

Biomass aerogel based on chitosan and bayberry tannin for uranium recovery from aqueous solution

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Abstract

Many adsorbents have been developed for uranium recovery to ensure global energy and environmental security. However, most reported adsorbents involve complex preparation process and rely heavily on petrochemical feedstocks, which undoubtedly increases carbon emissions from production in the nuclear industry. Here, a biomass aerogel (CS-BT) is prepared by the facile cross-linking of chitosan and bayberry tannins with glutaraldehyde. U(VI) can be adsorbed by hydroxyl groups on CS-BT aerogel via chelation, and the maximum adsorption capacity of the obtained aerogel to U(VI) is ~140 mg g⁻¹ and the removal rate reaches up to 99% (at 298.15 K, pH = 5.0). The pseudo-second-order kinetics model and Freundlich model can better match the adsorption process of CS-BT aerogel, implying that its adsorption is a chemical adsorption process dominated by multilayer adsorption. The thermodynamic results show that the adsorption process of U(VI) by CS-BT aerogel is spontaneous and exothermic. Hence, our biomass aerogel can effectively extract uranium from water, contributing to the sustainable development of the nuclear industry.

Full Text

Biomass Aerogel Based on Chitosan and Bayberry Tannin for Uranium Recovery from Aqueous Solution

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Abstract

Numerous adsorbents have been developed for uranium recovery to ensure global energy and environmental security. However, most reported adsorbents involve complex preparation processes and rely heavily on petrochemical feedstocks, which inevitably increases carbon emissions from production in the nuclear industry. Here, we prepared a biomass aerogel (CS-BT) through facile cross-linking of chitosan and bayberry tannins with glutaraldehyde. U(VI) can be adsorbed by hydroxyl groups on the CS-BT aerogel via chelation, achieving a maximum adsorption capacity of approximately 140 mg g⁻¹ and a removal rate of up to 99% (at 298.15 K, pH = 5.0). The pseudo-second-order kinetics model and Freundlich model provide the best fit for the adsorption process, indicating that adsorption occurs through a chemical process dominated by multilayer adsorption. Thermodynamic results demonstrate that U(VI) adsorption by the CS-BT aerogel is spontaneous and exothermic. Therefore, our biomass aerogel can effectively extract uranium from water, contributing to the sustainable development of the nuclear industry.

Keywords: biomass aerogel, chitosan, bayberry tannin, uranium recovery

Introduction

Limited resource availability, uncontrolled population growth, and global environmental degradation have intensified interest in sustainable clean energy resources. Nuclear fuel based on uranium represents a new clean energy source whose fission releases far more energy than coal combustion while effectively avoiding greenhouse gas emissions [1, 2]. However, uranium-containing wastes from the nuclear fuel cycle or nuclear accidents are both radioactive and chemically toxic [3]. With a half-life of millions of years, uranium can easily migrate into aquatic environments following slight environmental changes, causing chronic pollution [4]. Simultaneously, large uranium reserves are essential for energy security and greenhouse gas mitigation. Therefore, developing efficient technologies for removing and recovering uranium from nuclear industry

wastewater and other solutions is critical for ensuring environmental safety and building nuclear energy reserves.

Current research efforts heavily focus on extracting U(VI) (the predominant aqueous species of uranium) from diverse uranium-laden solutions, including nuclear industry wastewater and seawater, primarily through adsorption [5, 6], ion exchange [7], membrane separation [8, 9], and solvent extraction [10]. Among these methods, adsorption is the most common and effective approach for uranium extraction and removal of other contaminants due to its cost-effectiveness and operational simplicity [11-15]. Various adsorbents have been successfully developed, including metal/covalent organic frameworks [6, 16], synthetic organic polymers [17], and carbon-based materials [18]. However, most reported adsorbents are synthetic materials derived from petrochemicals, which increase global carbon emissions during nuclear industry production.

In response to increasingly serious environmental problems, researchers are shifting focus from petrochemical products to biomass materials, which offer unique advantages such as eco-friendliness, abundant availability, and numerous reactive groups that can interact with target adsorbates [19, 20]. Consequently, biomass-based adsorbents are emerging as promising alternatives [21-23]. For example, Wang et al. synthesized amidoximated cellulose fibers by grafting polyacrylonitrile onto cellulose fibers for uranium extraction from seawater [24]. Ye and colleagues designed a novel uranium extraction material by grafting phosphoric acid and amidoxime groups onto skin collagen fibers [25]. Meanwhile, aerogel adsorption materials have attracted extensive attention due to their high porosity, large specific surface area, and exceptional adsorption performance for uranium extraction from aqueous solutions [26]. Chen et al. developed carbon-encapsulated zero-valent iron nanoparticles using konjac glucomannan-derived carbon aerogel for reduction-assisted uranium extraction [27]. Other biomass aerogels, such as feather keratin [28], chitosan [29], and microalgae aerogels [30], have also been reported for uranium recovery. However, despite using biomass materials, these adsorbents require complex multi-step chemical reactions and substantial petrochemical feedstocks. To pursue a greener, lower-carbon uranium extraction process, developing biomass aerogels through facile methods using predominantly biomass feedstocks is highly significant.

Tannin, a naturally occurring polyphenol extracted from plants, ranks among the most abundant biomass feedstocks in nature. Plant tannins contain abundant phenolic hydroxyls that can form five-membered rings to chelate multiple metal ions [31, 32], making polyphenol-based adsorbents promising for uranium extraction. However, tannins cannot be directly applied due to their high water solubility and must be embedded within a solid matrix. Chitosan is another biomass material with abundant active groups such as -OH and -NH₂ that has been used to prepare adsorbents for metal ions [33-35]. Since chitosan is water-soluble under acidic conditions, it also requires solidification. Based on these considerations, we believe that preparing biomass aerogels with a 3D network structure through facile cross-linking of tannins with chitosan shows promise

for uranium extraction from seawater, potentially meeting the green demands of uranium recovery processes.

Herein, we fabricated a biomass aerogel by facile cross-linking of chitosan and bayberry tannins with glutaraldehyde, maximizing the use of animal- and plant-derived biomass feedstocks. The resulting CS-BT aerogel features a 3D porous network structure with numerous reactive groups for uranium interaction, enabling uranium adsorption from aqueous solutions. We conducted adsorption experiments to evaluate its performance, performed kinetic and thermodynamic studies to propose appropriate models, and summarized the adsorption mechanism through multiple characterization methods. This work contributes to reducing chemical feedstock usage in uranium recovery, thereby decreasing carbon emissions and promoting environmental sustainability.

2.1 Materials

Uranyl nitrate (99%, AR) was provided by Hubei Chushengwei Chemical Co., Ltd. Glutaraldehyde (50 wt% aqueous solution, AR) and acetic acid (98%, AR) were purchased from Chengdu Kelong Chemical Co., Ltd. Chitosan (CS, BR, 85%, deacetylation degree 90%, Mw: ~100000) was obtained from Shanghai Macklin Biochemical Technology Co., Ltd. Bayberry tannin (BT, industrial grade, 66%) was sourced from Guangxi Baise Forest Chemical Factory. Arsenazo III was acquired from Shanghai Linen Technology Development Co., Ltd. Uranium standard solution was provided by Beijing Institute of Chemical Metallurgy for Nuclear Industry.

2.2 Preparation of CS-BT Aerogel

First, 3 g of BT was dissolved in 600 mL of deionized water in a 1 L beaker with stirring (300 r/min at 25 °C) for 2 h. Then, 3 g of CS and 6 mL of acetic acid were added and continuously stirred (300 r/min at 25 °C) for 2 h. After complete dissolution, 1.5 mL of glutaraldehyde was added, and the mixture was placed in a thermostatic shaking box (150 r/min at 45 °C) for 24 h. The prepared sol was transferred to a plastic petri dish, washed three times with deionized water to remove unreacted reagents, frozen at -80 °C for 12 h, and freeze-dried for 72 h to obtain the CS-BT composite aerogel.

2.3 Characterizations of CS-BT Aerogel

X-ray photoelectron spectroscopy (XPS) analysis was performed on a Thermo Fisher Kalpha spectrometer (USA) with monochromatic Al X-ray radiation. Fourier transform infrared (FT-IR) spectra were recorded from 4000 to 500 cm⁻¹ on a Thermo Fisher Nicolet iS50 spectrometer (USA) using potassium bromide pellets. Microscopic morphology was examined by field emission scanning electron microscopy (FE-SEM, Carl Zeiss Ultra55, Germany) at 200 kV. Nitrogen adsorption-desorption isotherms were measured at liquid nitrogen temperature

using a Micromeritics ASAP 2460 automatic surface area and porosity analyzer (USA).

2.4.1 Test Methods for Uranium Ion and BT

Uranium standard solution (1000 mg L^{-1}), dilute hydrochloric acid (0.1 mol L^{-1}), and arsenazo III solution (1 g L^{-1}) were used to prepare uranium test solutions with concentrations of 0, 1, 2, 3, 4, and 5 mg L^{-1} at various pH levels. Absorbance measurements were performed on a Molecular SpectraMax 190 enzyme marker (USA) to establish a uranium content standard curve. For BT analysis, solutions with concentrations of 0.02, 0.04, 0.06, 0.08, 0.1, 0.12, and 0.14 g L^{-1} were prepared and measured using a Hitachi UV-3900 spectrophotometer (Japan) from 200 to 400 nm to establish a BT content standard curve.

2.4.2 Effects of Solution pH and Adsorbent Mass on Adsorption

Uranyl nitrate solutions (20 mg L^{-1}) were prepared at pH values of 3.0, 4.0, 5.0, 6.0, 7.0, and 8.0. Each solution (50 mL) was combined with varying masses of aerogel (0.005, 0.01, 0.02, 0.03, 0.04, 0.06, and 0.08 g) in a conical flask and shaken in a thermostatic shaking box (150 r/min at 45°C) for 24 h. After filtration, U(VI) concentration was measured. All experiments were performed in triplicate to minimize error. Adsorption capacity (Ac , mg g^{-1}) and removal rate (R_r , %) were calculated using:

$$Ac = (C_0 - C) / m = (C_0 - C) / C_0 \quad (\text{Eq. 1})$$

$$(\text{Eq. 2})$$

where C_0 (mg L^{-1}) and C (mg L^{-1}) are initial and final U(VI) concentrations, V is solution volume (L), and m is adsorbent mass (g).

2.4.3 Cyclic Adsorption

Uranium-loaded CS-BT aerogel (20 mg) was placed in a 150 mL conical flask with 50 mL of 0.1 mol L^{-1} HCl solution for desorption at 25°C for 24 h. After desorption, the aerogel was washed three times with 100 mL distilled water and dried at 45°C . This cycle was repeated five times, with adsorption capacity calculated after each cycle.

2.4.4 Adsorption Kinetics

Kinetic data were obtained from 20 mg L^{-1} uranyl nitrate solution and fitted to pseudo-first-order and pseudo-second-order models:

$$\ln(q_0 - q) = \ln q_0 - K_1 t$$

$$t/q = 1/(K_2 q_0^2) + t/q_0 \quad (\text{Eq. 3}) \quad (\text{Eq. 4})$$

where K_1 and K_2 ($\text{g mg}^{-1} \text{ min}^{-1}$) are rate constants, t is adsorption time, and q_0 and q (mg g^{-1}) are adsorption capacities at time t and equilibrium, respectively.

Isothermal data were fitted to Langmuir and Freundlich models:

$$q = \frac{q_m C}{1 + K C} \quad (\text{Eq. 5}) \quad (\text{Eq. 6})$$

where C (mg L^{-1}) is equilibrium concentration, q is maximum adsorption capacity, K (L mg^{-1}) is the Langmuir constant, $K_f ((\text{mg g}^{-1}) \cdot (\text{L mg}^{-1})^{1/n})$ is the Freundlich constant, and n is an empirical constant.

2.4.5 Adsorption Thermodynamics

Isothermal data were obtained from uranyl nitrate solutions (20, 80, 140, 240, and 300 mg L^{-1}) across a temperature range and fitted using:

$$\ln q = -R \times 1$$

$$\Delta G = -RT \ln K$$

$$K = q \times K_f \quad (\text{Eq. 7}) \quad (\text{Eq. 8}) \quad (\text{Eq. 9})$$

where R is the ideal gas constant ($8.314 \times 10^{-3} \text{ kJ mol}^{-1} \text{ K}^{-1}$), T is temperature (K), K (mL g^{-1}) is the equilibrium constant, and ΔG , ΔH , and ΔS are Gibbs free energy, enthalpy, and entropy changes, respectively.

2.4.6 Effects of Coexisting Ions on Adsorption

A 20 mg L^{-1} uranyl nitrate solution containing coexisting ions (2 mg L^{-1} Cd^{2+} , Ni^{2+} , Fe^{3+} , and Pb^{2+}) was prepared. The effect of individual coexisting ions on adsorption was investigated over 2 h by measuring the adsorption rate.

3.1 Preparation and Characterizations of CS-BT Aerogel

The preparation schematic for CS-BT aerogel is shown in Fig. 1a [Figure 1: see original paper]. Initially, CS and BT dissolved completely under stirring to form a complex solution. Glutaraldehyde was then added as a chemical cross-linking agent, facilitating grafting and crosslinking between BT and CS. During this process, glutaraldehyde underwent a Schiff base reaction with $-\text{NH}_2$ groups on CS and a Mannich reaction with C6 and C8 reactive hydrogens on BT, forming covalent bonds that ensured successful functionalization of CS by BT [32].

FTIR spectra of CS-BT aerogel and CS/BT mixture are presented in Fig. 1b. The band at 3418 cm^{-1} corresponds to $-\text{NH}_2$ and $-\text{OH}$ stretching vibrations, while the band at 2917 cm^{-1} relates to C-H stretching. Benzene skeleton vibrations from BT appear at 1632 cm^{-1} and 1567 cm^{-1} . Compared with the CS/BT mixture, the CS-BT aerogel shows significantly enhanced peak intensity at 1632 cm^{-1} , which shifted to 1619 cm^{-1} , attributed to C=N formation via Schiff base reaction between $-\text{NH}_2$ and aldehyde groups [36-38]. Since C=N bonds are unstable under strongly acidic conditions, BT leaching experiments were conducted at different pH levels. As shown in Fig. 1c, BT leaching was less than 1% at $\text{pH} > 3.0$, confirming successful BT grafting to CS.

SEM characterization revealed the microscopic morphology of CS-BT aerogel. As shown in Fig. 2a [Figure 2: see original paper]-b, the interior is loose and porous with a three-dimensional network structure and pore sizes between ten and twenty microns, indicating lightweight properties, high porosity, and large specific surface area that enhance contact with metal ions and provide abundant adsorption sites. Nitrogen adsorption-desorption isotherms (Fig. 2c) exhibit typical type IV behavior, with gradual nitrogen adsorption at low pressure and sharp increases at high pressure, suggesting the presence of both micro- and mesopores [17]. The BET-specific surface area was $26.06 \text{ cm}^2 \text{ g}^{-1}$ with an average pore size of 10.33 nm, providing favorable conditions for metal ion diffusion.

3.2.1 Effects of Solution pH on Adsorption

Solution pH strongly influences metal ion chemical activity and adsorbent functional group ionization [39]. As shown in Fig. 3a [Figure 3: see original paper], the adsorption rate of 0.06 g CS-BT aerogel for U(VI) increased gradually as pH rose from 3.0 to 5.0. Lower adsorption efficiency at low pH resulted from protonation of surface amino groups, creating positive charges that increased electrostatic repulsion with U(VI). When pH increased from 5.0 to 8.0, adsorption rate reached equilibrium at approximately 99% maximum efficiency, likely because uranyl ions exist as positively charged $[(\text{UO})_2(\text{OH})]^{+}$ species [40] while amino group protonation decreases, weakening electrostatic repulsion. Simultaneously, the aerogel became deprotonated, and more carboxyl groups dissociated into negatively charged carboxylate ions, providing additional active sites for U(VI) adsorption. Since adsorption rate was highest at pH 5.0, subsequent experiments used this condition.

3.2.2 Effect of CS-BT Aerogel Mass on Adsorption

Adsorbent mass determines the number of active adsorption sites. As shown in Fig. 3b, adsorption rate increased significantly with aerogel mass, reaching 98% at 0.02 g CS-BT aerogel—higher than previously reported aerogels such as feather keratin (92%) [28], chitosan/amidoxime polyacrylonitrile (91%) [29], and microalgae (85%) [30]. Further mass increases did not improve adsorption rate, while adsorption capacity decreased because higher adsorbent mass at constant U(VI) concentration reduced the capacity per unit mass. Considering both efficiency and cost, 0.02 g CS-BT aerogel was selected for subsequent experiments. Cyclic adsorption tests (Fig. 3c) showed only slight capacity reduction from 44.6 to 37.3 mg g^{-1} over five cycles, demonstrating promising reusability.

3.3 Adsorption Kinetics

Adsorption kinetics effectively assess adsorbent efficiency. As shown in Fig. 4a [Figure 4: see original paper], adsorption rate and capacity improved significantly with time, exceeding 80% after 180 min and reaching equilibrium at approximately 600 min with 95.09% removal and 45.4 mg g^{-1} capacity. Initially, U(VI) rapidly bound to surface active sites, followed by diffusion into the aerogel interior driven by high concentration gradients. As active sites became saturated, the rate plateaued, reaching equilibrium.

Kinetic data were fitted to pseudo-first-order and pseudo-second-order models (Fig. 4b-c, Table 1). The pseudo-first-order model yielded $R^2 = 0.957$ with theoretical equilibrium capacity (38.034 mg g^{-1}) much lower than experimental capacity (48.311 mg g^{-1}). In contrast, the pseudo-second-order model showed excellent fit ($R^2 = 0.999$) with theoretical capacity (48.309 mg g^{-1}) closely matching experimental data, indicating chemical adsorption dominated by multilayer adsorption [41].

3.4 Adsorption Isotherms

Adsorption isotherms for U(VI) on CS-BT aerogel are shown in Fig. 5a [Figure 5: see original paper]. Capacity increased rapidly with U(VI) concentration, then gradually slowed. At low concentrations, abundant active sites enabled efficient adsorption; as concentration increased, site saturation reduced the concentration gradient, slowing capacity increment until equilibrium. Capacity increased with temperature, reaching 390.11 mg g^{-1} at 328.15 K, indicating high temperature favored adsorption.

Isotherm data were fitted to Langmuir and Freundlich models (Fig. 5b-c, Table 2). The Freundlich model showed higher R^2 values (0.991) than the Langmuir model (0.988) at 328.15 K, suggesting multilayer adsorption on a heterogeneous surface [44].

3.5 Adsorption Thermodynamics

Temperature effects on equilibrium adsorption capacity are shown in Fig. 6a [Figure 6: see original paper]. Capacity increased significantly with temperature, though the thermodynamic analysis revealed the adsorption process is exothermic. Fig. 6b shows the linear fit of $\ln K$ versus $1/T$, with thermodynamic parameters summarized in Table 3. Negative Gibbs free energies at all temperatures indicated spontaneous, thermodynamically feasible adsorption. Chelation between -OH, -NH, and C=O groups with U(VI) drove the spontaneous process. Negative enthalpy change confirmed exothermic adsorption,

while positive entropy change indicated increased disorder at the solid-liquid interface [46].

3.6 Effect of Coexisting Ions

Practical uranium-containing solutions inevitably contain various coexisting ions that compete for adsorption sites, affecting U(VI) removal efficiency [47]. We investigated U(VI) removal efficiency in solutions containing Cd^{2+} , Ni^{2+} , Fe^{3+} , and Pb^{2+} (2 mg L^{-1} each) over 120 min. As shown in Fig. 6c, Pb^{2+} , Cd^{2+} , Ni^{2+} , Fe^{3+} , and VO exhibited minimal inhibitory effects. Although VO typically competes strongly for U(VI) adsorption, its effect was negligible here [45], possibly due to low affinity for CS-BT aerogel -OH groups. These results demonstrate excellent selectivity, endowing CS-BT aerogel with great potential for practical uranium recovery from wastewater and seawater.

3.7 Adsorption Mechanism

SEM characterization of uranium-loaded CS-BT aerogel (Fig. 7a [Figure 7: see original paper]-b) revealed blocky solid particles in pores and walls, identified as uranium aggregates. The energy spectrum (Fig. 7c) showed a uranium peak, with average elemental analysis (Table 4) indicating $15.36 \pm 5.20\%$ uranium content, confirming successful U(VI) loading.

FTIR and XPS characterized structural changes after adsorption. A new peak at 898 cm^{-1} (Fig. 7d) corresponds to U(VI) characteristic absorption. The -OH and -NH peak at 3419 cm^{-1} broadened, and phenolic -OH shifted from 1407 to 1389 cm^{-1} , indicating strong U(VI) interaction. XPS total spectra (Fig. 7e) showed a new U 4f peak, while O 1s high-resolution spectra (Fig. 6f) revealed C=O and C-O peaks shifted to higher binding energies (531.37 to 532.2 eV and 532.76 to 533.05 eV , respectively) [48], confirming CS-BT aerogel-U(VI) interaction. These results indicate adsorption occurs primarily through chelation between -OH groups and U(VI) [49]. A proposed mechanism is illustrated in Fig. 8 [Figure 8: see original paper].

4. Conclusion

We fabricated a porous biomass CS-BT aerogel by crosslinking chitosan and bayberry tannin with glutaraldehyde. The cross-linked aerogel exhibits excellent aqueous stability, overcoming the water solubility limitations of chitosan and bayberry tannin. With multiple -OH groups for U(VI) chelation, the CS-BT aerogel demonstrates excellent adsorption performance (maximum capacity $\sim 140 \text{ mg g}^{-1}$ at 298.15 K , $\text{pH} = 5.0$). Kinetic studies reveal chemical adsorption

dominated by multilayer adsorption, while thermodynamic analysis shows the process is spontaneous and exothermic. These results demonstrate CS-BT aerogel as a promising material for uranium extraction from natural seawater and nuclear industry wastewater. Unlike traditional adsorbents, this aerogel uses predominantly biomass feedstocks, reducing carbon emissions and promoting sustainability in the nuclear industry.

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Author Contributions

All authors contributed to study conception and design. Material preparation, data collection, and analysis were performed by Guiqiang He, Jinfan Ou, Aixia Lu, and Yanxia Wei. Guiqiang He wrote the first draft, and all authors commented on previous versions and approved the final manuscript.

Data Availability Statement

The data supporting this study are openly available at Science <https://cstr.cn/31253.11.sciencedb.28427> and <https://doi.org/10.57760/sciencedb.28427>.

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