



## Effect of pore structure on the adsorption of aqueous dyes to ordered mesoporous carbons



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### ABSTRACT

Three ordered mesoporous carbons, CMK-3, CMK-5 and its carbon/silica composite Si-CMK-5, with different pore structures were investigated for the adsorption of aqueous Acid Red 73 and Reactive Black 5. Among the adsorbents, CMK-5 shows the highest adsorption amount and fastest adsorption rate towards the two dyes, resulting from its double pore systems and large specific surface area. After normalizing by the specific surface area of the adsorbents, the adsorption of relatively small Acid Red 73 on the three adsorbents is close to each other. However, the adsorption of Reactive Black 5 on CMK-3 is stronger than on CMK-5 and Si-CMK-5, reflecting that the open and interconnected pores of CMK-3 are more favorable to adsorption of bulky dyes compared with the relatively closed and isolated pores of Si-CMK-5. The two dyes adsorption over these adsorbents can be well described using Langmuir model. CMK-5 adsorption toward Acid red 73 or Reactive black 5 is well fitted with double Langmuir model. Though the pore size of CMK-3 is smaller than that of Si-CMK-5, for a given adsorbate, the adsorption rate of CMK-3 is more rapid than that of Si-CMK-5 because of its open and interconnected pore features. In addition, the simulated results using Weber–Morris modal confirm that the adsorption processes of the dyes on the three adsorbents are affected by external mass transfer and intraparticle diffusion. External mass transfer is the slow step during adsorption process based on the results of Boyd model.

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## 1. Introduction

Dye wastewater is one of the representative effluents originating from chemical and textile industries. Synthetic dyes present in surface and ground water greatly influence both the aquatic ecosystem and human health. Most of synthetic dyes may induce carcinogenic and mutagenic activities [1,2]. Dye contamination also reduces species diversity via obstructing light penetration, thereby adversely affecting the photosynthesis of aquatic life [3]. Due to their synthetic source and complex structure, dyes are difficult to biodegrade. Therefore, it is of great importance to develop effective treatment techniques for the removal of aquatic synthetic dyes.

At present, several physical or chemical techniques, such as advanced oxidation [4], membrane separation [5], adsorption [6,7] and so on, are considered to be effective for synthetic dyes treatment. Among them, adsorption process provides a simple and

universal approach to effectively remove aqueous dyes. Activated carbon is one of the most used adsorbent in adsorption and separation processes due to its prominent adsorption capacity and adsorption affinity toward various organic pollutants in water. It should be pointed out that most synthetic dyes have bulky molecular structures in nature. However, conventional activated carbon, predominantly consisting of micropores, normally shows low adsorption capacity for bulky compounds because of the size exclusion effect [8,9]. In order to improve the adsorption ability of activated carbon toward organic pollutants with large molecular size, recent research attention has been focused on increase of mesopore proportion for activated carbon or synthesis of mesoporous carbonaceous materials with amended pore structures suitable for effective adsorption of bulky pollutants [10–17]. Our previous study found that mesoporous carbon, CMK-3, showed much stronger adsorption ability toward direct yellow 12 than commercial activated carbon, which could be ascribed to the higher mesopore fraction in CMK-3 compared to activated carbon [12]. The adsorption amount of Reactive Black 5 on carbon cryogel microspheres increased with an increase in mesopore sizes [13]. Ordered

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mesoporous carbon, with high specific surface area and large pore volume, shows excellent adsorption capacity and fast adsorption rate toward rhodamine B [14]. Additionally, super adsorption properties of ordered mesoporous carbon for bulky dyes were obtained by developing carbonaceous adsorbents with bimodal pores which have ultrahigh specific surface area and mesopore volume [15–17].

In summary, the current reports reveal that pore size combined with specific surface area and mesopore volume of activated carbonaceous materials play a crucial role in the adsorption of aqueous dyes with large molecular sizes, by reducing or eliminating the size exclusion effect on adsorption. Zhang et al. [18] pointed out that the molecular sieving effect may occur either when the shape of pores prevented the adsorbates from penetrating into micropores of adsorbents or when the pore size of adsorbents is narrower than the molecular size of adsorbates. Guo et al. [19] found that slit-shape pores of activated carbon could be more efficiently filled with polyaromatic compounds in comparison to ellipsoidal pores. Thus, it is evident that the pore structures, such as pore shape and network inter-connection as well as pore size play a crucial role in adsorption of organic pollutants by microporous activated carbon. However, as for mesoporous carbon adsorption toward bulky pollutants such as dyes, few relevant studies have been conducted thus far [20].

The overall goal of this study is to systematically investigate the influence of physical factors, particularly the pore shapes of mesoporous carbons and the molecular sizes, on the adsorption of aqueous dyes by different mesoporous carbons. Three mesoporous carbonaceous materials, CMK-5, CMK-3 and Si-CMK-5 composite, with different mesoporous structures, were synthesized and used as adsorbents. The schematic descriptions of the mesoporous structures for the materials were listed in Fig. 1. All of the selected adsorbents, prepared with the same template, carbon precursor, and carbonization procedure, are expected to have similar interactions with adsorbate molecules. Two commonly used dyes, Acid Red 73 and Reactive Black 5, were selected as adsorbates. They

differ greatly in molecular size and structure which were compiled in Fig. 2, and hence are likely to show varied adsorption behaviors, as regulated by adsorbent pore structures.

## 2. Experimental

### 2.1. Preparation of silica template

Mesoporous silica SBA-15 was synthesized according to the procedure described in the literature [21]. Typically, 30.0 g of Pluronic P123 (Aldrich) was dissolved in 1125 mL of 2.0 mol/L HCl solution at 40 °C and then 62.4 g of tetraethoxysilane (Shanghai Chemical Co., China) was added. After stirring at 40 °C for 24 h, the mixture was transferred to a Teflon-lined autoclave and heated at 100 °C for 48 h. The white precipitate was filtrated, washed with distilled water, and dried at 80 °C overnight. The product was obtained after calcination at 550 °C for 6 h to remove the organic surfactant Pluronic P123.

### 2.2. Preparation of ordered mesoporous carbons

The two ordered mesoporous carbons, CMK-5 and CMK-3, were prepared using SBA-15 as hard template and furfuryl alcohol (FA, Lingfeng Chemical Reagent Co., Shanghai, China) as carbon precursor [22,23]. Briefly, FA was dissolved in trimethylbenzene (TMB) (Haiqu Chemical Co., Shanghai, China) and oxalic acid (Sinopharm Chemical Reagent Co., China) was added as a polymerization catalyst. For CMK-5 and CMK-3, the volume fraction of FA in TMB was 35% and 100%, respectively. Then SBA-15 was impregnated with FA solution at room temperature, followed by polymerization at 50 °C for 24 h and at 90 °C for another 24 h under air. The solid composites were heated at 150 °C for 3 h and carbonized at 850 °C for 4 h under vacuum. The heating rate was 1 °C/min when the temperature was below 300 °C, and then increased to 5 °C/min over 300 °C. The samples were then treated with 10% (wt) aqueous HF solution at room temperature to remove the silica, washed with

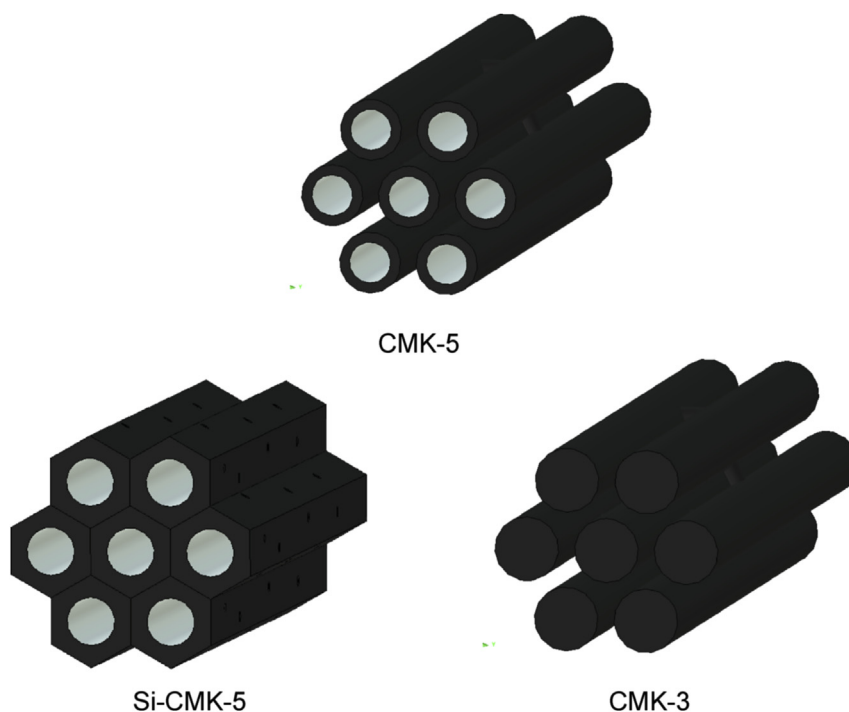


Fig. 1. Schematic description of pore structures for the three materials.

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