



Eco-friendly environmental trace analysis of thorium using a new supramolecular solvent-based liquid-liquid microextraction combined with spectrophotometry



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ABSTRACT

A novel green supramolecular solvent-based liquid-liquid microextraction (SsLLME) method was developed and validated for preconcentration of thorium(IV) in various environmental samples. The presented preconcentration method was based on using 1-decanol/THF as supramolecular solvent phase and quinalizarin as a chelating agent. Thorium(IV)-quinalizarin hydrophobic complex was obtained at pH 6.0, extracted into the supramolecular solvent phase, centrifuged and then the absorbance was measured spectrophotometrically at 590 nm. The impact of some variables on the efficiency of microextraction; pH, amount of quinalizarin, and supramolecular solvent type, ratio and volume, ultrasonication and centrifugation time, sample volume and influence of matrix ions; was optimized. Linearity ranged from 2.0 to 100 $\mu\text{g L}^{-1}$ with a limit of detection (LOD) of 0.40 $\mu\text{g L}^{-1}$. The preconcentration factor (PF) was found to be 40. The precision of the method was confirmed with its low RSD% (1.4). The developed method was applied to the analysis of real water and soil samples. The validity was checked with certified reference materials such as IAEA Lake Sediment SL-1 and IAEA-soil-7.

1. Introduction

Thorium [Th(IV)] is a naturally-occurring radioactive element present in low concentrations in the environment, nuclear fission products and the earth's crust. It has a wide application in industry, agriculture, and it is also used for production of electricity from nuclear energy in power plants. However, it has toxic effects and its compounds are hazardous and potentially carcinogenic causing irreversible or progressive renal injury [1, 2]. In various environmental, biological and geological samples, the development and application of a new, sensitive and accurate method for preconcentration, separation and determination of thorium at trace levels is of particular significance and interest [3].

Various analytical techniques were used for quantitation of Th(IV) such as optical sensor [4], inductively coupled plasma mass spectrometry (ICP-MS) [5, 6], spectrophotometry [7, 8], neutron activation analysis [9], resonance light scattering (RLS) spectra, second-order scattering (SOS) spectra and frequency-doubling scattering (FDS) spectra [10], Fourier transform infrared spectroscopy (FTIR), proton nuclear magnetic resonance (¹H NMR), and scanning electron microscopy (SEM) [11]. Several popular techniques were also reported for

separation and preconcentration of Th(IV) at trace levels including liquid-liquid [12–15] or solid-phase extraction [1, 2, 16–19], cloud point extraction [20–22] and flotation [23, 24]. These techniques [1, 2, 4–9, 12–24] have some disadvantages. They are time-consuming and labor-intensive. In addition, they have unsatisfactory enrichment factors, consume large amounts of organic solvents and require complicated sample-pretreatment steps.

Supramolecular solvent-based microextraction technique (SsLLME) is very important for the isolation and preconcentration of trace species from environmental samples prior to their determination because of its simplicity, fastness, accuracy, high preconcentration factor (PF), in addition to low cost, less consumption of all chemical reagents and being an environmental friendly technique [25–29].

Supramolecular (SM) solvents are nano-structures of water-immiscible liquids consisting of amphiphilic aggregates that are dispersed in a continuous phase and they are prepared through two steps of self-aggregation of the amphiphile. In the first step, the amphiphile molecules self-aggregate at a concentration that is above the critical aggregation concentration to form a reverse micelle. In the second step, the formed nano-structured aggregates undergo further aggregation to

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produce a water-immiscible phase by changing different agents including pH, solvent and amount of chelating agent. SM solvents can provide different kinds of interactions with the organic compound or with the metals-ligand complexes such as hydrogen bonding, hydrophobic interaction and dispersion forces. These interactions are essential for both increasing efficiency of analytes extraction from aqueous phase to SM solvent phase and decreasing the extraction time [30–35].

Quinalizarin is a chromogenic chelating reagent for trace metal ions. Chemically, it is 1,2,5,8-tetrahydroxyanthracene-9,10-dione. It was used by our research group for the separation and preconcentration of Cd(II), Ni(II), Cu(II), Zn(II) and Pb(II) on multiwalled carbon nanotubes. According to the literature survey, it has never been used in SsLLME studies.

The aim of the present work was to develop a novel SsLLME technique utilizing quinalizarin together with 1-decanol/THF SM solvents for the preconcentration and separation of Th(IV) in environmental samples followed by its determination spectrophotometrically.

2. Material and methods

2.1. Apparatus

For measuring the absorbance, a Varian UV–Vis spectrophotometer with a 1.0 cm quartz cell was used (Cary 100 Conc., Australia). A Hanna pH-meter (Portugal, HI:9321) equipped with a glass-calomel electrode was used for measurement of pH-values.

ALC PK 130 centrifuge (UK) was used for phase separation. Vortex

mixer (*i* S wix VT, Neuation Technologies Pvt. Ltd., India) was used to mix thoroughly the sample solutions. An ultrasonic water bath (Bandelin Sonorex Degitic, DT-255H, Berlin, Germany) was used for production of SM solvent and cloudy solution. All laboratory glass or plastic wares were kept in HNO₃ solution (10% v/v) for at least 12 h, then rinsed and cleaned with bidistilled water obtained from a Milli-Q purification device (Millipore, USA). All stock standard solutions were stored at 4 °C.

2.2. Materials and reagents

All reagents and chemicals were of analytical grade. All solutions were prepared in bidistilled water. Th(NO₃)₄·4H₂O was purchased from (E-Merck, Germany). Ethanol was obtained from BDH. A stock solution; containing 1000 mg L⁻¹ Th(IV); was prepared by dissolving Th (NO₃)₄·4H₂O in bidistilled water with the aid of few drops of nitric acid and then standardized applying the gravimetric iodide method. Preparation of the working standard solutions was done daily by further dilution of the metal stock solution. Quinalizarin, (Sigma-Aldrich, St. Louis, MO, USA), 5.0 × 10⁻⁴ mol L⁻¹ solution was prepared by dissolving 0.014 g in 100 mL of ethanol. A series of buffer solutions including acetate buffer solutions (pH 4.0–6.0), phosphate buffer solution (pH 7.0) and ammonical buffer solutions (pH 8.0–10.0) was used to adjust the pH value of the solution [36]. SM extraction solvents (1-decanol, undecanol) and THF were purchased from Merck (Darmstadt, Germany), while decanoic acid was supplied by Sigma-Aldrich (St. Louis, MO, USA). H₂O₂, 30% and 1.0% (v/v) and HNO₃, 65% and 1.0%

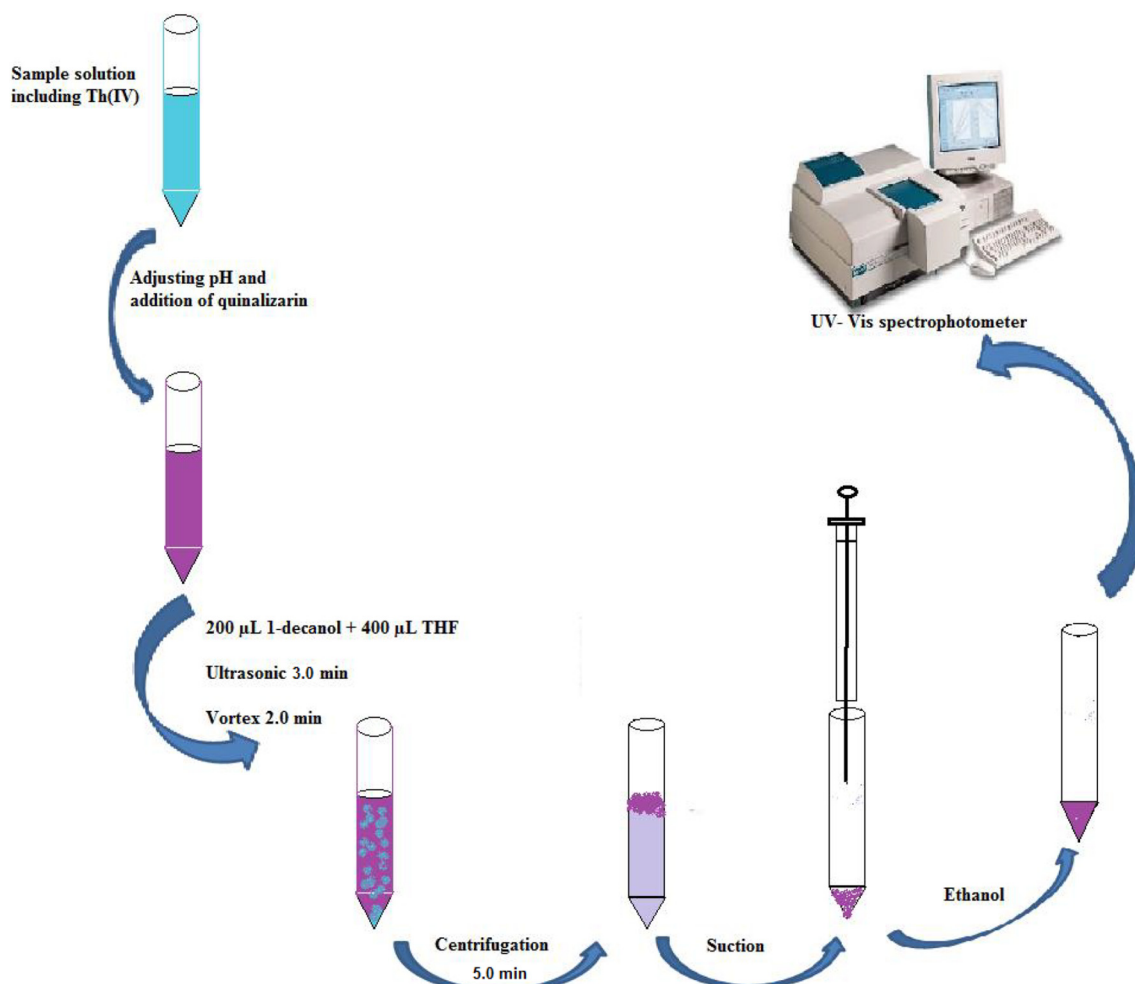


Fig. 1. The procedure of the supramolecular solvent-based microextraction technique.

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