



# Synthesis of magnetic biochar composites for enhanced uranium (VI) adsorption

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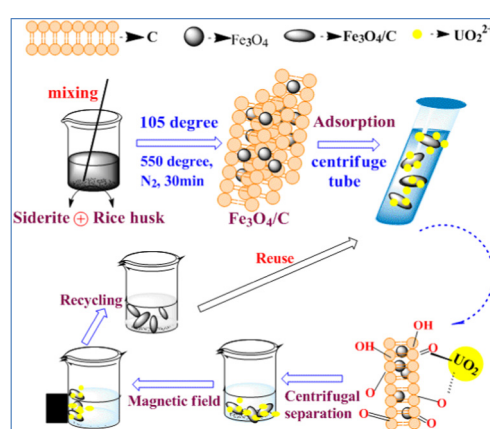
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## HIGHLIGHTS

- Magnetic biochar was obtained by pyrolysis the rice husk and siderite.
- The magnetic biochar has the advantages of low cost and high efficiency.
- The interaction mechanism of U(VI) on magnetic biochar was adsorption and reduction.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Magnetic biochar composites were successfully fabricated by pyrolysis of siderite and rice husk under N<sub>2</sub> condition. The results of a variety of characterization implied magnetic biochar displayed porous structures with larger specific surface area. The batch adsorption experiments showed high adsorption properties of magnetic biochar composites toward U(VI) (52.63 mg/g at pH 4.0), whereas U(VI) adsorption was significantly influenced by Na<sub>2</sub>CO<sub>3</sub> and HA. U(VI) adsorbed onto magnetic biochar was reduced to U(IV) by Fe<sub>3</sub>O<sub>4</sub> according to XPS and XANES analyses. In addition, no significant effect of ionic strength of NaCl and EXAFS results, illustrated the inner-sphere surface complexation of U(VI) on magnetic biochar. Owing to the simple synthesis procedure, low cost, high adsorption efficiency, easy separation and environmental friendly, magnetic biochar can be considered as a potential adsorbent for the purification of U(VI)-bearing wastewater in environmental remediation.

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## 1. Introduction

Nowadays, the increasing radionuclides will be released into the aquatic environments as a result of the development of long-term operation of nuclear energy and nuclear fuel cycle facilities (Borai et al.,

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2009; Fan et al., 2011; Li and Kaplan, 2012). Uranium(VI) is regarded as one of the most common radionuclides in the sewerage owing to its radioactivity and toxicity to human beings (Liu et al., 2017a). Hence, a variety of methods have been employed to remove uranium(VI) from aqueous solutions such as solvent extraction (Singh et al., 2004), chemical precipitation (Mellah et al., 2007), flotation (Rao et al., 2006), membrane dialysis (Omar et al., 2009), and adsorption (Abdel-Karim et al., 2016; Boeykens et al., 2017; Fuller et al., 2016). Among these approaches, adsorption has attracted much attention owing to the easy operation, low cost, high efficiency and environmental friendly (Hu et al., 2011).

Owing to the high surface area and considerable negative charge, biochar is ordinarily fabricated by the pyrolysis of organic materials under anoxic conditions (Zhang et al., 2013), which can reduce the mass/volume of waste materials. In recent years, a number of research efforts have been made to utilize biochar as an adsorbent in water cleanup applications for removing multiple contaminations, including heavy metals (Beesley et al., 2010), nutrients (Inyang et al., 2012) and organics (e.g. phenolics, halogenated compounds, solvents) (Yao et al., 2012). In these studies, it is demonstrated that interaction mechanism of biochar with these environmental contaminants by ion exchange, electrostatic attraction, physical sorption and precipitation. However, the difficult problem regarding the separation of biochar from solutions impeded the wide application into wastewater remediation (Jung et al., 2017).

In order to maximize the recycling of biochar from aqueous solution, magnetic biochar composites have been widely concerned. Surprisingly, a large number of reports showed that magnetic biochar not only achieved the purpose of recycling, but also greatly improved the adsorption capacity. (Han et al., 2016; Thines et al., 2017). However, these magnetic biochar were generally synthesized by the chemical precipitation of  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  or by a simplified aqueous phase approach using iron chloride on the surface of biochar under alkaline conditions. The reports on the synthesis of magnetic biochar from iron bearing minerals are quite rare. As is well known that, China is rich in siderite resources with nearly 2 billion tons, but the utilization of it was <10% of the total reserves. According to previous report (Xing et al., 2017), siderite can be transformed into magnetic minerals and exhibit a series of magnetic phenomena (Li et al., 2017a). In recent, Guo et al. (2007), Guo et al. (2008), Zhang et al. (2018) and Li et al. (2017b) had carried out in-depth studies on the removal of heavy metals from groundwater by siderite, but no report on the purification of other pollutants. Consequently, we synthesized magnetic biochar by siderite and rice husk in this study and further discussed the adsorption mechanism of U(VI) on it.

This study investigated the applicability of magnetic biochar for remediation of U(VI) species from contaminated waters. The main purposes are to (1) fabricate magnetic biochar composites by pyrolyzing mixture of siderite and rice husk under  $\text{N}_2$  conditions and characterize it by multiple methods; (2) discuss the effect of water conditions on U(VI) species adsorption by the target magnetic biochar composites by a combination of batch; (3) evaluate the adsorption mechanism of U(VI) species onto magnetic biochar composites using XANES, EXAFS and XPS analyses. This study highlights the utilization of waste to treat waste.

## 2. Experimental

### 2.1. Materials

The raw materials used in this study were natural siderite (Anhui, China) and rice husk were (Hefei, China), respectively. According to the result of X-ray fluorescence spectroscopy (XRF), the chemical composition of siderite mainly contains  $\text{Fe}_2\text{O}_3$  (60.22 wt%) and minor impurities such as MnO (3.55 wt%) and MgO (2.23 wt%, Table S1). The chemical compositions of rice husk were O (39.19 wt%), C (36.36 wt%),

H (5.34 wt%), N (0.61 wt%) and S (0.063 wt%, Table S2). The rice husk was pulverized and sieved to <180  $\mu\text{m}$ . U(VI) stock solution ( $1.0 \times 10^{-3}$  mol/L) was prepared by dissolving  $\text{UO}_2(\text{NO}_3)_2$  (spectrographic purity, Hubei Chemical Industry Co., Ltd.) into 0.02 mol/L  $\text{HNO}_3$  solutions. Other chemicals were all of analytical grade.

### 2.2. Preparation of magnetic biochar composites

The magnetic biochar composites were prepared by calcining the mixtures of siderite and rice husk under the protection of nitrogen. Briefly, 10.0 g of rice husk was primarily carbonized at 500 °C for 1 h under the protection of  $\text{N}_2$ . Next, 4.0 g of carbonized rice husk was mixed with 1.0 g of NaOH in 50 mL DI water under constant stirring conditions for 24 h and then washed to pH = 7. Afterward, 1.0 g of siderite was added into the suspension and stirred for 30 min, then was dried at 105 °C overnight. Magnetic biochar with 86 wt% of biochar and 14% wt% of  $\text{Fe}_3\text{O}_4$  was obtained by pyrolyzing at 550 °C for 0.5 h under  $\text{N}_2$  protection.

### 2.3. Characterization

CHN Elemental analyzer (Vario EL c) was used to analyze total carbon, nitrogen, oxygen and hydrogen content. Chemical composition of natural siderite was measured using an X-ray fluorescence (XRF) spectrometer (Shimadzu XRF-1800). The morphology of magnetic biochar was examined by scanning electron microscopy (JEM-2010, Japan) with dispersive X-ray spectroscopy (EDS). The mineralogy of magnetic biochar was analyzed by an X-ray diffractometer (Dandonghaoyuan 2700) equipped with a rotation anode using Cu  $K\alpha$  radiation at 300 mA and 45 kV. The surface structural groups of magnetic biochar were characterized using a Fourier transform infrared spectrometer (VERTEX-70). The zeta potential of each suspension was measured by a Malvern Zeta Potentiometer (Nano-ZS90). Surface elemental composition was analyzed by an X-ray photoelectron spectrometer (Thermo ESCALAB250Xi).  $S_{\text{BET}}$  was measured using  $\text{N}_2$  sorption using TriStar II 3020 instrument by Brunauer–Emmett–Teller (BET) method. Magnetization curve was examined by a magnetometer (Lakeshore Cryotronic, USA). U(VI) L3-edge EXAFS spectra were conducted at the BL14W1 beamline of the Shanghai Synchrotron Radiation Facility using a Si (111) double crystalline monochromator.

### 2.4. Batch experiment

The adsorption of U(VI) on magnetic biochar were carried out at 10 mL polycarbonate tubes ( $C_0 = 10$  mg/L,  $m/V = 0.2$  g/L). The procedure was performed as follows: the mixture of 0.6 mL 0.1 mol/L NaCl solution, 2.4 mL of Milli-Q water and 2.0 mL 0.6 g/L magnetic biochar suspension were pre-reacted for 24 h, after that 1 mL 60 mg/L U(VI) stock solutions was injected to the aforementioned solutions (total volume: 6 mL). The pH values of the suspensions were adjusted by 0.002–2.0 mol/L  $\text{HNO}_3$  and/or NaOH solutions. The solid-liquid phases were recovered from the supernatant using a magnet after shaken for 24 h. The blank tests were carried out simultaneously. The concentration of U(VI) was tested by a Spectrophotometric method (722E, China).

The regeneration of U(VI) species onto magnetic biochar composites was conducted to investigate the reusability in actual environments. After the adsorption process achieved equilibrium, the wet-solid was extracted from liquid-phase by centrifugation and then by washing with Milli-Q water for 3 times. Then 6 mL of 0.1 mol/L HCl solutions was added and reacted for 24 h. After desorption equilibrium, the samples were separated from the solution by 10,000 rpm centrifugation. Then the process of adsorption experiments mentioned above was repeated. The above adsorption-desorption procedure was cycled five times to test the reusability of magnetic biochar.

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