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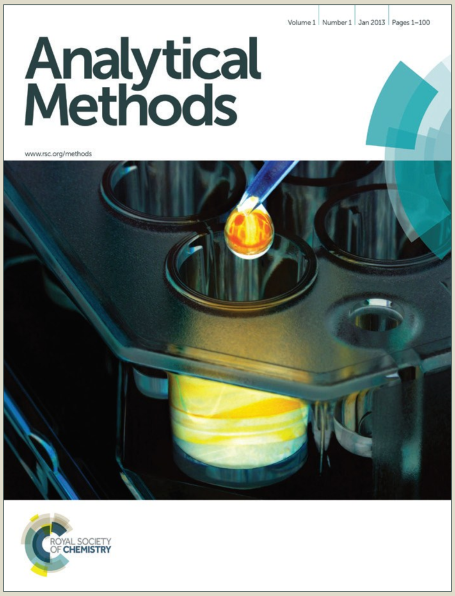

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Switchable Solvent Based Liquid Phase Microextraction of Uranium in Environmental Samples: A Green Approach

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Abstract

This paper described a new and green approach for the preconcentration and separation of U(VI) by switchable solvent based liquid phase microextraction (SPs-LPME) and its Uv-Vis spectrophotometric determination. 1-(2-pyridylazo)-2-naphthol (PAN) complexing agent was used. Conversion features of triethylamine (TEA) and protonated triethylamine bicarbonate (P-TEA-BC) as green and cheap switchable solvent pair was used in the presented work. Protonated triethylamine bicarbonate (P-TEA-BC) as polar form of switchable polarity solvent (SPs) were successfully synthesized from triethylamine (TEA) via proton transfer reaction and used for microextraction of hydrophobic U(VI)-PAN complex at pH 9.0. The relative standard deviation was 2.5 % for five repeated determinations of containing 6 µg L^{-1} of U(VI). The limit of detection (LOD), the limit of quantification and enhancement factor were 0.3 µg L^{-1}, 1.0 µg L^{-1} and 40, respectively. The accuracy of the method was evaluated by analyzing certified reference materials and addition-recovery tests. The method was successfully applied to determination of uranium in water, sediment, soil and rock samples.

Key Words: Uranium, Switchable solvent, microextraction, spectrophotometry, 1-(2-pyridylazo)-2-naphthol.

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1. Introduction

Uranium, a naturally occurring radioactive element on earth [1], has an important role in our daily life and the range of dangerous health problems can be caused by uranium is ranging from cancer to death [2-4]. Its potential for exposure has been increased by human activities such as mining, nuclear facilities and accidental releases [5-7]. Soil, rock, water and plant samples include low concentration of uranium and its compounds [8, 9]. Hence, we need to develop sensitive analytical methods for determining the low concentration of uranium in environmental samples.

Several expensive and difficult operated analytical methods, such as inductively coupled plasma mass spectrometry [10], adsorptive stripping voltammetry [11], neutron activation analysis [12], fluorimetry [13] laser-induced fluorescence [14] and x-ray fluorescence [15] have been used to determination of uranium.

In contrast, spectrophotometry is the most used simple and cheap technique. Spectrophotometry is distinguished by its low cost and the simplicity of its apparatus.[16-18] However, due to matrix effect and lower concentration of uranium in environmental samples than detection limit of spectrophotometry, development of sensitive, cheap and effective separation/preconcentration methods is a challenging task [19, 20].

The liquid–liquid extraction (LLE), cloud point extraction (CPE) and solid-phase extraction (SPE) are the most commonly used methods for the separation and preconcentration of uranium [21-23]. However, these methods suffers from the disadvantages of being time-consuming, expensive and requiring large and complex equipment, large volumes of both samples and harmful organic solvents which are often hazardous due to their high vapor pressure and produce secondary wastes along procedure [24-26].

Nowadays, in order to eliminate or decrease these disadvantages, a special attention has been focused on the development of green analytical methods. Novel solvent and equipment systems and miniaturized liquid phase sample pretreatment techniques ”liquid phase microextraction techniques” have been used to extend the concepts and practices of green chemistry [27, 28]. Liquid phase microextraction methods can be applied as different application mode such as dispersive liquid–liquid microextraction (DLLME) [29], single-drop microextraction (SDME) [30], supramolecular solvent based microextraction (Ss-ME) [31], hollow fiber liquid phase microextraction (HF-LPME) [32] and ionic liquid based microextraction (IL-ME) [25].

However, there are some drawbacks in these methods. The microextraction techniques consist of multistep; require different polarity organic solvents for each step to extract analyte components from matrix components, with different solubility’s in a sample. The use of different polarity organic solvents for each step of a microextraction technique render these techniques expensive, time consuming and harmful to worker due to use of these organic solvents in each step. When considering all of these disadvantages, the scientists have focused on the selection of the ‘‘right’’ solvent not only for an effective analyte extraction but also for the elimination of these disadvantages mentioned above. Imagine a solvent that can transform itself, changing its polarity properties to meet multiple applications. Such kinds of solvents are not more a dram but such solvents are a reality in the field of science.

A new generation solvents called switchable-polarity solvents (SPs), a unique class of solvents, has received attention as green solvents for a wide variety of chemical applications such as extraction and purification of compounds, chemical synthesis and catalysis reaction due to their reversibly change physical properties abruptly [33-35]. Switchable solvents are liquids that can be reversibly converted from one form to another, where the two forms differ in their physical properties. A switchable polarity solvent (SPs) is a solvent that is water-miscible (formation of hydrophilic form) in the presence of an atmosphere of CO_2 at 1 bar but separates from water when CO_2 is removed (formation of hydrophobic form) by addition of acids and bases, bubbling air, argon, nitrogen or other inert gas under heating. Polarity switching feature of these solvents provide simple, reliable and fast extraction method for scientists by decrease of the number extraction step and eliminate toxic solvents [33-37].

The aim of our work was to develop a novel, environmentally friendly and cheap homogeneous liquid phase microextraction application based on switchable solvent formation for the separation, preconcentration and of uranium in water and soil samples prior to the spectrophotometric detection. Switching feature of triethyl amine (TEA)-protonated triethylamine bicarbonate (TEA-BC) solvent pair was used for microextraction of uranium. Microextraction and spectrophotometric determination of uranium was conducted after chelation of uranium with 1-(2-pyridylazo)- 2-naphthol (PAN) as chelating agent.

2. Experimental

2.1. Reagents and standards

Ultrapure water obtained from a MilliQ Direct-16 purification system (18.2 MΩ cm, Millipore) and analytical grade reagents were used throughout. CO_2 (Dry ice) was provided by Ates Company (Kahramanmaras, Turkey). Analytical reagent grade standards for uranium (1000 mg L^{-1}) was provided by (High Purity Standards, Charleston, SC, USA) which was further diluted in ultrapure water (18.2 MΩ cm^{-1}, Millipore) for preparation of various standard solutions of U(VI).

0.01 % of 1-(2-Pyridylazo)-2-naphthol (PAN) solution was prepared by dissolving 0.01 g of PAN purchased from E. Merck (Darmstadt, Germany) in 100 mL of methanol (Sigma Aldrich).

Following combination of acid/salts and solutions were used to acquire the desired pH of solutions: phosphate buffer solution (pH 2–4, sodium dihydrogen phosphate/phosphoric acid), phosphate buffer solution (pH 5-7 sodium dihydrogen phosphate/disodium hydrogen phosphate), acetate buffer solution (pH 8 and 10 acetic acid/ammonium acetate) and phosphate buffer solution (pH 9 sodium phosphate).

Triethylamine, N,N dimethyl cyclohexylamine, hydrochloric acid (HCl), nitric acid (HNO_3), methanol (CH_3OH) and acetone (C_3H_6O) were also provided by E. Merck (Darmstadt, Germany). TMDA- 64.2 Lake Ontario water and HR-1 (Harbour river sediment) (National Water Research Institute, Ontario, Canada) and GBW07424 (GSS-10) soil (Institute of Geophysical and Geochemical Exploration, Langfang, China) were used as water certified reference material for trace analysis.

2.2. Instruments

A Hitachi 150-20 spectrophotometer with quartz micro–cell with path length of 10 mm and a volume of 700 µL was used for absorbance measurements. A pH-meter with, Nel pH 900 (Ankara-Turkey) Model glass-electrode was used for pH adjustment of all the sample solutions. Vortex mixer (Wiggen Hauser, Malaysia) was used for complete mixing of sample solution. Enhanced phase separation was achieved by using centrifuge with centrifugal vials (ALC PK 120 Model, Buckinghamshire, England).

2.3. The synthesis of hydrophilic form of switchable solvent

Protonated triethylamine bicarbonate and protonated N,N dimethyl cyclohexylamine bicarbonate forms of two different switchable extraction solvent pair were checked as extraction solvent. Protonated triethylamine bicarbonate (or protonated N,N dimethyl cyclohexylamine bicarbonate) was synthesized according to the procedure in the literature:[38, 39] 200 mL of triethylamine (or N,N dimethyl cyclohexylamine) and 200 mL of ultrapure water were mixed in a 1 L glass beaker on magnetic stirrer and a two phase system formed. After, Dry ice (~ 20 g) as CO_2 supplier was added gradually in the beaker, dissolution of CO_2 was completed and amine phase became cloudy. The addition of dry ice was repeated until obtain protonated triethylamine bicarbonate (or protonated N,N dimethyl cyclohexylamine bicarbonate) solution. Afterward, the mixture was stirred for 2 h at room temperature to ensure the complete protonation of triethylamine (or N,N dimethyl cyclohexylamine).

2.4. General procedure

The switchable solvent based liquid phase microextraction (SPs-LPME) for separation and preconcentration of uranium was carried out at 10 mL sample solution including 25 μg L^{-1} U (VI), 2 ml of pH 9 buffer solution and 20 μg of PAN in 50 ml of centrifuge tube. After 2 minutes for the hydrophobic U(VI)-PAN complex formation, 1.0 mL of protonated triethylamine bicarbonate (P-TEA-BC) synthesized as extraction solution was added in the solution by using an injector and a homogenous solution was obtained. Then, in order to convert hydrophilic protonated triethylamine bicarbonate to hydrophobic triethylamine (microsized extraction phase), 1.5 mL of 10 M NaOH solution was quickly added in the solution and a cloudy solution formed in the centrifuge tube. The solution was subjected to vortex having vortex speed of 40 × 100 rpm for 1 min to ensure the formation of TEA phase and extraction of U(VI)-PAN complex from water phase to in the fine droplets of TEA. For complete phase separation, the tube was put inside the centrifuge for 7 min. The aqueous phase was separated and discarded and the extraction phase was diluted to 0.5 mL using methanol. The concentration of the uranium in last volume was measured as soon as possible to avoid evaporation of methanol at 550 nm by using UV-Visible spectrophotometer. Schematic representation of the developed SPs-LPME steps was presented Figure 1.

2.5. Application to environmental samples

The proposed SPs-LPME method was applied to sea water (Marmara sea, Turkey), soil samples taken from Kayseri, Turkey and rock samples obtained from Erzincan, Turkey, while the accuracy of the method was certified by applying the method to TMDA-64.2 environmental water, GBW07424 (GSS-10) soil and HR-1 Harbour river sediment certified reference materials. The sea water sample were passes through a membrane with 0.45 µm size pore provided by (Millipore Corporation, Bedford, MA, USA) to get rid of suspended particles.

The soil and rock samples collected were dried at 80 ^{o}C in an oven and then, they were grinded and sieved to obtain homogenized small samples. Aqua-regia was used for wet digestion of soil, rock samples and certified reference materials, in which 24 mL of aqua-regia were added to each beaker having 0.25 g of soil samples (3 replicate) and 0.5 g of certified reference materials (3 replicate) each. All the samples were kept at room temperature for initial 30 min and then on hot plate at 95°C till dry residue were obtained. The obtained residues were again subjected to wet digestion method mention above. Final residues obtained were dissolved in 5 mL of distilled water and filtered through a Millipore blue filter paper. The clear solutions of the samples were subjected to the proposed SPs-LPME.

3. Results and Discussion

3.1. Effect of pH

In order to extract metal ions from donor phase to extraction phase by LPME, they must be converted into a hydrophobic complex in donor phase [31]. Hence, the pH value of the donor phase has a key role for the formation of a stable hydrophobic U(VI)-PAN complex and effective extraction of this complex. Hence, the effect of sample solution pH on the extraction of uranium was studied by varying the pH within the range of 6–12 and the results were shown in Figure 2. It could be seen that the recovery of uranium increased at the range of pH 6–10. When the pH further increased, the recovery of uranium decreased markedly. The quantitative results were obtained between pH 8 and pH 10. Based on these results, a pH of 9 was selected for subsequent works.

3.2. Selection of switchable extraction solvent

Selecting an effective extraction solvent is crucial in switchable solvent based extraction methods. For selection of the suitable extraction solvent, some properties must be considered such as (a) extraction capability of target compounds, (b) can be converted between the two forms by the addition or removal of CO_2 from the solvent system, (c) lower density than water for hydrophobic form of switchable solvent (d) high solubility in water for hydrophilic form and low solubility in water for hydrophobic form of switchable solvent that it can form a stable two-phase system.[33-35] Based on these criteria, in this study, triethylamine/protonated triethylamine bicarbonate and *N,N*-dimethyl-cyclohexylamine/ protonated *N,N*-dimethyl cyclohexylamine bicarbonate switchable extraction solvent pairs were checked for the extraction of hydrophobic U(VI)-PAN complex from sample solution. The recoveries obtained for triethylamine/protonated triethylamine bicarbonate and *N,N*-dimethyl-cyclohexylamine/protonated *N,N*-dimethyl-cyclohexylamine bicarbonate were 100±1 and 86±3, respectively. Regarding the recoveries, the triethylamine/protonated triethylamine bicarbonate (TEA/P-TEA-BC) switchable solvent pair was the best and selected as extraction solvent for subsequent work.

3.3. Selection of phase transition trigger

TEA (hydrophobic form of SPs) and P-TEA-BC (hydrophilic form of SPs) can be switched between the two forms by the addition or removal of CO_2 from the system. In order to convert polar P-TEA-BC (for deprotonation of protonated triethylamine bicarbonate) to micro volume apolar TEA phase, several an external stimulus, including physical and chemical methods such as addition of sodium hydroxide or hyrdochloric acid solution into the solution, passing of inert gas (Argon) in the solution, heating of the solution were checked. No formation of cloudy solution or phase separation was obtained by using of hydrochloric acid solution, heating or passing of inert gas (Argon) ways. Among all the procedures, the best result was obtained by using of a 10 M sodium hydroxide solution due to its efficiency. Because sodium hydroxide react with protonated amine and tear off hydrogen bond on amine and this reaction cause to formation of triethylamine. Hence, 10 M sodium hydroxide solution was used as phase transition trigger for subsequent work.

3.4. Effect of extraction solvent volume

After choosing TEA/P-TEA-BC switchable solvent pair as the extraction solvent system, it was necessary to optimize the P-TEA-BC volume, which is a miscible water form of switchable solvent system and its this feature provide the formation of homogenous extraction system for analytes. The effect of the volume of the protonated triethylamine bicarbonate as the extraction solvent on the recovery was investigated by fixing the volume of the 10 M NaOH at 1.5 mL. Figure 3 indicates that the recovery increased by increasing the volume of the P-TEA-BC to 1.0 mL and then remained quantitative by further increasing of its volume between 1.0 and 2.0 mL. Hence, 1.0 mL of P-TEA-BC was used in further experiments.

3.5. Effect of NaOH

The removing of CO_2 from polar protonated triethylamine bicarbonate causes the two phases including water and apolar TEA phases. In order to convert the P-TEA-BC form of switchable solvent to hydrophobic TEA phase and, also extract the U(VI)-PAN complex sample solution to TEA phase, 10 M of NaOH as phase transition trigger was used. The influence of the volume of 10 M NaOH was also studied and the results showed that the quantitative recoveries for uranium were obtained between 1.5 and 2.0 mL of 10 M NaOH. In this study, a 1.5 mL of 10 M NaOH was used for subsequent experiments.

3.6. Effect of complexing agent amount

PAN has been used as chromogenic reagent to form a highly stable uranium complex around basic pH [40] and this U(VI)-PAN complex can be extracted easily in extraction phase. The effect of the PAN amount on the extraction efficiency of uranium was studied, and the results were shown in Figure 4. The extraction recovery increased with an increase in the amount of PAN; more than 95 % of U(VI) was extracted with the use of more than 15 µg of PAN in sample solution. In this method, 20 µg of PAN was used.

3.7. Effect of sample volume

High preconcentration factor can be achieved, if the highest possible volume of sample is determined to which the proposed method can be successfully applied. Thus, the effect of the sample volume on recoveries of uranium was also studied. For this purpose, the develop SPs-LPME method was applied to different sample volume ranging from 5 mL to 25 mL as shown from the Figure 5. Quantitative recoveries for analyte could be obtained by using sample volume below 20 mL. Therefore, highest preconcentration factor of 40 could be achieved by this method by using 20 mL of sample volume with 0.5 mL of final volume.

3.8. Matrix effect

Due to the complex matrix nature of samples like soil, ore and water, preconcentration methods are greatly influenced by various coexisting ions present in the matrices of these samples with analyte metals.[40-44] The effect of coexisting ions on % recovery of uranium is of immense importance. In order to investigate the effects of matrix ions on SPs-LPME and spectrophotometric determination, the proposed microextraction method was carried in the presence of some interfering ions which are coexisting with analyte metal in samples matrices. The results given in the Table 1 show that there are no significant interferences by these interfering ions even in the presence of high concentration. This shows that the method is highly selective and free of interferences.

3.9. Analytical performance and comparison to other methods

All the analytical parameters were determined under optimized experimental condition. The detection limits of the analyte, which was calculated as 3 times the signal/slope (slope of calibration curve), was 0.3 μg L^{-1} for U(VI). The quantification limits, which was calculated as 10 times the signal/slope (slope of calibration curve), was and 1.0 μg L^{-1} for U(VI). The relative standard deviations (RSD, %), evaluated using the results of the analysis of five replicates containing 6 μg L^{-1} of U(VI) was 2.5. Enhancement factor (EF) was calculated (EF = ratio of the U(VI) concentration in the extraction phase to the initial concentration of U(VI) in the sample solution) was 40. Consumptive index (CIn) [45, 46] is another effective way to evaluate the analytical performance of preconcentration system. CIn, which calculated as (Cin = ratio of sample volume (in milliliters) to enhancement factor) was 0.5 mL.

A comparison of the developed SPs-LPME method with other reported preconcentration methods for uranium is given in Table 2. In comparison with the other reported methods, the SPs-LPME shows generally and comparatively low detection limit, low relative standard deviation and a high preconcentration/enhancement factor for uranium with some exceptions in a short time of extraction.

3.10. Analytical validation of the proposed method

The accuracy of the developed method was evaluated by applying the method to TMDA-64.2 environmental water, GBW07424 (GSS-10) soil and HR-1 Harbour river certified reference materials and the analytical results (% recovery with the standard deviations) are shown in Table 3. It was found that there is a close agreement between the results obtained and the certified results.

The accuracy of the develop method, was also determined by applying the method to the analysis of spiked (sea water and soil samples) and the results are given in Table 4. There is no significant difference between the added and recovered amount of U(VI). It can be concluded from results obtain for analyzing of certified reference materials and addition-recovery studies that the developed SPs-LPME method is accurate and can be successfully applied for the preconcentration of U(VI) at trace level and without effecting by complex matrix nature of the samples.

The proposed microextraction method was also applied for determination of uranium content of soil (Kayseri, Turkey) and rock (Erzincan, Turkey). The results are given in Table 5.

4. Conclusions

A method of switchable solvent based liquid phase microextraction (SPs-LPME) combined with spectrophotometric determination has been developed for the cheap, simple and sensitive determination of uranium in water, soil and sediment samples by considering the requirements of green chemistry. The novel SPs-LPME method for uranium microextraction and determination provides several advantages compared with the other preconcentration and detection methods (a) lower consumption of organic solvents and consequently less organic waste; (b) no expensive and complex chemicals, equipments and instrumentation is needed for availability of the method; (c) shorter extraction and analysis times; (d) Moreover, the performance of this method in the extraction and determination of uranium from different samples with various matrices is excellent.

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Figure captions

Figure 1. Schematic representation of the developed SPs-LPME steps

Figure 2. Effect of pH on the recovery of U(VI). (Experimental conditions; Concentration of U(VI): 0.4 mg L^{-1}, P-TEA-BC volume: 1.0 mL, amount of PAN: 20 µg, volume of the 10 M NaOH solution: 1.5 mL, sample volume: 10 mL, N=3).

Figure 3. Effect of extraction solvent volume on the recovery of U(VI). (Experimental conditions; pH: 9, Concentration of U(VI): 0.4 mg L^{-1}, amount of PAN: 20 µg, volume of the 10 M NaOH solution: 1.5 mL, sample volume: 10 mL, N=3).

Figure 4. Effect of complexing agent amount on the recovery of U(VI). (Experimental conditions; pH: 9, Concentration of U(VI): 0.4 mg L^{-1}, P-TEA-BC volume: 1.0 mL, amount of PAN: 20 µg, volume of the 10 M NaOH solution: 1.5 mL, sample volume: 10 mL, N=3).

Figure 5. Effect of sample volume on the recovery of U(VI). (Experimental conditions; pH: 9, Concentration of U(VI): 0.4 mg L^{-1}, P-TEA-BC volume: 1.0 mL, amount of PAN: 20 µg, volume of the 10 M NaOH solution: 1.5 mL, N=3).

Table 1. Effect of some matrix ions on extraction efficiency of U(VI) (Experimental conditions; pH: 9, P-TEA-BC volume: 1.0 mL, amount of PAN: 20 µg, volume of the 10 M NaOH solution: 1.5 mL, sample volume 10 mL, N=3).

Matrix ion	Amount added (mg L^{-1})	Added as	% Recovery
Na^+	2500	$NaNO_3$	98±5 [a]
K^+	1000	KCl	101±1
Ca^{2+}	100	$Ca(NO_3)_2.4H_2O$	102±3
Mg^{2+}	1000	$Mg(NO_3)_2.6H_2O$	94±2
Fe^{2+}	1	$Fe(NO_3)_3.4H_20$	96±0
Cd^{2+}	1	$Cd(NO_3)_2.\ 6H_2O$	98±3
Pb^{2+}	1	$Pb(NO_3).6H_2O$	96±0
Ni^{2+}	1	$Ni(NO_3)_2.\ 6H_2O$	97±2
Co^{2+}	1	$Co(NO_3)_2.6H_2O$	101±1
SO_4^{2-}	500	Na_2SO_4	97±0
CO_3^{2-}	500	Na_2CO_3	99±5
F^-	500	NaF	100±2
Cl^-	1000	KCl	101±1

[a] Mean ± standard deviation.

Table 2. Comparison of the SPs-LPME method with other methods for the determination of uranium in environmental samples

Method	Instrument	PF/EF[a]	Detection Limit μg L^{-1}	Sample	Ref.
Microwave-assisted of dispersive liquid–liquid microextraction	Spectrophotometry	135	6.7	Water	5
Solid phase extraction	Spectrophotometry	-	2.0	Soil, Sediment	47
Solid phase extraction	Spectrophotometry	500	2.0	Water	48
ionic liquid based liquid liquid microextraction	Spectrophotometry	448	2.4	Water	49
Dispersive liquid–liquid Microextraction	ICP-OES	11	2.0	Water	50
Dispersive liquid–liquid Microextraction	ICP-MS	25	0.03	Water	50
Solidified floating organic drop microextraction	Spectrophotometry	125	0.1	Water	51
Ionic liquid phase microextraction	Spectrophotometry	50	0.87	Ore	52
Cloud point extraction	Spectrophotometry	50.4	0.15	Water	53
Solid phase extraction	ICP-MS	30	0.0063	Environment	54
Switchable solvent based liquid phase microextraction	Spectrophotometry	40	0.3	Water, rock, soil	This study

[a] Preconcentration factor/enhancement factor

Table 3. The analysis results of certified reference materials (Experimental conditions; pH: 9, P-TEA-BC volume: 1.0 mL, amount of PAN: 20 µg, volume of the 10 M NaOH solution: 1.5 mL, sample volume 10 mL, N=3).

Certified Reference Material	**Certified value, µg g^{-1}**	**Found value, µg g^{-1}**	**Recovery, %**
GBW07424 (GSS-10) Soil	2.25	2.20±0.10 [a]	98
HR-1 Harbour River Sediment	1.99 [b]	1.95±0.10	98
Certified Reference Material	**Certified value, µg L^{-1}**	**Found value, µg L^{-1}**	**Recovery, %**
TMDA-64.2 Environmental water	142	142±2	100

[a] Mean ± standard deviation.

[b] Non-certified value.

Table 4. Addition and recovery test for SPs-LPME of uranium in sea water and soil samples (Experimental conditions; pH: 9, P-TEA-BC volume: 1.0 mL, amount of PAN: 20 µg, volume of the 10 M NaOH solution: 1.5 mL, sample volume 10 mL, N=3).

sample	Added, µg	Found, µg	Recovery, %
Soil	0	0.09±0.01 [a]	-
	3	3.17±0.02	103
	6	5.93±0.01	97
Sea Water	0	0.08±0.01	-
	3	3.42±0.07	102
	4	4.19±0.02	103

[a] Mean ± Standard deviation.

Table 5. The application of the suggested SPs-LPME method for determination of uranium in soil and rock samples (Experimental conditions; pH: 9, P-TEA-BC volume: 1.0 mL, amount of PAN: 20 µg, volume of the 10 M NaOH solution: 1.5 mL, sample volume 10 mL, N=3).

Samples	**Found, mg kg^{-1}**
Soil-I	3.91±0.20
Soil-II	3.83±0.15
Soil-III	2.05±0.14
Rock-I	2.60±0.06
Rock-II	BDL [a]

[a]: Below the detection limit

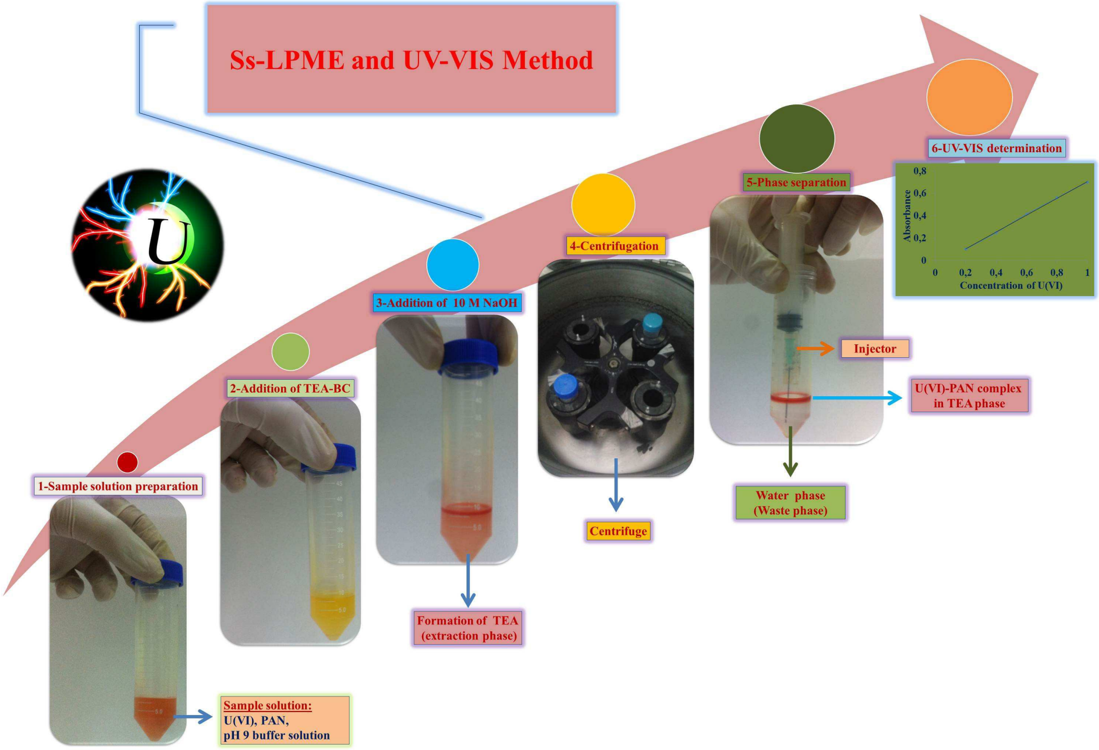
Ss-LPME and UV-VIS Method
U
1-Sample solution preparation
Sample solution:
U(VI), PAN,
pH 9 buffer solution
2-Addition of TEA-BC
3-Addition of 10 M NaOH
Formation of TEA
(extraction phase)
4-Centrifugation
Centrifuge
5-Phase separation
Injector
U(VI)-PAN complex
in TEA phase
Water phase
(Waste phase)
6-UV-VIS determination
Absorbance
0,8
0,6
0,4
0,2
0
0
0,2
0,4
0,6
0,8
1
Concentration of U(VI)

Figure 1.

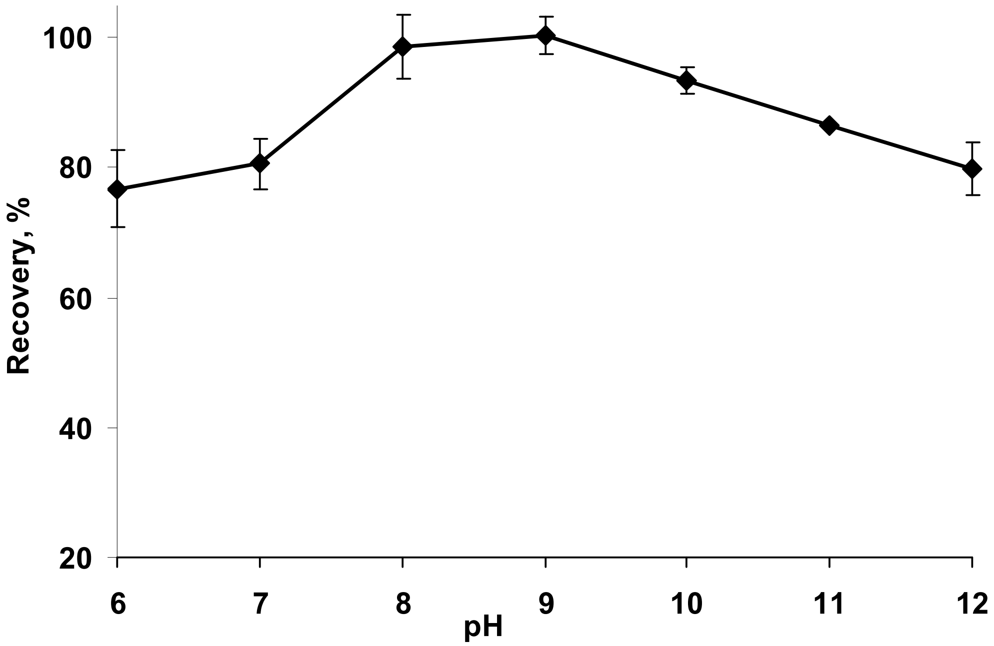

Figure 2.

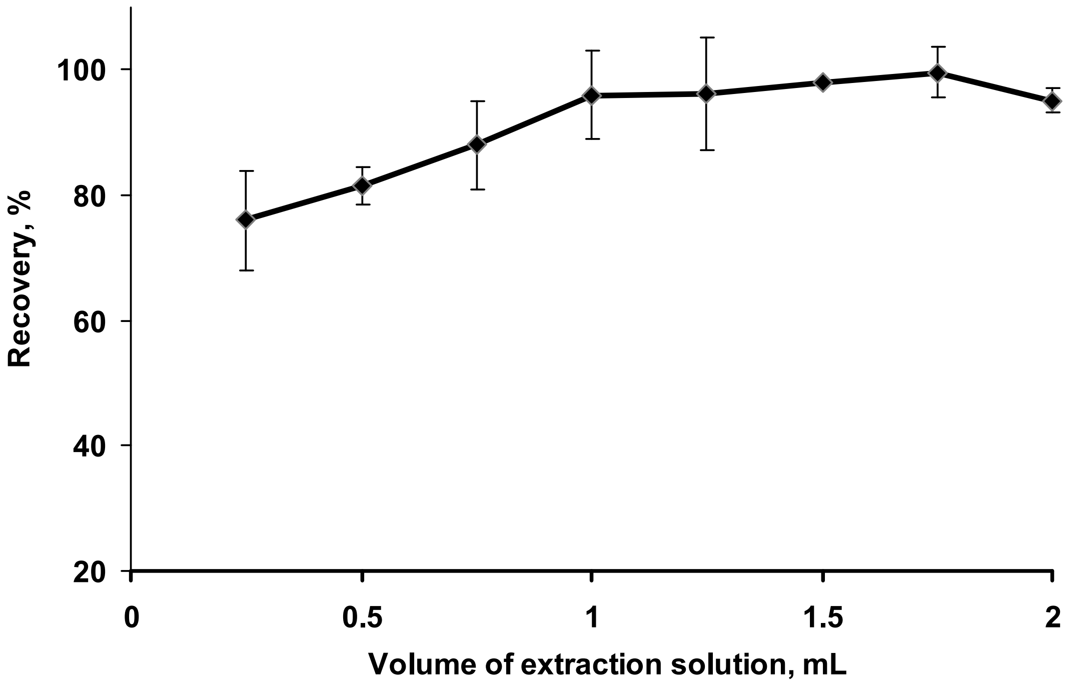
Recovery, %
100
80
60
40
20
0
0.5
1
1.5
2
Volume of extraction solution, mL

Figure 3.

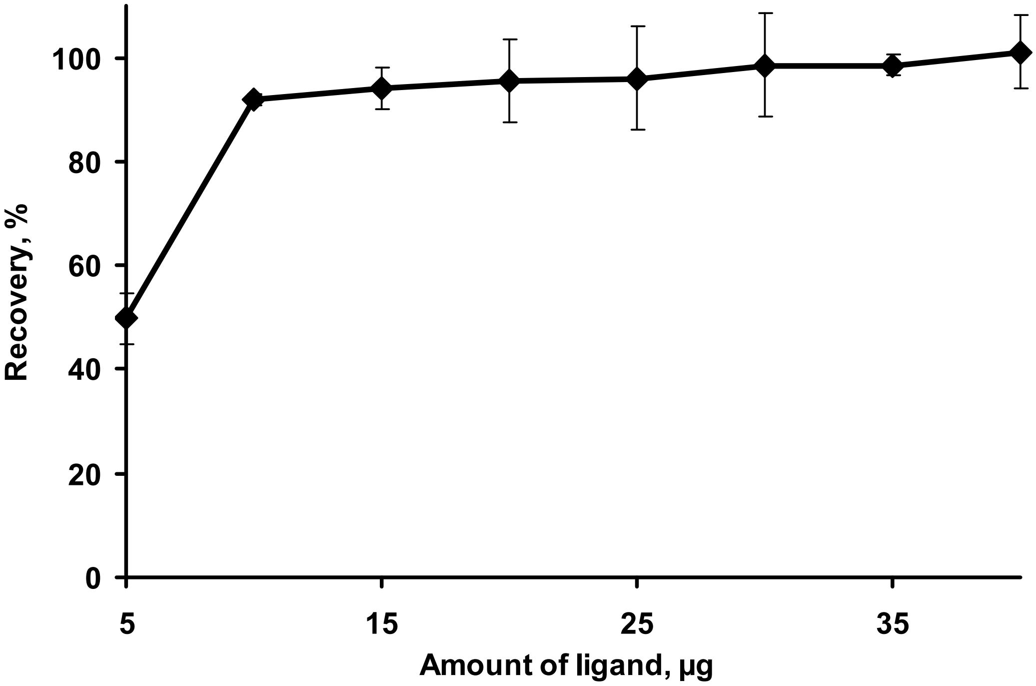

Figure 4.

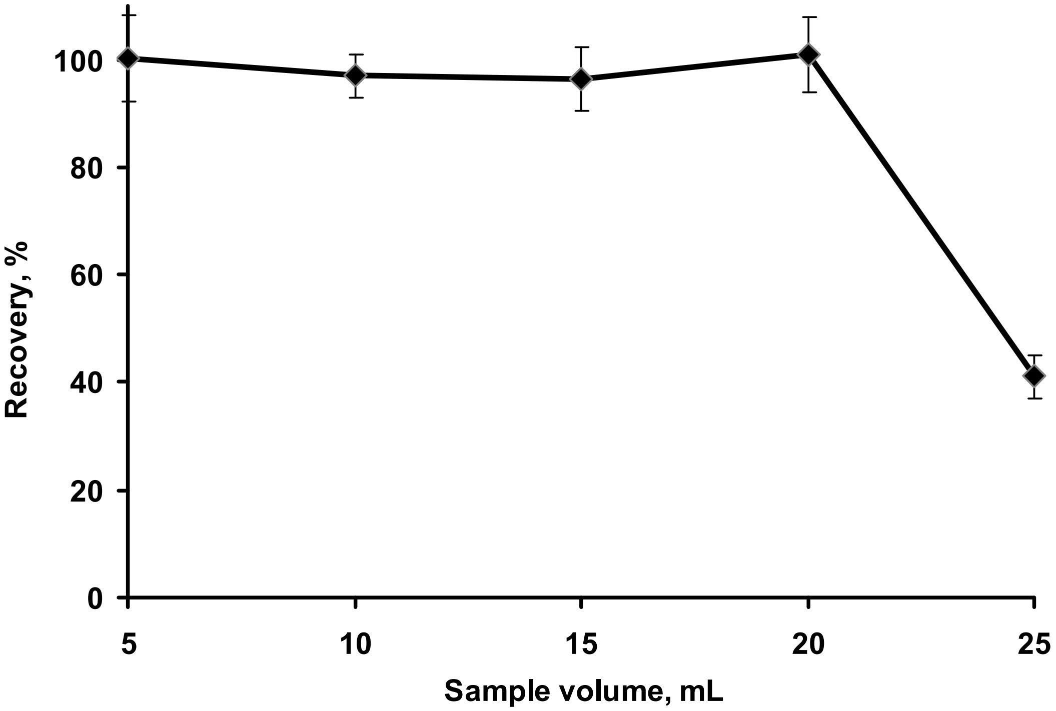

Figure 5.

Switchable Solvent Based Liquid Phase Microextraction of Uranium in Environmental Samples: A Green Approach

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