HE-β-CD).

Adsorption isotherm studying

Adsorption isotherms play a significant part in the study of adsorption mechanism and practical application. Figure 7 shows the adsorption isotherms of four cyclodextrin polymers toward the four naphthalene derivatives at 25 °C. The uptake capacity increases with the increase of adsorption equilibrium concentration as demonstrated in Fig. 7. The nonlinear Langmuir, Freundlich, Redlich-Peterson, and Sips adsorption isotherm models (Foo and Hameed 2010) (Eqs. 5–8) were used to fit the above adsorption isotherms.

$$q_e = \frac{q_m K_L C_e}{1 + (K_L C_e)}$$  \hspace{1cm} (5)

$$q_e = k_F C_e^{1/n}$$  \hspace{1cm} (6)

$$q_e = \frac{K_R C_e}{1 + \alpha_R C_e^\beta}$$  \hspace{1cm} (7)

$$q_e = \frac{q_m K_S C_e^{d_s}}{1 + K_S C_e^{d_s}}$$  \hspace{1cm} (8)
where $K_L$ (L·mg$^{-1}$) is a Langmuir constant that represents the affinity between solute and adsorbent and $K_F$ (mg·g$^{-1}$) and $n$ are the Freundlich constants related to the adsorption capacity and intensity, respectively, $q_m$ (mg·g$^{-1}$) is the maximum Sips adsorption capacity; $K_S$ (L·mg$^{-1}$) is the Sips constant at equilibrium, and $\beta_S$ is the Sips exponent describing the homogeneity and heterogeneity of an adsorption mechanism.

The fitting results are shown in Table 3. Comparing the square of the correlation coefficient ($R^2$) and chi square coefficient ($\chi^2$) of four adsorption isotherm models, it is found that the three-parameter Redlich-Peterson or Sips adsorption isotherm model can better fit the experimental data, suggesting a mixed process of single- and multilayer adsorption (Yang et al. 2020). In addition, Fig. 7 also shows that the order of adsorption effect of the four cyclodextrin polymers for 1-naphthylamine and 2-naphthylamine is poly(TFPPN-β-CD) > poly(TFPPN-HE-β-CD) > poly(TFPPN-HP-β-CD) > poly(TFPPN-Me-β-CD), while the order of adsorption effect for 1-naphthol and 2-naphthol is as follows: poly(TFPPN-Me-β-CD) > poly(TFPPN-HP-β-CD) > poly(TFPPN-β-CD) > poly(TFPPN-HE-β-CD). This is consistent with the results of kinetics studying.

For some adsorbents, there is a peculiar but interesting phenomenon. The adsorption ratio first rises and then

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**Table 1** The $Q_t$ at 15 min vs. the $Q_e$ of the four CD polymers toward the naphthalene derivatives, %

| CD polymers       | 1-Naphthylamine | 2-Naphthylamine | 1-Naphthol | 2-Naphthol |
|-------------------|-----------------|-----------------|------------|------------|
| Poly(TFPPN-β-CD)  | 94              | 96              | 97         | 89         |
| Poly(TFPPN-HE-β-CD) | 94            | 96              | 93         | 87         |
| Poly(TFPPN-HP-β-CD) | 97            | 97              | 100        | 90         |
| Poly(TFPPN-Me-β-CD) | 96            | 97              | 99         | 90         |

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Fig. 5 The influence of time on the adsorption of naphthalene derivatives by the four CD polymers: a 1-naphthylamine, b 2-naphthylamine, c 1-naphthol, d 2-naphthol
Table 2 The fitting parameters of kinetic models for the adsorption process of the four CD polymers toward the naphthalene derivatives

| Naphthalene derivatives | Polymers              | $Q_e^{exp}$ (mg/g) | PFO model | PSO model |
|-------------------------|-----------------------|--------------------|-----------|-----------|
|                         |                       | $Q_e$ (mg/g)       | $k_1$ (min$^{-1}$) | $R^2$     | $Q_e$ (mg/g) | $k_2$ (g·mg$^{-1}$·min$^{-1}$) | $R^2$ |
| 1-Naphthylamine         | Poly(TFPPN-β-CD)      | 32.46              | 7.23      | 0.05      | 0.87       | 32.60       | 0.03 | 1.00     |
|                         | Poly(TFPPN-HE-β-CD)   | 20.03              | 4.30      | 0.03      | 0.72       | 20.07       | 0.06 | 1.00     |
|                         | Poly(TFPPN-HP-β-CD)   | 27.74              | 3.74      | 0.03      | 0.50       | 27.74       | 0.07 | 0.99     |
|                         | Poly(TFPPN-Me-β-CD)   | 28.61              | 4.44      | 0.03      | 0.52       | 28.60       | 0.06 | 0.99     |
| 2-Naphthylamine         | Poly(TFPPN-β-CD)      | 39.09              | 6.17      | 0.04      | 0.74       | 39.21       | 0.04 | 0.99     |
|                         | Poly(TFPPN-HE-β-CD)   | 24.46              | 4.93      | 0.05      | 0.77       | 24.56       | 0.06 | 0.99     |
|                         | Poly(TFPPN-HP-β-CD)   | 32.29              | 7.09      | 0.06      | 0.84       | 32.37       | 0.05 | 0.99     |
|                         | Poly(TFPPN-Me-β-CD)   | 35.94              | 6.55      | 0.03      | 0.66       | 36.00       | 0.04 | 0.99     |
| 1-Naphthol              | Poly(TFPPN-β-CD)      | 32.60              | 4.05      | 0.03      | 0.54       | 32.68       | 0.04 | 0.99     |
|                         | Poly(TFPPN-HE-β-CD)   | 27.67              | 7.73      | 0.05      | 0.87       | 27.84       | 0.03 | 0.99     |
|                         | Poly(TFPPN-HP-β-CD)   | 33.13              | 1.80      | 0.02      | 0.13       | 33.22       | 0.27 | 0.99     |
|                         | Poly(TFPPN-Me-β-CD)   | 34.24              | 2.59      | 0.04      | 0.31       | 34.33       | 0.10 | 0.99     |
| 2-Naphthol              | Poly(TFPPN-β-CD)      | 33.07              | 11.18     | 0.05      | 0.93       | 33.42       | 0.02 | 0.99     |
|                         | Poly(TFPPN-HE-β-CD)   | 26.05              | 8.76      | 0.03      | 0.80       | 26.21       | 0.02 | 0.99     |
|                         | Poly(TFPPN-HP-β-CD)   | 34.38              | 14.98     | 0.07      | 0.97       | 34.76       | 0.02 | 0.99     |
|                         | Poly(TFPPN-Me-β-CD)   | 35.92              | 11.66     | 0.04      | 0.82       | 36.22       | 0.02 | 0.99     |

Fig. 6 The linear fitting of pseudo-second-order kinetic model for the adsorption process of the four CD polymers toward the naphthalene derivatives: a 1-naphthylamine, b 2-naphthylamine, c 1-naphthol, d 2-naphthol
decreases with the increase of the initial concentration of adsorbate. For high initial concentration of adsorbate, the adsorption ratio can reach 100%, but the adsorption of low concentration of adsorbate is not complete (Hu et al. 2019; Li et al. 2014; Zhou et al. 2020). This feature is the opposite of that of most adsorbents. Therefore, it is also important to pay attention to the adsorption ratio at low concentrations. Herein, the adsorption ratio of the four CD polymers toward the naphthalene derivatives has a significant increase with the decrease of the initial concentration of pollutants as displayed in Fig. 8. When the initial concentration \( C_0 \) of the pollutant is 10 mg/L, the adsorption ratio is up to 85.3% for the uptake of 1-naphthylamine by poly(TFPPN-β-CD), 94.8% for the uptake of 2-naphthylamine by poly(TFPPN-Me-β-CD), 94.0% for the uptake of 1-naphthol by poly(TFPPN-Me-β-CD), and 90.9% for the uptake of 2-naphthol by poly(TFPPN-Me-β-CD). When the \( C_0 \) of the contaminant is 5 mg/L, the adsorption ratio is increased to 95.6% for the uptake of 1-naphthylamine by poly(TFPPN-β-CD), 96.6% for the uptake of 2-naphthylamine by poly(TFPPN-Me-β-CD), 96.3% for the uptake of 1-naphthol by poly(TFPPN-Me-β-CD), and 95.2% for the uptake of 2-naphthol by poly(TFPPN-Me-β-CD). This phenomenon suggests that the four CD polymers are applicable to removal of naphthalene and naphthol with low concentration, especially for micropollutant.

### Study of temperature on naphthalene derivative removal by CD polymers

The impact of temperature on the adsorption of 1-naphthylamine, 2-naphthylamine, 1-naphthol, and 2-naphthol by the four CD polymers were studied ranging from 15 to 35 °C. Figure 9 shows that high temperature is conducive to the above adsorption process. Only rising temperature from room temperature 25 to 35 °C, the adsorption ratio can be improved by 6 ~ 16% for 50 mg/L pollutants. Based on this feature, whether can the adsorption ratio of pollutants with low initial concentrations be increased to 100% by slightly
### Table 3 The fitting parameters of the four adsorption isotherms for the adsorption process of the four CD polymers toward the naphthalene derivatives

| Polymer          | 1-Naphthylamine | 2-Naphthylamine | 1-Naphthol | 2-Naphthol |
|------------------|-----------------|-----------------|------------|------------|
|                  | Langmuir isotherm | Freundlich isotherm | Redlich-Peterson isotherm | Sips isotherm |
| **Poly(TFPPN-β-CD)** | $R^2$ | $\chi^2$ | $q_m$ (mg/g) | $K_L$ (L/mg) | $R^2$ | $\chi^2$ | $K_F$ (mg/g) | $l/n$ | $n$ | $R^2$ | $\chi^2$ | $K_R$ (L/mg) | $\alpha_R$ (mg$^{-1}$) | $\beta$ | $R^2$ | $\chi^2$ | $q_m$ (mg·g$^{-1}$) | $K_S$ (L·mg$^{-1}$) | $\phi$ (mg·g$^{-1}$) |
| **Poly(TFPPN-HE-β-CD)** | 0.999 | 0.004 | 197.6 | 0.004 | 0.999 | 0.044 | 1.063 | 0.883 | 1.133 | 1.000 | 0.003 | 0.775 | 0.0002 | 1.583 | 1.000 | 0.004 | 154.9 | 0.005 | 1.041 |
| **Poly(TFPPN-HP-β-CD)** | 0.992 | 0.256 | 112.4 | 0.016 | 0.996 | 0.120 | 3.141 | 0.716 | 1.397 | 0.996 | 0.100 | 6.089 | 1.180 | 0.365 | 0.996 | 0.114 | 1613.2 | 0.002 | 0.729 |
| **Poly(TFPPN-Me-β-CD)** | 0.974 | 0.628 | 7.4 | 0.507 | 0.977 | 0.938 | 7.407 | 0.507 | 1.972 | 0.982 | 0.528 | 7.208 | 0.398 | 0.688 | 0.980 | 0.601 | 114.3 | 0.053 | 0.706 |
| **Poly(TFPPN-β-CD)** | 0.990 | 1.269 | 69.5 | 0.150 | 0.967 | 1.137 | 15.624 | 0.380 | 2.629 | 0.990 | 0.613 | 14.540 | 0.354 | 0.864 | 0.993 | 0.278 | 82.8 | 0.162 | 0.769 |
| **Poly(TFPPN-HE-β-CD)** | 0.994 | 0.06 | 44.8 | 0.066 | 0.965 | 0.788 | 6.554 | 0.425 | 2.352 | 0.995 | 0.059 | 3.215 | 0.091 | 0.945 | 0.995 | 0.061 | 46.8 | 0.070 | 0.940 |
| **Poly(TFPPN-HP-β-CD)** | 0.966 | 1.614 | 62.0 | 0.060 | 0.974 | 0.468 | 7.884 | 0.464 | 2.156 | 0.976 | 0.409 | 13.939 | 1.189 | 0.682 | 0.977 | 0.406 | 126.8 | 0.054 | 0.616 |
| **Poly(TFPPN-Me-β-CD)** | 0.982 | 1.809 | 62.6 | 0.150 | 0.979 | 0.732 | 14.588 | 0.365 | 2.743 | 0.991 | 0.559 | 19.415 | 0.733 | 0.783 | 0.994 | 0.295 | 83.5 | 0.161 | 0.670 |

- **Poly(TFPPN-β-CD)**
  - **Poly(TFPPN-HE-β-CD)**
  - **Poly(TFPPN-HP-β-CD)**
  - **Poly(TFPPN-Me-β-CD)**
increasing the temperature or not? Thus, the adsorption experiments about the temperature effect were carried out at the initial concentration of 5 mg/L. As shown in Table 4, only a slight effect occurs, but the adsorption ratio indeed could be improved by increasing temperature.

**Reutilization performance studying**

The polymers with excellent reusability will have good practicability. In this study, methanol was selected as the desorption agent to desorb the four cyclodextrin polymers with 1-naphthylamine, 2-naphthylamine, 1-naphthol, and 2-naphthol according to the results of exploratory experiment as depicted in Table 5. As depicted in Fig. 10, poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) have a small decrease in the adsorption ratio of four naphthalene derivatives after five adsorption–desorption processes, but not more than 5%. In detail, for 1-naphthylamine, the adsorption ratio of poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) decreased by 3.38%, 2.39%, 3.46%, and 2.47%, respectively. For 2-naphthylamine, the adsorption ratio of poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) decreased by 2.49%, 3.50%, 3.46%, and 2.49%, respectively. For 1-naphthol, the adsorption ratio of poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) decreased by 2.46%, 5.46%, 6.24%, and 4.24%, respectively. For 2-naphthol, the adsorption ratio of poly(TFPPN-β-CD), poly(TFPPN-HE-β-CD), poly(TFPPN-HP-β-CD), and poly(TFPPN-Me-β-CD) decreased by 4.24%, 3.34%, 2.38%, and 3.28%, respectively. The results show that the four cyclodextrin polymers have excellent recycling performance on the adsorption of 1-naphthylamine, 2-naphthylamine, 1-naphthol, and 2-naphthol.

**Fig. 8** The effect of the initial concentration of naphthalene derivatives on the adsorption of the four CD polymers: a 1-naphthylamine, b 2-naphthylamine, c 1-naphthol, d 2-naphthol
In addition, the comparison of adsorption performance of various polymers toward naphthol and naphthylamine is shown in Table 6. The results indicate the prepared polymers in this work are a good candidate for naphthol and naphthylamine removal.

### Conclusion

β-CD polymer, HE-β-CD polymer, HP-β-CD polymer, and Me-β-CD polymer were successfully prepared by solvothermal method with tetrafluoroterephthalonitrile as

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**Table 4** The effect of the temperature increased from 25 to 35 °C on the adsorption ratio of naphthalene derivatives in 5 mg/L

| Adsorbent          | Adsorbate   | Adsorption ratio at 25 °C | Adsorption ratio at 35 °C |
|--------------------|-------------|---------------------------|---------------------------|
| Poly(TFPPN-β-CD)   | 1-Naphthylamine | 95.6%                     | 96.5%                     |
| Poly(TFPPN-Me-β-CD)| 2-Naphthylamine | 96.6%                     | 98.5%                     |
| Poly(TFPPN-Me-β-CD)| 1-Naphthol    | 96.3%                     | 98.0%                     |
| Poly(TFPPN-Me-β-CD)| 2-Naphthol    | 95.2%                     | 97.6%                     |

**Table 5** Desorption ratio of the adsorbed CD polymers using methanol as desorption agent

| Polymers          | 1-Naphthylamine | 2-Naphthylamine | 1-Naphthol | 2-Naphthol |
|-------------------|-----------------|-----------------|------------|------------|
| Poly(TFPPN-β-CD)  | 91%             | 97%             | 97%        | 98%        |
| Poly(TFPPN-HE-β-CD)| 92%            | 99%             | 99%        | 95%        |
| Poly(TFPPN-HP-β-CD)| 89%            | 99%             | 98%        | 96%        |
| Poly(TFPPN-Me-β-CD)| 97%            | 96%             | 99%        | 98%        |
cross-linking agent. The four cyclodextrin polymers have rough surface morphology, uneven distribution of pores, similar specific surface area, and similar pore structure. The four cyclodextrin polymers have good thermal stability, and their thermal decomposition temperatures are higher than 200 °C. The four cyclodextrin polymers have fast adsorption

Fig. 10 The reusability of the four CD polymers toward naphthalene derivatives: a 1-naphthylamine, b 2-naphthylamine, c 1-naphthol, d 2-naphthol

| Adsorbent                | Adsorbate    | Maximum adsorption capacity | Adsorption time | Reusability | Reference               |
|--------------------------|--------------|----------------------------|-----------------|-------------|-------------------------|
| O-MWNT                   | 2-Naphthol   | 72 mg/g                    | 7 days          | No data     | Hou et al. (2013)       |
|                          | 1-Naphthylamine | > 143 mg/g                | 7 days          | No data     |                         |
| TCBD/D318                | 1-Naphthol   | > 165 mg/g                 | 360 min         | Good        | Yang et al. (2020)      |
| MWCNTs/iron oxides/CD    | 1-Naphthylamine | 200 mg/g                  | 120 h           | No data     | Hu et al. (2011)        |
| β-CDP                    | 2-Naphthol   | 74 mg/g                    | 20 min          | Good        | Hu et al. (2020)        |
| Fe₃O₄@polyaniline        | 1-Naphthol   | 23.09 mg/g                 | 400 min         | No data     | Zhou et al. (2016)      |
|                          | 2-Naphthol   | 28.74 mg/g                 |                 |             |                         |
| Poly(TFPNP-β-CD)         | 1-Naphthylamine | 189.9 mg/g                | 30 min          | Good        | This work               |
| Poly(TFPNP-β-CD)         | 2-Naphthylamine | 82.8 mg/g                 | 15 min          | Good        | This work               |
| Poly(TFPNP-Me-β-CD)      | 1-Naphthol   | 137.7 mg/g                 | 15 min          | Good        | This work               |
| Poly(TFPNP-Me-β-CD)      | 2-Naphthol   | 88.7 mg/g                  | 60 min          | Good        | This work               |
rate for 1-naphthylamine, 2-naphthylamine, and 1-naphthol and could reach more than 93% of the equilibrium adsorption within 15 min. The uptake rate of 2-naphthol is slower, but it could also reach about 90% of the adsorption equilibrium within 15 min. The kinetics of the adsorption processes could be depicted well by the pseudo-second-order kinetic model, indicating that chemisorption is involved in the adsorption processes. The adsorption processes of the four cyclodextrin polymers on these four naphthalene compounds are endothermic, and the adsorption isotherms were fitted to three-parameter Redlich-Peterson model or Sips model. The prepared polymers have excellent reusability and are expected to be applied to the removal of naphthylamine and naphthol compounds in industrial wastewater.

Author contribution Weifeng Xu: Conceptualization, methodology, project administration, writing—review and editing, funding acquisition.

Xiang Liu: Investigation, data curation, writing—original draft.

Jianzhe Cai: Investigation, data curation, formal analysis.

Tiemeng Xue: Validation.

Kewen Tang: Conceptualization, methodology, project administration.

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