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Results from a round robin test for the ecotoxicological evaluation of construction products using two leaching tests and an aquatic test battery

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HIGHLIGHTS

- A European round robin test has been carried out with 17 laboratories from 5 countries.
- Two construction products were eluted in an on-stage batch test or a tank test.
- Ecotoxicity against algae, daphnia, luminescent bacteria and zebrafish eggs was determined according to ISO standards.
- The more toxic the eluates, the higher was the variability.
- The inter-laboratory variability of the ecotoxicological characterization of construction products in eluates and bioassays was acceptable.

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ABSTRACT

A European round robin test according to ISO 5725-2 was conceptually prepared, realised, and evaluated. The aim was to determine the inter-laboratory variability of the overall process for the ecotoxicological characterization of construction products in eluates and bioassays. To this end, two construction products BAM-G1 (granulate) and HSR-2 (roof sealing sheet), both made of EPDM polymers (rubber), were selected. The granular construction product was eluted in a one stage batch test, the planar product in the Dynamic Surface Leaching test (DSLT). A total of 17 laboratories from 5 countries participated in the

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round robin test: Germany (12), Austria (2), Belgium (1), Czech Republic (1) and France (1). A test battery of four standardised ecotoxicity tests with algae, daphnia, luminescent bacteria and zebrafish eggs was used. As toxicity measures, EC50 and LID values were calculated. All tests, except the fish egg test, were basically able to demonstrate toxic effects and the level of toxicity. The reproducibility of test results depended on the test specimens and the test organisms. Generally, the variability of the EC50 or LID values increased with the overall level of toxicity. For the very toxic BAM-G1 eluate a relative high variability of CV = 73%–110% was observed for EC50 in all biotests, while for the less toxic HSR-2 eluate the reproducibility of EC50 varied with sensitivity: it was very good (CV = 9.3%) for the daphnia test with the lowest sensitivity, followed by the algae test (CV = 36.4%). The luminescent bacteria test, being the most sensitive bioassay for HSR-2 Eluate, showed the highest variability (CV = 74.8%). When considering the complex overall process the reproducibility of bioassays with eluates from construction products was acceptable.

1. Introduction

Construction products that come into contact with rain, seepage water or groundwater in their intended use may release hazardous substances through leaching. The use of waste materials in construction has been recognized as a relevant source of heavy metal pollution (Cenni et al., 2001; Flyhammar and Bendz, 2006; Galvín et al., 2012). Leaching methods have been standardised and regulations drawn up to address the issue (Eikelboom et al., 2001; Hage and Mulder, 2004; Susset and Grathwohl, 2011; Nebel and Spanka, 2013). So far construction products releasing mainly hazardous organic substances have received much less attention (Burkhardt et al., 2011; Wangler et al., 2012; Baderna et al., 2015). The identification of hazardous substances by chemical analysis may not cover all contaminants present in leachates from construction products. With bioassays the joint effects of ingredients are detected by their effects to living organisms. Bioassays are especially suited to assess effects of organic substances for which reference or limit values for water quality often do not exist like for heavy metals. This has been acknowledged in research work addressing waste and road runoff (Pandard et al., 2006; Waara and Färm, 2008).

The variety of different construction products on the market is huge. For both the manufacturers and the users of construction products it is important that reliable and easy to use methods for the assessment of the products' environmental assessment and comparison of product performance are available. Thus, a test battery has been elaborated for the ecotoxicological characterization of eluates from construction products (Gartiser et al. submitted). Such a harmonised test battery is intended to facilitate the development of a common understanding for the assessment of leaching from the building sector for both regulatory purposes and voluntary initiatives by manufacturers or ecolabels.

By the combination of leaching tests with ecotoxicity tests the overall variability of the ecotoxicity test results is expected to increase. However, the extent of this variability was not known so far. Therefore, an interlaboratory round robin test with 17 laboratories and two construction products relevant for leaching of organic contaminants in their intended use (one granulate and one sheet like) was carried out in 2015, while using biotests with algae, daphnia, fish eggs, and luminescent bacteria. The testing strategy is also in-line with the technical CEN guidance CEN/TR 17105 (draft) on the use of ecotoxicity tests applied to construction products currently being developed by the European Committee for Standardization (CEN). The International Organization for Standardization has published several biological methods under the technical committee ISO/TC 147 for testing ecotoxicological effects.

An adequate reproducibility of ecotoxicity data is a prerequisite for the validation of the methodology before it can be used for regulatory purposes. Standardised performance tests for construction products have been mandated by the European Commission to facilitate the removal of technical barriers for trade. The objective of the round robin test was to obtain quantitative figures about the reproducibility and robustness of data obtained from ecotoxicity testing of eluates.

2. Materials and methods

2.1. Participating laboratories and organization of the ring test

A total of 17 laboratories from 5 countries participated in the round robin test: Germany (12), Austria (2), Belgium (1), Czech Republic (1) and France (1). The laboratories belong to governmental institutes, contract laboratories, research institutes and one university (see attribution of authors to their institutes, laboratory codes L01 – L18 are anonymised). Most laboratories maintain a quality assurance system, although the studies themselves have not been subjected under these systems. Because only data fulfilling all validity criteria of the different tests were considered in the

Table 1
Chemical parameters of eluates.

Lab code	BAM-G1				HSR-2		
	pH	Conductivity [µS/cm]	TOC [mg/L]	Pre-treatment	pH	Conductivity [µS/cm]	TOC [mg/L]
L01	8,2	163	48,4	s/c	6,9	8,6	1,5
L02	7,4	180	43,0	s/c	6,6	7,6	2,9
L04	7,7	180	N/A	s/c/f	6,4	9,0	N/A
L05	8,3	190	43,6	s/c	6,6	9,2	2,1
L06	7,9	189	45,8	s/c	7,1	8,5	1,8
L07	8,2	204	62,6	f	6,9	13,6	1,8
L08	8,1	183	N/A	s/c	6,1	10,3	1,8
L09	8,1	194	48,0	s/c	7,1	7,5	1,8
L10	8,1	179	47,1	s/c	6,7	8,5	1,5
L11	8,3	183	56,7	f	7,1	20,0	2,2
L12	7,9	202	54,0	s/c	7,2	10,2	1,4
L13	8,5	195	44,0	s/f	7,4	8,8	1,5
L14	8,2	195	N/A	c	7,1	22,0	N/A
L15	7,5	246	N/A	s/c	7,9	24,2	N/A
L16	7,5	200	51,0	s/f	6,8	9,0	1,9
L17	8,1	182	N/A	c	7,9	10,0	N/A
L18	7,8	191	N/A	s/c	6,9	8,8	N/A
N	17	17	11		17	17	12
Mean	8,0	191,6	49,5		7,0	11,5	1,8
Std	0,3	17,3	6,1		0,5	5,3	0,4

N/A: not analysed.
Pretreatment: s = sieve; c = centrifugation; f = filtration.