



Influence of porosity and surface modification on the adsorption of both cationic and anionic dyes



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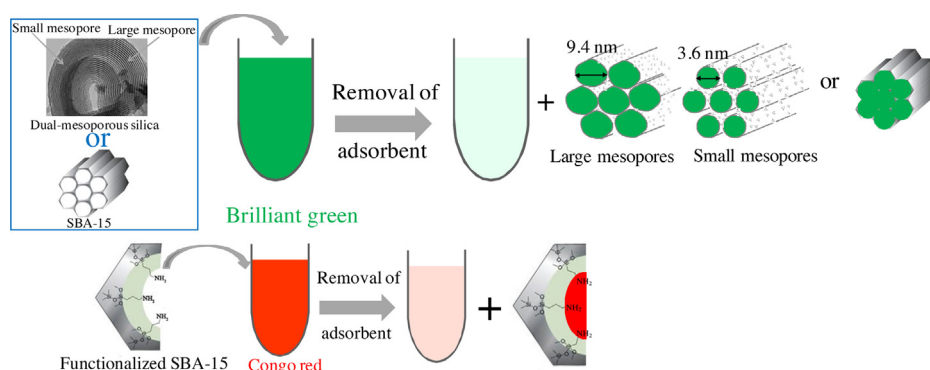
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HIGHLIGHTS

- Peculiar efficiency of bare silica mesoporous materials for cationic dye adsorption.
- Strong uptake of anionic dye adsorption by functionalization of the adsorbent.
- Dual mesoporosity positively affect cationic dye adsorption under acidic conditions.
- Adsorption kinetics determined from the pseudo-second order equation.
- Adsorption isotherms describe by Langmuir and Freundlich models.

GRAPHICAL ABSTRACT



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ABSTRACT

We report here the use of porous materials for the adsorption of both anionic and cationic dyes. We have investigated the effect of the pH and of the presence of a dual mesoporosity on the adsorbents capacity. The results show that, regardless of porosity, the bare mesostructured silicas are particularly efficient for the adsorption of the cationic dye, brilliant green.

The adsorption isotherms have been fitted by using the Langmuir and the Freundlich models. Under acidic conditions, the dual-mesoporous material presents a better affinity and a slightly higher maximum adsorption capacity for congo red than the mono-modal mesostructure. Concerning the adsorption of brilliant green, no significant effect of porosity is noted and the fitting parameters indicate that similar dye-adsorbent interactions occur.

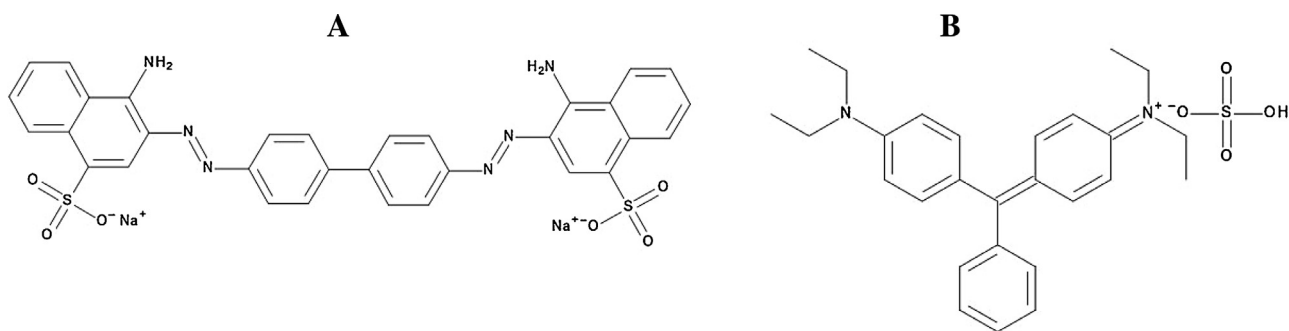
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1. Introduction

There are many different types of substances that may pollute water and contamination of surface water, groundwater and

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Scheme 1. Molecular structure of congo red (A) and brilliant green (B).

soil is still a major problem [1]. Because of their carcinogenic and toxic effects on human health [2,3], among the different pollutants such as detergents, fertilizers, volatile organic compounds (VOC), halogenated organic compounds (HOCs), dyes are a major environmental concern. For example, brilliant green is toxic for lungs through inhalation and congo red, a derivative of benzidine and naphthoic acid, metabolizes to carcinogenic products. Both colorants are hazardous when in contact with skin or eyes. Dyes are mainly discharged from pharmaceuticals, printing, food coloring, cosmetics, paper, textiles and many others industries [4,5], involving an environmental pollution. Because of their toxicity for human health, many efforts have been devoted to remove these pollutants from the effluents and various methods such as filtration, gravity separation, flotation, enzymatic decomposition and photocatalysis have been developed [6–8]. However, due to its high efficiency, adsorption is the cheapest employed process [6] and different kinds of absorbents have been designed, in particular for the adsorption of congo red and brilliant green [9–13].

For the past few years, surfactants templated silica mesoporous materials have attracted much research attentions [14–16] and a series of compounds labeled, for example MCM (Mobil Crystalline Materials), SBA (Santa Barbara), MSU (Michigan State University), have been prepared by different groups. The synthesis of these compounds combines the sol–gel chemistry and the use of assemblies of surfactant molecules as framework templates. Surfactant micelles are commonly used as self-assembly templates for the development of new mesoporous silicas with a variety of textures and structures [14–16]. Another approach to the preparation of ordered mesostructures uses liquid crystal phases and is labeled as the direct liquid crystal templating (LCT) pathway [17,18]. In this case, larger surfactant concentration is necessary and the structure of the recovered materials can be designed a priori based on the corresponding surfactant phase diagram. These materials have potential applications in many fields, such as catalysts, host matrices for electronic and photonic devices, drug delivery and sensors [19,20]. Thanks to their properties, such as large surface area and pore volume, ordered mesopores with tuned sizes, mesoporous silica materials are supports of choice for dyes adsorption, responding to both environmental and technical requirements [21–24]. For example, Lee et al. [21] have shown that MCM-41 may be an effective absorbent for basic dyes removal from aqueous solution. In addition, the nature and the strength of the interaction between the host molecules and the adsorbent can be tuned by functionalizing the surface of these supports [25,26]. The functionalization can occur either through the post synthesis grafting of an organic compound, such as an organoalkoxysilane, on the supports or by the co-hydrolysis and polycondensation of an alkoxysilane, for instance tetramethylorthosilicate (TMOS) and an organoalkoxysilane. This second pathway involves a covalent link between both moieties. For example, Ma et al. [25] have modified MCM-41 by introducing ammonium group, according to the post-synthesis way, for the

adsorption of anionic dyes. Authors have evidenced that the functionalized MCM-41 material has a high affinity for the considered dyes and that the electrostatic interaction was responsible of the colorants adsorption.

Besides these mono-modal mesostructures, more recently dual mesoporous materials have attracted much attention as they are of particular interest for catalysis and for the engineering of pore systems [27]. Indeed, it was reported that a hierarchical combination of mesopores reduces transport limitations, resulting in higher activities and better controlled selectivity [27]. One strategy to prepare these bimodal mesoporous materials consists in using mixtures of templates [28–31]. In our group we have used mixtures of Pluronic P123 and fluorinated surfactant $C_8F_{17}C_2H_4(OC_2H_4)_9OH$ [$R^F_8(EO)_9$] to design such bimodal mesoporous silicas [30,31]. Here we have investigated the ability of the obtained materials to be used as absorbents for the removal of congo red, an anionic dye and brilliant green, a cationic dye. The structure of both dyes is given in Scheme 1. The efficiency of the dual-mesoporous silica is compared with the one of SBA-15, which is a mono-modal mesostructure prepared by using Pluronic P123 as template. The SBA-15 has also been functionalized with 3-aminopropyl-triethoxysilane (APTES) in order to investigate the effect of the surface state on the adsorption of both the cationic and the anionic dyes.

2. Materials and methods

The used fluorinated surfactant, which was provided by DuPont, has an average chemical structure of $C_8F_{17}C_2H_4(OC_2H_4)_9OH$. It is labeled as $R^F_8(EO)_9$. The hydrophilic chain moiety exhibits a Gaussian chain length distribution and the hydrophobic part is composed of well defined mixture of fluorinated tails. The selected triblock copolymer, Pluronic P123 [(EO)₂₀(PO)₇₀(EO)₂₀], 3-aminopropyl-triethoxysilane (APTES), tetramethoxysilane (TMOS) and the dyes were purchased from Aldrich. Deionized water was used to prepare the various samples.

2.1. Absorbents preparation

To prepare the dual-mesoporous material: 0.9 g of $R^F_8(EO)_9$ and 0.1 g of P123 are dissolved in a hydrochloric acid solution (pH 0.3) to form a micellar solution containing 10 wt.% of surfactant. Then 0.32 g of tetramethoxysilane (TMOS), used as the silica source, is added dropwise into the micellar solution at 25 °C and let under gentle stirring (150 rpm) for 1 h. The obtained samples are sealed in Teflon autoclaves and heated at 80 °C for 1 day.

SBA-15 has been synthesized according to a procedure reported in literature [32]: 1.33 g of P123 are dissolved in 50 mL of hydrochloric acid solution (pH 0.3). Then 2.07 g of TMOS are added. After 30 min under gentle stirring (300 rpm) at 25 °C, the obtained samples are sealed in Teflon autoclaves and heated at 40 °C for 24 h, and then at 100 °C for 24 h.

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