



Leaching experiments on the release of heavy metals and PAH from soil and waste materials

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ABSTRACT

Leaching tests are fundamental tools for the assessment of long-term impact of contaminated waste materials on the soil-groundwater pathway. Experiments were carried out in the framework of standardization and validation of column percolation and batch test procedures, in particular concerning the stipulation of the experimental setup. The colloid release of column and batch experiments was compared and the influence of different column filling heights (12.5–50 cm) on the release of polycyclic aromatic hydrocarbons (PAH) from soil was studied, as well as the effect of varying contact times (2.5–16 h) on the release of chromium from construction and demolition (C&D) waste and municipal solid waste incineration (MSWI) bottom ash. The results indicate that filtration of the eluate, which is required for batch tests, does not always allow the simulation of the actual colloid amount in soil pore water. Medium column heights four times the inner diameter of the column seemed to provide reasonable equilibrium adjustment conditions and avoid major biodegradation. The release of chromium was only marginally affected by the contact time, varied between 0.115 and 0.150 mg/kg for demolition waste eluate at a liquid-to-solid ratio of approximately 5 L/kg.

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1. Introduction

Construction and demolition (C&D) waste and municipal solid waste incineration (MSWI) bottom ash constitute a significant mass percent of the waste materials produced every year around the world. In Germany alone, about 73 of the 240 million tons of mineral waste produced per year are C&D waste, with incineration and power plant ash accounting for about 15 million tons, thus representing the largest contingents of waste after excavated soil materials. Since C&D waste and MSWI bottom ash are often contaminated with inorganic and/or organic pollutants, the risk assessment of materials containing these substances is of particular importance. Laboratory leaching tests are common tools to aid the assessment of long-term impact of contaminated materials on the soil-groundwater pathway, as they determine the source term as an expression of release potential of water soluble contaminants during the use or disposal of waste materials. Different leaching tests have been developed to characterize and assess the constituents that can be released from waste materials [1–6].

Whereas column tests provide a flow-through pattern similar to that found in field conditions and enable basic characterization of waste materials, batch or tank tests present a snapshot at a certain liquid-to-solid (or to-surface area) ratio [7,8].

With the designated establishment of a new Ordinance on Waste Utilization and the amendment of the German Ordinance on Soil Protection and Contaminated Sites, leaching tests are expected to become a more important methodology for waste materials source term determination [9].

Waste materials should be tested under conditions similar to their actual application in re-use scenarios to achieve reliable results. Since the standards for leaching procedures available at an international level so far are either not validated or considered inadequate in respect to coarse-grained materials, new standards had to be developed which are applicable to coarse-grained waste materials and suitable to the new German regulations concerning waste management. Two standards, a column test (DIN 19528 2009 [10]) and a batch test (DIN 19529 2009 [11]), were developed by the responsible standardization committee of the German Standardization Organization DIN; these standards were recently validated by an interlaboratory comparison performed by BAM [12,13]. Following the outcome of the joint project “Seepage prognosis”, an agreement was reached concerning the liquid-to-solid ratio (L/S) for compliance testing: the L/S was stipulated to be set at 2 L/kg. Whereas batch tests are only suitable for compliance testing, column tests allow for both compliance testing and basic characterization.

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Table 1
Test conditions for compliance tests according to DIN 19528 and DIN 19529 using BA RM and CS1 RM.

Test conditions DIN 19528	BA RM (≤32 mm)	CS1 RM (≤10 mm)
Internal diameter of the column (cm)	10.00	5.86
Sample filling height in column (cm)	40.0	25
Filling volume (cm ³)	3.142	674
Dry mass (kg)	5.2	1.2
Particle density (g/cm ³)	2.50	2.60
Bulk density (g/cm ³)	1.65	1.77
Porosity <i>n</i> (-)	0.35	0.33
Contact time eluent/sample (h)	5.0	5.0
Flow rate (ml/min)	3.56	0.75
Eluate volume up to L/S 2 (L)	10.4	2.4
Test duration until L/S 2 (h)	48.5	52.8
Test conditions DIN 19529		
Dry mass (kg)	2.5	0.25
Eluate volume at L/S 2 (L)	5	0.5
Agitation duration (h)	24	24

As opposed to CEN/TS 14405 (2004) [14] and ISO/TS 21268-3 (2007) [15], the new column test procedure stipulates a certain contact time between leachant and sample material instead of a fixed flow rate. This new approach enables the use of different column dimensions by applicants depending on their specific requirements and most importantly, on the maximum grain size of the materials under investigation.

Additionally, DIN 19528 and DIN 19529 allow the testing of materials with a maximum grain size of 32 mm, which is not considered in other existing standards (CEN/TS 14405 (2004) [14], ISO/TS 21268-3 (2007) [15], EN 12457-1 2002 [16]; ISO/TS 21268-1 2007 [17]).

The new batch test procedure [11] has been validated only for inorganic substances. An analogous standard for batch testing is being prepared by DIN, which stipulates a special procedure for the liquid/solid separation step in the case of organic compounds. BAM currently conducts ruggedness testing for this batch test procedure taking into account regulated organic contaminants of high priority.

In addition to the validation interlaboratory comparisons, investigations on the variation of boundary conditions for batch and column tests were carried out for several years. Exemplary results concerning the stipulation of the experimental setup are presented. The colloid amount of the eluates from column and batch experiments considering different sample pre-treatment procedures are compared. The influence of test duration of column experiments in terms of equilibrium establishment between sample and eluate, as well as biodegradation using PAH contaminated soil, was studied. The impact of contact time on heavy metal release from C&D waste and MSWI bottom ash in column experiments was investigated.

2. Experimental

The test materials were obtained from materials previously prepared for two different interlaboratory comparisons [12,13]. CS1 RM (contaminated soil, reference material), BA RM (MSWI bottom ash, reference material), and DW1 RM (construction and demolition waste, reference material) were taken from the former, and CS2 RM (contaminated soil, reference material) and DW2 RM (construction and demolition waste, reference material) from the latter interlaboratory comparison.

Batch and column percolation tests were carried out according to [10] and [11] respectively using CS1 RM, CS2 RM, BA RM, DW1 RM, and DW2 RM. Experimental conditions for compliance testing of two different materials are given in Table 1.

Batch test were performed by agitating sample and demineralized water for 24 h in an end-over-end tumbler followed by sample preparation consisting of settling, centrifugation, and pressurized filtration through a glass fiber filter.

Column tests were performed with glass columns of 5.86 cm internal diameter. The test samples were filled into the columns and compacted slightly so that the bulk density was very similar for all columns. On bottom and top of the columns a quartz sand filter layer was placed (0.6–1.2 mm grain size). The columns were saturated with demineralized water within 2 h directly prior to the test.

Additionally, the column test allows basic characterization experiments, whose conditions differ from those for the compliance tests. Instead of one eluate, four fractions at different L/S ratios (0.3, 1, 2, and 4) have to be collected. The results for the respective compliance test can be calculated by cumulating the released amount of the first three fractions for the components analyzed and back-calculating it as a concentration at L/S 2 L/kg.

Fig. 1 shows the workflow for compliance testing and the relevant experimental conditions. The pre-treatment of the eluates prior to the analysis depends on whether organic or inorganic substances will be analyzed.

The contact time between eluent and test material (t_c [h]) is calculated according to [10] by Eq. (1):

$$t_c = \frac{V \cdot n}{q \cdot 60} \quad (1)$$

where V (mL) is the filling volume of the column, n (-) is the porosity, q (mL/min) is the applied flow rate and 60 is used as time unit conversion factor from minutes to hours.

The turbidity of the eluates was measured with a Hach 2100 IS turbidity meter according to DIN EN ISO 7027 [18]. PAH concentrations were measured by liquid-liquid extraction of the eluates followed by HPLC with fluorescence detection, according to DIN EN ISO 17993 [19]. The HPLC was a Waters 625 system, equipped with a Waters 470 fluorescence detector. The column used was a Supelcosil LC-PAH of 5 μm particle size, 25 cm length, and an internal diameter of 4.6 mm. A mixture of acetonitrile and water was used as mobile phase. After 5 min of 40% acetonitrile fraction, the gradient was changed within 25 min to 100% acetonitrile, which was kept for 6 more minutes. The PAHs investigated were those identified by the United States Environmental Protection Agency (US-EPA) as priority pollutants. Since acenaphthylene is not accessible through fluorescence detection, all results refer to the remaining 15 of the 16 EPA-PAH. The concentrations of chromium were measured with Inductively Coupled Plasma Optical Emission Spectrometry (ICP OES), following DIN EN ISO 11885 [20] and using a Thermo Scientific Iris Intrepid II XSP spectrometer.

3. Results and discussion

In order to explain the selection of the new specifications in the German leaching standards recently published, examples including interlaboratory test results are given below.

Batch tests often lead to enhanced mobilization of colloids due to the agitation. Those particles often contain colloidal-linked pollutants [21], and therefore the eluate has to be centrifuged and/or filtrated in order to avoid an overestimation of contaminant release. However, determining the cut-off for filtering and/or centrifugation to simulate realistic soil water remains a difficult step. In the case of column tests, the packing of the sample material and quartz sand filter layers results in a self-filtration capacity that usually renders unnecessary an additional subsequent sample preparation. Fig. 2 compares the turbidity of DW2 RM and CS2 RM eluates obtained from column tests without subsequent filtration and from batch tests using a 0.45-μm mem-

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