



Bioassembly of fungal hyphae/carbon nanotubes composite as a versatile adsorbent for water pollution control

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HIGHLIGHTS

- Fungus was used as a template for the assembly of CNTs.
- Fungal template enhanced the dispersity and stability of CNTs.
- FH/CNTs exhibited high sorption capacity for U(VI) (187.26 mg/g), MV (43.99 mg/g) and CR (20.89 mg/g).
- FH/CNTs possessed satisfactory regeneration performance and reusability.
- FH/CNTs has a good selectivity on U(VI).

ARTICLE INFO

Keywords:

Fungal hyphae
Carbon nanotubes
Adsorption
Uranium
Dye

ABSTRACT

In this paper, a biological assembly method was used to fix carbon nanotubes (CNTs) onto fungal hyphae (FH) to prepare sphere FH/CNTs composite as a versatile adsorbent for water pollution control. Physico-chemical characterization results showed that FH/CNTs composites had a wire stacking surface morphology, showed a typical diffraction peak of CNTs, was rich in functional groups, and was negatively charged under pH 3 to 10. Adsorptions of uranium (U(VI)), anionic (Congo red (CR)) and cationic (methyl violet (MV)) dyes under various conditions were investigated to elucidate their adsorption performances and mechanisms. Results showed that the composites could efficiently remove U(VI), CR and MV from solutions. For example, the maximum adsorption amounts of FH/CNTs composites for U(VI), CR and MV reached 187.26, 43.99 and 20.89 mg/g, respectively. The adsorption process was fitted better by pseudo-second order model, while both of Langmuir and Freundlich models were well fitted to the adsorption isotherms for these pollutants. Moreover, the composites could be easily separated after adsorption and efficiently reused. Thus FH was an efficient platform for the assembly of CNTs, and the as-prepared FH/CNTs composites had the potential application in water pollution control.

1. Introduction

Nanomaterials are known for their unique physicochemical properties. For example, small size effects bring significant optical, thermal and mechanical properties to nanomaterials. However, nanomaterial is not convenient to use [1], and can pose a potential risk when it's utilized as environmental material, which limit its practical application [2]. Thus there is a need for macro-assembly of nano-units. At present,

various techniques have been developed for the assembly of nano-units, such as layer-by-layer deposition, vacuum filtration assisted assembly, freeze-drying, interface-assisted assembly, and Langmuir–Blodgett technique [3–5].

Inspired by nature, an advanced nano-particle assembly (i.e., bioassembly) method is developed by the researchers. Attributed to its strong life activity, high reproduction rate, unique structure and cheap source, microorganisms (e.g., bacteria, fungi, and viruses) as vectors of

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micro/nano particles have been widely concerned [6–8]. Combining micro-organisms and micro/nano particles, micro/nano particles are involved in the growth of micro-organisms, and then the controllable assembly of 3D macro materials will be achieved through this interaction [9–11]. Furthermore, fungal hyphae (FH) with a certain shape and size is promising as functional material [12]. It can achieve the orderly assembly of micro/nano particles through its rapid growth process, making the synthesis of specific functional biosynthetic materials becomes a reality [12].

Environmental pollution, especially water pollution, has become a global environmental problem. Wastewater, especially from industry, contains a variety of pollutants, such as radionuclides and dyes. Adsorption is one of the most common-used strategies for the removal of these pollutants, and many adsorbents have been developed to remove these pollutants [13,14]. CNTs have great efficiency in water treatment due to their high surface area and ease of engineering with specific functionality [1]. Herein, we loaded CNTs on FH to prepare a composite of FH/CNTs through FH mediated bioassembly for the removal of uranium (U(VI)), cationic and anionic dyes [15]. The CNTs added in the FH growth environment could be deposited on the cell wall. Physico-chemical properties of the as-prepared composites were characterized. Furthermore, batch adsorption experiment was conducted to investigate their adsorption performances and mechanisms under various conditions, thus to demonstrate the effectiveness of this hyphae mediated assembly of CNTs and reveal the application potential of as-prepared FH/CNTs in water pollution control.

2. Materials and methods

2.1. Materials

Xylaria was obtained from Microbiology Department of East China Institute of Technology. Multi-walled carbon nanotubes (CNTs), purchased from XF NANO Material Technology Co., Ltd (XFM07), were produced by chemical vapor deposition (Nanjing, China). According to the manufacturer's specifications, the average diameter and length of CNTs were 15 nm and 50 nm, respectively, with 95% purity. Methyl violet (MV) and Congo red (CR) were purchased from Tianjin Chemical Reagent Institute Co., Ltd (Tianjin, China). Uranyl nitrate, sodium hydroxide, glucose, yeast extract, nitric acid, and peptone were supplied by Chengdu Kelong Chemical Co., Ltd.

2.2. Preparation of FH and FH/CNTs spheres

In a typical process, The fungi strains of *Xylaria* were incubated in 100 mL medium culture (g/L, peptone 2.5, glucose 20.0, Yeast extract 2.5, at pH 5.5), at 28 °C in Erlenmeyer flasks on an orbital shaker (Kuhner, Switzerland ISF-1-W) at 180 rpm for 4 d. The fungal biomass was harvested by filtration using nylon cloth, washed with generous amounts of deionized water and then immersed in 1 wt% aqueous NaOH and HCl at 20 °C for 3 h to inactivate the fungi and remove their residues, respectively, and finally washed with distilled water until neutral pH.

For FH/CNTs spheres preparation, the CNTs were treated by nitric acid, causing oxidation on the side walls of the nanotubes to obtain dispersal oxidized CNTs aqueous solution (5 mg/mL). FH/CNTs composite with different CNTs content (FH/CNTs-0.5, FH/CNTs-1 and FH/CNTs-2) was obtained by adding the CNTs solution (0.5, 1 and 2 mL) into the nutrient culture medium at 180 rpm for 4 d and the same treatment as pure fungus biomass. Both of FH and FH/CNTs were freeze-dried into the final hybrid spheres.

2.3. Characterization

The structural characteristics of samples were studied by X-ray diffraction (XRD, Panalytical X'Pert Pro) using Cu K α radiation. Fourier

transform infrared (FT-IR, PE Spectrum One) spectrometer with a resolution of 4 cm⁻¹ in the range of 4000–500 cm⁻¹. Thermal gravimetric analyses (TGA) was carried out using a thermal gravimetric analysis instrument (Q5000, TA, Newark, NJ, USA) under the nitrogen at heating rate of 10 °C/min. Zeta potential (zetaPALS, Brookhaven) was used to analyze the change of surface charge between FH and FH/CNTs. The surface morphology and cross-sectional features of the fibers cutting by liquid nitrogen were characterized by scanning electron microscopy (SEM; Zeiss Ultra 55). To determine U(VI) and dye content, UV–vis absorption spectrophotometer (UV-3150, Shimadzu) was used for optical absorbance measurements.

2.4. Adsorption experiments

The experiments evaluated the effects of pH, temperature, time and initial concentration by adding 10 mg of samples to 100 mL Erlenmeyer flasks containing 20 mL solution. In this study, the initial concentration of U(VI) and dye solution was controlled by the standard solution and the target concentration was obtained by quantitative dilution. The concentrations of U(VI) or dye in the solutions before and after adsorption were determined by a double beam UV–vis spectrophotometer (Shimadzu, Japan) at the wavelength of 651.8 nm (U(VI)), 497 nm (CR) and 579 nm (MV), respectively. All the experiments were conducted in triplicates and results were averaged.

For the effects of pH, the initial pH of U(VI) solutions (120 mg/L) were 2, 3, 4, 5, 6, 7 and 8, respectively; the initial pH of CR solutions (10 mg/L) were 5, 6, 7, 8, 9, 10 and 11, respectively; while the initial pH of MV solutions (4 mg/L) were 3, 4, 5, 6, 7, 8, 9 and 10, respectively. The expected pH was obtained by adjustment with 1 M HCl and 1 M NaOH solution using a pH meter (PHS-3G, pH meter precision). For the effects of temperature, the adsorption was conducted under temperatures from 20 to 40 °C and other variables were remained the same.

Kinetic experiments were conducted to determine the time needed to reach equilibrium and adsorption rates for U(VI) and dye. The initial concentrations of U(VI), CR and MV on adsorption kinetics were 120 mg/L, 10 mg/L and 4 mg/L, respectively. Samples were taken at different time intervals. All of the bottles were placed in a water bath at 25 ± 1 °C and shaken for 3 h.

For equilibrium adsorption, 5 mg adsorbent were added to 20 mL solutions with a initial pH of 5, 6.33 and 9.13 for U(VI), CR and MV, respectively, and incubated at 25 °C. The initial concentrations for U(VI) were 20, 50, 80, 100, 120, 150, 180 mg/L, respectively; for CR, were 5, 10, 15, 20 and 25 mg/L, respectively; for MV, were 2, 4, 6, 8, 10 mg/L, respectively. The adsorption amount of adsorbent was calculated through the Eq. S1.

To further clarify the mechanism of U(VI) sorption on FH and FH/CNTs-2, the pseudo-first-order (Eq. S2) and pseudo-second-order (Eq. S3) kinetics models were applied to study the adsorption kinetics of U(VI), CR and MV on FH and FH/CNTs [12,16]. The equilibrium adsorption data were fitted with Langmuir and Freundlich models (Eqs. S4–S7) [17,18]. The interpretation of equations used for fitting kinetics and isotherms were presented in supporting information (Eqs. S2–S7).

2.5. Co-existence of ions experiments

The co-existence ions experiments based on the adsorption process by adding different anionic sodium salt or cationic chlorate, and taking the suspension after adsorption equilibrium to measure the specified ion concentrations.

2.6. Desorption experiments

After 24 h, the concentration adsorbed was reached equilibrium. FH and FH/CNTs hybrid sphere loaded with U(VI) or dye could be separated from solution by a simple process. In general, the bulk material of FH and FH/CNTs sphere hydrogel would be held back by a glass rod

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