ULTRASOUND-ASSISTED METAL EXTRACTIONS

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Introduction

Ultrasonic energy has been used for a wide variety of applications in industry, medicine and science. In the analytical chemistry field, most applications lie in the ability of ultrasound to extract compounds from the solid matrix. Solid–liquid extraction with the use of ultrasonic energy (i.e. ultrasound-assisted extraction) has been successfully applied for many years as a sample pretreatment method to extract organic compounds from matrices to which they are weakly bound (e.g. environmental samples). Sonication methods have been compared to other methods for pretreatment of solid samples (e.g. Soxhlet extraction, accelerated solvent extraction and supercritical fluid extraction), being competitive to them owing to its simplicity, efficiency and ease of use. Moreover, sonication methods do not involve the use of high temperatures, pressures or concentrated and harmful chemicals. Usually, ultrasound has been applied to the sample with the use of ultrasonic cleaning baths. Ultrasonic cleaning baths are readily available, large numbers of samples can be simultaneously treated and low-cost instrumentation is involved, but they lack the capability of transmitting sufficient ultrasonic power to produce the desired effects on the sample.

More recently, ultrasound-assisted extraction has been applied to the separation of inorganic compounds and metal ions from the matrix, to facilitate their analytical determination, and to avoid traditional sample pretreatment methods such as dry or wet ashing, which involve tedious and time-consuming treatments with corrosive reagents.

Other application areas of ultrasound-assisted extraction include the selective extraction of different physicochemical forms of elements for speciation. In this case, advantage is taken of the nondestructive character of ultrasound treatments which, under suitable conditions, maintain the integrity of the extracted species.

Finally, ultrasound can accelerate many sequential extraction schemes which are traditionally applied for metal partitioning in environmental samples such as soils, sludges and sediments.

Ultrasound-Assisted Extraction for Metal Determination

Intensive sample pretreatment of biological, environmental and industrial samples is frequently a necessary requirement for elemental analysis, so that ideally a matrix-free solution is obtained. Typically, dry ashing or wet ashing methods involve the use of high temperatures or corrosive reagents, usually under pressure, which demands very stringent safety conditions. However, a simple analyte separation without matrix decomposition is enough for many analytical techniques. Thus, atomic absorption spectrometry (mainly with the use of electrothermal atomization) allows analytical determinations to be carried out with minimum sample pretreatment owing to the low dependence of the analytical signal on the accompanying matrix as compared with other techniques for elemental analysis.

Thus, in the authors' laboratory, a number of elements have been quantitatively extracted from a large variety of matrices when probe-type sonicators operated under optimized conditions are employed. Toxic metals such as Cd and Pb can be easily extracted from mussel tissue and other biological samples, the exact extraction conditions depending on the metal. Cadmium could be quantitatively extracted from a sample mass of 10 mg slurried in a 1.5 mL volume. The sample has to be previously ground, the particle size being a critical parameter in the case of Pb since extraction efficiency diminishes for a particle size large than 150 μm. For Cu and Cd, extraction can be achieved for a particle size larger than 200 μm. The presence of an acidic medium is an essential requirement for quantitative extraction to be attained. For analytical techniques such as electrothermal atomic absorption spectrometry (ETAAS), nitric acid is recommended since unlike hydrochloric acid it does not form volatile compounds with analytes, which are the origin of interferences. In addition, nitric acid combined with the ultrasonic action promotes matrix oxidation so that analyte extraction is facilitated. Minimum acid concentration used for quantitative extraction depends again on the analyte to be extracted. A nitric acid concentration as low as 0.05% v/v is sufficient for quantitative extraction of Cd, whereas Pb requires at least 1% v/v nitric acid. Additional parameters controlling the amount of ultrasonic power delivered to the sample such as sonication time and vibrational amplitude of the probe (expressed as a percentage of the nominal power) should be optimized for best performance. Metals which are easy to extract, such as Cd, require very short sonication times, typically less than 1 min while stronger bound metals such as Pb require 3–5 min. When using a 100 W probe sonicator, at least a 10% amplitude is necessary for extraction of Cd, while a 60% amplitude is required for Pb. Sample mass is also an important variable; extraction is usually quantitative for a mass of less than 20 mg suspended in 1.5 mL volume. Although the preparation of suspensions in larger volumes with larger amounts of ground material is also feasible, preparation of suspensions in autosampler cups is a more convenient way for ETAAS when sample homogeneity is not a limiting factor. An experimental design applied to the extraction process of Cd and Pb confirmed that soft sonication conditions (minimum sonication time and amplitude) along with maximum particle size (e.g., >200 μm) could be used for quantitative solid–liquid extraction of Cd provided that maximum acid concentration was used (e.g., 3% v/v). On the other hand, Pb needed maximum sonication time, amplitude and acid concentration together with minimum particle size. The concentration of nitric acid proved to be the most critical factor for achieving quantitative extraction.

A study carried out with Pb as target analyte and certified reference materials has shown the importance of using the appropriate ultrasonic processor so that quantitative extraction is attained. Thus, an ultrasonic cleaning bath is not suitable since only a fraction of the analyte is brought into solution even using long sonication times (e.g., 60 min). When comparing two probe-type sonicators (50 versus 100 W), quantitative extraction was observed with the 100 W sonicator for all biological materials attempted. The explanation for the above results could lie in the greater ability of probe-type sonicators to cause cavitation in the liquid medium, which results in a more efficient disruption of solid particles, so facilitating metal extraction.

Incomplete extraction was observed for Pb and Cd from sediments, thereby indicating that matrix–analyte binding plays an important role in the solid–liquid extraction process. This may be due to particle disruption being more difficult with hard materials such as sediments, so that unless the analyte is adsorbed on the surface the fraction of analyte occluded inside the solid particles will not be brought into solution, hence resulting in incomplete extraction.

Usually, the ultrasonic action will cause the matrix to be partly extracted into the liquid medium, but background absorbance caused by the small amount of matrix released can be easily handled by the background correction system (**Table 1**).

Table 1 Percentage of metal extracted from certified reference materials using ultrasound irradiated with a probe ultrasonic processor

Certified sample	*% Extraction*			
	Cd[a]	*Cu*[b]	*Cr*[c]	*Pb*[c]
BCR 278 Mussel tissue	101.8	82.4	42.0	94.2
NRCC DORM-2 Dogfish muscle	93.0	93.2	2.7	–
NRCC DOLT-2 Dogfish liver	91.6	–	32.9	–
BCR 60 Aquatic plant	101.4	102.4	30.2	101.2
BCR 145 R Sewage sludge	56.3	–	46.6	104
BCR 320 River sediment	75.4	–	15.0	69.0
NRCC TORT-2 Lobster hepatopancreas	–	–	69.3	–
BCR 482 Lichen	–	–	23.7	–
GBW07605 Tea leaves	–	–	-	95.5

[a] Capelo JL, Lavilla I and Bendicho C (1998) *Journal of Analytical Atomic Spectrometry* 13: 1285–1290.

[b] Capelo JL, Filgueiras AV, Lavilla I and Bendicho C (1999) *Talanta* 50: 905–911.

[c] Capelo JL, Lavilla I and Bendicho C (1999) *Journal of Analytical Atomic Spectrometry* 14: 1221–1226.

The use of other analytical techniques for detection after ultrasound-assisted extraction has also been reported. For instance, Ashley has studied the extraction of Pb from several standard reference materials (SRMs) such as lead-based paint, urban particulate and river sediment followed by anodic stripping voltammetry (ASV). Analytical results were satisfactory after ultrasonic extraction for 30 min using a 10% v/v nitric acid solution. ASV has been also used for determination of Pb in workplace air samples collected in the field using cellulose ester membrane filters. The filters were subjected to ultrasound under the conditions given above for SRMs. An advantage of ultrasound-assisted extraction methods over methods involving matrix decomposition (e.g., microwave-assisted digestion) is the ability to use them in the field, hence facilitating on-site analysis with portable instruments.

The use of diluted acids for extraction can also offer a simplified methodology for determination of metals by flame atomic absorption spectrometry (FAAS). In a comparison of five methods for pretreatment of plant samples, Matejovic and Durackova found that extraction of metals could be accomplished with 1 M hydrochloric acid in an ultrasonic bath. After sonication the extracts were filtered so that no particulate material could clog the nebulizer. In this case, the use of a nonoxidizing and complexing acid such as hydrochloric acid is perhaps more convenient than other acids, since it avoids a final evaporation step to remove the excess of acid as is necessary when concentrated acids are used for mineralization in conventional digestion procedures. Incomplete release of P bound into organic compounds and Fe was observed with this procedure.

Leaching of heavy metals from aquatic plants used as environmental biomonitors has been performed by ultrasound-assisted extraction with a 1% w/w $HCl + 15\%$ w/w HNO_3 mixture. In this case, two consecutive extractions were needed to quantitatively extract Mn, Cu and Zn, the recovery of Cu being only about 75%, with RSDs lower than 2.5%. In order to obtain good analytical performance when applying ultrasound-assisted extraction, all the variables influencing the process should be borne in mind: concentration of the suspension (i.e. sample mass and extraction volume), particle size, sonication time, sonication amplitude, type of acid and its concentration, and temperature. This last variable is seldom considered for its influence on ultrasonic extractions. Since most ultrasonic cleaning baths warm up slowly during operation, many applications reported with these devices for extraction use a pre-heated liquid so that temperature is constant, hence improving reproducibility. On the other hand, acoustic cavitation is diminished on increasing the temperature above 50°C, and consequently extraction efficiency is also diminished. Thus, some workers have found only partial extraction for some elements when using a pre-heated ultrasonic bath or allowing the bath to warm up during operation to a temperature higher than 50°C. Other workers have reported quantitative extraction of metals such as Cd, Cu, Pb and Mn from powdered biological samples when sonication is carried out at 40°C. Other extractants succesfully employed for solid–liquid extraction with an ultrasonic cleaning bath include dilute HCl, HNO_3 and H_2O_2. Some procedures employing ultrasonic baths for sample pretreatment were aimed at complete digestion of the sample by the use of concentrated acids, and therefore cannot be regarded as extraction procedures.

Applications of Ultrasound-Assisted Extraction for Element Speciation

Ultrasound extraction shows advantageous features for element speciation. Organometallic species can be extracted without changes in their integrity under suitable extraction conditions. Both organic and aqueous extraction media have been used for separation of organometallic and inorganic species from the solid matrix, most applications using ultrasonic cleaning baths for extraction.

A recent application of ultrasound-assisted extraction with the use of a probe-type sonicator has been reported for mercury speciation in combination with flow injection–cold vapour–atomic absorption spectrometry (FI-CVAAS) for detection. In this case, a 400 mg portion of sample and 1–7 mL of 0.5–7 M acid were placed in a centrifuge tube and sonicated at a fixed ultrasound amplitude for 1–5 min. Selective extraction of methylmercury required less than 5 mL of 2 M HCl, the extraction being quantitative ($>95\%$) when the HCl volume was higher than 2 mL. The extraction could be accomplished using ultrasound amplitude in the range 20–70% for 2–5 min. The optimization procedure was addressed to selectively extract methylmercury from slurried biological samples such as mussel tissue; inorganic mercury extraction required higher HCl concentrations. Both mercury species could be extracted with 5 mL of 5 M HCl and sonicating at 20–70% amplitude for 3–5 min. Methylmercury was determined using sodium tetrahydroborate(III) as reducing agent whereas inorganic mercury was determined by selective reduction with stannous chloride in the extracts containing both species. The limits of detection were 11 and 5 $ng\,g^{-1}$ for methylmercury and inorganic mercury, respectively. The repeatability (between-batch precision), was in the range 5–10% for both mercury species.

In a study on As extraction, similar distributions of arsenicals (e.g., arsenobetaine, arsenocholine and dimethylarsinic acid) were found in a comparison between accelerated solvent extraction and sonication. Nonpolar As is extracted with acetone whereas polar As is extracted with 50% w/w methanol.

Cr(VI) has been extracted from industrial hygiene samples with an ultrasonic cleaning bath at 40–50°C for 1 h using alkaline solutions containing 0.05 M $(NH_4)_2SO_4$–0.05 M NH_3. The Cr(VI) was separated from other cations present in the extract by retention with an anion-exchange resin. Elution of Cr(VI) from the resin was performed with a buffer solution at pH 8. The eluate was acidified with HCl and the complex between Cr(VI) and 1,5-diphenylcarbazide was measured by flow injection–UV/VIS detection. Determination of total Cr following ultrasonic extraction was also feasible using a prior oxidation step with Ce(IV) so that Cr(III) is converted into Cr(VI). This simple and effective preparation method compared favourably with other methods employing intensive treatments leading to matrix decomposition (e.g., acid digestion) for determination of total Cr in fly ash, paint chips, etc.

Sequential Extraction of Metals from Environmental Samples

The bioavailability and mobility of trace metallic and metalloid elements in the environment depend on the chemical form of the element and the type of binding to the matrix. Sequential extraction schemes, although far from being perfect, have the ability to extract elemental species from particular solid phases in sediments, soils and sewage sludge. However, application of these schemes entails a difficult experimental task owing to the large number of slow and tedious stages. For instance, the Tessier scheme apportions metal distribution in four different stages: (1) exchangeable, (2) associated to carbonates, (3) associated to Fe and Mn oxides and (4) associated to organic matter and sulfides. For dissolving a particular solid phase, chemical extractants are applied successively to the solid sample, each follow-up treatment being more drastic in chemical action or different in nature from the previous one. Thus, for the phases mentioned above, an $MgCl_2$ solution, an NaOAc solution, an $NH_2OH.HCl$ solution, and an $HNO_3 + H_2O_2$ solution are used sequentially. The Tessier scheme requires an overall operation time of about 18 h. Ultrasonic energy from a probe-type sonicator has been employed for acceleration of the sequential chemical extraction of Cu, Cr, Ni, Pb and Zn from sediment and sewage sludge samples. Conventional and ultrasound-accelerated Tessier extraction schemes offered similar partitioning patterns for the two first fractions (i.e., exchangeable and carbonate-bound) when applied to a sewage sludge sample. However, significant differences in metal extractability were observed for some metals when applying the ultrasound-accelerated Tessier scheme to river sediments. On the other hand, a good agreement for the total extractable contents (i.e., sum of metal contents found in each stage) was seen for Ni, Pb and Zn in sewage sludge and Cr, Ni, Pb and Zn in river sediment, meaning that the ultrasound methodology could be useful for fast screening of extractable

Table 2 Analytical results obtained by applying the conventional and the modified Tessier sequential extraction schemes for metal partitioning in a river sediment and a sewage sludge

Fraction	*Element*	*River sediment*[a]			*Sewage sludge*[b]		
		Conventional method (X ± SD)[c]	*Ultrasound method (X ± SD)*[c]	*Recovery*[d] *(%)*	*Conventional method (X ± SD)*[c]	*Ultrasound method (X ± SD)*[c]	*Recovery*[d] *(%)*
Exchangeable	Cu	2.17 ± 0.05	1.90 ± 0.1	87.6	18.4 ± 0.18	18.2 ± 0.12	98.9
	Cr	ND	ND	–	ND	ND	–
	Ni	12.2 ± 0.3	12.1 ± 0.4	99.3	9.51 ± 0.18	9.24 ± 0.23	97.2
	Pb	9.83 ± 0.16	9.73 ± 0.34	99.0	10.9 ± 0.26	10.7 ± 0.26	97.6
	Zn	14.2 ± 0.3	14.0 ± 0.4	98.6	96.7 ± 2.1	96.2 ± 3.7	99.5
Carbonate-bound	Cu	15.5 ± 0.47	4.21 ± 0.27	27.2	8.16 ± 0.11	8.1 ± 0.12	98.7
	Cr	ND	ND	–	ND	ND	–
	Ni	14.1 ± 0.57	14.0 ± 0.21	99.3	6.35 ± 0.09	6.16 ± 0.25	97.0
	Pb	41.1 ± 0.37	40.6 ± 0.83	98.9	13.7 ± 0.24	13.6 ± 0.19	99.8
	Zn	70.8 ± 1.34	69.2 ± 1.43	97.7	80.0 ± 1.1	78.6 ± 1.6	98.2
Fe–Mn oxide-bound	Cu	7.71 ± 0.35	18.7 ± 0.28	242	10.3 ± 0.16	26.1 ± 0.27	253
	Cr	7.06 ± 0.11	2.85 ± 0.1	40.4	ND	ND	–
	Ni	6.0 ± 0.23	6.0 ± 0.3	100	4.58 ± 0.30	4.42 ± 0.14	96.3
	Pb	165.4 ± 3.7	134 ± 1	81.2	19.7 ± 0.71	19.2 ± 0.16	97.7
	Zn	130 ± 3	106 ± 1	81.3	397 ± 3	393 ± 3	99.1
Organic matter-bound	Cu	152 ± 2	149 ± 4	98.3	165 ± 3	46.3 ± 0.43	28.0
	Cr	3.92 ± 0.04	ND	0.0	8.31 ± 0.23	ND	0.0
	Ni	ND	ND	–	6.00 ± 0.12	5.97 ± 0.32	99.5
	Pb	5.60 ± 0.36	34.6 ± 0.63	618	16.0 ± 0.46	15.6 ± 0.47	97.4
	Zn	15.4 ± 0.24	32.2 ± 1.0	210	90.0 ± 2.0	58.4 ± 2.0	64.9

[a] Pérez-Cid B, Lavilla I and Bendicho C (1999) *International Journal of Environmental Analytical Chemistry* 73: 79.

[b] Pérez-Cid B, Lavilla I and Bendicho C (1999) *Fresenius Journal of Analytical Chemistry* 363: 667.

[c] Average of three determinations (expressed as $\mu g\, g^{-1}$) ± standard deviation.

[d] The recovery was calculated in the following way: [metal leached using the accelerated method/metal leached using the conventional method] × 100.

ND, non detected.

metals in solid environmental samples. The operation time per sample was 20 and 28 min for sewage sludge and river sediment, respectively, when ultrasound was used for the Tessier scheme (**Table 2**).

The sequential extraction scheme, proposed by the Community Bureau of Reference (BCR), now the Standards, Measurement and Testing Programme, consists of three stages: acid-soluble, reducible and oxidizable. The reagents employed are a HOAc solution, an $NH_2OH.HCl$ solution and an H_2O_2 solution, respectively. Despite using a stage less than the Tessier scheme, its operation time is much longer (about 51 h per sample). Application of the BCR scheme to sewage sludge showed that a drastic shortening in time from 51 h to about 22 min per sample could be achieved by the use of ultrasonication. In this case, a much better agreement between the conventional and the ultrasound-accelerated BCR schemes was found in all fractions, so that information concerning extractable metal contents from sewage sludge was virtually the same.

Conclusions

Ultrasound-assisted extraction can be used as an alternative to traditional sample preparation methods for elemental analysis and speciation where matrix separation rather than complete matrix elimination is performed. Sonication methods usually involve mild treatments which meet an important requirement for speciation, i.e., extraction of the species of interest without changes in their integrity. As a result of the decreased amount of matrix released during sonication treatments, matrix interferences can also be reduced. Additionally, ultrasonic treatments provide a significant speeding up of those methods requiring long and tedious extractions (e.g., sequential extraction of metals from solid environmental samples). So far, analytical results obtained on applying ultrasound for sample preparation are very promising, and new developments are expected on the topics addressed in the present work. On-line solid–liquid extraction with the use of ultrasound will require specially designed ultrasonic cells to further simplify sample treatment.

See also: **II/Extraction:** Analytical Inorganic Extractions. **III/Microwave-Assisted Extraction: Environmental Applications.**

Further Reading

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VENOMS: CHROMATOGRAPHY

See **III / NEUROTOXINS: CHROMATOGRAPHY**

VETERINARY DRUGS: LIQUID CHROMATOGRAPHY

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For the analysis of residues of veterinary drugs, liquid chromatography (LC) is of increasing importance: some of these molecules are polar, heat-sensitive and/or difficult to analyse by gas chromatography–mass spectrometry (GC-MS). Moreover, LC is the method of choice for components of high molecular mass. Since the introduction of benchtop LC-MS instruments, there has been an increasing number of publications on the application of this technique in the field of residue analysis.

Equipment

In LC a large variety of packed columns are in use but most residue separations are carried out with some kind of reversed-phase material based on modified silicas (RP-18, RP-8, etc.). Hitherto, in our laboratory, a particle size of 5 μm with column dimensions 150 × 2.1 mm has been commonly used. For a laboratory involved in residue analysis under accreditation, the daily reproducibility of the chromatogram from column to column is very important (see section on quality criteria, below). In the future, column material of smaller particle sizes (3 μm) may be used routinely, allowing faster separation, higher sample throughput and better limits of detection.

The nature of the mobile phase depends on the column used. In most cases a mixture of water and an organic solvent such as methanol or acetonitrile is used. Special LC grades of solvents are necessary. For analysis of residues, gradient elution is a must. In most cases the column has to be cleaned from interfering components after each run by a gradient. As well as organic solvents, a number of chemicals may be added to the mobile phase (buffers and chelating agents) but the compatibility of these products with the detector should be checked. For LC-MS only volatile components (e.g. trifluoroacetic acid) can be used and this limitation sometimes hinders the transformation of an LC into an LC-MS^n method.

Autoinjection is a must for the routine analysis of residues of veterinary drugs, not only for higher sample throughput but also for reproducibility in the validation of the results. However, particular attention should be drawn to the danger of cross-contamination with such injectors, especially in combination with LC-MS which has low detection limits.

Detectors

For screening purposes universal detectors such as UV and light-scattering detectors are used. However, for the confirmation of suspect samples more is required than just retention time and detector response. Since the results of laboratory analysis may have a serious impact on individuals and companies, false positives must be avoided at any price. For example, a sample of poultry feed, analysed by ion chromatography, was suspected to contain KSCN (a thyreostatic drug). Both the retention time and co-chromatography met the quality criteria. However, the presence of KSCN was so unlikely that the effluent was collected and mixed with Fe^{3+} (to give a red colour with SCN). This test was negative. Later on, it was found that the sample contained acetylsalicylic acid, which is often used in poultry rearing, and that the two molecules are not separated in the chromatographic system used.