

Synergistic flotation of U(VI)–alizarin complex with some diamines followed by spectrophotometric determination of U(VI) using 4,4'-diaminophenylmethane

Mohammad Saeid Hosseini*, Heidar Raissi, Hamid Reza Yavari

Department of Chemistry, Faculty of Science, Birjand University, Birjand, P.O. Box 79, Iran

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Abstract

In this work, synergistic flotation of U(VI)–alizarin complex at the presence of some diamine compounds was firstly investigated by the spectrophotometric method. The flotation process was carried out on aliquots of 100 ml of U(VI) solutions containing alizarin and the diamine at pH of 5.00 using *n*-heptane. The floated layer was then dissolved in acetonitrile and its absorbance was measured. Since the synergistic effect of 4,4'-diaminophenylmethane (dapm) was much more than the others, it was used for the determination of U(VI) by this method. Beer's law was obeyed ($\lambda_{\max} = 591 \text{ nm}$) in the range of 5×10^{-7} to $1 \times 10^{-5} \text{ mol l}^{-1}$ with the apparent molar absorptivity of $1.12 \times 10^6 \text{ l mol}^{-1} \text{ cm}^{-1}$, and a correlation coefficient of 0.9991. The detection limit ($n=7$) was $1 \times 10^{-7} \text{ mol l}^{-1}$, and the R.S.D. ($n=7$) obtained for $4 \times 10^{-6} \text{ mol l}^{-1}$ of U(VI) was 2.23%. Except that only a few analogous cations, which could be masked by EDTA, no interference was observed at the presence of various conventional ions, even at high concentrations. The reliability and applicability of the method were confirmed using some geological standard reference materials and spiked synthetic water samples, respectively.

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1. Introduction

Uranium is an important element in view of industry, especially in view of nuclear energy production. Mining, nuclear fuel preparation and waste management require a number of chemical methods for purification, enrichment and its determination. Widely used methods for uranium determination in environmental samples are inductively coupled plasma atomic emission spectrometry [1], inductively coupled plasma-mass spectrometry (ICP-MS) [2,3], neutron activation analysis (NAA), gamma and alpha spectrometry [4–6] and electroanalysis using ion-selective electrode [7–9]. Although, these methods benefit a high considerable sensitivity and favorable detection limits, but except that the last, their application require to rather expensive equipment and undergo a higher running cost. Moreover, utilizing from some methods needs preliminary separation steps, such as extraction and ion-exchange procedures for sample prepara-

tion [10]. Disadvantages associated with these procedures include the use of large volumes of organic solvents and acids resulting in large volumes of generated waste.

Generally, spectrophotometric determinations are widely used to determine of uranium [11–15]. Achievement to more sensitivity and selectivity should be feasible, when complementary techniques, such as liquid or solid phase extraction are coupled with it prior to the measurements [16–19]. Previously, synergistic extraction of uranium with β -diketones assisted with a number of individual agents was reported [20]. The systematic studies concerning on synergistic phenomenon were conducted without following the extraction process [21]. It was found that the synergistic phenomenon occurs in aqueous phase and provides a new possibility for improving the detection limit in determination of RE(III) ions via the fluorescence enhancement of some RE(III) complexes with certain β -diketones using various types of nitrogen-involving neutral bidentate ligands, such as 1,10-phenanthroline (phen) and 2,2'-bipyridine (bpy) [22,23],

One of the interest methods for separation and preconcentration of trace amount of metal ions is flotation spectrophotometry in which the organic solvent, on the contrary of solvent extrac-

* Corresponding author. Tel.: +98 561 2224803; fax: +98 561 2230009.
E-mail address: MSHosseini@birjand.ac.ir (M.S. Hosseini).

tion methods, is not polluted during the performance so as can be used over again [24–28]. In the previous work, the synergistic flotation of RE(III) ions with alizarin at the presence of phen was investigated and found that the absorbance was considerably increased for majority of these ions in acidic media [29]. By considering to those observations, it is expected the selective determination of individual lanthanide or actinide ions can be feasible using the synergistic flotation method. In this work, initially the synergistic flotation behavior of U(VI)–alizarin complex in the presence of certain diamine compounds, as the nitrogen-involving neutral bidentate ligands, was investigated and then the determination of U(VI) was carried out using dapm, which its tendency to enhance the flotation efficiency was obviously considerable in comparison with the others.

2. Experimental

2.1. Material and apparatus

All the reagents were of analytical grade, obtained from Merck, Germany and used without further purification. All of the solutions were prepared with fresh double distilled water. Alizarin solution ($2.0 \times 10^{-3} \text{ mol l}^{-1}$) was prepared by dissolving the required amount of in methanol. The diamine compounds were used as 0.1 mol l^{-1} solutions prepared similarly in methanol. A standard stock $1.0 \times 10^{-3} \text{ mol l}^{-1}$ U(VI) solutions was prepared by dissolving the appropriate amount of its nitrate salt in 50 ml of 2 mol l^{-1} nitric acid and diluting to the mark (250 ml) with water. The working solution with concentration of $1 \times 10^{-5} \text{ mol l}^{-1}$ was prepared by using the stock solution. Ammonium nitrate (0.5 mol l^{-1}), nitric acid (0.25 mol l^{-1}) and ammonia (0.25 mol l^{-1}) solutions were used to adjust the pH and ionic strength of the working solutions to the desired values.

A Shimadzu model 160 A double beam spectrophotometer and a Corning model 130 pH-meter were used for absorption and pH measurements, respectively.

2.2. Procedure

To a separating funnel containing 100 ml of $4.0 \times 10^{-6} \text{ mol l}^{-1}$ U(VI) solution, 1.2 ml of the diamine compound, 2 ml of the ammonium nitrate and 0.1 ml of the alizarin solutions were added and its pH was adjusted to the desired value using ammonia. After addition of 10 ml *n*-heptane, the funnel was stoppered and vigorously shaken for 20 min, then left to rest for 30 min to appear a floated layer at the interface of aqueous/organic layers. By slowly opening the stopcock of the funnel, both the aqueous/organic layers were removed and the floated layer that was adhering to the inner wall was dissolved in 5 ml of acetonitrile, then its absorbance was measured at the maximum wavelength (591 nm) against a reagent blank, prepared by the same manner.

In order to determine of U(VI) in the solutions, the flotation process was carried out similarly on aliquots of the solutions (100 ml) containing up to 0.238 mg U(VI) ion at pH of 5.00 and at the presence of $1.0 \times 10^{-5} \text{ mol l}^{-1}$ of dapm as the synergistic agent.

3. Results and discussion

3.1. Flotation process

Initially, a number of parameters were investigated that influence on performance of flotation the binary U(VI)–alizarin complex formed in acidic media. Before that, the effect of pH was examined on efficiency of the flotation process over a range of 3.00–6.00 by adjusting the pH of the solution just before the organic phase addition. As shown in Table 1, the absorbance measurements represent a maximum at pH of 5; hence, the future studies to optimize the other parameters were carried out at this pH. Evidently, by increasing the pH, the flotation efficiency should be increased owing to increase the dissociated form of alizarin, Hz^- , but the absorbance decreasing at the pHs more than 5 is obviously attributed to change the structure of the complex.

The effect of alizarin concentration was studied over a range of 4.0×10^{-6} to $6.0 \times 10^{-5} \text{ mol l}^{-1}$. Fig. 1 indicates a maximum absorbance at the concentrations more than $1.4 \times 10^{-5} \text{ mol l}^{-1}$. Since, increasing of the concentration of

Table 1

The absorption values of the floated U(VI)–alizarin complex after dissolving in 5 ml of acetonitrile, obtained from the solutions with various pH values

pH	λ_{max}	Absorbance ^a
3.0	–	–
3.5	591	0.065 (± 0.008)
4.0	591	0.252 (± 0.007)
4.5	591	0.349 (± 0.006)
5.0	591	0.383 (± 0.010)
5.5	591	0.356 (± 0.016)
6.0	591	0.330 (± 0.009)
6.5	586	0.321 (± 0.007)
7.0	581	0.316 (± 0.006)
7.5	581	0.308 (± 0.009)
8.0	581	0.301 (± 0.012)

^a The results are reported as the average values from five samples measurements.

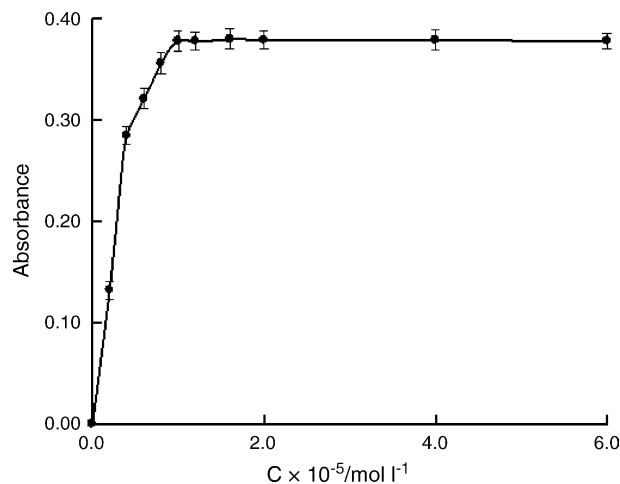


Fig. 1. Effect of alizarin concentration on the flotation process. The pH of the solutions was adjusted to 5.00; $C_{\text{U(VI)}} = 4 \times 10^{-6} \text{ mol l}^{-1}$ and ionic strength is 0.01 mol l^{-1} in all of the examinations.

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