

Environmental characterisation of Reclaimed Asphalt

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Abstract

From a European perspective, the share of recycling of reclaimed asphalt (RA) that is reused in new asphalt surface courses is rather lower than what can be technically achieved. The use of RA in pavement construction can conserve natural resources and reduce environmental impacts (e.g. CO₂ emissions, depletion of natural resources etc). However, for RA to be used to its full potential it must first be fully characterised. This would include an environmental assessment to determine whether or not the RA contains any contaminants associated with its previous use as a pavement material. Without this information the handling and reuse of RA could cause adverse environmental and health effects due to new or higher emissions of potentially hazardous substances. To increase the reuse of RA, and to ensure safe handling during the whole life cycle, it is thus important to correctly characterise these materials and to quantify potential emissions that arise at different stages (e.g. during production, processing and handling etc.). This study outlines a methodology for the environmental characterisation of RA, allowing the identification of potentially hazardous compounds in RA. This includes:

- GC-MS screening to identify potentially hazardous organic compounds susceptible to leaching from RA;
- Identification and evaluation of leaching tests (both with respect to characterization and compliance testing);
- Ecotoxicological testing of leachates, using a biotest battery (freshwater algae, terrestrial plant, water arthropod and fish) that represent different trophic levels.

The study is part of the European FP7 project Re-Road – end of life strategies of asphalt pavements.

Keywords: Reclaimed Asphalt; Characterisation; Leaching; Ecotoxicity; Airborne emissions;

1 Introduction

For recycled asphalt to be successfully reused in high value recycling applications, such as the reuse of asphalt surfacing back into a new asphalt surface, it is necessary to ensure that the environmental characteristics of the material are fully understood. Poor understanding will constrain the reuse of RA as highway authorities will control environmental risks by limiting use. To gain this understanding will involve conducting both risk assessment and life cycle analysis of the various stages of the recycling process. To allow for meaningful analysis it is of course important that the environmental assessment is based on relevant and valid test data. An important task of this work is thus to search for and generate the best possible data and confirm its relevance, i.e. to verify that the data is obtained by the use of valid methods on representative samples of RA. All of these issues are addressed within Re-Road, a European Framework Program (FP7) project (2009-2012), which this study is part of.

The overall research program of Re-Road encompasses the whole life cycle of RA such as production (e.g. milling), processing and handling (e.g. crushing, screening and storage), mixing, use in

pavement, and recycling. In order to conduct exposure assessment, a central task of Re-Road is to model the release, transport and distribution of contaminants due to the different emissions that arise during the life cycle of RA (e.g. particles, fumes, water-borne emissions). This study was outlined to support this work, focusing on water-borne emissions from RA, and on identifying methods that could be used to characterize these emissions.

1.1 Objectives and outline of the study

The overall aim of this study is to provide guidance on the selection of appropriate methods for environmental characterization of water-borne emissions from RA. The specific objectives were to:

- i. identify potential hazardous organic compounds susceptible to leach from RA;
- ii. identify and evaluate methods suitable to assess leaching of hazardous compounds from RA and
- iii. to further characterise these leachates with suitable ecotoxicity tests.

Three different methods to assess leaching were chosen for evaluation. The evaluation comprised:

- assessment of the repeatability of the methods,
- assess the effect of increased leaching time on the leached concentrations, and
- compare levels of leached concentrations of the different methods.

2 Materials and Methods

In total 10 different RA materials were selected for the different experiments of this study. For a more detailed description see Table 1.

Table 1. Materials tested

Sample name	Description	Used in
W-045_10	Open porous asphalt. Surface layer, Germany, ~10 years. Collected from stockpile. Reference material within Re-road.	Screening analysis
W-099_10	Open porous asphalt physically modified with SBS, Surface layer, France.	Screening analysis
E-094_10	Porous asphalts made from a penetration grade 70/100 bitumen, modified through addition of crumb tyre rubber. Sampled after production (i.e. not an RA).	Screening analysis
Contaminated-RA	Sample taken from a mixed source stockpile (containing tar-RA) in Sweden. Sample was crushed (<10mm) and well homogenised.	Leaching (Batch test) Leaching (ER-H test) Leaching (Percolation test) Ecotoxicity tests
RA repository	Sampled taken from mixed source stockpile in Czech Republic.	Leaching (Batch test) Ecotoxicity tests
Reference MIX 1	Re-Road reference material – an 11.2mm stone mastic asphalt containing 0% RA	Leaching (Batch test) Ecotoxicity tests
Reference MIX 2	Re-Road reference material – an 11.2mm stone mastic asphalt containing 15% RA (RA= W-045_10)	Leaching (Batch test) Ecotoxicity tests
Reference MIX 3	Re-Road reference material – an 11.2mm stone mastic asphalt containing 30% RA (RA= W-045_10)	Leaching (Batch test) Ecotoxicity tests
Irish RA	Sample taken from a mixed source stockpile in Ireland. The maximum particle size is 20mm.	Leaching (Batch test) Ecotoxicity tests
50% RA Storbit	Asphalt mix containing 50% RA and the rejuvenator “Storbit”.	Leaching (Batch test) Ecotoxicity tests

2.1 Screening analyses of potential hazardous organic compounds

The aim of the screening analyses was to identify potential hazardous organic compounds that could occur in leachates from RA. Three different types of asphalt pavements, that are suitable for recycling, were used for identification of possible hazardous compounds that could be released and bring

contamination of waters in contact with these materials (Table 1). The three materials and one blank sample were leached in accordance with ISO/TS 21268-1:2007 (Table 2), but with the following modifications:

1. distilled water was used as leachant (no addition of NaN_3 or CaCl_2)
2. several subsamples were leached (in parallel) in order to generate in total >1.5L eluate/sample.

The leachates were extracted using dichloromethane and analysed by GC/MS. Mass spectrum was measured in SCAN mode (Start $m/z = 100$, End $m/z = 400$, Scan speed = 625).

2.2 Leaching tests

Detailed information on the chosen tests for the different experiments is given in Table 2. Below is a short description of the background to the choices of leaching methods that were incorporated into this study.

Table 2. Description of leaching methods and information on studies performed with these methods.

Method	Test design	Leachant	L/S	Duration time	Particle size	Separation method	Was used to study:
CEN/TC351 N0272 ¹	Up-flow percolation test (once-through column test)	0.001 M calcium chloride	From L/S=0-10. 7 eluates are collected	L/S=10 should be reached, thus approx. 1-2 months	The test should be carried out preferably on a sample in the condition as it was delivered to the laboratory.	Leachate is pre-filtrated through 1.5-20 μm in top of the column and off line through 0.45 μm (glass fibre filters).	Repeatability Levels of leached concentrations of 16PAH
ISO/TS 21268-1:2007 ²	One stage batch test	0.001M calcium chloride (and 0.1% NaN_3 if not to be used for ecotoxicological testing)	2	24 h	$\leq 4\text{mm}$	Centrifugation operating at 20000-30000g Or 2000-2500g with increased centrifugation time.	Repeatability Effect of increased leaching time Levels of leached concentrations of 16PAH Production of eluates for ecotox-test
ER-H ³	Recirculation column test	0.001M calcium chloride (and NaN_3)	2	7 days	4mm or 10mm We tested <10mm	No separation technique is used	Repeatability Levels of leached concentrations of 16PAH

¹CEN/TC351-N0272, 2010, Draft Generic horizontal up-flow percolation test for determination of release of substances from granular construction products. N0272.. 2010-01-13.

²ISO/TS 21268-1:2007. Soil quality - Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials - Part 1: Batch test using a liquid to solid ratio of 2 l/kg dry matter.

³ER-H (chemical Equilibrium Recirculation column test for Hydrophobic organic compounds). Preliminary Danish standard (Gamst et al., 2003; Gamst et al., 2007).

Reclaimed asphalt, intended as a component in new asphalt production, is a material that should be seen as a product and not waste. As a product used in a construction (e.g. as surface course in a road) RA falls under the construction product directive, (CPD), and not under the waste directive. Thus, testing of such RA-materials should be carried out by tests designed for construction products and not waste.

Methods for the assessment of release of dangerous substances from construction products are under development within the workgroup 1 (WG1) of CEN/TC351. Two methods that seem suitable for characterisation of RA are a surface leaching test (CEN/TC351, 2009) and a test for granulates;

percolation test (CEN/TC351, 2010). The suggested methods are based on existing standards for leaching of waste and contaminated soil but modified in order to be applicable for characterization of leaching of both organic and inorganic compounds. For this study we have included the pre-standard percolation test to characterise leaching with increased liquid to solid ratio (L/S-ratio).

As a complement to the full characterisation test (i.e. the percolation test) a batch test was also included in the evaluation study. The batch test can serve as a compliance test when a material has previously been characterised but needs verification (to verify that the RA material is complying with reference values). The batch test is faster to conduct and less expensive. Batch tests are also commonly used to produce eluates used in ecotoxicity tests. Of available standards and technical specifications the batch test ISO/TS 21268-1, developed by ISO (2007), seemed most promising. This test is developed to be suitable for both inorganic and organic compounds and allow for subsequent ecotoxicity testing.

In addition, a recirculated equilibrium column leaching test, developed by Gamst et al. (2007), was included as a comparison to the batch test, since previous leaching studies on soil and waste materials claimed that this test generate more reliable and reproducible results than the batch tests (Hansen et al., 2004; Elert et al., 2008).

All tests were used to study repeatability and PAH leaching behaviour; in addition the batch test was used to study the effect of leaching time and produce eluates for eco-tox testing.

2.3 Ecotoxicity tests

Ecotoxicological tests were performed on leachates of the materials prepared in accordance with CEN ISO/TS 21268-1:2009 “Soil quality – Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials – Part 1: Batch test using a liquid to soil ratio of 2l/kg dry matter (ISO/TS21268-1:2007)”. The material was dried in laboratory under 20 – 23°C and prepared by sieving to contain particles less or equal to 4 mm. Larger particles were discarded. The leachate was centrifuged for 5 hours at 2500 g in glass bottles to remove suspended particles. The leachate was divided into 3 parts; 1 l for ecotoxicity testing, 1 l for PAHs analysis and 0.5 l for analysis of selected inorganic elements and turbidity.

An ecological test battery representing different trophic levels was used to evaluate the toxic effect of the leachates, see table 3.

Table 3. Description of ecotoxicity tests performed on the leachate.

Bioassay	Organism	Exposition duration	Measured parameters
EN ISO 6341 Mobility of <i>Daphnia magna</i>	<i>Daphnia magna</i>	48 hours	Immobilisation
EN ISO 8692:2004 Algal growth inhibition	<i>Desmodesmus subspicatus</i>	72 hours	inhibition / stimulation
ISO 7346-2 Acute lethal toxicity to a freshwater fish - Semi-static method	<i>Poecilia reticulata</i>	96 hours	mortality
OECD 208/1984 Terrestrial plant seedling emergence and seedling growth test	<i>Sinapis alba</i>	72 hours	inhibition / stimulation

3 Results and Discussion

3.1 Screening of potential hazardous compounds in RA leachates

Measured chromatograms were analyzed for individual m/z records to single compounds, compounds type (isomers of compound) and compounds groups (group of basic substance derivatives) identification. Standards of some PAHs, some Phthalates and n-Alkanes were used for comparisons to these compounds identification. For compounds where no standard was available comparison with MS spectrum library was used. Identified compounds are summarized in Table 4.

Table 4 Result of the screening analysis.

Compound	m/z record / retention time	X = identified in sample				Comparison with
		W-045_10	W-099_10	Blank	E-094_10	
Phenanthrene	178	X	X	X	X	Standard
Anthracene	178	X	X	X	X	Standard
Fluoranthene	202	X	X	X	X	Standard
Pyrene	202	X	X	X	X	Standard
Benz[a]anthracene	228	X	X	X	X	Standard
Chrysene	228	X	X	X	X	Standard
Benzo[b]fluorene,	252	X	X	X	X	Standard
Benzo[k]fluorene,	252	X	X	X	X	Standard
Benzo[e]pyrene	252	X	X	X	X	Standard
Benzo[a]pyrene	252	X	X	X	X	Standard
Indeno[1,2,3-cd]pyrene	276	X	X	X	X	Standard
Benzo[ghi]perylene	276	X	X	X	X	Standard
Dibenz[ah]anthracene	278	X	X	X	X	Standard
Coronene	300	X				Standard
n-Alkanes	113	X	X	X		Standard
Adipates	129				X	MS spectrum library, 82 % similarity
Phthalates (Diethyl-, Bis(2-methylpropyl)-, Dibutyl-, Benzyl butyl-, Bis(2-ethylhexyl)-, Di-n-octyl phthalate	149	X	X	X	X	Standard
Dimethyl phthalate	163		X			Standard
Benzothiazole	135				X	MS spectrum library, 98 % similarity
Methylacetophenone/Alkylbenzene izomers	119				X	MS spectrum library, 96 % similarity
1-Indanone	8.768 min	X			X	MS spectrum library, 95 % similarity
9-Fluorenone	14.629 min	X			X	MS spectrum library, 72 % similarity
2-Methylbenzothiazole	9.006				X	MS spectrum library, 84 % similarity
3,5-di-tert-butyl-4-hydroxy benzaldehyde	14.768				X	MS spectrum library, 94 % similarity

As expected, PAHs belonging to the group of 16 EPA-PAH were identified in all analyzed samples, but fewer compounds were found in eluates from the virgin asphalt material containing rubber compared to the studied RAs. In addition, the PAH compound coronene was identified in one of the tested open porous asphalt materials (W-045_10). This PAH is produced dominantly by fuel combustion in vehicles engines and is often used as tracer for PAH emissions from motor vehicles and in particularly of gasoline fueled vehicles (Ramdahl et al., 1982; Freeman et al., 1990). Contamination of RA during pavement life (through e.g. exhaust gasses, wear from tires and brakes) is a question of

concern, especially for porous asphalt pavements where more deposits can find space in the open graded mixture. The identification of coronene indicates that exhaust gasses could accumulate in porous asphalt into amounts that causes detectable concentrations in leachates.

Group of n-alkanes was identified in the leachates from the studied RAs (W-045_10 and W-099_10) but also in the blank sample. However, no n-alkanes were found in E-094_10. These compounds are common in chemical processing as raw materials for production of olefins, alcohols, acids, tensides, plasticizers for plastics, lubrication additives, synthetic oils etc. and are also applied as component of degreasing and cleaning media. The n-alkanes can be regarded as non-toxic substances but are ranged into the group of biological active compounds with regard to the effect on human organism (Irwin, 1997).

Group of adipates was identified only in the sample E-094_10. This material is a rubber asphalt which contains rubber from tires. Adipates are used as additives (plasticizers) in PVC products and small quantities of these compounds are used also in tyres. Only very few information about their influence on the environment and human health are available.

Group of phthalates was identified in all samples, and especially in the blank. Phtalates are used also as additives (plasticizers) in PVC products and in rubbers. Phtalates are less volatile relative to adipates so this could be the reason of their occurrence in more samples compared to adipates. Only very few information about their influence on the environment and human health are available.

High amount of benzothiazole was identified in the rubber-asphalt sample (E-094_10). Benzothiazole-based thiazoles are used in the rubber vulcanization process. Existing data for these compounds indicate that they are of concern for aquatic toxicity, irritation/allergic reaction, and low concern for mammalian toxicity and carcinogenicity (ECHA 2012).

Methylacetophenone was detected only in E-094_10. However, it was not possible to confirm, whether it is methylacetophenone isomers, or alkylbenzene isomers (tetramethylbenzene, tert. butylbenzene, dimethylethylbenzenes etc.), since the similarity of all of these compounds was greater than 90%. No data was available about their effects on human health. Some data in literature describes neurotoxic and sensory respiratory irritation effects on mice and rats.

In addition, the rubber asphalt (E-094_10) also contained 2-Methylbenzothiazole and 3,5-di-tert-butyl-4-hydroxy benzaldehyde which is probably product of oxidation of butylated hydroxytoluene.

1-Indanone and 9-Fluorenone were contained in E-094_10 and W-045_10 samples. These compounds are products of PAHs degradation and are thus markers of primary PAHs content in the material.

In conclusion, the results from the blank test (leaching conducted with no material) show that n-alkanes, phthalates and several PAHs are ubiquitous compounds in a laboratory environment. This proves the importance to perform blanks in future testing with quantitative analyses in order to determine background concentrations.

3.2 Evaluation of leaching tests

3.2.1 Repeatability of the methods

The repeatability (Table 5) of the tested methods was calculated as:

$$\text{Repeatability} = \frac{Sdev}{Mean\ value} * 100$$

where the *Mean value* is the arithmetic mean of n=3. In general, the batch test showed the best repeatability of the studied methods for PAHs with low or medium molecular weight (1-11%). However the repeatability of the ER-H method and the percolation test for these compounds must also be considered as acceptable (1-27% for ER-H and 0-61% for the percolation test) considering the heterogeneity of the studied material. The repeatability of the methods when studying PAHs with high

molecular weight where less good (maximum values were 40%, 43% and 47%, respectively, for the batch test, ER-H and percolation test). This can be explained by the higher hydrophobicity of these compounds and their affection to particles and colloids in the eluates. Since none of these methods allows filtration by filters there will inevitably be some particles or colloids in the analysed eluate that consequently will affect the results.

Table 5. The repeatability of the evaluated methods show in % (calculation based on n=3).

PAH	Batch L/S=2	ER-H L/S=2	Percolation Min/max ^a	PAH	Batch L/S=2	ER-H L/S=2	Percolation Min/max ^a
Naphthalene	1	27	0/61	Benzo(b)fluoranthene	32	0	0/47
Acenaphthylene	4	3	1/10	Benzo(k)fluoranthene	30	-*	-*
Acenaphthene	2	18	2/7	Benzo(a)pyrene	32	43	0/43
Fluorene	1	11	0/19	Dibenzo(ah)anthracene	33	-*	-*
Phenanthrene	3	13	0/8	Benzo(ghi)perylene	40	-*	-*
Anthracene	2	16	4/18	Indeno(123cd)pyrene	35	-*	-*
Fluoranthene	7	14	0/11				
Pyrene	11	14	4/12	PAH, sum 16	2	13	1/8
Benzo(a)anthracene	18	13	4/18	PAH, sum carcinogenic	24	16	6/26
Chrysene	21	20	0/27	PAH, sum other	2	12	1/8

*Not calculated due to only one available value above detection limit or values very close to detection limit.

^aMinimum and maximum values for accumulated L/S-values 0.1-10 L/kg

3.2.2 Effect of increased leaching time on the leached concentrations

To evaluate if the default value of 24 h of shaking is enough time to reach maximum concentration of PAHs in the eluate (i.e. if chemical equilibrium or values “near equilibrium” is reached) the time of the test was increased to 48h and 168h (Figure 1).

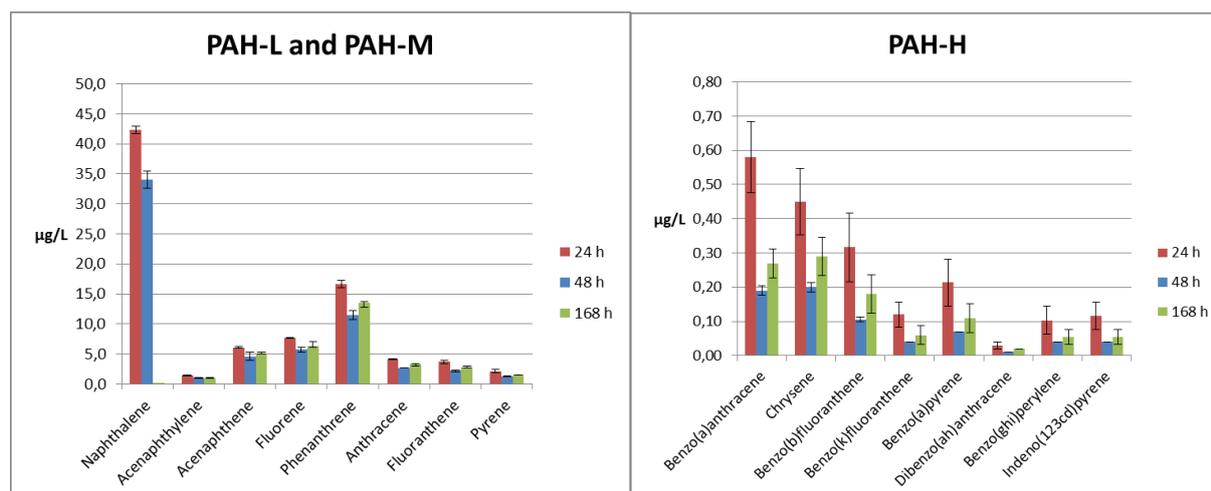


Figure 1 Effect of prolonged leaching time of the batch test. Shaking for 24h (default value) compared to shaking in 48 and 168h at L/S=2.

No trend of increased leaching with increased time could be seen. In opposite to the expected, highest concentrations were obtained in the test running for 24h, however, for several compounds the difference could not be significantly determined from the values obtained at 168h. It must also be

noted that the error of the measurements (calculated as standard deviation from n=3 or n=2) can be much higher than the here presented value (error-bars in Figure X), due to round off errors for several of the compounds that were detected in low amounts.

Consequently, it was concluded that increased time of shaking did not provide higher concentrations in the eluates. However, since the number of tests performed was very limited, and only one material has been studied no conclusions about if chemical equilibrium is reached or not can be draw. In addition, the evaluation of “accumulated leached amounts” (see section 3.2.3) indicates that the leaching of PAH-H may be primarily governed by particulate matter in the leachate. If this is true, the batch test can be ruled out as a test aiming to assess freely dissolved concentrations at chemical equilibrium.

3.2.3 Comparison of the different methods – accumulated leached amounts

In order to compare the levels of leached PAHs between the different methods the accumulated leached amount of individual PAHs at the L/S ratio of 2 was calculated. The results are presented in Figure 2 as $\mu\text{g PAH/kg TS}$.

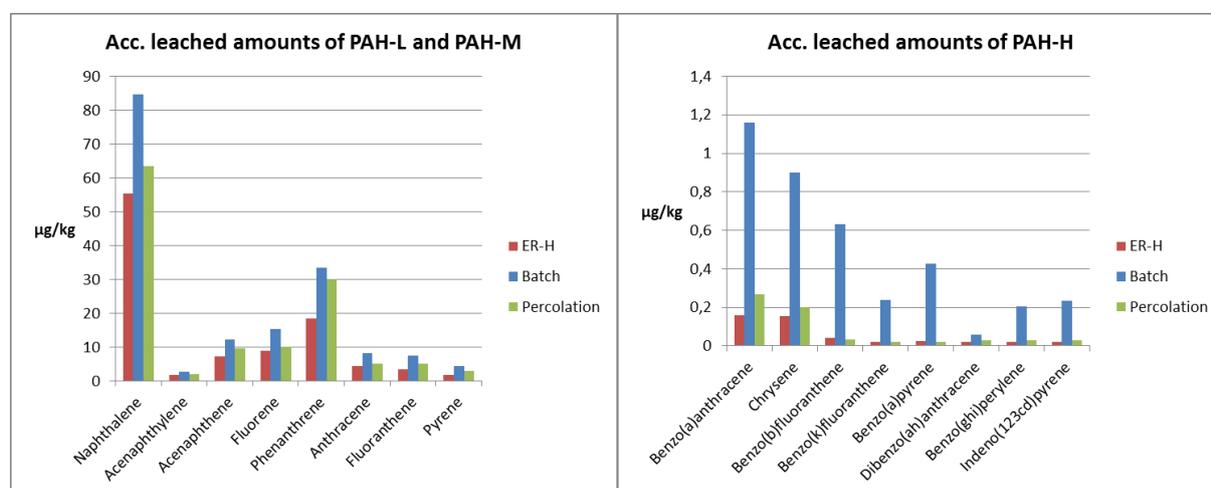


Figure 2. Accumulated leached amounts at L/S=2 obtained by the three different leaching methods.

The results from the batch leaching test indicated that the material was disaggregated, due to the grinding effect when the asphalt-water slurry was shaken in the batch test. In addition, the recommended separation technique (centrifugation at 2000g in 5h) did not result in a successful separation of the two phases and the supernatant had thus a higher content of colloids/particles compared to leachates obtained with the other methods. This was also confirmed by the findings of higher turbidity in the leachates from the batch test; 20.4 FNU compared to 1.2 and 2.0 FNU in leachates from the ER-H and the percolation test (calculated as mean of n=3). Hence, the leachates from the batch reactor test contained up to 20 times higher concentrations of PAH-H than the leachates from the percolation test. For PAH-L and PAH-M this effect was not so pronounced. This is due to the fact that PAHs with high molecular weight are more hydrophobic and they are thus distributed to a higher content to the particulate phase compared to PAHs with lower molecular size. This result was also in agreement with previous findings for leaching of PAHs from contaminated soil using batch tests (e.g. Gamst et al. 2003; Bergendahl, 2005).

In conclusion, the batch test could probably be used as compliance test to the percolation when studying PAH-L and PAH-M but will overestimate the leaching of PAH-H. For this purpose the ER-H method may be a better choice. However, the ER-H method produced lower leached amounts of PAH-L and PAH-M compared to percolation test. To verify these findings more materials should be tested.

3.3 Evaluation ecotoxicity tests

The analysis of the leachates showed that the concentration of inorganic substances (Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, V, Zn) were low and in the same order of magnitude for all leachates. Only a few substances showed an evident variation and concentrations above 10 µg/l (Ba ≤ 30 µg/l, Mn 1-50 µg/l, Zn ≤ 13 µg/l and V 1-20 µg/l). None of the inorganic substances occur in concentrations that are expected to trigger toxic response in the bioassays used.

The concentration of PAH are essentially the same in all leachates and the Σ-PAH-16 varies between 0.8-1 µg/l. The exception being the leachate from the contaminated RA where the levels of acenaphthylene, acenaphthene, fluorene, anthracene and fluoranthene are noticeably raised but Σ-PAH-16 are still not higher than 3.2 µg/l. PAH in the leachates are not expected to trigger toxic response in the bioassays used even if additive effects are taken in account.

Results from the bioassays are shown in Table 6. Animal bioassays show a low response for the leachates and toxic effects are only detected for Repository and Contaminated RA (*Daphnia magna* immobilisation). Plant bioassay also shows a low response for the leachates. Both positive response (stimulation of growth) and negative response (inhibition of growth) was registered. There is a strong correlation ($y = 0,99x - 0,009$, $R^2 = 0,91$) between the response in the aquatic plant bioassay (*Desmodemus subspicatus*) and the terrestrial plant bioassay (*Sinapis alba*).

No relation between analysed inorganic and organic components in the leachates and the response in bioassays could be identified.

Table 6. Response in the bioassays exposed to leachates

Leachate	Animal bioassay		Plant bioassay	
	<i>Daphnia magna</i> immobilisation	<i>Poecilia reticulata</i> mortality	<i>Desmodemus subspicatus</i>	<i>Sinapis alba</i>
Repository RA	10%	No effect	6.1% stimulation	8.1% stimulation
Contaminated RA	5%	No effect	7.5% stimulation	4.7% stimulation
Ref. mix 1	No effect	No effect	2.3% inhibition	3.4% inhibition
Ref. mix 2	No effect	No effect	1.4% stimulation	0.1% stimulation
Ref. mix 3	No effect	No effect	1.1% stimulation	0.1% stimulation
Irish RA	No effect	No effect	1.8% inhibition	3.8% inhibition
50% RA+storbit	No effect	No effect	6.8% inhibition	6.8% inhibition

4 Conclusions

The main conclusions from the screening analysis are:

- PAH were identified in all analyzed samples except coronene that was identified only in the material with 10 years in operation.
- Group of n-alkanes was contained in the most of analysed samples except rubber asphalt leachate.
- Adipates, benzothiazole, 2-Methylbenzothiazole, 3,5-di-tert-butyl-4-hydroxy benzaldehyde, methylacetophenone were contained only in the rubber asphalt leachate.
- 1-Indanone and 9-Fluorenone were identified in rubber asphalt leachate and in leachate of open porous asphalt that was in operation for 10 years.
- Group of phthalates was contained in all samples.
- Results from the blank test (leaching conducted with no material) show that n-alkanes, phthalates and several PAHs are ubiquitous compounds in a laboratory environment. This proves the importance to perform blanks in future testing.

The main conclusions from the evaluation of leaching tests are:

- Although based on a very limited number of samples the conclusion was that all the tests had a sufficient repeatability for use on contaminated RA.

- The prescribed shaking time (24h) of the batch test (ISO/TS 21268-1) appears to be enough to reach maximum concentrations i.e. increased time of shaking did not provide higher concentrations in the eluates. However, the concentrations of PAH-H appear to be more a result of prevalence of particulate matter in the leachate than of reaching concentrations near chemical equilibrium.
- The batch test could possibly be used as compliance test to the percolation method when studying PAH-L and PAH-M, but will overestimate the leaching of PAH-H. For this purpose the ER-H method may be a better choice.

The main conclusions from the evaluation of ecotoxicity tests are:

- A low toxic response for the leachates were detected by freshwater algae, terrestrial plant, water arthropod and fish bioassays.
- No relation between analysed inorganic and organic components in the leachates and the response in bioassays could be identified.

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