

ЭФФЕКТИВНОЕ ПОЛУЧЕНИЕ ПЛЕНКИ ИЗ КОСТНОГО УГЛЕРОДА С СЕЛЕКТИВНОЙ СОРБЦИЕЙ U(VI) ИЗ ВОДНОГО РАСТВОРА МЕТОДОМ РУЛОННОЙ ПЕЧАТИ

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Многие соединения урана (VI) хорошо растворимы в воде и легко мигрируют в геологическую среду. Подобно накоплению тяжелых металлов, U (VI) также может накапливаться в пищевой цепи, вызывая опасные заболевания, включая повреждение почек, печени, легких, мышц и даже рак или смерть. В ходе нашего предыдущего исследования обнаружено, что пленка из субмикронных волокон/измельченного костного угля (CL/MB), полученная методом электроформования, может быть хорошо диспергирована в растворителе для электроформования. Мембрана CL/MB может эффективно адсорбировать U(VI) и обладает хорошей избирательностью к взаимодействию сосуществующих ионов в имитируемой воде. В этом исследовании костный уголь, полученный в результате пиролиза остатков костей животных при температуре 350 °C, 500 °C и 650 °C был превращен в чернила из костного угля на спиртовой основе путем добавления поверхностно-активных веществ и дисперсных средств, препятствующих осаждению. Методом рулонной печати чернила из костного углерода на спиртовой основе наносятся на полиэтилен-терефталат (ПЭТ). В моделируемой морской воде двухвалентные катионы оказывают большее влияние на субстрат для формирования костной углеродной пленки (BCF). Двухвалентные катионы, особенно Ba^{2+} , оказывают большее влияние на BCF, при этом избирательная сорбция водного U(VI) достигает (81,4±1,6)%. CO_3^{2-} и SO_4^{2-} могут взаимодействовать с U(VI) в воде, что приводит к их значительному уменьшению (CO_3^{2-} снижается на (70,5±2,4)%; SO_4^{2-} снизился на (62,4±4,8)%). Это исследование открывает путь для практического применения рулонного промышленного производства BCF с селективной сорбцией водного U(VI) (скорость лабораторной печати 5-8 м/мин, ширина пленки ≤600 мм).

Ключевые слова: рулонная печать, чернила из костного угля, пленка из костного угля, механизм

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EFFICIENT PREPARATION OF BONE CARBON FILM WITH SELECTIVE SORPTION OF AQUATIC U(VI) BY ROLL-TO-ROLL PRINTING METHOD

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The highly soluble oxidation state of uranium (U (VI)) in an aqueous solution is the most dangerous, as it easily migrates into the geological environment. Similar to the accumulation of heavy metals, uranium U(VI) can also accumulate through the food chain, causing harmful diseases, including damage to the kidneys, liver and lungs, muscles, and even cancer or death. In our previous research, we found that the submicron fiber/ball ground bone charcoal (CL/MB) film prepared by electrospinning technology can be well dispersed in electrospinning solvent. CL/MB membrane can adsorb U(VI) efficiently, and has good selectivity to the interference of coexisting ions in simulated water. In this study, the bone carbon obtained from the pyrolysis of animal bone residues at 350 °C, 500 °C and 650 °C was prepared into alcohol-based bone carbon ink by adding surfactants and dispersed anti-settling agents. By roll-to-roll printing, alcohol-based bone carbon ink is printed onto polyethylene terephthalate (PET) substrate to form bone carbon film (BCF). In simulated seawater, bivalent cations especially Ba^{2+} , have a greater effect on BCF with selective sorption of aquatic U(VI) reaches (81.4±1.6)%. CO_3^{2-} and SO_4^{2-} can interact with U(VI) in water, resulting in significant leads to their significant reduction leads to their significant reduction: decreased by (70.5±2.4)%; SO_4^{2-} decreased by (62.4±4.8)%. This study paves the way for the feasible application of roll-to-roll industrial efficient production of of BCF with selective sorption of aquatic U(VI) (laboratory printing speed 5-8m/min, film width ≤600mm).

Keywords: roll-to-roll printing, bone charcoal ink, bone charcoal film, mechanism

INTRODUCTION

The highly soluble oxidation state of uranium (U(VI)) in an aqueous solution is the most dangerous, as it easily migrates into the geological environment [1, 2]. Similar to the accumulation of heavy metals, U (VI) can also accumulate through the food chain, causing harmful diseases, including damage to the kidneys, liver and lungs, muscles, and even cancer or death [3, 4]. At the same time, the recovery of U(VI) from seawater is a critical issue, as it is crucial for the development of nuclear power [5]. From the point of view of radionuclide management and nuclear energy sustainability, the efficient capture of U(VI) from aqueous solutions

is an urgent issue that deserves urgent research. Conventional methods for removing aquatic U(VI) are inefficient because they require large amounts of organic solvents and metal extractants or solvent extraction using selective ligand extractants is limited. Therefore, U(VI) adsorption is considered to be an economical, easy to operate and promising environmental remediation strategy [6, 7]. However, the traditional adsorbents often have the problems of weak stability, narrow utilization rate and low absorption capacity [8, 9]. The products in this study may be widely used due to the advantages of industrial production, low cost of main raw materials (animal bone and PET), and good dispersion of adsorbent.

The roll-to-roll printing technology is currently one of the most efficient printing methods, it will be continuous base material (Polymer composites, such as PET, PE, etc.) are rolled into huge spool or core and fed into the printer for printing [10, 11]. The roll-to-roll printing machines usually use letterpress printing, gravure printing, flexographic printing or inkjet printing and other technologies, through digital control equipment, to achieve high-speed, high-precision printing [12]. Especially suitable for large-scale continuous production, can achieve high production efficiency. This study mainly uses inkjet printing, so the ink modulation is particularly critical. We introduce binder, dispersant, surfactant, leveling agent, humectant, thickener, curing agent and other additives to improve the ink performance, and use the mixture of *n*-heptane (40%) and 2-propanol (60%) as the solvent, to prepare a uniform dispersion, stable performance, strong adhesion to PET substrate and does not affect the bone charcoal on aquatic U(VI) highly selective adsorption ink [13]. In this study, BCF was prepared by roll-to-roll printing technology, Fig. 1.



Fig. 1. Film printing equipment by roll-to-roll mode

Рис. 1. Оборудование для печати на плёнке рулонным методом

Bone char dispersed in the ink was derived from sheep bone residue pyrolyzed at 350 °C, 500 °C and 650 °C respectively, and the preparation was successful through characterization of morphology, crystal structure and surface functional groups [14-16]. At the same time, the selective sorption of aquatic U(VI) of BCF under various hydrochemical conditions (including contact time, solution PH, ionic strength, humic acid (HA), coexistence of cations and anions, ambient temperature) showed that U(VI) could be adsorbed with fast kinetics (170.0 min), excellent capacity (317.6 mg/g at pH = 5.00 and T = 293 K) and selectivity against the interference of coexisting ions in artificial wastewater and simulated seawaters. By combining the spectroscopic analysis of BCFs after the sorption of U(VI), the multiple sorption mechanisms

were elucidated, such as functional group complexation, phosphorus atom bonding to form precipitation and electrostatic adsorption [17].

MATERIAL AND METHODS

Materials

Residuals of sheep bone were obtained from the local slaughter house of Chongqing (China). All the chemicals and reagents including binder (DJB-823), surfactant (Zonyl@FSO), dispersant (ethanolamine), flattening agent (ethylene glycol dibutyl ether: propylene glycol methyl ether-1:1), humectant (propylene glycol), antifoam agent (2-hexyl ethanol), solvent (*n*-heptane: 2-propanol-2:3) were purchased from Aladdin Chemistry Co. Ltd. (Shanghai, China). These were of analytical grade and used without further purification. Deionized (DI) water (resistance 18.2 MΩ·cm, Milli-Q Plus, Merck Millipore Co., Germany) was used in all experiments.

Preparation of milled bone charcoals

The preparation process was illustrated in Fig. 1. The collected raw sheep bone residuals were pyrolyzed in a tube furnace at 350 °C, 500 °C and 650 °C for 2 h with a continuous N₂ flow of 200 sccm. 1.0 g bone chars were treated by ball milling with the addition of 20.0 g DI water and 100.0 g agate balls (φ 1-3 mm). The milled duration was 12 h at 500 rpm with a changed rotation direction each 3 h. The obtained milled chars were named as milled bone charcoal (MBC) including MBC350, MBC500 and MBC650 according to the pyrolysis temperature of pristine bone charcoal.

Preparation of bone charcoal ink

The solid protective agent DJB-823 was ground into powder. 100 ml of mixed solvent (40% *n*-heptane, 60% 2-propanol) was added to its 1.5 g sample. The resulting mixture was heated in a water bath to 55-650 °C with gentle stirring for 10 minutes until the protective agent was completely dissolved in a clear solution. According to the determined formula composition of milled bone charcoal 1%, surfactant 0.05%, small molecule dispersant 1%, small molecule levelling agent 1.5%, small molecule humectant 3%, small molecule defoamer 1% and solvent 92.45%, the required amount of each substance was calculated. The remaining ingredients, except the protective agent, were then mixed well and stirred for 20 min. Then 0.15% of the protective agent was added to the stirred ink and stirring continued for 30 min [18, 19]. Finally, the final ink was filtered by a filter membrane with a aperture of 1 μm.

Preparation of bone charcoal film

The finished ink was added to the roll-to-roll printing machine, and the base film was selected to be

a PET roll with a thickness of 20 μm and a width of 600 mm. The bone charcoal was printed on the PET base film by spraying and baked at 80 $^{\circ}\text{C}$ for 2 min to obtain the bone charcoal film as it was shown in the Fig. 1.

Characterization

A coating of gold (4 nm of thickness) was coated onto the surface of bone charcoal films by a spraying device (Mind in China). Then the morphology of samples was observed by scanning electron microscopy (SEM) equipped (Carl Zeiss NTS GmbH, Oberkochen, Germany). The crystalline structure of bone charcoal films was recorded using X-ray diffractometry (XRD) with a Cu K-alpha radiation at 40 kV ($\lambda = 0.15418$ nm). The surface functional groups of samples were recorded on a Spectrum One Autoimage spectrometer (PerkinElmer, U.S.A.) in the range of 400–4000 cm^{-1} with the addition of KBr pellets. Zeta potential of sample surfaces in the pH range of 2.0–7.0 was obtained via a Zetasizer Nano ZS90 Analyzer.

RESULTS AND DISCUSSION

Characterization of electrospun bone charcoal films

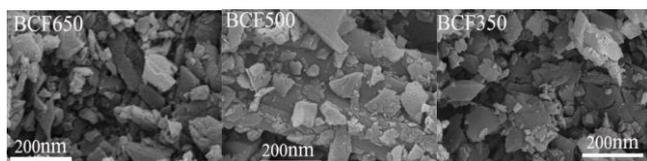


Fig. 2. SEM images of BCF350, BCF500, BCF650
Рис. 2. СЭМ-изображения BCF350, BCF500, BCF650

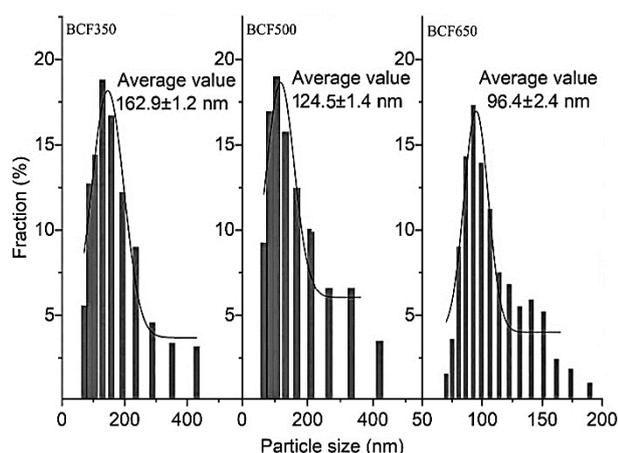


Fig. 3. Particle size of BCF350, BCF500, BCF650
Рис. 3. Размер частиц BCF350, BCF500, BCF650

Fig. 2 shows the SEM microscopic image of bone carbon membranes (BCFs). Irregular bone carbon particles with a diameter of about 100 nm are evenly distributed on PET. The particle size of bone char decreased with the increase in pyrolysis temperature. For

further proof, we measured the particle size of the fractured bone char and found that its average diameter decreased from 153.9 ± 1.2 nm (BCF350) to 96.4 ± 2.4 nm (BCF650), as shown in Fig. 3.

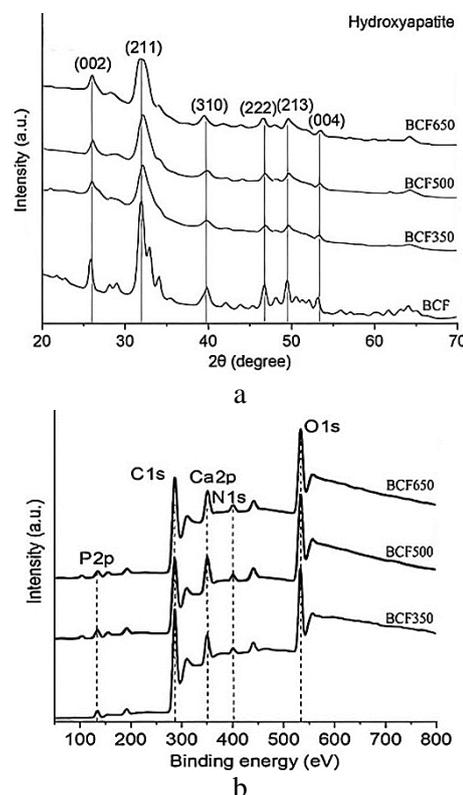


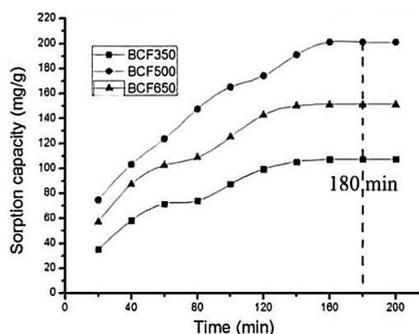
Fig. 4. XRD patterns (a) and Survey XPS spectra (b) of BCFs
Рис. 4. Рентгенограммы (а) и обзорные спектры XPS (b) BCF

XRD patterns of BCFs were shown in Fig. 4a. Six diffraction peaks corresponded to (002), (211), (310), (222), (213) and (004) of BCF component (JCPDS PDF 00-001-1008) were located at 25.6 $^{\circ}$, 31.6 $^{\circ}$, 39.4 $^{\circ}$, 46.5 $^{\circ}$, 49.4 $^{\circ}$, and 53.0 $^{\circ}$ respectively [20]. Considering the same peak positions of BCF350, BCF500, BCF65 with pristine BCF, it is indicated that the crystal structure of BCF was not damaged by the roll-to-roll printing.

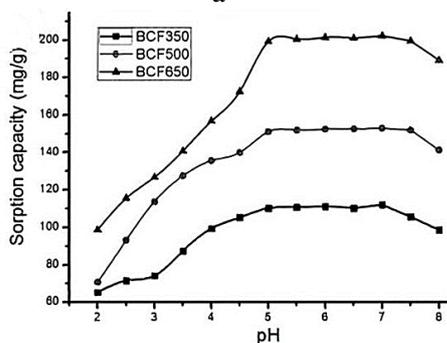
The surface elemental compositions and their corresponding chemical states of BCFs were shown in Fig. 4b, elements of P, C, Ca, N and O were observed in the survey XPS spectra. Since raw bone powder is a by-product of sheep slaughtering plants, it is believed that N element on the surface of BCFs should be attributed to the organic and inorganic elements existed in the sheep bone residuals. Bone char particles after grinding induced the exposure of oxygen- and nitrogen-containing groups from blocked pores, allowing the modification of carbon component. The bone chars were composed of porous carbon matrix and hydroxyapatite (HAP). Meanwhile, HAP component of bone

chars induced with the sizes of sub-micrometer and nanometer has been reported as an efficient sorbent for the capture of aquatic radionuclides through precipitation and cation exchange. By combination of bone chars with PET films, it would be desired to be applied of separation materials for the elimination of heavy metals from aqueous solutions.

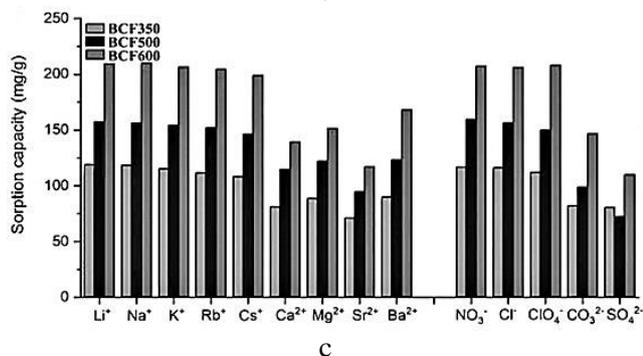
Effect of various hydrochemical conditions



a



b



c

Fig. 5. Effects of contact time (a), solution pH (b), and coexisting cations and anions (c) on the sorption of U(VI). $C = 50.0 \text{ mg/L}$, $T = 293 \text{ K}$

Рис. 5. Влияние времени контакта (а), pH раствора (б) и сосуществующих катионов и анионов (с) на сорбцию U(VI). $C = 50,0 \text{ мг/л}$, $T = 293 \text{ К}$

As it shown in the Fig. 5a, the adsorption capacity of BCFs depends on the contact time and consists of three cycles, the value of which increases rapidly in the first cycle of 100 min, which should be due to the abundant adsorption sites and sufficient U(VI) in the aqueous solution. The second contact time is 100

~ 160 min, and the value increases with the decrease in ascending gradient due to the enhancement of U(VI) enriched BCFs diffusion resistance. The U(VI) value on BCFs reached the third stage at the high stage after 160 min, and the adsorption equilibrium was reached at 180 min.

It exists in aqueous solution ($C[\text{U(VI)}]_{\text{initial}} = 50.0 \text{ mg/L}$, $T = 293 \text{ K}$) under given experimental conditions. In aqueous solution with pH value of 2.0-5.0, the adsorption capacity of BCFs for U(VI) showed an increasing trend with the gradual increase in pH value. The adsorption capacity of BCFs to U(VI) tends to be stable in aqueous solution with $\text{pH} = 5.0-7.0$. When $7.0 < \text{pH} < 8.0$, the adsorption capacity of BCFs for U(VI) decreased and increased with the gradual increase in pH value, Fig. 5b.

As shown in Fig. 5c, the monovalent cations (Li^+ , Na^+ , K^+ , Rb^+ , Cs^+ , etc.) in the solution BCFs has a little influence on the absorption rate of U(VI) [21]. The adsorption of BCFs on U(VI) was more obvious in divalent cations (Ca^{2+} , Mg^{2+} , Sr^{2+} and Ba^{2+}) solutions, especially in Ba^{2+} solution, the effect of BCFs on the adsorption rate of U(VI) was as high as $(81.4 \pm 1.6)\%$. This can be attributed to the formation of $(=\text{SO})_2\text{-M}^{2+}$, which occupied twice binding sites than monovalent cations. Meanwhile, the divalent cation also presented smaller ionic radius (Ba^{2+} (1.34 Å)) than the monovalent cation (Na^+ (1.02 Å)) in the same period [22]. Secondly, a neutral alkaline earth metal-uranium carbonate complex $\text{Ba}_2\text{UO}_2(\text{CO}_3)_{30}$ is formed.

In addition, even if carbonates or bicarbonates are not directly added to the solution, the exchange of CO_2 molecules in the aqueous solution with the atmosphere will affect the total amount of dissolved inorganic carbon in the solution. The effect of neutral or alkaline chemical environment on the uranium absorption rate of BCFs cannot be ignored, because U(VI), CO_3^{2-} and alkaline earth metals (Mg^{2+} , Ca^{2+} , Sr^{2+} , Ba^{2+}) form ternary complexes [23, 24]. The effect of BCFs on uranium absorption in anion solutions (NO_3^- , Cl^- , ClO_4^- and BO_3^{3-}) is negligible because of their low affinity for U(VI). However, since CO_3^{2-} and SO_4^{2-} can interact with aquatic U(VI) by forming $\text{UO}_2\text{CO}_3(\text{aq})$ and $\text{UO}_2\text{SO}_4(\text{aq})$, the addition of CO_3^{2-} and SO_4^{2-} to the solution interferes significantly with the adsorption of U(VI) ($70.5 \pm 2.4\%$ of CO_3^{2-} . SO_4^{2-} decrease rate is $(62.4 \pm 4.8)\%$ [25, 26].

CONCLUSIONS

In summary the bone carbon obtained from the pyrolysis of animal bone residues at 350 °C, 500 °C and 650 °C was prepared into alcohol-based bone carbon ink by adding surfactants and dispersed anti-settling agents. By roll-to-roll printing, alcohol-based

bone carbon ink is printed onto PET substrate to form BCF. The systematically study of sorption behaviors under various hydrochemical conditions showed that U(VI) could be adsorbed by BCF. The U(VI) adsorption value of BCF pair reached the highest at about 160 min, and the adsorption equilibrium was reached at 180.0 min. Excellent capacity (309.8 mg/g at pH = 5.00 and T = 293 K) and selectivity towards coexisting multiple ion interferences in artificial wastewater and simulated seawater were achieved.

In simulated seawater, bivalent cations have a greater effect on BCF with selective sorption of aquatic U(VI) than monovalent cations, especially in Ba²⁺, BCF with selective sorption of aquatic U(VI) affects up to (81.4±1.6)%. The effect of neutral or alkaline chemical environment on BCF with selective sorption of aquatic U(VI) cannot be ignored. The effect of anions on BCF with selective sorption of aquatic U(VI) was negligible. However, CO₃²⁻ and SO₄²⁻ can interact with U(VI) in water, resulting in significant interference with selective sorption of aquatic U(VI) (CO₃²⁻ decreased by (70.5±2.4)%; SO₄²⁻ decreased by (62.4±4.8)%).

Radioactive waste disposal: Uranium-saturated adsorbent films are slurried in water under stirring. Add Ca₁₀(PO₄)₆(OH)₂ was added to form the precipitate of Ca(UO₂)₂(PO₄)₂(H₂O)₃ [27, 28]. The solid residue is immobilized in a polymer matrix, packaged in a labeled container, and transferred to a certified radioactive waste disposal agency for final disposal.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

The authors declare the absence of a conflict of interest warranting disclosure in this article.

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