

Fig. 2 (color online) The gathering of MoO_3 and TeO_2 .

References

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2 - 33 Synthesis of Amidoximated Silica and Its Application for Uranium Adsorption from Saline Lake Brine

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Sorption is an effective method for uranium recovery from saline lake brines for the benefits of simplicity, rapidity and easy recycling. To make uranium extraction feasible, solid adsorbents which are effective under saline lake brine conditions should be developed. In present work, MCM-41 type silica adsorbents with different content of amidoximated groups were prepared via co-condensation method according to the following steps: 1 g of CTAB was dissolved in 135 mL of deionized water and then 102 mL of ammonia was added to the solution. After stirring for 0.5 h, CTES and 5 mL of TEOS were mixed and added to the homogeneous solution. The mixture was stirred for 2 h at room temperature. The powder was recovered by filtration, purified with plenty of 10/90 HCl/C₂H₅OH and dried at 60 °C. The specific molar ratio of CTES/TEOS was obtained by controlling the quantity of CTES. The obtained silica grafted with nitrile(-CN) named CMCM-X (X = 0, 0.2, 0.4 or 0.6) were treated with 3 g NH₂OH·HCl in 100 mL 50/50 H₂O/CH₃OH solution for 72 h at 80 °C. Sodium carbonate was used to adjust the solution pH to neutral. The final products, denoted as AMCM-X, were washed with deionized water and dried at 60 °C.

Fig. 1 showed the FTIR spectra of CMCM-0, CMCM-0.4, AMCM-0.4 and AMCM-0.6. The large broad band at 3 427 cm⁻¹ and the band at 1 630 cm⁻¹ were attributed to the stretching vibration of Si–OH group and the adsorbed water. The bands at 1 085, 800 and 470 cm⁻¹ were related to the stretching vibration, typical symmetric and bending vibrations Si–O-Si in the silica, respectively. The stretching band of the free silanol groups at 960 cm⁻¹



was also observed. Furthermore, the undesirable faint peaks at 2 927 and 2 856 cm⁻¹, which were attributed to the characteristic asymmetric and symmetric vibration of CH₂ bands, demonstrated the trace residue of CTAB on CMCM-0. In comparison with the spectrum of CMCM-0, the characteristic stretching vibration absorption band of cyano groups at 2 253 cm⁻¹was observed in the spectrum of CMCM-0.4. After amidoximation, the band of cyano groups extremely weakened. The vibration absorption of C=N (expected at 1 660 cm⁻¹) and N–OH (expected at 944 cm⁻¹) in the amidoxime group on AMCM-0.4 could not be confirmed directly by FTIR spectrum due to overlap of the strong O–H band and the free silanol groups, respectively. In fact, the broad peak at 1 651 cm⁻¹ in the spectrum

of AMCM-0.4 was the combination of the peaks of O-H and C=N groups. These confirm that cyano groups were converted to amidoxime groups by treatment with hydroxylamine. The absorption band at 2 253 cm⁻¹ in the spectrum of AMCM-0.6 illustrate that only a portion of the cyano groups on CMCM-0.6 were converted to amidoxime groups in spite of sufficient of hydroxylamine and adequate reaction time. CTES could not disperse sufficiently in TEOS during preparation of CMCM-0.6 and the cyano groups on CMCM-0.6 were clustered resulting in the partial amidoximation of the cyano groups. The above information confirmed the successful synthesis of amidoximated silica.

The effect of solution pH on uranium sorption was discussed in detail. Sorption kinetic and isotherms were investigated. The results indicated that chemisorption was dominated in the uranium sorption and the uranium formed a monolayer on the adsorbent surface. XPS spectra of sorbent and uranium loaded sorbent indicated that the sorption was attributed to the interaction between N in amidoxime groups and uranium. Both N atoms of -C=N-OH and $-C-NH_2$ interacted with uranium as electron donors during the sorption.



Fig. 1 (color online) Uranium sorption from saline lake brine samples by AMCM-0.4 (

Amidoximated silica could efficiently absorb the naturally occurring uranium in the saline lake brine. Fig.2 shows the uranium adsorption capacity from the saline lake brine by AMCM-0.4. The amount of uranium adsorbed at 1 and 7 d were comparable demonstrated the adsorption reached equilibrium within 1 d. Such fast adsorption kinetics was essential to uranium extraction from saline lake. Great amount of positive ions in saline lake brine compete with uranium for adsorption sites may be the dominant factor for low sorption capacity. The fast adsorption kinetic and acceptable uranium sorption capacity demonstrated that AMCM-X could be a promising adsorbent for uranium extraction from saline lake brine.

Sample	Uranium concentration/($\mu g/L$)	pH
YT-1	177 ± 3	7.0
YT-2	520 ± 20	6.1
YT-3	$450{\pm}10$	6.0

Table 1 Uranium concentration and pH of saline lake brine.