

Preparation of porous magnesium oxide foam and study on its enrichment of uranium

Yunsheng Wang, Yuantao Chen*, Chen Liu, Fang Yu

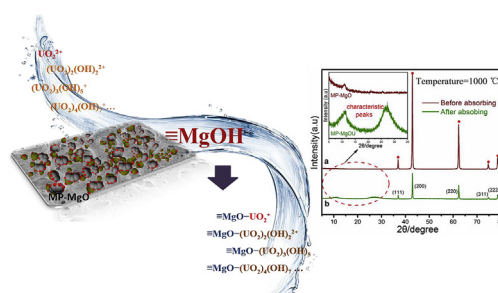
School of Chemistry and Chemical Engineering, Qinghai Normal University, Xining, Qinghai, 810008, PR China



HIGHLIGHTS

- Porous magnesium oxide was prepared by in situ carbonized.
- When the initial pH was from 3.0 to 4.5, the adsorption capacity was more than 2500 mg g⁻¹.
- The time of adsorption equilibrium was shorter than that of pH reach a relatively stable state.
- The adsorption process was spontaneous and exothermic.

GRAPHICAL ABSTRACT



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ABSTRACT

Herein, we prepared porous magnesium oxide (MP-MgO) by in situ carbonization and using Mg(NO₃)₂ as precursor along with P123 as soft template. The BET surface area, and total pore volume of MP-MgO were found to be 14.76 m² g⁻¹, 0.15 cm³ g⁻¹, respectively. The adsorption behavior of U (VI) by the porous MgO was studied by static adsorption experiments, and also the effects of adsorption time, pH of wastewater and U (VI) concentration were discussed. The results indicated that the optimum pH for MP-MgO was 3.0–4.5, the removal of uranium in this case was mainly due to surface complexation. Through the study, we found that the adsorption capacity of MP-MgO for uranium was more than 2500 mg g⁻¹, the adsorption equilibrium time was about 150 min. Moreover, the kinetic study showed that the process followed the pseudo-first-order model, and the adsorption process was spontaneous and endothermic.

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1. Introduction

Uranium is a natural radioactive element, and has been widely used in various fields. Militarily, uranium is mainly used to produce nuclear fuel and manufacture nuclear weapons. In the field of energy, it has been mainly used in nuclear power plants. Compared with hydropower and thermal power, nuclear power has the

advantages of low cost, safety and environmental friendliness. Therefore, nuclear power is developing all over the world, especially in developed countries. Now, uranium is obtained mainly by in situ uranium mining, which has a large amount of wastewater [1–4].

There is a series of nuclear processes that may cause radioactive contamination, such as nuclear power plant spent fuel, uranium mining, nuclear tests, nuclear power plant accidents and various types of nuclear facilities are likely to discharge wastewater containing radionuclides. In some cases, it may get concentrated because of evaporation of active liquid wastes [5,6]. High levels of

* Corresponding author.

E-mail address: chenyt@qhnu.edu.cn (Y. Chen).

uranium in water may increase the risk of cancer and kidney damage [7]. Therefore, the removal of uranium (VI) ion from waters is conducive to reducing the uranium contamination, maintaining the natural balance, and promoting the recovery of uranium resources [8,9].

Traditional methods for the removal of radionuclides include ion exchange, surface precipitation, solidification, membrane filtration, and sorption [10–17]. Among these methods, sorption technique has been widely utilized. It has been proved to be an effective and simple separation process for removing heavy metals or enriching rare metals [18]. Regularly morphological materials with high surface areas and porous structures have potential applications in physical and chemical fields. Magnesium oxide with large specific surface area is a promising sorbent for chemisorption and destructive adsorption of various pollutants. However, most of the MgO materials prepared by conventional routes such as thermal-decomposition of magnesium hydroxide or carbonate are characterized with a small surface area. Many other methods like chemical gas phase deposition, hydrothermal, combustion aerosol synthesis [19–22] etc. have been reported for the synthesis of MgO. However, these methods appear to be rather expensive for large-scale applications.

In our previous work, urchin-like MgO have been synthesized by using urea as precipitating agent [23]. Studies have shown that magnesium oxide has excellent adsorption properties for Uranium (VI). In order to further increase the adsorption capacity of magnesium oxide, in this work, we prepared porous magnesium oxide. And, we try a route of in situ carbonization to fabricate pore in magnesia by using $Mg(NO_3)_2$ as precursor along with P123 as soft template. Due to space-limited growth of MgO crystallites within and/or around the organic templates, many pores and defects are generated in the foam-like magnesia so it will possess both large surface area and high basic sites density, exhibiting high capacity adsorption of U (VI).

2. Materials and method

2.1. Materials

Pluronic P123 ($EO_{20}PO_{70}EO_{20}$, $M_n = 5800$, Energy Chemical, Shanghai, China). Magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$), and ethanol (C_2H_5OH) of AR grade were obtained from Tianjin Kemiou Chemical Reagent Co., China. The uranyl nitrate hexahydrate ($UO_2(NO_3)_2 \cdot 6H_2O$, HuBei ChuShengWei Chemistry Co., Ltd, China) was dissolved in deionized water as the source of uranium (VI). Deionized (DI) water (Shandong Xinrui Analytical Instruments Co., Ltd, China) was used throughout the experiment.

2.2. Preparation of MP-MgO

The porous magnesium oxide was synthesized by a sol-gel route with block copolymer (P123) as soft template, by adapting a process developed by Han et al. [24]. for mesoporous magnesium-alumina oxides composites. As a typical synthesis, a solution was prepared by dissolving 10.0 g of P123 in 200.0 mL of ethanol, and the mixture vigorously stirred for 60 min at room temperature. Then 0.1 mol of $Mg(NO_3)_2 \cdot 6H_2O$ and 20 mL of 69 wt% nitric acid were added with vigorous stirring. After stirring for 10 h, the mixture was transferred into a 500 mL beaker which was put in an oven at 40 °C to undergo solvent evaporation. After 72 h, a white solid gel was obtained.

Then the obtained sample was heated to 600 °C in a furnace at a slow ramp rate of 2 °C min^{-1} and kept at 600 °C for 3 h in air. To obtain the crystalline products, further high temperature calcination at 1000 °C was carried out in air for 2 h using a rapid ramp rate

of 10 °C min^{-1} . After naturally cooling to room temperature, the porous magnesium oxide was collected and kept in an oven at 60 °C. And the calcined samples before and after U (VI) adsorption were designated as MP-MgO, MP-MgOU respectively.

2.3. Characterization

X-ray diffraction (XRD) patterns of the samples were recorded using a Shimadzu XRD-6000. Fourier transform infrared (FTIR) spectra were taken using a FTIR spectrometer (Nicolet iS50 FT-IR, Thermo Scientific America). Surface morphology of the samples was investigated by scanning electron microscopy (SEM, Hitachi SU8010, Japan). X-ray photoelectron spectroscopy (XPS) of the fresh and used adsorbents were performed by using a ULVAC-PHI PHI Quantera SXM. The specific surface area and pore size were calculated using the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) method (Quantachrome Instruments, America).

2.4. Adsorption experiments

Different concentrations of U (VI) aqueous solutions were prepared using $UO_2(NO_3)_2 \cdot 6H_2O$ as the sources of uranium. The pH value of the individual solutions was adjusted by adding small volumes of HNO_3 and/or $NaOH$. To determine the effect of pH on the U (VI) sorption capacity of MP-MgO, experiments were carried out using solutions of initial pH values ranging from 2.5 to 11, 10 mg of MP-MgO was added into 100 mL U (VI) solutions ($300 mg L^{-1}$) and then the solutions were shaken (180 rpm) for 12 h at 25 °C.

For the adsorption kinetic study, the pH of the each solution was adjusted to 4, MP-MgO (10 mg) was added into U (VI) solutions (100 mL, $300 mg L^{-1}$). This process was repeated for sampling times 10 min, 30 min, 120 min and 4 h, and then the solid was separated from aqueous solution by centrifugation at 8000 rpm for 5 min and then the supernatant was separated by using 0.45- μm polyether-sulfone membrane filters. The uranium (VI) solution was measured by inductively coupled plasma atomic emission spectrometer analysis (ICP, Shimadzu Corp., ICPE-9000). Particularly, since uranyl ions were easily complexed with carbonate ions, we had chosen to do adsorption experiments in iodine flasks rather than in erlenmeyer flasks to avoid the effects of large amounts of carbonate on adsorption experiments. The adsorption capacity at equilibrium (C_s , $mg g^{-1}$) could be calculated by the mass balance equation [25–28]:

$$C_s = (C_0 - C_e)V/m \quad (1)$$

Herein, C_0 ($mg L^{-1}$) is the initial concentration of U (VI), and C_e ($mg L^{-1}$) is the final concentrations of U (VI) in aqueous solution after sorption equilibration ($mg L^{-1}$); V (L) is the solution volume, m (g) denotes the mass of the MP-MgO.

3. Results and discussion

3.1. Morphology and characterization

As shown in Fig. 1, porous magnesium oxide can keep its monolith morphology to a certain extent, and it is easily to be fragmented into particles having a size of 100–200 μm . MP-MgO sample has the foam-like morphology with macropores of different pore diameters, which is well consistent with the result of BET analysis. As revealed in the SEM images with higher magnification, MP-MgO sample possesses the three-dimensional (3D) interconnected porous architecture (Fig. 1a and b and Fig. 2), and the sample has rough surface with specific cellular structure that

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