Influence of mechanochemical and microwave modification on ion-exchange properties of tin dioxide with respect to uranyl ions

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Abstract

Wet gel and dried xerogel of precipitated tin oxy-hydroxide were subjected to mechanochemical and microwave treatments (MChT and MWT, respectively). The prepared samples were studied using DTA-TG, XRD, FTIR, nitrogen adsorption–desorption. MChT and MWT cause improvement of crystal structure, removal of structural OH-groups and increase mesopores diameter as well as form the meso-macroporous structure. As a result, drastic increase of uranyl-ions sorption for all modified samples is observed. Sorption centers can be divided into three groups: selective cation-exchange, non-selective cation-exchange and anion-exchange.

Keywords SnO_2 · Mechanochemical and microwave treatments · Ion-exchange properties

1 Introduction

Tin dioxide SnO_2 is a versatile material, which is in demand in many industries, namely: the production of gas sensors, catalysis and photocatalysis, and others (Miller et al. 2006; Adnan et al. 2010; Teterycz et al. 2011). At the same time, it is promising as an ion exchanger (Petro et al. 1990). It is used for the removal of cations (Co (II), Th (IV), Cr (VI), U (VI) and others) and anions (F (I), I (I), IO_3 (I), Sb (OH) (VI)) (Misak et al. 1992; Nilchi et al. 2013; White and Rautiu 1997). The crystal and porous structure as well as design of surface are the most important parameters ensuring its effective application (Naushad 2009; Coker 2000). There are many methods for synthesis of tin dioxide, among others sol–gel and precipitation (Ivanenko et al. 1999; Zhang and Liu 1999; Nilchi et al. 2013; Gavrilov 2000). These techniques allow to prepare tin dioxide of a large specific surface area. However, a large content of micropores limits accessibility of a large part of the surface for hydrated ions. It is possible to obtain the mesoporous structure, which is

necessary for sorption processes, by means of mechanochemical (MChT) and microwave (MWT) treatment. These methods allow to regulate effectively physical–chemical parameters which were demonstrated for many oxides and hydroxides (Buyanov et al. 2009; Sepelak et al. 2012; Tompsett et al. 2006) including SnO_2 (Khalameida et al. 2017). However, the ion-exchange properties of the oxides, including SnO_2 modified in this way, have not been previously studied. Thus, the aim of this paper is investigation of the effects of mechanochemical and microwave modification on the cation- and anion-exchange properties of tin dioxide in relation to uranyl ions and relationship of sorption properties with physical–chemical characteristics of SnO_2 samples.

2 Experimental

2.1 Preparation and modification of samples

Initial tin dioxide was prepared by heterogeneous precipitation. The tin tetrachloride $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ was used as the starting material: into 600 mL of its 0.5 M aqueous solution was added under stirring 10% aqueous solution ammonia hydroxide until the pH was 4.0. After aging in the mother liquor (25 h), the obtained precipitate was washed with distilled water to remove Cl^- ions and filtered. One part of the

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precipitate was used as a wet gel, and the other was dried at 20 °C for xerogel preparation.

Wet gel and dried xerogel were subjected to mechanochemical and microwave treatments (MChT and MWT), respectively. Mechanochemical treatment was carried out using a planetary ball mill “Pulverisette-7” (“Fritsch”, Germany) in air and water for 0.5 h at 300 and 500 rpm. The high-pressure reactor “NANO 2000” (“Plazmotronika”, Poland) was used for microwave treatment. MWT carried out at 175 °C for 2 h, and at 235 °C for 1 h.

2.2 Characterization of initial and modified samples

Thermal analysis was performed on an apparatus Derivatograph-C (F.Paulik, J.Paulik, L.Erdey) under the following conditions: the heating rate 10°/min, temperature range of 20–700 °C. FTIR spectra were recorded using the spectrophotometer “Spectrum-One” (Perkin-Elmer, USA) in the range of 4000–400 cm^{−1} in the reflection mode. The adsorption–desorption isotherms of nitrogen were recorded using the analyzer ASAP 2405N (“Micromeritics Instrument Corp”). The surface areas S , sorption pore volume V_s , micropores volume V_{mi} , mesopores volume V_{me} were calculated from the isotherms using the BET, t - and the BJH methods. The total pore volume V_Σ was determined by ethanol impregnation of the samples granules dried at 150 °C. The volume of macropores V_{ma} was calculated as the difference between V_Σ and V_s . The mesopores diameter d_{me} was calculated from the pore size distribution (PSD) curves constructed using the desorption branches of isotherms.

2.2.1 Sorption experiment

The initial and modified samples were tested as an ion-exchanger of uranyl-ions under different conditions. Thus, in order to study the cation exchange properties, sorption of ions U (VI) was performed in the most favorable conditions, namely at pH 5.4 in the absence of background. The studies were also carried out under different conditions (pH value, presence of background and other cations):

- (i) sorption of U (VI) ions under optimal pH 5.4 in the absence of background;
- (ii) pH 5.4 but in the presence of NaCl;
- (iii) pH 5.4 in the absence of background but in the presence of Cs(I) and Sr(II) cations which often accompany the U (VI) ions when liquid radioactive wastes are cleaned;
- (iv) sorption of U (VI) ions at pH 3.0, which corresponds to acid mine waters and acid radioactive wastes, in the absence of background but in the presence of Cs⁺ and Sr²⁺ cations; The third and fourth experimental solutions ((iii) and (iv)) contained a mixture of U (VI), Cs

(I) and Sr (II) ions in the quantities of 1:0.2:0.2 mEq/L, respectively.

- (v) the pH value was 8.2 during sorption of ions U (VI) on the background of 0.1 M NaHCO₃; these conditions corresponds to composition of “block” waters of the Chornobyl Nuclear Power Plant (ChNPP) and waste in storage of tailings (Kornilovych et al. 2018); under these conditions U (VI) is present in the solution as anionic carbonate complexes (Odintsov et al. 2009).

All studies were carried out under static conditions, the solid:liquid weight ratio was 1:2000. The uranium concentration in solution was determined using the KFK-3 photometer (Russia). The method for determining U (VI) is based on formation of a stable coloured complex with arsenazo III at pH 1.0–3.0 by UO₂²⁺ ion (Upor et al. 1985). Using the obtained data, the sorption capacity A and the distribution coefficient K_d were calculated according to the formulas 1 and 2:

$$A = \frac{(C_0 - C_{\text{equilibrium}}) \times V}{135 \times m_{\text{sorbent}}} \quad (1)$$

$$K_d = \frac{A}{C_{\text{equilibrium}}}, \quad (2)$$

where C_0 is the initial concentration of ions U (VI); $C_{\text{equilibrium}}$ the equilibrium concentration of ions U (VI); V the volume of solution of ions U (VI); m_{sorbent} the weight of sorbent; A is the sorption capacity.

3 Results and discussion

Figure 1 and Table 1 present the results of DTA-TG for the initial and modified samples. The largest mass loss (Δm) is detected from 20 to 200 °C for all samples. This is associated with desorption of physically adsorbed water. At the same time Δm in the range 200–700 °C corresponds to removal of OH groups. This parameter was calculated for a dry weight - without physically adsorbed water. Thus, the initial xerogel corresponds to the composition of tin oxy-hydroxide (Petro et al. 1990; White and Rautiu 1997). The mass loss from 200 to 700 °C, when structural OH-groups are removed, is 5.73%. This corresponds to ratio OH/Sn 1.15. MChT and MWT result in partial removal of structural OH groups and partial transformation of tin oxy-hydroxide into SnO₂ (Fig. 1, curve b, c). The ratio of the number of structural OH-groups to Sn practically does not change after MChT of xerogel but sharply decreases after MWT of gel (Table 1). The latter is a consequence of the improvement of the crystal structure SnO₂ under hydrothermal conditions

Fig. 1 TG curves for tin oxy-hydroxide: initial xerogel (a), gels after MChT at 300 rpm (b) and after MWT at 235 °C (c)

Table 1 The data of DTA-TG for initial and modified samples

Sample	$\Delta m_{\text{exper } 200-700^\circ\text{C}}$ (%)	The number of structural OH groups/Sn
Tin oxyhydroxide		
Initial	5.73	1.15
MChT xerogel air	5.64	1.13
MChT xerogel H ₂ O	5.61	1.12
MChT gel 300 rpm	7.25	1.45
MWT gel 235 °C	2.63	0.53

(Khalameida et al. 2017). This ratio increases to 1.45 only for sample milled in the form of wet gel (Fig. 1, curve b). These data correlate with the results of FTIR-spectroscopy.

The FTIR spectra registered for all samples in the range 4000–400 cm^{-1} (Fig. 2) show several absorption bands (a.b.) attributed to the surface O–H vibrations, namely: the stretching vibrations at 3000–3500 cm^{-1} and the bonding vibrations at 1235 cm^{-1} . The intensity of a.b. is decreased for MChT and MWT. Besides the absorption bands assigned to O–Sn–O, Sn–O–Sn and Sn–O(H) vibrations: at 660, 611 and 575 cm^{-1} , respectively, can be identified (Zhang and Liu 1999; Orel et al. 1994).

The initial sample is characterized by high values of specific surface area (S) and a large content of micropores and the smallest mesopores (with the diameter 2.2 nm) (Table 2). This is evidenced by the isotherms of nitrogen adsorption-desorption which are close to type I according to the IUPAC classification (Fig. 3). Moreover, this sample has a wide pore size distribution without maximum (inset to Fig. 3). During MChT and MWT, formation of a meso-macroporous or more uniform mesoporous structure takes place, which is determined by the processing conditions (Fig. 3). The

Fig. 2 FTIR-spectra of samples of the initial tin oxy-hydroxide as well as those after MChT of xerogel in water at 300 rpm, in air at 300 rpm and MChT of gel at 300 rpm

porous structure parameters of all samples are given in Table 2. There is some reduction in the specific surface area for the modified samples. However, the total pore volume (V_{Σ}) and the volume of mesopores (V_{me}) increase significantly. As a result, diameter of mesopores (d_{me}) increases almost twice (to 4.1–4.3 nm) (inset to Fig. 3; Table 2). As can be seen, the PSD curves with the maxima in the region of larger mesopores were obtained for the modified samples. Furthermore, formation of the secondary porosity presented by meso- and macropores is a peculiarity of milling in water. The macropores volume (V_{ma}) for this sample is about 60% of the total pore volume (V_{Σ}) (Table 2). On the whole, the porous structure of modified samples becomes more open and thus more accessible to various ionic species of U (VI).

Combining practical and scientific interests, sorption properties of the initial and modified samples were studied for the ions of stable isotopes U (VI). Its radioactive isotopes are present in the “block” waters of the ChNPP (Odintsov et al. 2009) and interfere with their processing according to the conventional technological scheme used at the ChNPP to deactivate other liquid radioactive wastes. It should be noted that uranium belongs to the group of so-called naturally occurring radioactive materials (NORM) that can be found in natural water (Panias et al. 2005; Timoshenko et al. 2009), as well as in mine waters.

U (VI) has features that make it difficult to extract. In particular, this is the variety of forms in solution (Fig. 4). Up to pH 5.0 U (VI) is present in the solution mainly in the form of uranyl ions (UO_2^{2+}), at higher pH - in the form of hydroxides and anionic carbonate complexes, the vast majority of which are polymeric (Kobets et al. 2012).

Table 3 summarizes the results of investigations of sorption properties for the initial and modified samples of tin oxy-hydroxide under different conditions. In order to

Table 2 Porous structure parameters of tin oxy-hydroxide samples

Modification conditions	S (m ² /g)	V _Σ (cm ³ /g)	V _s (cm ³ /g)	V _{mi} (cm ³ /g)	V _{me} (cm ³ /g)	V _{ma} (cm ³ /g)	d _{me} (nm)
Tin oxy-hydroxide							
Initial xerogel	178	0.05	0.05	0.03	0.02	0	2.3
MChT xerogel air 300 rpm	138	0.08	0.08	0.04	0.03	0	4.1
MChT xerogel H ₂ O 300 rpm	163	0.33	0.1	0.04	0.05	0.23	4.3
MChT gel 300 rpm	180	0.17	0.09	0.04	0.04	0.08	3.4
MChT gel 500 rpm	98	0.08	0.08	0.02	0.04	0	3.4
MWT xerogel 2 h 175 °C	92	0.14	0.14	0	0.11	0	4.7
MWT gel 1 h 235 °C	156	0.25	0.25	0	0.25	0	4.3

Fig. 3 Isotherms of nitrogen adsorption–desorption and curves of PSD for the samples of tin oxy-hydroxide **Fig. 4** The forms of uranyl ions depending on pH of the solution

establish the maximum sorption capacities of the samples under study, the sorption of U (VI) ions was conducted in the most favorable conditions at pH 5.4 and in the absence of the background. As can be seen, all types of treatment increase considerably the sorption capacity of tin oxy-hydroxide accessible to the ions of U (VI) to the level of

the most common cation exchange resins (Özeroğlu and Keçeli 2009). The greatest increase was observed for the sample after the MChT of gel, the sorption capacity of which increases almost three times. Xerogels milled in water and air, as well as the gel after MWT show magnitudes of sorption capacities for U (VI) ions which are close to each other (Table 3). Favorable effect of MChT is associated with formation of a mesoporous structure with larger pore size and more uniform PSD during modification which is more accessible to various ionic forms of U (VI), whose size is within 0.4–1.0 nm (Persson 2010; Marcus 1988).

The influence of MChT on the kinetics of ions U (VI) sorption can be estimated from the corresponding kinetic curves (Fig. 5). These data are fully consistent with the consideration of the sorption capacities obtained related to the U (VI) ions (Table 3). In particular, all samples are divided into three groups:

- (1) The initial sample is characterized by the lowest capacity and fast sorption kinetics. Since the first experimental point was taken after 850 h, the moment of equilibrium could have occurred earlier. Accessible active centers for the existing U (VI) forms are filled relatively quickly under these conditions.
- (2) After MChT of gel, the sample is characterized by the highest capacity and fast sorption kinetics. Due to the changes in the porous structure, MChT makes the U (VI) ions accessible to active centers that were inaccessible in the initial sample.
- (3) There are two areas on the kinetic curves of all other samples: quick filling of easily accessible active centers (first 1000 h) and slow diffusion of ions U (VI) into the depth of the sorbent grain. The sorption capacity obtained for this group of samples tends to the values typical for the sample after MChT of gel. It can be assumed that MChT of xerogel does not create porous structure open for diffusion of all U (VI) forms to active centers as it is observed for the sample after MChT of gel which is evidenced in the case of xerogel after MChT in water. If for the other two samples in this

Table 3 Sorption properties of initial and modified samples of tin oxy-hydroxide

Exp. no.	Sample	Solution	U(VI)			
			C_0 (mEq/L)	C_{eq} (mEq/L)	A (mEq/g)	K_d (L/g)
1	Initial	No background, pH 5.4	1.5	1.09	0.82	0.75
2	MChT gel 300 rpm			0.06	2.9	51.5
3	MChT air 300 rpm			0.42	2.1	5.02
4	MChT H ₂ O 300 rpm			0.38	2.3	6.09
5	MWT gel 1 h 235 °C	0.1 M NaHCO ₃	1	0.54	1.9	3.24
6	Initial			0.92	0.31	0.34
7	MChT gel 300 rpm			0.85	0.43	0.51
8	Initial	0.1 M NaCl	1.5	1.03	0.85	0.85
9	MChT gel 300 rpm			1.06	0.85	0.81
10	Initial			0.79	0.41	0.52
11	MChT gel 300 rpm			0.77	0.44	0.56
12	MChT gel 500 rpm	0.2 mEq/L CsCl + 0.2 mEq/L SrCl ₂	1	0.8	0.37	0.46
13	MWT xerogel 2 h 175 °C			0.54	0.83	1.54
14	Initial			0.65	0.61	0.95
15	MChT gel 300 rpm			0.33	1.26	3.84

Fig. 5 Kinetic curves of U(VI) ions sorption in the absence of the background

group equilibrium is reached after 4000 h, this sample carries on absorbing the ionic forms of U (VI). This may be due to the pores shape. The pore entrances for this sample may be commensurated with hydrated uranyl-ions. It should be noted that sorption of U (VI) is characterized by a long duration, since it is associated with slow intra-diffusion processes as this previously observed (Um et al. 2007).

Since the actual uranium-containing media contain impurities of extraneous cations and anions, the influence of the background presence in the solution on the sorption capacity of the studied samples with respect to the U (VI)

ions was determined. 0.1 M NaHCO₃ was used as the first background. In the first approximation, this solution simulates the “block” water of the ChNPP and has a pH value of 8.2. Under these conditions, U (VI) is present in the form of anionic carbonate complexes (Odintsov et al. 2009) and the sorbent exhibits its anion exchange properties which are determined by the number of anion exchange centers, presumably Sn-OH groups. In these circumstances a positive effect was obtained from MChT (Table 3), in particular: the sorption capacity of the sample milled in the form of gel at 300 rpm increases by 43% compared to that of the initial sample. This result correlates with the fact that the content of structural OH-groups for this sample increases (Table 1). Therefore this sorbent can remove both cationic and anionic forms of U (VI) ions from the solution (exhibits amphotytic properties). This makes it more versatile, for example, in comparison with inorganic cationites (for example, titanium phosphates), cation exchange or anion exchange resins.

The analysis of the data on the sorption of U (VI) ions (Table 3) allows to note the total effect of 3 factors: (i) the parameters of porous structure (Table 2), (ii) the number of sorption active OH-groups (Table 1), (iii) nature of the latter. The sorption data in this stage of research is preliminary. However, with some degree of confidence, one can trace the following patterns.

All active centers of tin oxy-hydroxide can be divided into cation-exchange (selective and non-selective), as well as anion-exchange. The latter occur during the sorption from carbonate solutions of pH ≈ 8.2 (Table 3). The separation of cation-exchange centers into selective and non-selective follows from analyzing the data on the sorption of U (VI) ions in the presence and absence of competing

background cations (Table 3) based on the example of the initial sample and the sample after MChT of gel at 300 rpm.

The MChT of gel at 300 rpm leads not only to changes of the parameters of the porous structure (first of all, increasing the volume and size of pores), but also to a significant increase in the number of structural OH-groups which are the main centers of sorption. As a result, considerable increase in the sorption capacity for the U (VI) ions in the absence of background is observed.

Conversely, a significant reduction in the number of sorption active OH-groups for the sample after MWT of gel at 235 °C coincides with the deterioration of its sorption properties. This loss of sorption sites is not compensated even by an increase in the mesopore volume and size as compared with the sample after MChT of gel at 300 rpm. These data demonstrate evidently the influence of such factor as the number of active sorption centers on the sorption of U (VI) ions.

Based on the values of sorption capacities for U (VI) ions, obtained both in the presence and in the absence of background (Table 3, experiments 1 and 9, respectively), it can be stated that the number of active centers for the initial sample is the same in both cases. This indicates that all its sorption centers are selective. The sorption capacity of the sample after MChT of gel at 300 rpm in the solution of 0.1 M NaCl within the experimental error also coincides with the sorption capacity of the initial sample. It can be assumed that during MChT, the number of OH-groups increases (Table 1); however, in terms of sorption properties, they have a different nature (non-selective sorption centers).

This is confirmed by the data obtained during the sorption of U (VI) ions from the solution containing simultaneously 0.2 mEq/L CsCl and SrCl₂ as the background electrolytes. Under these conditions, where the sorbed U (VI) ions compete with 0.2 mEq/L Cs⁺ and 0.2 mEq/L Sr²⁺, the sorption capacities of the initial sample and sample after MChT of gel at 300 rpm also coincide (≈ 0.4 mEq/g UO₂²⁺).

Interesting observations about the nature of functional groups can also be obtained from the results of sorption of U (VI) ions from the solution containing U (VI) + Cs⁺ + Sr²⁺ at pH ≈ 3.0 (Table 3). Under these conditions one can see that the sorption capacities of the initial and modified samples with respect to the U (VI) ions is even higher than at pH ≈ 5.4 , which is unusual for the sorption of U (VI) ions. It can be assumed that most of the centers of the initial sample are strongly acidic. Therefore they are capable of active sorption at pH ≈ 3 . Under these conditions U (VI) is in the form of a UO₂²⁺ ion with a size slightly smaller than 0.4 nm, which is less than its hydrated forms (Fig. 4). This greatly facilitates their diffusion to the sorption centers, and the more developed porous structure of modified sample allows to achieve even better results (Table 3).

4 Conclusions

MChT and MWT of precipitated tin oxy-hydroxide cause increase in the mesopores diameter and formation of meso-macroporous structure, improvement of crystal structure and removal of structural OH-groups (milling of xerogel) or increase of their content (milling of gel). As a result, an increase in the surface accessibility to hydrated uranyl-ions (with a diameter 0.4–1.0 nm) occurs, although the total specific surface area measured using nitrogen adsorption is somewhat reduced, as a result of modification. Therefore the analysis of data on the sorption of U (VI) ions from the tin oxy-hydroxide samples allows to suggest that three factors influence on sorption processes: the parameters of the porous structure, the number of sorption active OH-groups and their nature. The obtained results indicate that the sorption centers are not of the same effect. They can be divided into three groups: (1) selective cation-exchange; (2) non-selective cation exchange; (3) anion exchange.

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