



Adsorption, desorption and condensation of nitrobenzene solution from active carbon: A comparison of two cyclodextrins and two surfactants

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ABSTRACT

The adsorption of nitrobenzene on active carbon was researched. The experimental results shown the adsorption of nitrobenzene on active carbon can be described by Freundlich's adsorption model. On the other hand, β -cyclodextrin (β -CD) and hydroxypropyl- β -cyclodextrin (HP- β -CD) can react with nitrobenzene to form inclusion complex, which will enhance the aqueous solubility of nitrobenzene. By using different concentrations of β -CD and HP- β -CD as extractants, nitrobenzene on active carbon has been desorbed statically. As a comparison, surfactant CTAB and SDS were also selected as extractants. The desorbing of mechanism has been discussed. The low concentration of NB was effectively condensed by enrichment of active carbon and desorption of HP- β -CD. HP- β -CD is regarded as a potential extractant to deal with soil, sediment or active carbon.

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1. Introduction

Hydrophobic organic pollutants such as aromatic nitro compounds, polycyclic aromatic hydrocarbons (PAH) are of special interest because they are strongly sorbed by soils or sediments [1,2]. Aromatic nitro compounds are receiving increasing attention because of their toxic, mutagenic, and highly carcinogenic properties and their continuous release in the environment through human activities associated with producing intermediate of dye, pesticide, and detonator. Environmental Protection Agency (EPA) regulates that nitrobenzene, 2,4-dinitromethylbenzene and 2,6-dinitromethylbenzene are prior for detection. The contamination of soil and water by aromatic nitro compounds is a widespread environmental problem. Various physical, chemical, biological, and their combined technologies have been attempted to remediate organic-contaminated soils and water [3–5].

The microbial degradation of hydrophobic organic pollutants in situ is limited by their low bioavailability and low water-solubility. Improvement of desorption efficiency from soils, mobility and bioavailability in the aqueous phase, of organic pollutants are essential to the remediation of contaminated soils and groundwa-

ter. In order to enhance the desorption rate of organic pollutants, various extractants have been used: supercritical CO₂ extraction [6], cyclodextrin extraction [7–9] and surfactant extraction [10–12] have been proposed for the prediction of bioavailability of hydrophobic organic pollutants.

More recently, cyclodextrins (CDs) have been proposed as an alternative agent in order to enhance water solubility of hydrophobic compounds [12–14]. Cyclodextrins are cyclic oligosaccharides made up of six to twelve α -D-glucopyranose monomers connected at 1 and 4 carbon atoms. CDs with 6–8 α -D-glucopyranose units are denoted as α -, β -, and γ -CDs, respectively. They have the property of forming inclusion complex with various guest molecules with suitable polarity and dimension because of their special molecular structure—hydrophobic internal cavity and hydrophilic external surface [15,16]. It has been demonstrated that several weak forces, including van der Waals, hydrophobic, dipole–dipole, and hydrogen bonding interactions, cooperatively determine the inclusion complex behavior of the cyclodextrin host. Cyclodextrins can enhance solubility of contaminations [12,13,17], reduce their toxicity to animalcule [18], and catalyze their decomposition [19,20]. Cyclodextrins present several advantages over solvents and non-ionic surfactants such as their lower toxicity and their higher biodegradability. Chen et al. [21] studied the formation reaction for inclusion compound between β -CD and nitrobenzene. The results showed that the ratio of host and guest in the inclusion compound is 1:1 and the dissociated constant of the inclusion com-

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pound is $6.5 \times 10^{-3} \text{ mol L}^{-1}$. Recently, hydroxypropyl-cyclodextrins (HP-CDs) have received attention because of their amorphous property and higher solubility (>50%), compared with parent CD. Such characteristics of hydroxypropyl-CD are particularly useful for enhancing solubility of contaminations and extracting contaminations from polluted soils or sediments [22–24].

In this article, to research the mechanism and the kinetics for the extracting reaction, active carbon is used as sorbent and nitrobenzene (NB) is chosen as an example of pollutant. Active carbon is used to absorb and enrich nitrobenzene, β -CD and HP- β -CD are used as extractants to extract and condense NB. As comparing, a similar experiment is carried out by using surfactants, such as hexadecyltrimethylammonium bromide (CTAB) and sodium dodecylsulfate (SDS) as extractants. The results show that β -CD and HP- β -CD extractants are of more advantages comparing to surfactants.

2. Materials and methods

2.1. Reagents

β -Cyclodextrin (β -CD, Shanghai Chemical Reagents Company) was twice purified by recrystallization before using; hydroxypropyl- β -cyclodextrin (Yiming Chemical Reagents Factory, Jiangsu); nitrobenzene (NB, Shanghai Chemical Reagents Company); grainy active carbon (particle diameter: 0.4–0.9 mm, Liyang Active Carbon Factory, Jiangsu); hexadecyltrimethylammonium bromide (CTAB, Aldrich, 99+%), sodium dodecylsulfate (SDS, Aldrich, 99+%) and other chemical reagents were analytical reagent grade and used as commercial. Double distilled and sterilized water was used to prepare all solution.

2.2. Apparatus

The UV–vis spectrum was recorded on a UV-2550 (Shimadzu, Japan) double-beam spectra photometer equipped with a stoppered quartz cell with 1.0 cm optical path length. Sorption and desorption experiments were carried out on a laboratory vibrator HZ-9211 K (Jiangsu, China).

2.3. Enhancement of solubility of NB by β -CD, HP- β -CD and surfactants

The solubility of NB in different concentration of β -CD and HP- β -CD was determined by putting excess NB into an aqueous solution with different concentration of β -CD or HP- β -CD, respectively. Then the solution was shaken at least 24 h intensively on laboratory vibrator (180 rpm) at 25 °C. The solution was centrifuged. Supernatant liquid was taken out and diluted with 1:1 CH_3OH aqueous solution to make the inclusion compound decompose completely [25] so that the solubility of NB in different concentration of β -CD or HP- β -CD can be determined by UV–vis spectrum. Similarly, the solubility of NB in different concentration of CTAB and SDS can be obtained.

2.4. Sorption and desorption of NB

To research the sorption of NB on active carbon, fresh active carbon was put into a solution contained different concentration of NB. The clean solution was obtained by filtrating and centrifuging after the solution shaken 24 h by laboratory vibrator (180 rpm) at 25 °C. The amount of NB absorbed by active carbon can be determined by checking the residual NB in the solution by UV–vis.

The behavior for the desorption of NB from active carbon was also studied with similar method described above except for using extractants instead of NB aqueous solution. To research the desorption behavior of NB from active carbon, the fresh active carbon was first put into 0.55 mM NB aqueous solution. Then the solution with active carbon was shaken by laboratory shaker (160 rpm) 5 h at 25 °C to make the absorption of NB on active carbon arrive at equilibrium. Then the active carbon covered with NB was taken out by filtrating and given an airing at room temperature for further use. The amount of NB absorbed by active carbon is 2.6 mmol g^{-1} . The desorption kinetics of NB from active carbon in different extractants was determined by putting the active carbon covered with NB in extracting agent solution. At different desorption time, the concentration of NB in liquid phase was measured by UV–spectrum as previously described.

The desorption efficiency of different extractants was measured. By using H_2O , different concentrations of β -CD, HP- β -CD, CTAB and SDS as extractants, nitrobenzene on active carbon was desorbed statically. In a typical experiment, 100 mg active carbon covered with NB was dipped in turn in fresh extracting agent for five times. Each time, the amount of extracting agent is 50 mL. To make desorption arrive at the equilibrium, the desorption system was shaken by laboratory shaker at 25 °C for 5 h. The amount of NB desorped from active carbon was determined by analyzing the concentration of NB in the liquid phase.

2.5. Condensation of nitrobenzene solution from active carbon

The condensation of NB by HP- β -CD Laboratory column studies was carried out using 10 mm diameter and 150 mm long glass columns packed with the active carbon tested. Glass wool was placed on the base of each column, minimizing the dead end volume. The column was packed using 1 g of active carbon, which resulted in an active carbon column of 4 cm length and uniform bulk density of 0.318 kg/L. After packing, the active carbon column was saturated with 5 L 0.2 mM NB solution by a peristaltic pump until the concentration of NB in the leachate became constant. NB was almost absorbed on the active carbon. Then, the columns were eluted with 300 mL of solutions at the following concentrations: 0, 1, 2, 5 mM for HP- β -CD by a peristaltic pump. A flow rate of 1.0 mL/min was used for all miscible displacement experiments. Leachate was collected in 10 mL portions. The concentrations of NB in aliquots were determined by UV–vis.

3. Results and discussion

3.1. Dissociated constant of inclusion compound between NB and HP- β -CD

In the previous article, the dissociated constant for the inclusion compound of NB and β -CD was reported. Similarly, between NB and HP- β -CD an inclusion compound can also be formed. However, the dissociated constant of inclusion compound between NB and HP- β -CD is different from that between NB and β -CD. As previous article described, the dissociated constant of the inclusion compound can be determined by UV–vis spectroscopy [21]. In neutral solution (pH 7.0), the UV–vis spectra at a given concentration of NB with different amount of HP- β -CD was shown in Fig. 1. The peak position of the spectra was independent of the concentration of HP- β -CD. However, the peak intensity increased with the concentration of HP- β -CD, which confirmed the formation of the inclusion compound between NB and HP- β -CD. The molar absorption coefficient of the inclusion compound was larger than that of NB itself. As previously described [21], if one makes assumptions of 1:1 ratio

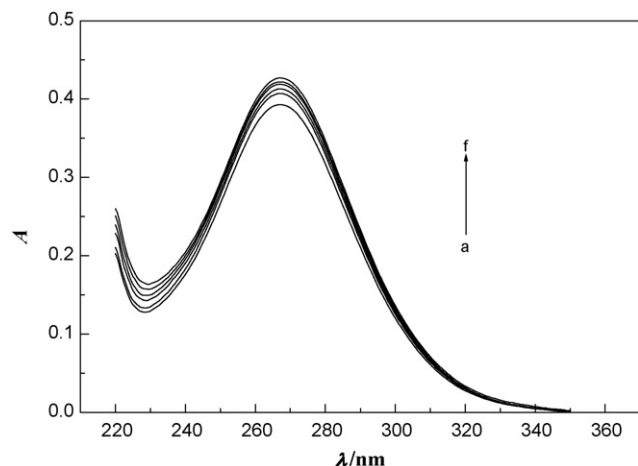


Fig. 1. At 25 °C, UV-vis spectra of 50 μM NB aqueous solution (pH 7.0) with different concentrations of HP-β-CD. $c_{\text{HP-}\beta\text{-CD}}$ (mM): a:0; b:1.0; c:2.0; d:3.0; e:4.0; f:5.0.

of NB to HP-β-CD in inclusion compound and $[H]_0 \gg [G]_0$, where $[H]_0$ and $[G]_0$ are initial concentrations of HP-β-CD and NB, respectively, the relationship of the peak intensity and the concentration of HP-β-CD can be deduced as follows in Eq. (1).

$$\frac{[H]_0[G]_0}{\Delta A} = \frac{[H]_0}{\Delta \epsilon} + \frac{K_D}{\Delta \epsilon} \quad (1)$$

Where ΔA is difference of peak intensity of NB with and without HP-β-CD, $\Delta \epsilon$ is the difference of molar absorption coefficient between inclusion compound and NB, K_D is the dissociated constant of the inclusion compound.

According to Eq. (1), the plot of $[H]_0[G]_0/\Delta A$ versus $[H]_0$ should be a straight line. The experimental result was shown in Fig. 2. A well behaved linear relationship between $[H]_0[G]_0/\Delta A$ and $[H]_0$ showed that the assumption of 1:1 ratio of NB to HP-β-CD in inclusion compound was correct. From the slope and the intercept of the line, the dissociated constant, K_D was evaluated as 2.2 mM, which is smaller than 6.5 mM, the dissociated constant of inclusion compound between NB and β-CD [21]. The result shows that the inclusion compound of NB and HP-β-CD is more stable than that of NB and β-CD.

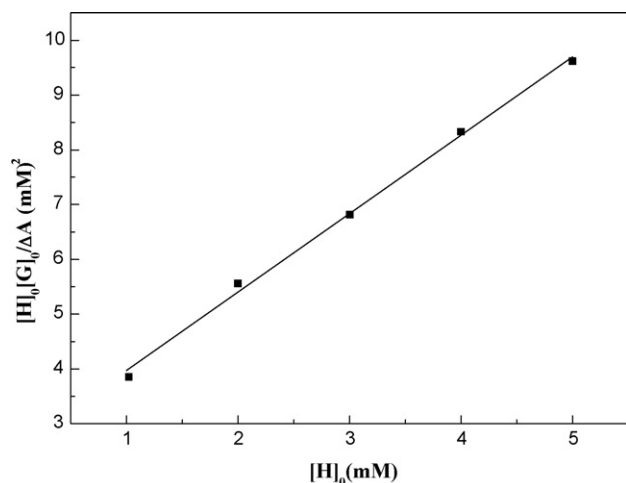


Fig. 2. The plot of $[H]_0[G]_0/\Delta A$ versus $[H]_0$.

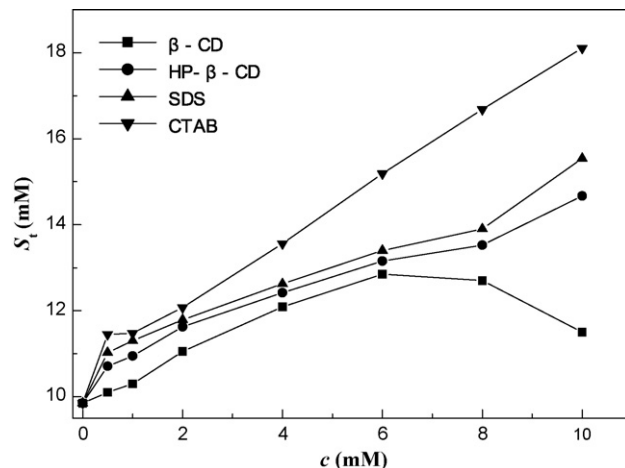


Fig. 3. At 25 °C and pH 7.0, the plot of S_t versus different concentration c of extractants.

3.2. Solubility of NB in different extractants

As previously described that CD can increase the solubility of organic compound in water [12–14] because of the formation of inclusion compound. Therefore, if β-CD or HP-β-CD is put into a solution with excess NB, the solubility of NB will increase, which is similar to that of surfactant [26,27]. At 25 °C, the solubility, S_t of NB in water was measured as 9.8 mM which is near to that reported in ref. [28]. The relation between the solubility of NB and the concentration of different extractants was shown in Fig. 3. Generally speaking, the solubility of NB increases with the concentration of extractant. At a given concentration of extractant, the solubility of NB increases in series of β-CD, HP-β-CD, SDS, and CTAB. The solubility of NB in HP-β-CD is near to that in SDS. However, for β-CD when its concentration is more than 6 mM, the solubility of NB decreases with the concentration of β-CD increasing, which may be due to analogously homology ion effect [25].

3.3. Adsorption of NB by active carbon

At equilibrium, Q , the amount of NB absorbed by active carbon depends on c , the concentration of NB in aqueous solution. Fig. 4 shows the relationship of $\log Q$ and $\log c$ at different temperatures.

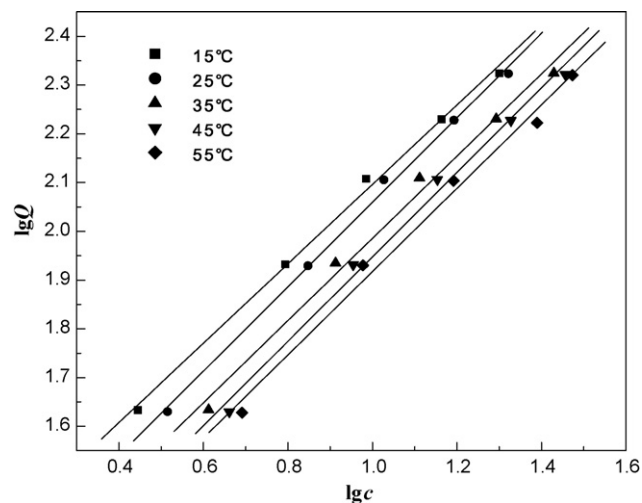


Fig. 4. The plot of $\log Q$ versus $\log c$.

Table 1
At different temperatures adsorption constant, K_F , and constant, n

T ($^{\circ}\text{C}$)	15	25	35	45	55
K_F	19.1	15.5	13.8	12.0	11.5
n	1.22	1.16	1.19	1.16	1.18

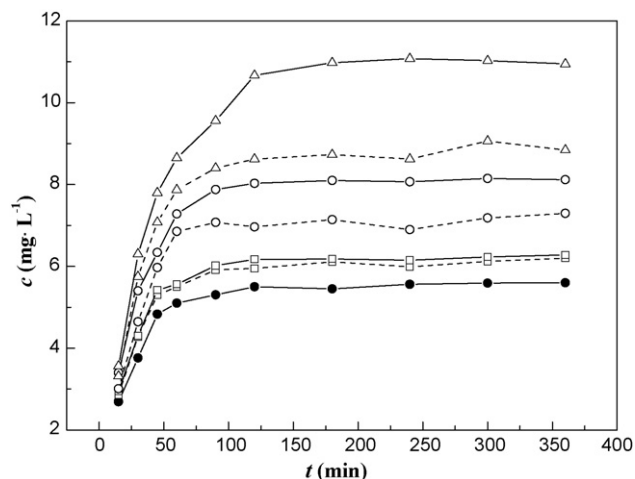


Fig. 5. Plot of c , the concentration of NB in both β -CD (dash line) and HP- β -CD (solid line) versus desorption time, t . The concentration of extractants (mM): (●) 0; (□) 0.5; (○) 1.0; (Δ) 2.0.

A well behaved linear relationship shows that adsorption of NB on active carbon obey Freundlich's model:

$$\log Q = \log K_F + \frac{1}{n} \log c \quad (2)$$

where K_F and n are constant at given temperature. According to the slope and the intercept of the straight line, both K_F and n can be evaluated. Table 1 listed the values of K_F and n at different temperatures. From Table 1, n is almost independent of temperature. However, K_F decreased slightly with temperature increasing, which means that it is disadvantaged for NB adsorbed by active carbon at high temperature.

3.4. Desorption of NB from active carbon

The desorption kinetics of NB from active carbon by different extractants was determined by putting the active carbon covered with NB in extracting agent solution. At different desorption time, the concentration of NB in solution phase was measured by UV-spectrum as previously described. The concentration of NB in different concentration of extractants are shown in Fig. 5, where solid line is measured in HP- β -CD extracting agent, and dashed line is measured in β -CD. It is clear that desorption efficiency

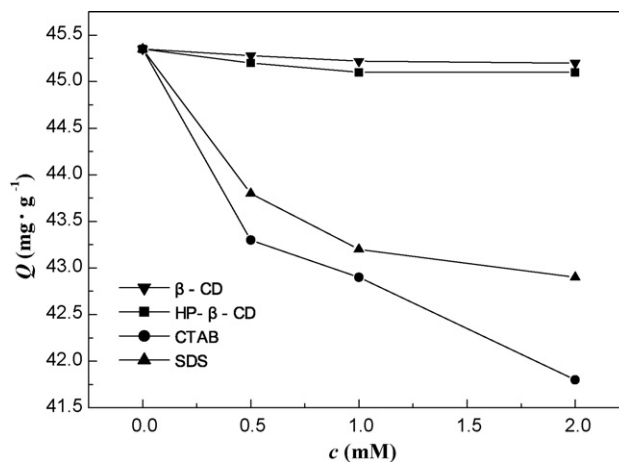


Fig. 6. The plot of the adsorptive capacity of active carbon Q versus the different concentration c of extractants.

increases with the concentration of β -CD or HP- β -CD. At given concentration of extracting agent, desorption efficiency of HP- β -CD is greater than that of β -CD. For the more, the difference of desorption efficiency between HP- β -CD and β -CD is more significant at high concentration of extracting agent. However, at any cases, desorption equilibrium are observed at ca. 100 min.

As previously described, surfactant, such as CTAB, SDS is also good extracting agent to organic pollution. For comparison, H₂O, and different concentration of β -CD, HP- β -CD, CTAB, and SDS were used as extractant. Table 2 lists the desorption rate defined as a ratio of amount of NB desorped from active carbon to the total initial amount of NB covered on 100 mg active carbon in different extractants at 25 $^{\circ}\text{C}$. General speaking, the desorption efficiency for different extractants changes in an order of H₂O < β -CD < HP- β -CD < CTAB < SDS. Comparing with the solubility experiment showed in Fig. 3, the order of desorption efficiency of the extractant is same as that of the solubility of NB in extractant except for that in CTAB and SDS. In fact, the desorption efficiency depends on the coverage of NB on active carbon and the solubility of NB in extracting agent, which depends on the interaction between NB and extracting agent. At a given coverage of NB, the desorption efficiency mainly depends on the solubility of NB in the extractant. Therefore, it is reasonable for that if higher solubility of NB in an extracting agent is observed, the extracting agent should have higher desorption efficiency, which is confirmed by the experimental results in first desorption operation when the concentration of extracting agent is more than 1 mM.

To understand in detail, the absorption of extractant on active carbon was researched. First, the fresh active carbon was marinated in solution contained different concentration of extracting agent until to absorption equilibrium. Then the active carbon absorbed by extracting agent was used to make an experiment of absorp-

Table 2
The rate of desorption (%) in different extractants

Desorption times	H ₂ O	Concentration of extractants (M)											
		5×10^{-4}				1×10^{-3}				2×10^{-3}			
		β -CD	HP- β -CD	CTAB	SDS	β -CD	HP- β -CD	CTAB	SDS	β -CD	HP- β -CD	CTAB	SDS
1	8.5	10.1	13.3	13.2	13.6	10.7	13.9	16.3	14.5	12.5	14.3	17.7	15.4
2	5.1	7.7	9.9	9.1	10.3	8.2	10.4	10.2	11.6	9.5	11.4	11.8	13.7
3	3.6	6.3	7.1	6.3	8.2	7.1	7.2	6.9	8.8	7.2	7.9	8.1	9.5
4	3.0	5.2	5.2	6.2	6.7	5.3	5.4	6.7	7.4	5.7	6.2	7.0	7.8
5	2.4	4.7	3.9	5.4	6.7	4.8	4.1	5.8	7.2	5.3	5.3	6.6	7.5
Total	22.6	34.0	39.4	40.2	45.4	36.1	41.0	45.9	49.5	40.2	45.1	51.2	53.9

tion to NB. The experimental results were shown in Fig. 6. It is evident that the absorption capacity decreased significantly when the active carbon treated previously by surfactants. However, the absorption capacity kept almost constant when the active carbon was treated previously by β -CD or HP- β -CD. The results show that the absorption of surfactants is more significant comparing with β -CD or HP- β -CD. Little β -CD or HP- β -CD can be absorbed by active carbon. The fact that β -CD and HP- β -CD did not absorbed by active carbon is very important for environmental scientists. If one uses β -CD or HP- β -CD as extracting agent to make the contaminated material desorb from active carbon, the active carbon will reproduce simultaneously. However, if surfactants are used as extractant, although they also can make the contaminated material desorb from active carbon, but the active carbon lost its active sites because of the absorption of surfactants. Another operation should be taken if one tries to make the active carbon renew. Therefore, as an extracting agent, β -CD or HP- β -CD is better than surfactants.

On the other hand, comparing with CTAB, SDS is a better extracting agent. Although the solubility of NB in CTAB is greater than that in SDS, the total desorption efficiency of SDS is greater than that of CTAB, which may be due to the interaction between CTAB and NB on active carbon. Comparing to surfactant, the absorption of β -CD and HP- β -CD on active carbon is much weaker, therefore, the desorption ability for two kinds of extractants relies on their ability to increase the solubility of NB, which relies on the dissociated constant, K_D . The smaller the K_D , the larger the desorption ability is.

The conclusion is very important. Although the desorption efficiency of β -CD and HP- β -CD solution is smaller than surfactant, however, if the active carbon absorbed with NB is treated by β -CD or HP- β -CD extracting agent, the active site will partly recover. Therefore, the active carbon can be used directly to treat another sample. If surfactant is used as extracting agent, the active site will be covered largely by the adsorption of surfactant when NB is desorbed. Therefore, the active carbon cannot be used directly. It should be regenerated before using. In overall view, HP- β -CD solution is better extracting agent.

3.5. Temperature dependence of the desorption

The amount of NB desorbed from active carbon depends on the temperature. Fig. 7 showed the rate of desorption at different temperature in different extractants. From bottom to top, the extractants are H_2O , $2 \times 10^{-3} M$ β -CD, and $2 \times 10^{-3} M$ HP- β -CD, respectively. It is clear that the rate of desorption of NB from active

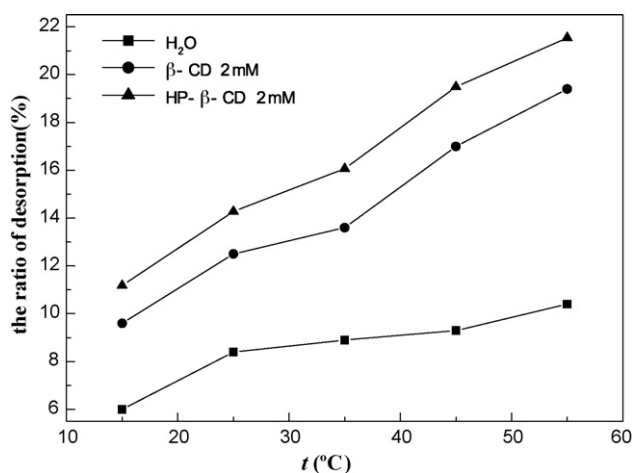


Fig. 7. The plot of the ratio of desorption at different temperature.

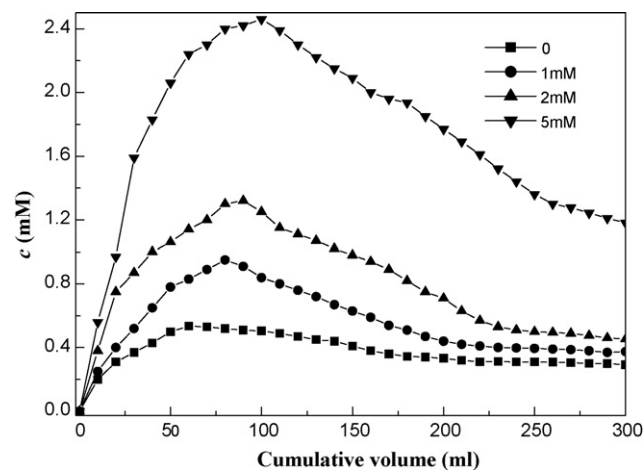


Fig. 8. Plot of c , the effluent concentration of NB in versus cumulative volume of HP- β -CD.

carbon increased with temperature for all extractants. However, it is more sensitive to temperature for β -CD and HP- β -CD solution comparing to water.

3.6. Condensation of nitrobenzene solution from active carbon

The condensation of NB in a flow-through system was studied using the miscible displacement techniques. Treatment without HP- β -CD was done as a control for the HP- β -CD-amended experiments. Effluent curves for NB in HP- β -CD-treated and untreated active carbon columns are shown in Fig. 8. The curves are plotted as a function of total effluent volumes. The highest effluent concentration of NB treated with water was 0.53 mM, when the cumulation volume reached 70 mL. The concentrations of NB in the column effluent increase sharply when flushed with various concentrations HP- β -CD solution. With the concentrations of HP- β -CD increasing, the effluent concentrations of NB were increased simultaneously. It can be concluded that the magnitude of the enhanced-condensation effect depends on the mass of HP- β -CD in solution. When the concentration of HP- β -CD increased to 5 mM, the total effluent concentration of NB by 300 mL HP- β -CD was 1.9 mM, which was nearly ten times than the initial concentration of NB (0.2 mM). At the same time, the volume of the NB solution was decreased from 5 L to 300 mL. The low concentration of NB was condensed effectively through enrichment by active carbon and desorption by HP- β -CD. Therefore, this method may be used as to deal with the low concentration organic wastewater. Because of decreasing the volume of organic wastewater through enrichment and condensation, the high concentration of organic wastewater may be treated conveniently by electrochemistry oxidation, photochemical catalytic oxidation or microorganism decomposition.

4. Conclusion

A 1:1 inclusion complex between HP- β -CD and NB formed at room temperature. At pH 7.0, the dissociate constant of the inclusion complex is 2.2 mM. β -CD and HP- β -CD can be used as extractants to extract organic compound absorbed by active carbon. Desorption ability for different kinds of extractants used in our experiments not only depends on the ability to increase the solubility of NB, but also depends on the absorption ability of the extracting agent onto active carbon. Although the desorption efficiency of β -CD or HP- β -CD is smaller than that of surfactant, they are still a better selection of the extracting agent because of their

weaker absorption on active carbon. When the concentration of HP- β -CD was 5 mM, the total effluent concentration of NB was nearly ten times than the initial concentration of NB. The low concentration of NB was condensed effectively through absorption by active carbon and desorption by HP- β -CD.

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