Crown ether-type organic composite adsorbents embedded in high-porous silica beads for simultaneous recovery of lithium and uranium in seawater

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Fig. 1

: Li (ref. 7) □: U 20 4000 (Li) P 10 ٢ ф I 2000 Ţ I  $\square$ Ð  $\square$  $\oplus$ Φ Φ 0 0 ⊕ B A Е F С D G

 $\frac{6}{7}$ 

1

 $\frac{2}{3}$ 

4

 $\mathbf{5}$ 





Fig. 3

 $\mathbf{2}$ 



 $\frac{2}{3}$ 

4



Fig. 4

3





 $\frac{2}{3}$ 



Fig. 6





 $\frac{6}{7}$ 

1

 $\frac{2}{3}$ 

4

 $\mathbf{5}$ 

- 10
- 1-
- 11
- 12











 $\mathbf{7}$ 

1  $\mathbf{2}$ Figure captions: 3 Fia. 1 Structural formulas, cavity sizes, and values of hydrophobicity of synthesized 4  $\mathbf{5}$ crown ether adsorbents. 6  $\overline{7}$ Fig. 2 Plots of  $K_d$  values vs. seven kinds of crown ether adsorbents. Temp. = room temp. 8 Particle size = 100 - 250 mesh, adsorbent = 500 mg, solution volume = 10 mL. 9 A: BC12, B: DBC14, C: BC15, D: BC18, E: DBC18, F: DBC21, G: DBC22. 10 11 12Fia. 3 Plots of  $K_d$  values vs. various types of adsorbents. Temp. = room temp. Adsorbent 13= 500 mg, solution volume = 10 mL. A: WA10, B: WA20, C: WA30, PA308, E: 14PA312, F: PA316, G: WK10, H: WK40L, I: PK208, J: PK216, K: PK220, L: PK228, 15M: PK228L, N: SK112L, O: BT-AG, P: CR10, Q: BC15, and R: BC18. The data 16of Q and R were referred from our previous work [7]. 1718 19Fig. 4 20Distribution diagram of U(VI) species as a function of pH at 298 K. The stability constants between U(VI) $O_2^{2+}$  and OH<sup>-</sup>, Cl<sup>-</sup>, CO<sub>3</sub><sup>2-</sup>, and CO<sub>2</sub>(g) [17]. (a): UO<sub>2</sub><sup>2+</sup>, 21(b):  $UO_2CI^+$ , (c):  $(UO_2)_2(OH)_2^{2+}$ , (d):  $(UO_2)_{11}(CO_3)_6(OH)_{12}^{2-}$ , (e):  $(UO_2)_2CO_3(OH)_{3^-}$ , (f):  $(UO_2)_2(OH)^{3+}$ , (g):  $UO_2(CO_3)_3^{4-}$ , (h):  $(UO_2)_3(OH)_4^{2+}$ , (i):  $(UO_2)_4(OH)_6^{2+}$ , (j): 2223(UO<sub>2</sub>)<sub>3</sub>(OH)<sup>5+</sup>. With respect to the fraction of other species, UO<sub>2</sub>Cl<sub>2</sub>, UO<sub>2</sub>OH<sup>+</sup>, 24UO<sub>2</sub>(OH)<sub>3</sub><sup>-</sup>, (UO<sub>2</sub>)<sub>3</sub>(OH)<sub>7</sub><sup>-</sup>, (UO<sub>2</sub>)<sub>4</sub>(OH)<sub>3</sub><sup>5+</sup>, UO<sub>2</sub>CO<sub>3</sub>, UO<sub>2</sub>(CO<sub>3</sub>)<sub>2</sub><sup>2-</sup>, (UO<sub>2</sub>)<sub>3</sub>(CO<sub>3</sub>)<sub>6</sub><sup>6-</sup> 25are excluded because of their small fraction of U(VI) species. Ionic strength = 0.5. 26 $[CO_3^{2-}]_T = 2.4 \times 10^{-3} \text{ M}$  [6].  $[CI_T]_T = 6.1 \times 10^{-1} \text{ M}$  [6].  $[U]_T = 4.2 \times 10^{-7} \text{ M}$ .  $[CO_2(g)]_T$ 27= 8.9 × 10<sup>-3</sup> M [40]. 2829Fig. 5 30 Plots of  $K_d$  values vs. pH values. Temp. = room temp. Particle size = 100 - 250 31mesh, adsorbent = 500 mg, solution volume = 10 mL.  $\circ$ : Li / BC15,  $\triangle$ : U / BC15, 3233 □: Li / BC18, ∇: U / BC18. 3435Fig. 6 Plots of  $K_d$  values vs. CFs. Temp. = room temp. Particle size = 100 - 250 mesh, 36 adsorbent = 500 mg. Solution volume = 10 mL.  $[Li]_i = (8.0 \pm 0.9) \times 10^{-5} M. [U]_i =$ 37(8.2 ± 2.5) × 10<sup>-7</sup> M. 3839 40 Fig. 7 Plots of ln K<sub>d</sub> values vs. 1 / T values. Temp. = 278 - 333 K. Particle size = 100 -41 250 mesh, adsorbent = 0.50 g for U ion and 1.0 g for Li ion, solution volume = 10 42mL.○•: Li / BC15, □: U / BC15, Δ: U / BC18, ∇▼: Li / BC18. 434445Fig. 8 Chromatogram of Li and U ions using BC15 and BC18 adsorbents at 298 K. 46 Particle size = 100 - 250 mesh. : Li for BC15, : Li for BC18, : pH for Li with 47BC15, ◆: pH for Li with BC18, △: U for BC15, ⊽: U for BC18, ×: pH for U with 48

49 BC15, +: pH for U with BC18.

- $\mathbf{2}$ Fig. 9
- SEM images of BC15 and BC18 adsorbents. (A-1) BC15 adsorbent before test, 3
- 4
- M = 50× (A-2) BC15 adsorbent before test, M = 2000× (A-3) BC15 adsorbent after test, M = 50× (A-4) BC15 adsorbent after test, M = 2000× (B-1) BC18  $\mathbf{5}$
- adsorbent before test, M =  $50 \times (B-2)$  BC18 adsorbent before test, M =  $2000 \times (B-2)$ 6
- 3) BC18 adsorbent after test, M = 50× (B-4) BC18 adsorbent after test, M = 2000×.  $\overline{7}$
- 8
- 9