ORIGINAL PAPER

Investigation on extraction behavior and extraction mechanism of uranium(VI) with DGA derivative into organic system from aqueous solution

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Abstract

The extraction behavior and mechanism of uranium(VI) from aqueous solution with the multidentate ligand *N,N,N''',N'''*-tetraoctyl-*N'',N''*-ethidene diglycolamide (TOE-BisDGA) as extractant were investigated. The extraction percentages of uranium(VI) were found to increase with increasing aqueous nitric acid solution. The extraction process was deduced to proceed by neutral extraction mechanism or ion association extraction mechanism, and the TOE-BisDGA extractant formed a major 1:1 complex with uranium(VI) in xylene system. The possible molecular structure of extracted pieces were presumed to be $[UO_2(NO_3)_2(TOE\text{-}BisDGA)]$ or $[UO_2(TOE\text{-}BisDGA)](NO_3)_2$ or $[UO_2(NO_3)TOE\text{-}BisDGA)](NO_3)$ by ESI–MS and extraction mechanism.

Keywords Bisdiglycolamide · Extraction behavior · Uranium(VI) · Extraction mechanism · Extracted pieces

Abbreviations

TOE-BisDGA	*N,N,N''',N'''*-tetraoctyl-*N'',N''*-ethidene bisdiglycolamide
TBP	Tri-*n*-butylphosphate
PUREX	Plutonium uranium recovery by extraction
DHOA	Dihexyloctanamide
D2EHIBA	Isobutyramide
DGA	Diglycolamide

Introduction

Uranium is a naturally occurring radioactive element in the earth's crust and present in the various forms of isotopes and minerals such as uranite, pitchblende, coffinite, brannerite, davidite, thucholite, and thucholite along with other secondary uranium minerals. (Rajendran et al. 2018; Kaoru and Yusuke 2019; Mumtaz et al. 2018). Uranium and mixed oxide (MOX) fuels were used as a source by most of the nuclear energy plants for power generation. Therefore, nuclear power plant and uranium deposit are the main sources for potential release of uranium into the environment. Uranium accident or unaware consumption poses a potential risk for human health and affects all forms of life due to the radiation impact from uranium isotopes and its decay elements (Gupta and Singh 2003). An inducing carcinogenic activities such as mining and processing of uranium ores, nuclear weapon test sites and natural leaching process (such as weathering of rocks and soil bed into aquifers) have become pressing issues throughout the world (Child and Hotchkis 2013). It is acknowledged that lung cancer is the king of carcinoma, which seriously threatens human health and may be caused by inhaling of uranium decay products. Therefore, it is necessary to extract and recycle uranium from different matrices to achieve maximum use of nuclear energy while protecting the environment. There has been continued scientific interest towards developing technologies for decontaminating and extracting uranium from nuclear wastes and uranium ores (Abdi et al. 2017; Shen and Schäfer 2014; Li et al. 2017).

Uranium recovery can be achieved by kinds of technical means including extraction, precipitation, ion exchange, electrochemical techniques, adsorption, membrane separation (Beltrami et al. 2014). Among these methods, liquid–liquid extraction using extractants is the most commonly method for uranium processing operation due to its available

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continuous operation, high throughout and solvent recycling (Boltoeva et al. 2018; Wehbie et al. 2017; Ansari et al. 2017). Many extractants were synthesized for the extraction of actinides and lanthanides from spent fuel (Swami et al. 2017; Gujar et al. 2016; Ansari et al. 2012). Organophosphorous compound tri-*n*-butylphosphate (TBP) dissolved in *n*-dodecane is used for extraction of uranium(VI) and plutonium(V) in PUREX process at industrial scale (Kumari et al. 2012). Large number of eco-friendly new ligands with mono as well as bifunctional donor group based on amides have been reported, such as dihexyloctanamide (DHOA), isobutyramide (D2EHIBA) (Manchanda and Pathak 2004), malonamide (Rout et al. 2012) and diglycolamide (Ansari et al. 2012) ligands have been evaluated as alternative extractants for the extraction and separation of actinides. Compared with phosphorus-containing extractant, the amide ligands have several outstanding advantages such as facile preparation, high chemical and radiolytic stability, complete incineration which dramatically reduces the amounts of waste (Mallampalli et al. 2017). In contrast with monoamides, polydentate diamide ligands have attracted more attention due to its stronger complexation and better extraction ability. Among these amides, the diglycolamide (DGA) ligands were ultimately found to have the best extraction behavior by further structural optimizations in the family of amides. The DGAs were assumed to associate with metal ions to form two bridged five-membered rings based on three oxygen atoms of one ether and two amide groups, and expected to increase the affinity with metal ions as tridentate ligands (Shanmugaperumal et al. 2008).

Based on the previous reports, the different structural backbone or substituent group of the DGA diamides has an impact to the actinide extraction abilities in liquid–liquid extraction (Iqbal et al. 2010; Sasaki and Tachimori 2002). In our previous work, the new multidentate ligand BisDGAs have been synthesized for actinides extraction, and which show strong extraction capacity for thorium(IV) in dodecanol from nitric acid solution (Ren et al. 2017). To further demonstrate the potential of the BisDGAs extractants in the spent fuel recycle. Here in, the extraction behaviors and the detailed extraction mechanism of uranium(VI) with TOE-BisDGA (Fig. 1) were investigated, the possible molecular of extracted pieces were also deduced on the basis of different extraction mechanisms.

Experimental

Materials and solutions

All chemicals and reagents used for organic synthesis and solvent extraction were of analytical grades and used without any further purification. $UO_2(NO_3)_2$ in nitric acid solution was prepared from analytical grade U_3O_8. The extractant *N*,*N*,*N‴*,*N‴*-tetraoctyl-*N″*,*N″*-ethidene bisdiglycolamide (TOE-BisDGA) (Fig. 1) was synthesized and purified in our laboratory according to the previous literature (Ren et al. 2017). ^{1}H NMR(400 M) and ^{13}C NMR(400 M) spectra of extractant prepared were recorded with a Varian Mercury-400 spectrometer in $CDCl_3$ using TMS as the internal standard. The extracted pieces were further characterized by mass spectrometry (MS) using a Bruker Esquire 6000 mass spectrometer.

Methods

The extractant TOE-BisDGA solution of a certain concentration was prepared by dissolving in organic solvent, which was used for the further extraction experiments after proper dilution. The organic phase was initially pre-equilibrated with the aqueous phase containing the desired concentration of nitric acid after dilution to achieve the equilibrium acidity. The extraction experiments of uranyl ion were carried out in a rotation speed of 2000 rpm by shaking 1.0 mL of the organic phase with 1.0 mL of an nitric acid solution acid solution containing 5.0×10^{-4} M $UO_2(NO_3)_2$ in airtight polypropylene tubes for about 20 min at room temperature. The concentration of nitric acid in the initial aqueous phase was varied from 1.0 M to 6.0 M, which is adjusted with commercial nitric acid (69%). After extraction, the organic phase and aqueous phase were separated by centrifugation at 2000 rpm for 5 min. The concentration of uranyl ion in the aqueous phase was examined by the Arsenazo-III spectrophotometric method using a 723 N model UV–Vis spectrophotometer (Shanghai, China), which was equipped with a 1 cm path length quartz cell for the acquisition of visible spectra (wavelength range 325–1000 nm). The absorption wavelength was set to 652 nm for the extraction of uranyl

Fig. 1 Extractant used in the present study
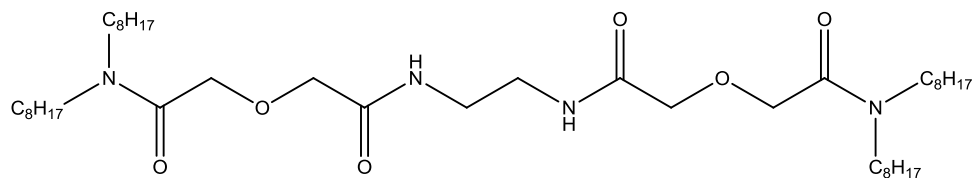
TOE-BisDGA

ion. The concentration of uranyl ion in the organic phase was calculated by mass balance. The data detailed in this paper are average of triplicate measurements and the reproducibility in mass balance was $\pm 5\%$. The distribution ratios (D) of uranium were calculated using the following equation:

$$D = \frac{\left[\mathrm{UO}_2^{2+}\right]_{\mathrm{org}}}{\left[\mathrm{UO}_2^{2+}\right]_{\mathrm{aq}}}, \tag{1}$$

where $\left[\mathrm{UO}_2^{2+}\right]_{\mathrm{org}}$ and $\left[\mathrm{UO}_2^{2+}\right]_{\mathrm{aq}}$ are the concentrations of the uranyl ion in the organic phase and the aqueous phase at equilibrium, respectively. The extraction percentage ($E\%$) was written by the following equation:

$$E = \frac{D}{D + 1}. \tag{2}$$

Results and discussion

Extraction behavior

The effect of the equilibration time on the extraction process was investigated at 6.0 M nitric acid solution with measuring the extraction percentages of uranyl ion as a function of contacting time of two phases. Figure 2 shows that the extraction kinetics for a 80-min period. The extraction efficiency of uranium(VI) increases rapidly with the increasing of time to reach a plateau in xylene and 1,2-dichloroethane system after 20 min. For instance, the extraction percentages of uranyl ion reached 85.5% in xylene system and 73.7% in 1,2-dichloroethane system after 20 min. In addition, further extending the two phases contact time to 30 min, no significant change in the extraction efficiency of uranium(VI) was observed. Therefore, a 20-min period is sufficient to
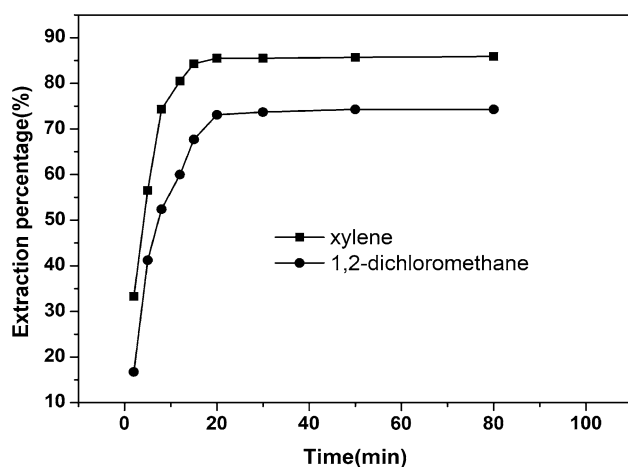
achieve quantitative extraction which will be employed for the further extraction process.

Based on a certain extractant TOE-BisDGA concentration, the extraction behavior of uranium(VI) in different aliphatic and aromatic diluents (xylene, 1,2-dichloromethane, dodecane, chloroform and dodecanol) was studied as a function of the nitric acid in the aqueous phase. As shown in Fig. 3, it is obvious that the concentration of nitric acid has a great influence on extraction percentages of uranyl ion, the data of extraction percentages increase with the increase of nitric acid concentration for all the solvents used. Although there are high values of extraction percentages in dodecane diluent, the formation of third phase was found in extraction of uranyl ions in extraction of uranium(VI) in 5.0 M and 6.0 M HNO₃. Compared with other diluents investigated, the reasonable extraction behavior of uranium(VI) in xylene diluent within the range of 1.0–6.0 M of nitric acid solution was observed. Therefore, xylene was chosen as the preferable diluent for further extraction investigation in this work.

In a further step, we studied the extraction efficiency starting from different initial concentrations of TOE-BisDGA in the organic phase ranging from 0.02 to 0.15 M. Figure 4 shows that the extraction efficiency of uranium(VI) gradually increases from 65 to 90% with the initial TOE-BisDGA concentration increases up to 0.1 M. Nevertheless, the extraction efficiency reaches to 90% if the TOE-BisDGA molar quantity exceeds 0.1 M. This shows that small amounts of TOE-BisDGA extractant are sufficient to quantitatively extract uranium(VI) from aqueous solution at the following conditions: (0.1 M TOE-BisDGA dissolved in 1 mL xylene/1 mL of uranium(VI) solution: $[\mathrm{UO}_2^{2+}] = 5.0 \times 10^{-4}$ M).

The effect profiles of interfering elements on extraction of uranyl ion, such as Fe(III), Sr(II) and Cs(I) were
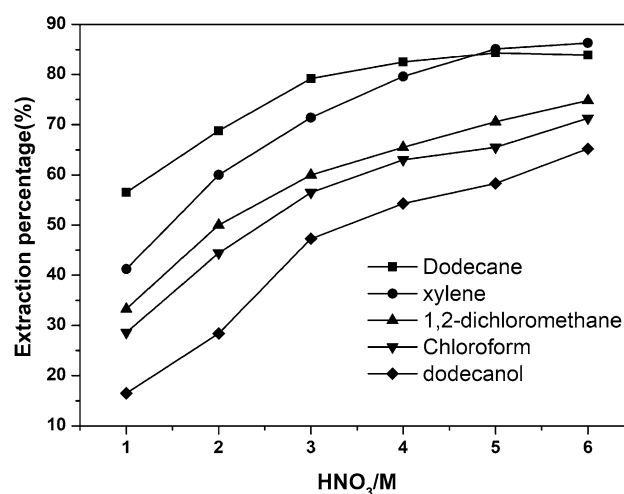

Fig. 2 Extraction kinetics of uranium(VI) at 25 ± 1 °C from 6 M HNO₃ with 0.07 M TOE-BisDGA in xylene/1,2-dchloroethane

Fig. 3 Effect of different diluents on uranium(VI) extraction; equilibration time: 30 min; organic phase: 0.07 M TOE-BisDGA in different diluents, T: 25 ± 1 °C
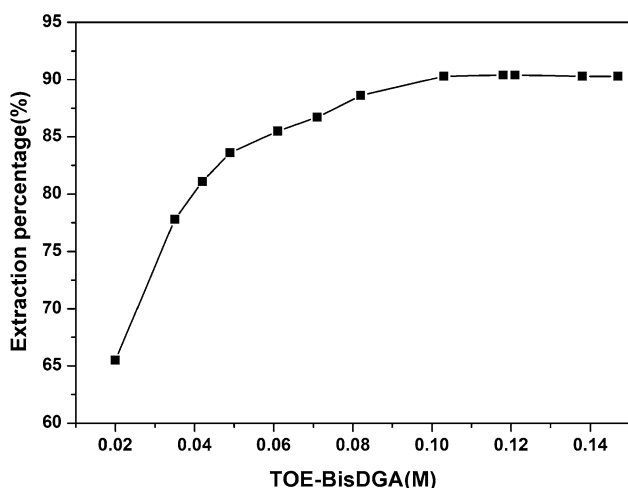
Fig. 4 Effect of different concentrations of TOE-BisDGA on uranium(VI) extraction; equilibration time: 30 min; T: 25 ± 1 °C

studied from pure nitric acid employing TOE-BisDGA/xylene. Figure 5 shows the variation in extraction percentages of uranyl ion in the presence of varying concentration of Fe(III), Sr(II), and Cs(I). The results show that even the presence of 5.0×10^{-4} M of Cs(I) dose not affect the extraction behavior of uranyl ion. This is attributed to the higher extraction of U(VI) as compared to as reflected in the batch studies. In case of Sr(II), the distribution values of U(VI) decreased sharply with increase in the Sr(II) concentration. It is attributed to the fact that the extractant TOE-BisDGA was involved in the extraction process of Sr(II). Figure 5 also shows that there is slightly decrease in the extraction of U(VI) even in the presence of 5.0×10^{-4}M of Fe(III) investigated at 6 M HNO$_3$, which suggested that Fe(III) may slightly affect the recovery of U(VI) with TOE-BisDGA. It
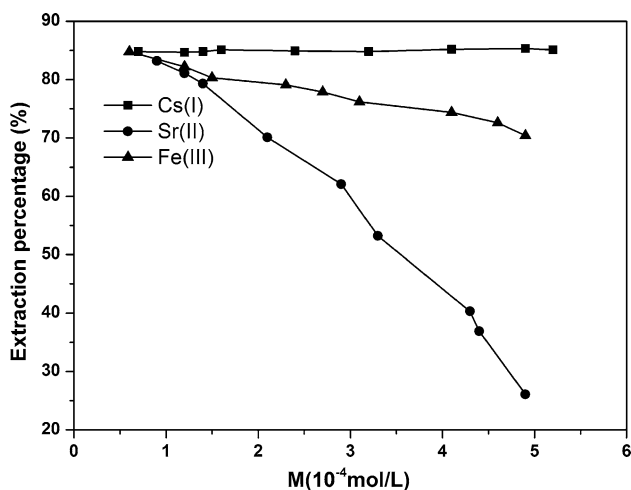
is related to the fact that whereas TOE-BisDGA has weak extraction ability for Fe(III).

Extraction mechanism

To examine the fundamental stoichiometry of extracted complexes, slope analysis method was carried out with TOE-BisDGA extractant in xylene system, where D values of the uranium(VI) were plotted as a function of the equilibrium concentration of TOE-BisDGA at 6.0 M HNO$_3$. As seen in Fig. 6, the linear relationships of log$D$ and log[TOE-BisDGA] were obtained in a slope of ca.1. It was assumed that one molecular extractant could be associated with one molecular uranyl ion to form a 1:1 complex during the extraction process.

In general, the extraction process in conventional organic phase proceeded by neutral extraction mechanism (Iqbal et al. 2010). As shown in Fig. 3, the extraction efficiency of uranium(VI) increased with the increase of nitric acid concentration. It suggested that nitrate anion may be involved in extraction of uranium(VI). To obtain a detailed insight into the role of the nitrate anion in the extraction process, an attempt was made by adding sodium nitrate or sodium chloride into aqueous phase during the extraction process to evaluate their effect on the distribution ratio of uranium(VI) while keeping the initial constant uranium(VI) concentration in the aqueous phase (Fig. 7). The results show that the distribution ratios increased with the increase of inorganic salt concentration for all cases. It is worthy of note that the addition of sodium nitrate into aqueous solution has a more significant impact on the distribution ratios of uranium(VI) than adding sodium chloride into aqueous solution. The evidence implied that sodium nitrate is a very strong salting-out reagent for the present extraction system.
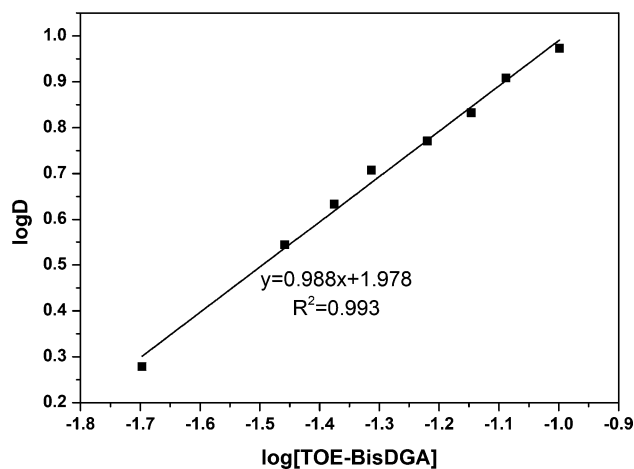

Fig. 5 Effect of different concentrations of interfering elements on uranium(VI) extraction; equilibration time: 30 min; T: 25 ± 1 °C

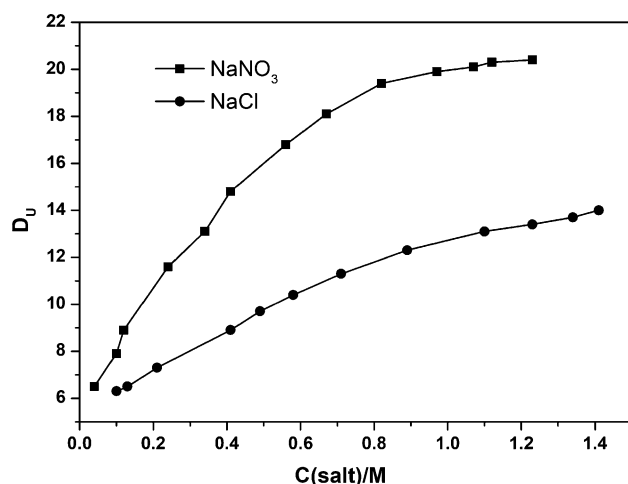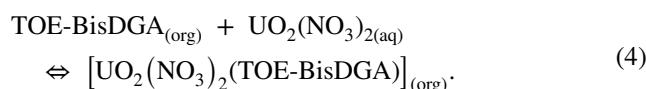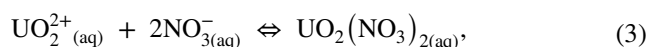Fig. 6 Plots of log$D$ versus log[TOE-BisDGA]; [UO$_2^{2+}$]=5.0×10^{-4} M; T: 25 ± 1 °C

Fig. 7 Influence of NaNO₃ and NaCl on the extraction of uranium(VI) from 6.0 M nitric acid solution with 0.07 M TOE-Bis-DGA in xylene. $[UO_2^{2+}] = 5.0 \times 10^{-4}$ M; T: 25 ± 1 °C

Besides the above slope analysis method, the ESI–MS data of organic phase were also performed after extraction of 0.03 M uranium(VI) with 0.1 M TOE-BisDGA ligand in xylene. The data were acquired on a Bruker Model maX-is4G system. In positive ion mode of mass spectrometry with TOE-BisDGA (Fig. 8), the peaks at *m/z* 1070.6234 and *m/z* 1007.6295 can be observed, which are corresponding to the representative extracted species [(TOE-BisDGA)(UO₂)NO₃]⁺ (calcd.1070.65) and [(TOE-BisDGA)(UO₂)-H]⁺ (calcd.1007.66), respectively. The strong peak observed at 739.6093 is corresponding to the representative extracted species [(TOE-BisDGA)+H]⁺ (calcd.739.63). As described above, that the 1:1 extracted pieces formed with the present extractant TOE-BisDGA and uranium(VI) were proved.
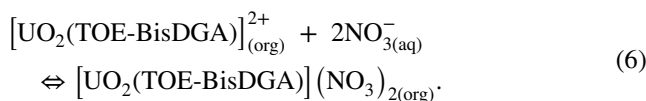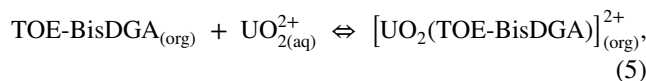
The extraction mechanism is crucially influenced by the solvation state of uranium(VI) in nitric acid solution. Uranium(VI) can form some complexes such as $UO_2(NO_3)^+$ and $UO_2(NO_3)_2$, which are depending on the concentration of nitrate ion in aqueous solution. Among these species, $UO_2(NO_3)_2$ is the easier complex to form since it is neutral (Houwer and Görller-Walrand 2001). In addition, $UO_2(NO_3)_2$ is more highly hydrophobic than the complex $UO_2(NO_3)^+$ in aqueous phase. Indeed, the neutral complex is easily formed because it requires less free energy. Therefore, the multidentate coordinate dinitrato complex is regarded as the most probable structure for the uranyl-nitrate complex species (Ikeda-Ohno et al. 2009). Several researchers studied the speciation of uranium(VI) in concentrated HNO₃ aqueous solutions (Ikeda-Ohno et al. 2009; Ikeda et al. 2008). Overall, they showed that the cationic form UO_2^{2+} and $UO_2(NO_3)^+$ exist in nitric acid low concentration solutions. The neutral form $UO_2(NO_3)_2$ is predominant until 8.0 M.

According to the conclusions reported in above literature, the log*D*–log*C* slope analysis, ESI–MS data as well as the salting-out effect, the extraction reaction mechanism must take into account of positive ion and uranyl-nitrite complexes. Therefore, the extraction mechanism at lower concentrated nitric acid solution can predominantly proceed with ion association extraction mechanism, at higher concentrated nitric acid solution can predominantly proceed with neutral extraction mechanism. Obviously, both extraction mechanisms coexist at the same time in the whole extraction process with 1.0–6.0 M concentration nitric acid solution.

As we known, uranium has a coordination number of 6 (Stephon et al. 1991). The extractant TOE-BisDGA containing six oxygen atoms of amide, hence, it may shows multidentate features. Meanwhile, according to the neutral extraction mechanism, the structure of the possible complex of uranium(VI) with TOE-BisDGA extractant as depicted in Fig. 9. Obviously, the extractant TOE-BisDGA only provides one of the carbonyl oxygen atoms and one of the ether oxygen atoms in coordination with the uranyl ion, while the remaining unoccupied orbitals of uranyl ion require nitrate anions to participate in coordination. As shown in Fig. 9. The uranyl ion forms a five-membered ring with the extractant TOE-BisDGA and forms two four-membered rings with the nitrate anions. The complex has two structures due to the five-membered ring formed with the extractant TOE-BisDGA which has two positions. Therefore, the extraction reaction of uranyl ion with TOE-BisDGA can expressed by Eqs. (3) and (4).

$$UO_2^{2+}{}_{(aq)} + 2NO_3^-{}_{(aq)} \Leftrightarrow UO_2(NO_3)_{2(aq)}, \tag{3}$$

$$TOE\text{-}BisDGA_{(org)} + UO_2(NO_3)_{2(aq)} \\ \Leftrightarrow \left[UO_2(NO_3)_2(TOE\text{-}BisDGA)\right]_{(org)}. \tag{4}$$

According to the ion association extraction mechanism, the structure of the possible complex of uranium(VI) with TOE-BisDGA extractant is depicted in Fig. 10. As shown Fig. 10a, the extractant TOE-BisDGA provides all of the carbonyl oxygen atoms and the ether oxygen atoms to form four five-membered rings and a big nine-membered ring with uranium(VI). Obviously, the nitrate anion does not participate in the coordination with uranium(VI), and which only acts to neutralize the charge of the central atom. Therefore, the extraction reaction of uranium(VI) with TOE-BisDGA can expressed by Eqs. (5) and (6):
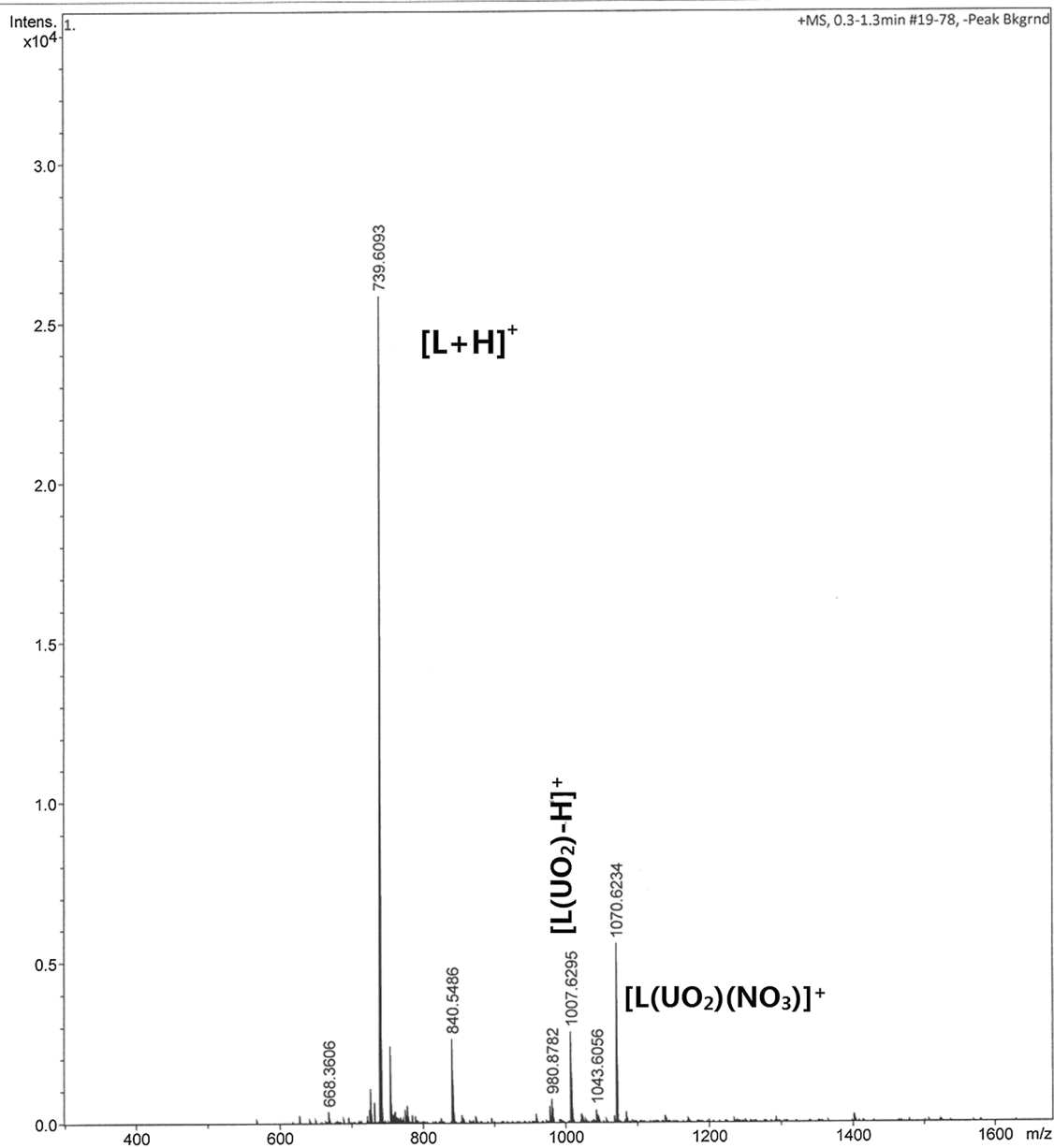
$$TOE\text{-}BisDGA_{(org)} + UO_2^{2+}{}_{(aq)} \Leftrightarrow \left[UO_2(TOE\text{-}BisDGA)\right]^{2+}_{(org)}, \tag{5}$$

$$\left[UO_2(TOE\text{-}BisDGA)\right]^{2+}_{(org)} + 2NO_3^-{}_{(aq)} \\ \Leftrightarrow \left[UO_2(TOE\text{-}BisDGA)\right](NO_3)_{2(org)}. \tag{6}$$

Fig. 8 ESI–MS spectrum after extraction of uranium(VI) with TOE-BisDGA in xylene. (*L* stands for the extractant TOE-BisDGA)

As shown Fig. 10, the extractant TOE-BisDGA provides two carbonyl oxygen atoms and two ether oxygen atoms in the middle of the molecular structure to participate in

coordination with uranyl ion. The uranium(VI) forms two five-membered rings and a big nine-membered ring with the extractant TOE-BisDGA. Obviously, there are still two unoccupied orbitals of uranium(VI). Then, one molecule of

Fig. 9 The structure of the possible complex of uranium(VI) with TOE-BisDGA extractants which can proceed with neutral mechanism
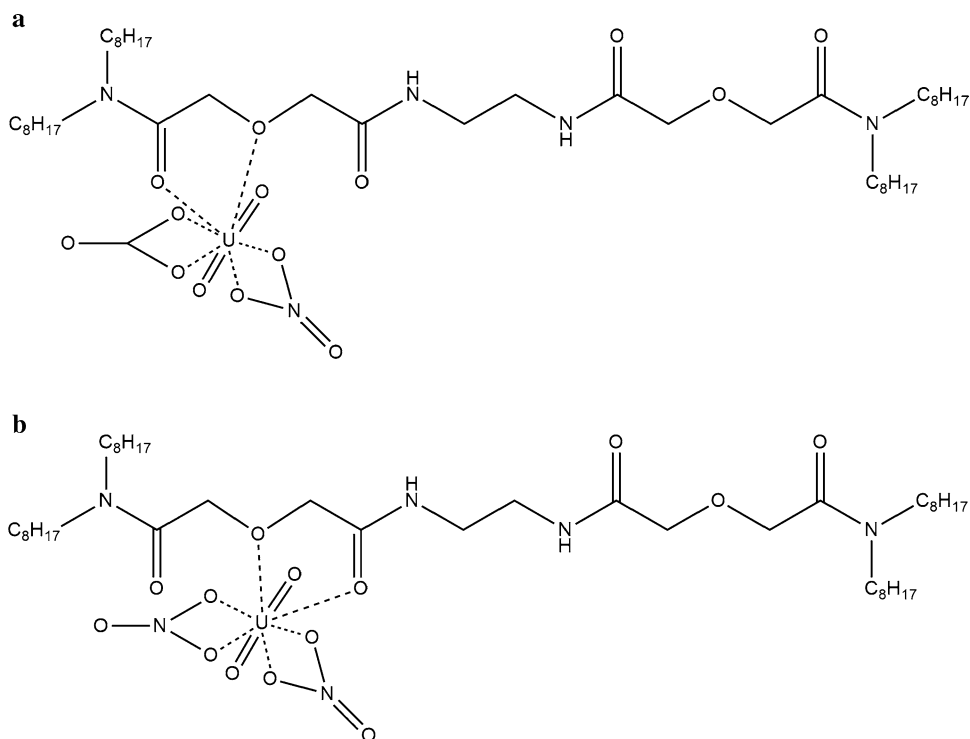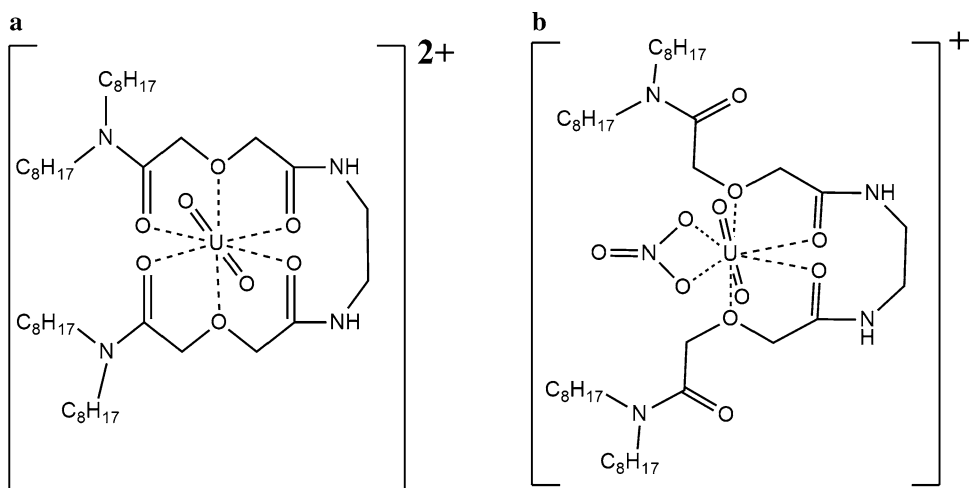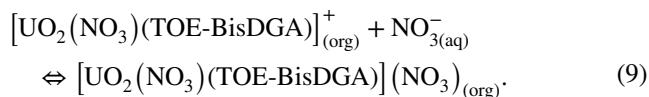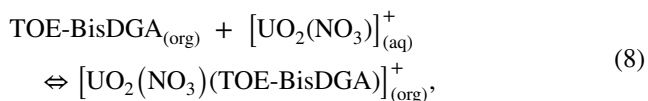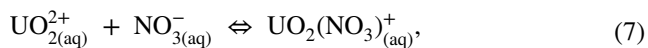

Fig. 10 The structure of the possible complex of uranium(VI) with TOE-BisDGA extractant which can proceed with ion association extraction mechanism

nitrate anion participates in coordination with uranium(VI), which formed a four-membered ring. On the other hand, one molecule of nitrate anion acts to neutralize the charge of the central atom. Therefore, the extraction reaction of uranium(VI) with TOE-BisDGA can be expressed by Eqs. (7)–(9):

$$UO_{2(aq)}^{2+} + NO_{3(aq)}^{-} \Leftrightarrow UO_2(NO_3)_{(aq)}^{+}, \tag{7}$$

$$TOE\text{-}BisDGA_{(org)} + \left[UO_2(NO_3)\right]_{(aq)}^{+}$$
$$\Leftrightarrow \left[UO_2(NO_3)(TOE\text{-}BisDGA)\right]_{(org)}^{+}, \tag{8}$$

$$\left[UO_2(NO_3)(TOE\text{-}BisDGA)\right]_{(org)}^{+} + NO_{3(aq)}^{-}$$
$$\Leftrightarrow \left[UO_2(NO_3)(TOE\text{-}BisDGA)\right](NO_3)_{(org)}. \tag{9}$$

Conclusions

In conclusion, solvent extraction of uranium(VI) from aqueous solution with TOE-BisDGA as the extractant was investigated. The extraction mechanism of uranium(VI) with TOE-BisDGA closely depends on the solvation state of uranium(VI) in nitric acid solution. Based on the relationships between $\log D_U$ and $\log[TOE\text{-}BisDGA]$, ESI–MS

spectra as well as the salting-out effect, the extraction process of uranium(VI) was deduced to proceed by neutral extraction mechanism or ion association extraction mechanism, and TOE-BisDGA extractant formed a 1:1 complex with uranium(VI). Furthermore, the possible molecular structures of the extracted pieces and the different extraction reaction were inferred according to the neutral mechanism and the ion association extraction mechanism.

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