



Microorganism-derived carbon microspheres for uranium removal from aqueous solution



Feize Li^a, Dongmei Li^a, Xiaolong Li^a, Jiali Liao^{a,*}, Shoujian Li^b, Jijun Yang^a, Yuanyou Yang^a, Jun Tang^a, Ning Liu^{a,*}

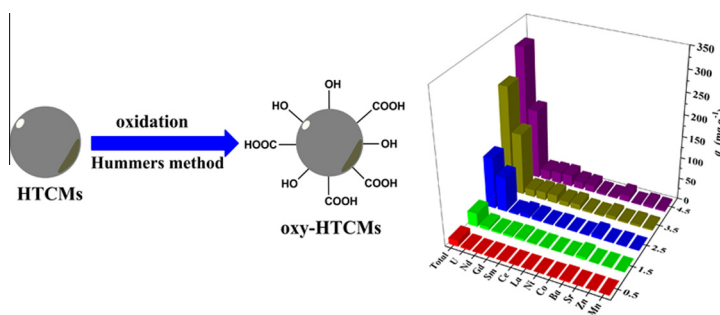
^a Key Laboratory of Radiation Physics and Technology, Ministry of Education, Institute of Nuclear Science and Technology, Sichuan University, Chengdu 610064, PR China

^b College of Chemistry, Sichuan University, Chengdu 610064, PR China

HIGHLIGHTS

- Microbial cells were applied to hydrothermal carbonization.
- Hummers method was employed to oxidize microorganism-derived hydrothermal carbon.
- Functional hydrothermal carbon was obtained without chemical grafting.
- The sorbent exhibits high sorption capacity for uranium.

GRAPHICAL ABSTRACT



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ABSTRACT

Saccharomyces cerevisiae, a typical industrial microorganism, was employed as a precursor to directly synthesize carbon microspheres. After a low temperature hydrothermal treatment (180 °C, 12 h), the microbial cells were successively transferred to carbon microspheres that had a diameter of 3–5 μm. The Hummers method was used to chemically modify the hydrothermal carbon materials to obtain functional materials for uranium removal. The characterizations and analysis showed that the morphology of the hydrothermal carbon was partially destroyed after functionalized activation, and abundant oxygen-containing groups were introduced onto the material surface. The results of the sorption behavior showed that the uranium (U) sorption capacity of the modified sorbent reached up to 183.4 mg g⁻¹, which was an improvement of approximately 4.6 times relative to the raw carbon materials. The thermodynamic and kinetic parameters demonstrated the removal process to be spontaneous, endothermic and pseudo-second-order chemisorption. The selective sorption of U(VI) from the simulated nuclear effluent at different pH values suggested that the sorbent displayed a desirable selectivity for the U(VI) ions over the other 11 competitive cations.

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1. Introduction

Uranium, one of the most important source materials for nuclear energy, has a specific commercial use as fuel for electricity

* Corresponding authors at: Sichuan University, No. 29, Wang Jiang Road, Chengdu, Sichuan Province, PR China. Tel.: +86 28 85412613; fax: +86 28 85412374.

E-mail addresses: liaojiali@scu.edu.cn (J. Liao), nliu720@scu.edu.cn (N. Liu).

generation [1]. Unfortunately, uranium is a non-renewable resource and the expected shortage of which in the near future may hinder the sustainable development of nuclear energy. Meanwhile, uranium is an element that can affect the ecosystem due to its radiochemical and toxicological effects on living components. Hence, the highly efficient separation and recovery of uranium from nuclear industrial effluent and contaminated water is of great significance not only for the full utilization of uranium resources

but also the protection of human health and ecological security [2,3].

To achieve the removal of uranium from aqueous media, one of the most important tasks is to obtain excellent separation materials with desirable characteristics, such as flexibility, high enrichment factors and minimal solvent consumption. Among the various materials, functional hydrothermal carbon (HTC), a newly developed solid-phase extractant, has attracted considerable attention over the last decade because of its outstanding thermal and radiation stability, pronounced acid–base resistance and high enrichment capacity [4,5]. However, because there are only a few functional groups on the material surface [5], unmodified HTCs always exhibit relatively low selectivity and poor sorption capacity toward uranium. Therefore, the surface modification of HTC via the introduction of various functional groups is often required to enhance the enrichment capacity and performance.

Generally, control of the surface functionality of HTC materials can be achieved by two main approaches. The first one is to incorporate a specific polymer with desirable functional groups during the carbonization process [4], which involves the protection of functional compounds, demanding critical selectivity of the starting materials and the strict control of the synthesis conditions. Another commonly employed approach is chemical modification using a post-treatment process. By taking advantage of the covalent bonds, hydrogen-bonding interactions and electrostatic effects, functional components, such as amidoxime, 5-azacytosine and salicylideneimine [6,7], could be grafted onto carbon matrix. Such chemical modification requires the choosing or synthesizing of specifically functional compounds with a high affinity for the target metal ions. Subsequently, the fixation of the functional components on the HTC matrix is also required. The additional chemical modification would make a complicated and multi-step synthesis procedure.

Thus, to directly introduce functional groups on the HTC matrix and to avoid chemical grafting of the selected components, further study is required. The surface oxidation via the Hummers method may provide another technically optional and economical approach to obtain functional HTC for uranium separation. First, as a classical approach to prepare graphite/graphene oxide [8,9], the Hummers method can effectively produce abundant oxygen-containing groups on raw carbon material surfaces. Furthermore, as suggested [10], graphene oxide nanosheets produced via this method can be used for uranium adsorption from aqueous solutions, and the abundant oxygen-containing functional groups have been demonstrated to play crucial roles in the sorption process. Additionally, previous studies have also employed this method to treat styrene–divinylbenzene copolymer microparticles for the preparation of polyphenolic hydroxyl functionalized uranium-selective chelating sorbent [11]. It has been demonstrated that benzene and furanic rings are typically present in HTC materials [12–14], which indicates the possibility of their oxidation similar to graphite/graphene and the styrene–divinylbenzene copolymer. Therefore, we hypothesize that the oxidation of HTC materials using the Hummers method could be a feasible and an effective way to obtain a surface-modified carbonaceous sorbent for uranium enrichment.

On the other hand, to date, general types of starting materials for HTC include pure carbohydrates (glucose, sucrose, cellulose, starch, lignin, chitosan, etc.) [15–18] or crude plant materials (pericarps, leaves, shrimp shells, pine needles, etc.) [19–21]. Nevertheless, there is a lack of research on the use of microorganism cells as carbon precursors to prepare functional HTCs. In a previous work, we reported a direct synthesis of carbon-based microtubes by hydrothermal carbonization of a soil microorganism biomass (*Streptomyces sporverruosus dwc-3*) [22]. As the natural structure of the soft biomass had also been well inherited during the

hydrothermal carbonization process, we proposed that novel carbon materials with various structural features could be prepared employing different microorganisms as precursors. Herein, *Saccharomyces cerevisiae* (*S. cerevisiae*), an industrial microorganism used in food and beverage production [23], were employed as precursors to synthesize hydrothermal carbon microspheres (HTCMs) in this work. To achieve the surface functionality, for the first time, the HTC materials were further modified using the Hummers method. Our goal was to prepare carbon materials with improved thermal and radiation stability on a large scale and at a low cost, which could serve as a promising sorbent in uranium pollution clean-up and uranium enrichment.

2. Experimental section

2.1. Reagents

The chemicals and reagents, including KMnO_4 , H_2SO_4 , NaOH , HNO_3 , HCl , glucose, glutaraldehyde and H_2O_2 were purchased from Chengdu Kelong Chemical Reagent Co., Ltd., China. All metal oxides or nitrates were purchased from Aladdin Chemistry Co., Ltd., China. All reagents were of AR grade and were used without further purification. The strain of *S. cerevisiae* was provided by the College of Life Science at Sichuan University and cultivated as described in the [supplementary material](#).

2.2. Characterization

The morphology of the materials was observed by scanning electron microscopy (SEM, S-4800, Hitachi, Japan). The elemental chemical states on the surface of the produced materials were measured by X-ray photoelectron spectroscopy (XPS, XSAM800, UK). Fourier transform infrared spectra (FT-IR) were obtained using a PerkinElmer IR-843 spectrometer (USA). The potentiometric acid–base titration was performed using a G20 Compact Titrator (Mettler Toledo, Switzerland).

2.3. Synthesis of HTC using *S. cerevisiae* cells as the carbon source

In the typical HTC synthesis procedure, the wet *S. cerevisiae* cells (5 g) were first dispersed in a 6% (v/v) glutaraldehyde aqueous solution (30 mL) as homogeneously as possible. The suspension was then transferred to a 50 mL Teflon-lined stainless steel autoclave that was then sealed and heated to 180 °C at a rate of 1.5 °C min^{-1} and maintained for 12 h. After allowing the suspension to cool down to room temperature, a brown powder was obtained via vacuum filtration, which was washed alternatively using deionized water and ethanol until the filtrate was colorless. It was then dried at 60 °C in a vacuum overnight. The obtained materials were denoted as raw-HTCMs.

2.4. Preparation of the functionalized HTC

The chemical modification of the HTC was achieved via the Hummers method, as described in a previous report [9]. Generally, 1.0 g of the raw-HTCM was dispersed into 30 mL of concentrated sulfuric acid in a round-bottomed flask, then the mixture was magnetically stirred at room temperature for 30 min. Then, 2.5 g of potassium permanganate was added to the flask slowly, and the mixture was stirred continually at 40 °C in a water bath for 6 h. The suspension was diluted to approximately 100 mL with deionized water and allowed to cool to room temperature. Finally, the product was treated using 5 mL of 30% H_2O_2 to reduce the residual permanganate and manganese dioxide in the mixture. The yellowish-brown hydrophilic powder was obtained by vacuum fil-

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