

Solvent extraction of Thorium(IV) from chloride acidic solution using Schiff base

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Abstract

Thorium(IV) is extracted from chloride medium by solvent extraction using a prepared Schiff base chelating agent, (E)-4-(2-hydroxy phenyl imino) pentane-2-one (AcPh) that is synthesized and characterized by FT-IR, ¹H-NMR and elemental analysis. The obtained optimum extraction parameters were 0.02M AcPh/chloroform and diethyl ether ratio mixture, pH 6.5, 3:1 aqueous to organic phase ratio for 5 min. contact time at room temperature. The thorium(IV) was then stripped into aqueous media containing 0.5 M HNO₃ solution using of 1:2 aqueous to organic phase ratio for 7 min. contact time at room temperature.

1. Introduction

Thorium has a huge important in the nuclear energy field. In the context of growing world population whose demand for energy is increasing, and the threat of global warming due to carbon dioxide emissions (caused by coal and hydrocarbon burning), the nuclear technology may be an attractive option especially in the developing countries (Manchanda, 2004). Thorium element is used in several of industrial and technological applications. Thorium is widely distributed in small amounts over the earth surface. It occurs in several minerals, the most common ones are monazite sand, thorianite, thorite and thoganite (Masson, et al., 2015).

Extensive efforts were recently focused on the extraction of thorium ions from its sources, some of the commonly employed separation methods; which precipitation (Armarego and Perrin, 2000), electrophoresis (Yekta, et al., 2014), electrodialysis (Mohammadi, et al., 2005), chromatography (Sharma and Sharma, 2007; Pathak and Argekar, 1992), flotation (Shiri, et al., 2011), ion exchange separation (Chen, et al., 2016; Metwally, et al., 2005) and solvent extraction (Patkar, et al., 2007) were studied. However, solvent extraction method has some advantages that are large capacity, simple equipment, quick and safe operation and low cost. Therefore it has been widely employed for the separation of precious metals from their solutions or real solution (Sole, et al., 2005).

Various extractants had been employed for the extraction of thorium, such as phosphoric acids, neutral phosphates, amines, Schiff bases, oximes, isoxazolones, crown ethers, ionic liquids, and so on (Li, et al., 2012).

Thorium(IV) was quantitatively extracted at pH 7.5 with 0.0001M of hexaacetate calix(6)arene in toluene and stripped with 0.05 M nitric acid (Malkhede, et al., 1999). Solvent extraction of thorium ion from perchlorate solution using di-dodecyl phosphoric acid, DDPA, in chloroform diluent was also carried out (Khalili, et al., 2003). A calixarene derivative, 5,11,17,23 tetra (diethoxyphospho-ryl)-25,26,27,28-tetra acetoxycalix-4-arene (L) used for the separation of thorium and rare earths from nitrate medium (Yanling, et al., 2012). The extraction of thorium by β -hydroxy naphthaldoxime in presence of neutral donors and the effects of neutral organo-phosphorous compounds on the extraction of thorium by β -hydroxy naphthaldoxime in o-xylene were studied (Banerjee, et al., 2001). The extraction of thorium(IV) from nitric acid solutions by di-n-butyl sulfoxide (DBSO) in o-xylene was evaluated and the extracted species were supposed to be Th(NO₃)₄·2DBSO wherein extraction was found to be almost independent of thorium concentration in the range between 4.3×10^{-4} - 4.3×10^{-2} M and inversely dependent on the temperature factor (Khan, et al., 1995).

The present study aims to the synthesized chelating agent Schiff base for the extraction and separation of Th(IV) from its acidic solutions. The effects of various experimental parameters are studied on the

extraction of Th(IV) from a synthetic solution. These factors include pH, extractant concentration, contact time, A/O ratio and temperature, while the stripping parameters include stripping concentration, contact time, phase ratio and reaction temperature.

2. Experimental

2.1. Reagents, Solutions and Instruments

All the chemicals and reagents used in all different parts of this work are analytical grade and used without further purification. A standard stock solution of 1000 mg/L thorium(IV) has been prepared by dissolving of 2.535 g thorium nitrate ($\text{Th}(\text{NO}_3)_4 \cdot 6\text{H}_2\text{O}$) from Sigma-Aldrich in 1000 ml acidified distilled water with 10 ml concentrated hydrochloric acid. This solution is actually used to determine the relevant factors of thorium extraction upon the prepared Schiff base.

A double beam recording UV/Visible spectrophotometer model 160-A, shimadzu, Japan, is used for measuring the absorbance of the investigated solutions and for the colorimetric determination of thorium(IV). The Fourier Transform Infrared spectrometer (FT-IR) (Thermo Scientific - NICOLET iS10 USA) spectrometer has been used to characterize the major functional groups of the synthesized extractant and the thorium complex.

2.2. Synthesis of Schiff Base

The Schiff base ligand (E)-4-(2-hydroxy phenyl imino) pentane-2-one (AcPh) is synthesized according to Ibrahim, et al., 1998 by refluxing ethanolic solution of 10 mmole of acetyl acetone with 10 mmole of 2-aminophenol for 6 h. Recrystallized from absolute ethanol, filtered, washed using ethanol and diethyl ether afforded pure products. The preparation of the ligand containing nitrogen and oxygen donor atoms is shown in Scheme 1.

Scheme 1: (E)-4-(2-hydroxy phenyl imino) pentane-2-one (AcPh) preparation

2.3. Experimental procedures

2.3.1. Extraction investigation

In the present work, the extraction of thorium ions from its aqueous solutions is carried out using the prepared (E)-4-(2-hydroxy phenyl imino) pentane-2-one (AcPh), the suitable amount of the studied extractant, is dissolved in a mixture of chloroform and diethyl ether (3:2 ratio) as diluent. The aqueous phase containing thorium(IV) is adjusted to the desired pH value using dilute solutions of HCl/NaOH and is equilibrated by shaking for a certain time in a glass separating funnel with an appropriate volume of the organic phase. The aqueous and organic phases are allowed to be settled, and after equilibration, they are separated completely by separatory funnel. The concentration of the Th(IV) in the aqueous phase after extraction is measured using the spectrophotometer technique by Thoron I reagent (Marczenko and Balcerzak, 2000).

The concentration of the extracted metal ions into the organic phase (C_o) is calculated based on the mass or material balance by subtracting the remainder concentration of metal ion in the aqueous phase

after extraction (C_A) from the initial concentration of metal ion before extraction (C_i) (Fouad, et al., 2015). The extraction distribution coefficient (D_E) is used as the most important index for determining the efficiency of solvent extraction process and is calculated by the following:

$$D_E = \left(\frac{C_i - C_A}{C_A} \right) \times \frac{V_A}{V_O} \quad (1)$$

where the extraction distribution coefficient (D_E) is defined as the concentration of species in the organic phase divided by the remained concentration in aqueous phase. The percentage of extraction (%E) is calculated accordingly from the equation:

$$\%E = \frac{100D_E}{D_E + \left(\frac{V_A}{V_O} \right)} \quad (2)$$

where V_A and V_O are the volumes of the aqueous and organic phases, respectively.

2.3.2. Stripping investigations

Stripping process (back-extraction) involves bringing the thorium ions from the organic extract back into the aqueous phase. Stripping experiments are carried out by shaking different volumes of the organic phase solutions containing the extracted metal after extraction and the aqueous solution volumes of the stripping agent (HNO_3) under study for contact time ranging from 1 to 15 min at room temperature. After equilibration and settling, complete separation of the two phases are obtained. Known aliquot portions are carefully withdrawn from the stripping aqueous phase for measuring the concentration of the stripped thorium ions. The stripping distribution coefficient (D_S) is used as the most important index for determining the efficiency of stripping process and is calculated by the following:

$$D_S = \left(\frac{C_A}{C_O - C_A} \right) \times \frac{V_O}{V_A} \quad (3)$$

where the distribution coefficient (D_S) is defined as the concentration of species in the aqueous phase divided by the remained concentration in organic phase after stripping process. The percentage of stripping (%S) is calculated accordingly from the equation:

$$\%S = \frac{100D_S}{D_S + \left(\frac{V_O}{V_A} \right)} \quad (4)$$

where V_O and V_A are the volumes of the organic and aqueous phases, respectively. All the extraction and stripping studies are carried out at ambient temperature ($25 \pm 1^\circ\text{C}$) (except when studying the effect of temperature).

The all experiments are repeated several times in order to confirm the correctness of the obtained results. The relative errors are no more than 5 %.

3. Results and Discussion

3.1. Characterization of the Prepared Ligand

The purity of the synthesized Schiff base is confirmed by melting point, elemental analysis, ^1H NMR and FT-IR analysis.

The yield of (E)-4-(2-hydroxy phenyl imino) pentane-2-one (AcPh) is 79.5% (15.9 g) that has 184 °C melting point and yellow color (Scheme 1). The elemental analysis: calculated % for C₁₁H₁₃NO₂: C; 69.08%, H; 6.87%, N; 7.33%. Found: C; 69.13%, H; 6.81%, N; 7.65%.

The ¹H NMR studies of AcPh ligand (DMSO-d₆) show a signal of two [-CH₃] protons at δ 2.0 ppm as singlet, the [=CH-] proton come to resonance at δ 5.19 ppm, and the 4-aromatic protons appeared at δ 6.72-7.11 ppm. The proton of -OH of the enol form resonate at δ 9.95 ppm, and the proton of -OH on the phenyl group come to resonance at δ 12.17 ppm. The protons of [=CH-], -OH on the phenyl group, and -OH of the enol form are exchangeable with D₂O, and the HOD signal appeared at δ 3.7 ppm (Salib, et al., 1991; Kwiatkowski and Kwiatkowski, 1980).

The IR spectra of the AcPh ligand gave medium and broad band centered at 3450 cm⁻¹, which could be assigned as stretching (sym and assym) vibration of the phenolic O-H group, which is slightly disappeared in Th(IV)-complex. The change in the intensity of the νO-H band could be denote the interaction of the phenolic O-H with the Th(IV) ion. The bands which appeared at 1630 and 1550 cm⁻¹ could be assigned as νC=O and νC=N bands respectively. These bands become very weaker and slightly shifted to lower wave number in the formed complex. The obtained results indicate the entrance of the C=O and C=N in the coordination system with metal ions. The strongly disappearance of the band O-H group in the complex may be due to the ionization of this group in complexation.

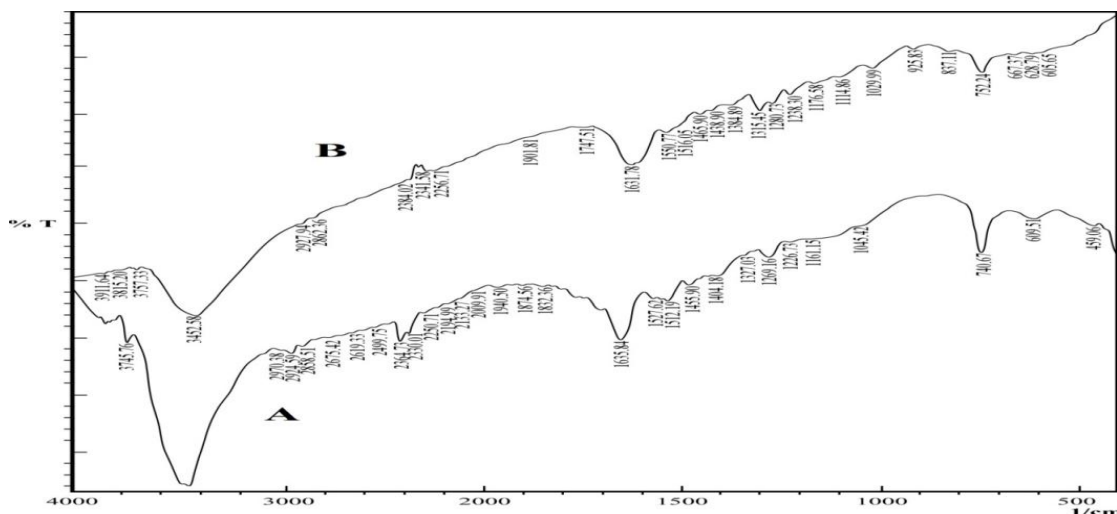


Fig. 1: FT-IR spectrum of AcPh (A) and its thorium complex (B).

3.2. Extraction studies of thorium

3.2.1. Diluent type

The dissolution of the studied Schiff base (AcPh) is performed using various aromatic and aliphatic organic diluents, such as benzene, toluene, petroleum ether, kerosene, chlorobenzene, methylene chloride, chloroform, and diethyl ether. From the obtained results, it is found that that AcPh is insoluble in benzene, toluene, petroleum ether, kerosene and methylene chloride, moreover, it is partially soluble in chlorobenzene, chloroform and diethyl ether, while it is completely dissolved in chlorobenzene on heating but it returns to deposit again on cooling. To solve this problem, two diluents are synergism with each other to dissolve the AcPh. The chloroform and diethyl ether are mixed with the ratio 3:2 to be completely dissolved of the AcPh.

3.2.2. Effect of pH

In order to study the influence of pH upon the extraction behaviors of thorium(IV) on the studied chelating extractant, several experiments are performed at different pH value ranging from 1 to 9 and other factors are fixed at 4.31 x 10⁻⁴ M (100 mg/L) thorium ions in the aqueous solution, 0.38% (0.02M)

AcPh/chloroform and diethyl ether mixture and 3:1 (A/O) aqueous to organic phase ratio for 7 min. contact time at room temperature. The pH of the studied thorium(IV) solutions are adjusted using 1M HCl or 1M NaOH. The concentration of the metal ions in the aqueous solution is spectrophotometrically determined after equilibrium. The results in Fig. 2, it is clearly evident that the thorium(IV) extraction efficiency on the studied extractant has gradually increased by increasing the pH till reaching maximum values at pH 6.5.

At low pH, there will be excess of H^+ ions in the acidic solution which could lead to the protonation of oxygen atoms of the ligand. Owing to the acidity increasing, the hydrogen ions compete for the thorium ions during the extraction process. Therefore, thorium extraction efficiency is decreased at low pH. With increasing pH of the aqueous solution, the concentration of hydrogen ions in aqueous phase decreases, therefore the reaction of hydrogen ions with internal oxygen atoms of AcPh is decreased and the Th(IV) complex is significantly formed, leading to higher extractability of Th(IV). Hence, the selected pH 6.5 is used for the quantitative extraction of thorium(IV) using the studied extractant which gives the highest uptake.

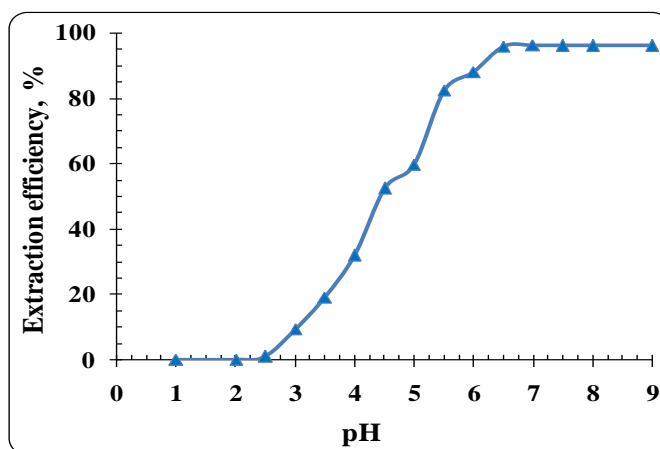


Fig. 2: Effect of pH upon the thorium extraction efficiency using AcPh extractant.

3.2.3. Effect of extractant concentration

The effect of AcPh/chloroform + diethyl ether mixture concentration on the extraction efficiency of thorium ions is studied in the range from 1.0×10^{-3} to 3×10^{-2} M. In these experiments, the extraction conditions are fixed at pH 6.5 for 7 min. shaking time and (3/1) A/O phase ratio at room temperature (Fig. 3). The obtained results revealed that the extraction efficiency of thorium ions is increased from 4.39 to 96.19% with increasing the concentration of acetylacetone phenolamine (AcPh) to 0.02 M (0.38%). Further increase in the extractant concentration up to 0.03M the extraction efficiency of thorium has not added any perceptible effect and showed a plateau due to the excess of free extractant. However, the 0.02M AcPh is the optimum concentration for the next experiments.

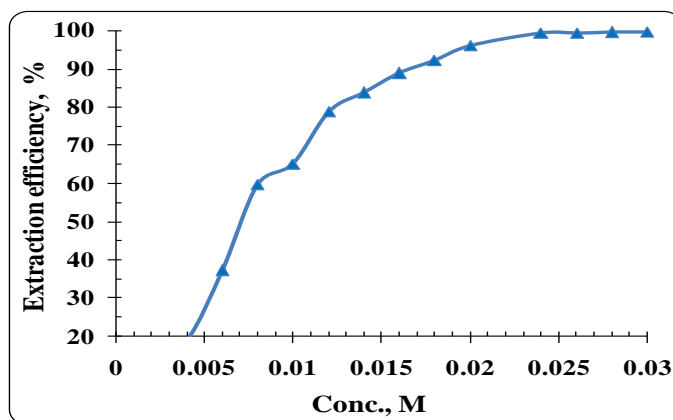


Fig. 3: Effect of AcPh Schiff base concentration upon the extraction efficiency of thorium.

3.2.4. Effect of contact time

A series of experiments have been applied to determine the effect of contacting time on Th(IV) extraction ranging from 1 to 10 min using extractant concentration of 0.02M (0.38%) AcPh/chloroform and diethyl ether mixture, pH 6.5 and 3/1 (A/O) ratio at room temperature. From the obtained data in Fig. 4 illustrate that, the Th(IV) extraction efficiency increases with contact time increases until 5 min for AcPh ligand and subsequently Th(IV) extraction efficiency then plateau. So a contacting time of 5 min. is enough to attain maximum extraction efficiency attained 96.2%. Therefore, the following experiments are performed by contacting the two phases for 5 min.

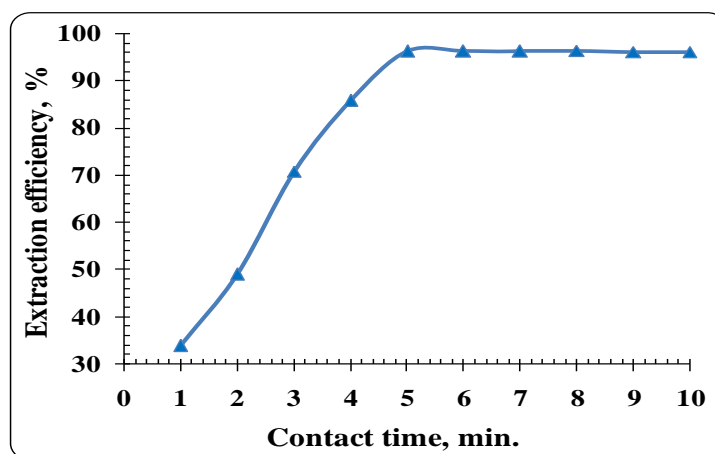


Fig. 4: Effect of contact time upon the extraction efficiency of thorium using AcPh extractant.

3.2.5. Effect of aqueous/organic phase ratio

Phase ratio (A/O) is one of the most important factors that affect the extraction efficiency. The effect of A/O ratio upon Th(IV) extraction efficiency is investigated in the range 7/1 to 1/4 using 0.02M (0.38%) AcPh/chloroform + diethyl ether extractant at room temperature for 5 min. contact time, after adjusting the aqueous solution to pH 6.5 and the thorium concentration is about 100 mg/L. Mixing of the aqueous solution with the organic phase is followed by the separation of the two phases in which the thorium concentration is determined in the aqueous solution and the obtained results are plotted in Fig. 5. The latter, it is quite clear that the maximum extraction efficiency is relatively remained constant at 3/1 until 1/4 A/O ratio while the maximum extraction efficiency is obtained at 3/1 A/O ratio for the AcPh Schiff base extractant. Therefore the 3/1 ratio is set to be the optimum A/O phase ratio.

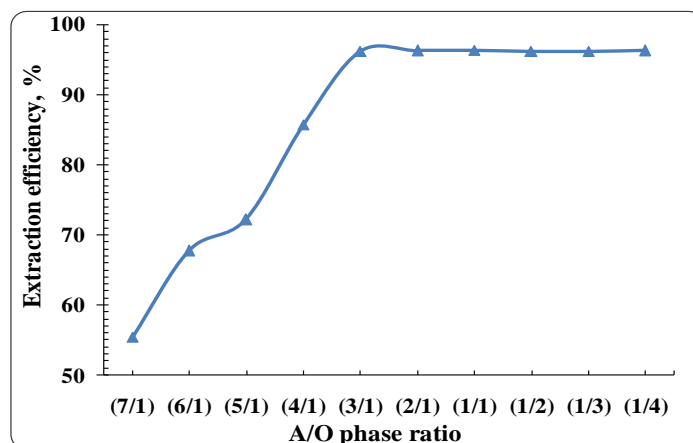


Fig. 5: Effect of A/O phase ratio upon the extraction efficiency of thorium using AcPh extractant.

3.2.6. Effect of temperature

The effect of temperature on Th(IV) extraction from aqueous solution is applied in the range of 25-55 °C using 0.02M (0.38%) AcPh/chloroform + diethyl ether extractant, 3/1 aqueous/organic phase ratio, pH6.5 and 100 mg/L thorium concentration for 5 min. contact time. From the obtained results in Fig. 7 give that the extraction efficiency of Th(IV) increase with the increase in temperature from 25 to 55 °C. According to the visibility study, the room temperature is best extraction temperature. Consequently, the maximum efficiency of thorium extraction upon the synthesized extractant AcPh in a mixture of chloroform + diethyl ether is 96.2 which is equivalent to 288.6 mg (Th)/L (organic phase). By the other words, the experimental thorium extraction capacity of AcPh extractant is 75.95 mg (Th)/g (extractant). However, the extraction capacity of AcPh is equivalent to 75.95 mg thorium ions per 1 g for AcPh extractant. Therefore, the extraction process is clearly favored at room temperature.

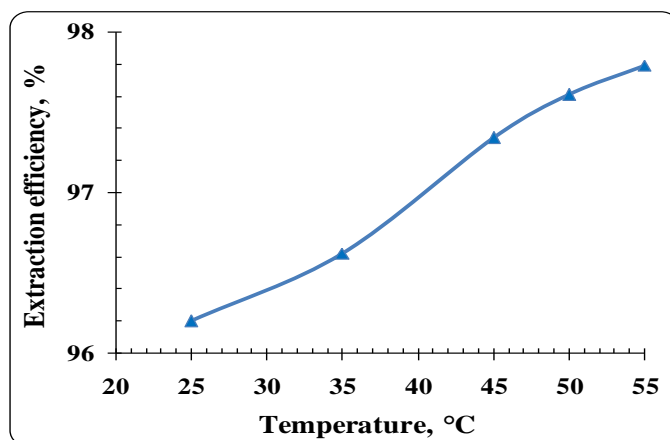


Fig. 6: Effect of temperature on the extraction efficiency of thorium using AcPh extractant.

3.3. Stripping studies

In the preliminary experiments aimed to strip Th(IV) from thorium loaded 0.02M AcPh in a mixture of chloroform + diethyl ether using nitric acid solution, series of experiments are carried out to back extract thorium ions into the aqueous phase from the loaded organic phases. For this purpose, some relevant parameters are applied to find out the stripping characteristics on the extracted thorium ions such as, stripping agent concentration, contact time, A/O phase ratio, temperature and the construction of the McCabe-Thiele diagram of the stripping process.

3.3.1. Effect of stripping agent concentration

To study the stripping agent concentration, Fig. 2 has actually illustrated that low Th(IV) extraction is observed at low pH values; a matter which implies that its stripping from the loaded 0.02M AcPh chelating ligand will occur with increasing the acid concentration. For this purpose, the different concentrations of nitric acid from 0.01 up to 1 M are examined while the other stripping conditions are fixed at an A/O ratio of 1/2 for 7 min. contact time at room temperature. From the obtained results given in Fig. 8, it is obvious that 0.5M HNO₃ has given the maximum thorium stripping efficiency of about 96.1 from the loaded AcPh. Therefore, 0.5 M nitric acid could be adequate and recommended for next studies.

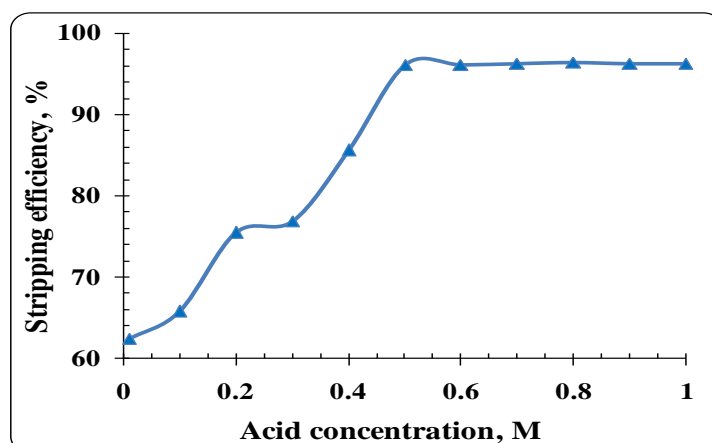


Fig. 7: Effect of nitric acid concentration on the stripping efficiency of Th(IV) from the loaded AcPh.

3.3.2 Effect of contact time

The effect of contact time upon thorium stripping efficiency by 0.5M HNO₃ from the loaded 0.02M AcPh in mixture of chloroform + diethyl ether is studied in the range from 1 to 15 min at the fixed conditions of A/O ratio of 1/2 at room temperature. From the results shown in Fig. 9, it is indicated that the stripping efficiency of thorium increased from 72.18 to 96.01% with increasing the contact time from 1 min to 7 min. After that, a contact time beyond 7 min did not perceptibly increase the thorium stripping from the loaded solvent beyond 96.01 for AcPh extractant. Therefore, a 7 min shaking time can be considered sufficient for quantitative stripping.

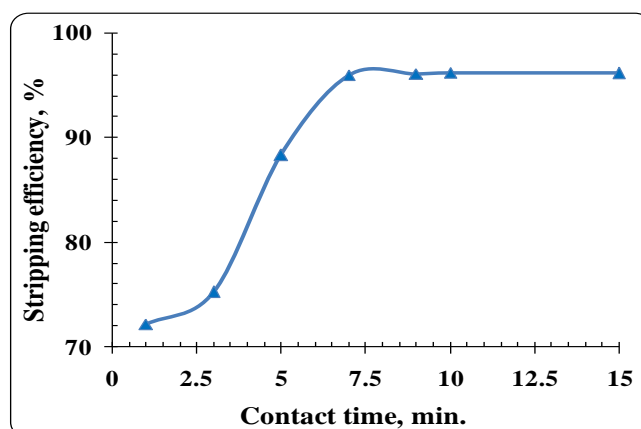


Fig. 8: Effect of contact time on the stripping efficiency of Th(IV) from the loaded AcPh.

3.3.3. Effect of aqueous to organic phase ratio

In order to study the effect of the A/O phase ratio on the thorium stripping from the loaded 0.02M AcPh in mixture of chloroform + diethyl ether (288.6 mg(Th)/L), several stripping experiments are performed at A/O ratio varying from 1/5 to 3/1 while the other stripping factors are fixed at 0.5M HNO₃ as stripping agent for 7 min contact time at room temperature. From the data given in Fig. 10, it is indicated that the maximum stripping efficiency from the loaded AcPh in its solvent is achieved at an A/O ratio of 1:2 and remained constant thereafter.

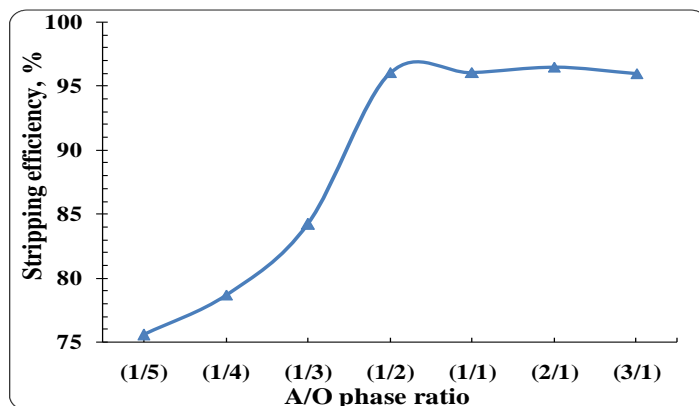


Fig. 9: Effect of A/O phase ratio on the stripping efficiency of Th(IV) from the loaded AcPh.

3.3.4. Effect of temperature

To investigate the effect of temperature upon the thorium stripping efficiency by 0.5M nitric acid from the loaded 0.02M AcPh in mixture of chloroform + diethyl ether, several experiments have been carried out in the range from 25 up to 55 °C. In these experiments, the other stripping conditions are fixed at 7 min contact time and an A/O phase ratio of 1/2. From the obtained results given in Fig. 12, it is clear that the temperature increased from 25 to 65 °C, the stripping efficiency is progressively decreased from 96.01 down to 80.4% for AcPh extractant. This result could be explained by the exothermic reaction of the stripping process. Therefore, the room temperature can be considered as the best temperature for the re-extraction process.

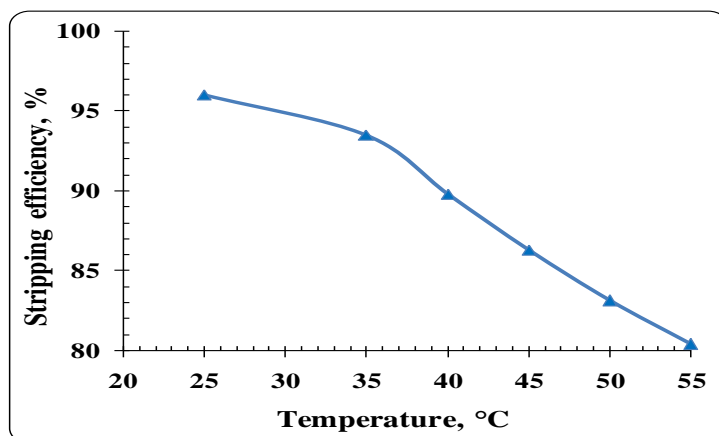


Fig. 11: Effect of temperature on the stripping efficiency of Th(IV) from the loaded AcPh.

3.4. Regenerations of organic extractant

For regeneration of the studied Schiff base extractant (0.02M AcPh in a mixture of chloroform + diethyl ether), the used organic phase is contacted with an equal volume of DI (De-ionized) water for 20

min. The phases are separated and the pH of the aqueous phase is measured. The organic phase is given repeated contacts with fresh DI water until there is no change in pH. The organic phase is then reused.

4. Conclusion

The extraction of Th(IV) ions using the synthesized (E)-4-(2-hydroxy phenyl imino) pentane-2-one (AcPh) Schiff base was studied from its synthetic solution. The results showed that the maximum extraction efficiency was obtained at 0.02M AcPh/chloroform and diethyl ether mixture, 3:1 A/O ratio for 5 min. contact time at room temperature. The thorium(IV) is then back-extracted into aqueous media containing 0.5 M HNO₃ using the optimum conditions 1/2 A/O ratio, 7 min. contact time at room temperature. The results of this study clearly showed the potential and versatility of (E)-4-(2-hydroxy phenyl imino) pentane-2-one for separation of Th(IV) with 96 % from its acidic solution.

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إستخلاص أيونات الثوريوم بإستخدام قاعدة شيف من المحلول الحامضي

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١ - قسم الكيمياء - كلية العلوم - جامعة قناة السويس - مصر

٢ - هيئة المواد النووية - مصر

الهدف من البحث إستخلاص أيونات الثوريوم من وسط حامضي بإستخدام تقنية الإستخلاص بالمذيب العضوي بقاعدة شيف المحضرة من تفاعل 10 مللي مول من 2- أمينو فينول مع 10 مللي مول أسيتيل أسيتون وتم توصيف الناتج (AcPh) بإستخدام التحليل العنصري و تحليل الأشعة تحت الحمراء و ¹H-NMR. وتم دراسة العوامل المؤثرة علي فصل وإستخلاص الثوريوم من المحلول القياسي الحامضي بإستخدام الهادة المحضرة وذلك للوصول للظروف المثلي لعملية الإستخلاص وهي تم إذابة قاعدة شيف المحضرة في مخلوط من الكلوروفورم و ثنائي إيثيل إيثير إذابة كاملة بنسبة 3:2، قيمة الأس الهيدروجيني هي 6,5 وحدة و 0,02 مول من قاعدة شيف الذائبة في الكلوروفورم و ثنائي إيثيل إيثير ونسبة الوسط المائي الي الوسط العضوي هي 1:3 لمدة 5 دقائق زمن التلامس عند درجة حرارة الغرفة. وكذلك أجريت عملية الإسترجاع للثوريوم من الوسط العضوي المحمل بالثوريوم للوصول للظروف المثلي وهي 0,5 مول من حامض النيتريك ونسبة الوسط المائي الي الوسط العضوي هي 2:1 في زمن تلامس 7 دقائق في درجة حرارة الغرفة وبالتالي أمكن إستخلاص وإعادة تركيز أيونات الثوريوم بنسبة 96% من محلوله الحامضي.