



Contents lists available at ScienceDirect

Microchemical Journal

journal homepage: www.elsevier.com/locate/microc

Study of ultrasound-assisted sequential extraction procedure for potentially toxic element content of soils and sediments

Katalin Kovács^{a,*}, Gábor Halász^a, Anita Takács^a, György Heltai^a, Éva Széles^d, Zoltán Györi^c, Márk Horváth^{a,b}

^a Szent István University, Department of Chemistry, Páter Károly Str. 1, H-2100 Gödöllő, Hungary

^b Szent István University, Regional Knowledge Center, Páter Károly Str. 1, H-2103 Gödöllő, Hungary

^c University of Debrecen, Nutrition Institute, Egyetem Space 1, H-4032 Debrecen, Hungary

^d Hungarian Academy of Sciences, Centre for Energy Research, Konkoly Thege Miklós Str. 29-33, H-1121 Budapest, Hungary

ARTICLE INFO

Article history:

Received 4 August 2016

Received in revised form 6 October 2016

Accepted 7 October 2016

Available online xxxx

Keywords:

BCR sequential extraction

Potentially toxic elements

Soil

Ultrasonic

ABSTRACT

For risk assessment of potentially toxic element (PTE) pollution of sediments and soils the fractionation by sequential extraction procedures are applied which require longtime leaching-shaking periods to achieve the dissolution equilibria. In this work an attempt is described for reduction the duration these leaching-shaking periods of the sequential extraction procedure proposed by Community Bureau of Reference (BCR) in 1993 by application of ultrasonic treatment. The fractionation of PTE content of BCR 701 certified reference sample was performed in three experiments: "A" experiment was the control, all extraction steps were performed with 16 hours shaking according to the BCR prescription; "B" experiment: in each extraction step was applied 1 hour preliminary ultrasonic treatment and after this 1 hour shaking; "C" experiment: only 1 hour ultrasonic treatment and no shaking was applied. It was experienced that by ultrasonic treatment can be efficiently increased the rate of dissolution when constant temperature of assisting medium was kept. 77% and 63% of element content extractable with the control "A" experiment can be extracted in "B" and "C" experiments, respectively. To achieve the 100% efficiency further experiments are in process.

© 2016 Published by Elsevier B.V.

1. Introduction, aim of the study

The growing industrialization and urbanization in the 20th century prompted biologists and environmental researchers to call for the mapping of biogeochemical cycles of various elements which is essential to ensure conditions for a healthy life in harmony with our environment. This revelation significantly affected the requirements to be met by the methods and instruments in the field of analytical chemistry [1]. Improvements in the speed and cost of the supplying of data as well as the accuracy and reliability of them are expected in analytics for several fields of industry, agrochemistry and environmental research [2–5].

Mobility of different elements in environmental systems determines their getting into the food chain, therefore the development of analytical methodologies for estimation of bioavailability has a great importance in practice, particularly in the field of agricultural and environmental chemistry. Aquatic sediments and soils can accumulate potentially toxic elements (PTE) from different pollution sources for many years in toxic amounts without realizing their acute toxicity [2,6,7]. For such kind of estimations in agrochemistry single step extractions are used modeling the plant uptake or for determination of total soluble element fractions [8–11].

Beside these particularly in studies dealing with environmental pollution the sequential extraction procedures play a growing part [7,12,13] which have the great advance of giving numerous chemical information using the same sample. For environmental risk assessment of PTE contaminated areas in Europe mostly the simplified extraction scheme proposed by Community Bureau of Reference (BCR) in 1993 is widely used, why only to this a certified sediment sample (BCR 701) is available, certified for fractionation of six elements (Cu, Cd, Cr, Ni, Pb, Zn) [13–16]. In this three-plus-one-step procedure the sample is treated with reagent-solvents sequentially, thereby chemical information is gained after each step on bonding-type of PTEs. Each extraction step is performed by batch leaching methodology with 16 hours shaking followed by phase separation with centrifugation. The time demand of the whole procedure therefore is about five working day and strongly limits the sample throughput which is particularly essential in case of risk assessment of havana events. Therefore the aim of present studies was how the duration of shaking period prescribed for each extraction step can be shortened or eliminated using preliminary ultrasonic treatment in ultrasonic bath. Ultrasonic treatment has been used for several years in analytical chemistry, especially for extraction of organic compounds because of its breaking and relaxing effect on the sample matrix. Ultrasonic irradiation decreases the stability of solid/aggregated particles, and formerly it was experienced that it helps to dissolve the different elemental species from sediment or soil matrix, too [17]. Practically

* Corresponding author.

E-mail address: Kovacs.Katalin.Eva@hallgato.szie.hu (K. Kovács).

all types of samples are suitable for ultrasonic treatment, however, special care should be taken to keep constant temperature. Without this the temperature of the transmitting water medium can rise as high as 40–45 °C degrees and it worsens the accuracy of the study, because element content released by the ultrasonic treatment and the emerged temperature cannot be distinguished subsequently. To avoid this effect temperature of the transmitting water bath has been kept constant. In present study the fractionation of PTE content of BCR 701 certified reference sample was performed in three experiments: (1) “A” experiment was the control, all extraction steps were performed with 16 hours shaking according to the BCR prescription; (2) In the “B” experiment in each extraction step was applied 1 hour preliminary ultrasonic treatment and after this 1 hour shaking; (3) In “C” experiment: only 1 hour ultrasonic treatment and no shaking was applied. Our point of view in selection of the duration of “B” and “C” experiments was whether the whole procedure should be performed within one day with acceptable results.

2. Material and methods

2.1. Sample

Commercially available BCR-701 sediment certified reference material (CRM) was used which is recommended for the validation of the BCR procedure. It is certified for extractable concentrations of six elements (Cd, Cr, Cu, Ni, Pb, Zn) by the 1st, 2nd and 3rd BCR extraction steps [15].

2.2. BCR sequential extraction procedure

The three-plus-one-step BCR sequential extraction procedure is presented in Table 1. For estimation of total soluble PTE content the microwave-assisted digestion with HNO₃/H₂O₂ mixture as described in STEP 4 was performed with the original BCR-701 sediment CRM sample in all experiments. It is signed in the followings as STEP 0.

The samples were weighed in to 50 mL Falcon-type centrifuge tubes, and all operation were performed in these pots.

Table 1
Scheme of the BCR sequential extraction procedure [8,9].

STEP 1	STEP 2	STEP 3	STEP 4
Sample weight: 1.0000 g Extractant: 0.11 M CH ₃ COOH (40 mL) Shaking: 16 h (480 min ⁻¹) ↓ Centrifugation: 20 min (4000 min ⁻¹) ↓ Filtration: filter paper with medium pore size (type 389) ↓ FILTRATE no. 1: fraction exchangeable and bound-to-carbonates	Sample: residue of Step 1 Extractant: 0.5 M NH ₂ OH·HCl (40 mL) Shaking: 16 h (480 min ⁻¹) ↓ Centrifugation: 20 min (4000 min ⁻¹) ↓ Filtration: filter paper with medium pore size (type 389) ↓ FILTRATE no. 2: fraction bound to Fe and Mn oxides and hydroxides	Sample: residue of Step 2 Oxidation: residue + 20 mL 30 w/w% H ₂ O ₂ ↓ Reaction with H ₂ O ₂ (85 °C, 8 h) Extractant: 1 M NH ₄ OAc (50 mL) Shaking: 16 h (480 min ⁻¹) ↓ Centrifugation: 20 min (4000 min ⁻¹) ↓ Filtration: filter paper with medium pore size (type 389)↓ ↓ FILTRATE no. 3: fraction bound to organic matter and sulfides	(Microwave-assisted digestion) Digestion scheme: Ramp time: 20 min, 180 °C, 450 PSI Hold time: 15 min, 180 °C, 450 PSI 800 W Relax time: 5 min, 0 W Hold time: 5 min, 180 °C, 450 PSI, 800 W Cool down: approx. 20 min Weigh in: residue of Step 3. (after air drying) Reagent: 5 mL 65 w/w% HNO ₃ , 2 mL 30 w/w% H ₂ O ₂ ↓ Filtration: filter paper with medium pore size (type 389) No. 4: fraction residual
Washing phase: residue was shaken with 20 mL of bidistilled water (480 min ⁻¹) for 15 min followed by centrifugation (400 min ⁻¹) for 10 min, then the supernatant was discharged.	Washing phase: residue was shaken with 20 mL of bidistilled water (480 min ⁻¹) for 15 min followed by centrifugation (400 min ⁻¹) for 10 min, then the supernatant was discharged.	Washing phase: residue was shaken with 20 mL of bidistilled water (480 min ⁻¹) for 15 min followed by centrifugation (400 min ⁻¹) for 10 min, then the supernatant was discharged.	

2.3. Ultrasonic treatment

Extraction pots was placed into ultrasonic water bath of type Transonic T460 with a working frequency of 35 kHz. Temperature of the water bath was kept between 20 and 21 °C by circulation of the water as it show on Fig. 1. Time of the preliminary treatment was 1 h in each BCR extraction steps.

2.4. Extraction experiments

Test “A”: BCR sequential extraction procedure 16 hours shaking in each steps, without preliminary ultrasonic treatment.

Test “B”: 1 hour preliminary ultrasonic treatment in each BCR extraction steps, 1 hour shaking in each BCR extraction steps.

Test “C”: 1 hour preliminary ultrasonic treatment in each BCR extraction steps, without shaking.

In all test the sequential extraction of BCR-701 CRM sample was repeated three times.

2.5. Element analytics

Element content of the extracted solutions was determined by ICP-OES using a HORIBA JOBIN YVON ACTIVA-M instrument. Matrix-matched calibration was applied during the measurements [18]. Multielemental calibration standards (MERCK CertiPUR 1.11355.0100 ICP multi-element standard solution IV.: 23 elements in diluted nitric acid - Ag, Al, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, In, K, Li, Mg, Mn, Na, Ni, Pb, Sr, Tl, Zn) were used in concentrations of 0 mg/L, 5 mg/L, 10 mg/L and 20 mg/L, dilution was performed by appropriate solvents used in the BCR extraction steps.

The following operational parameters were used:

- Incident power: 1200 W
- Plasma argon gas flow: 15.59 L/min
- Sheath argon gas flow: 0.32 L/min
- Auxiliary gas flow: 0.605 L/min
- Cyclone-type (Meinhard) nebulization

Download English Version:

<https://daneshyari.com/en/article/5138951>

Download Persian Version:

<https://daneshyari.com/article/5138951>

[Daneshyari.com](https://daneshyari.com)