Standardization of Release of Dangerous Substances in Construction Products - an Endless Circle?

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• Introduction on leaching
• Standardisation work in CEN/TC351
  • Leaching
  • Eluate and Content analysis
• Aspects of leaching from mortars
  • Sensitivity to pH
  • Leaching behaviour as a monolith
• Statistics, tiered approach and WFT Dossier
• Status of work in progress
• Conclusions
There are some 80 leaching tests described worldwide and still today new methods are presented.

Single step leaching tests like TCLP en DIN S4 have been applied extensively, mainly in relation to landfilling of waste.

Today simple tests are no longer suitable to deal with questions around recycling and reuse of alternative materials in construction where guarantees for long term performance are needed.

However, we do not need 80 tests to assess environmental behaviour of materials and products in intended use, reuse or recycling scenarios.
What is Leaching?

Process by which constituents of a solid material are released into a contacting water phase

**Percolation Release**
- Water passes thru material
- Equilibrium
- High concentration

**Mass Transfer Release**
- Water flows around material
- Diffusion to material surface
- Lower concentration
Monolith leach test CEN/TS-2, PrEN15863 and EPA Method 1315

Mass-Transfer Test
- Semi-dynamic tank leach test

Test Specifications
- Material forms
  - monolithic (all faces exposed)
- DI water so that waste dictates pH
- Liquid-surface area ratio (L/A) of 9±1 mL/cm²
- Refresh leaching solution at 8 cumulative times
  - Up to 36 or 64 days
- Reported Data
  - Refresh time
  - Eluate pH and conductivity
  - Eluate constituent concentrations

Flux and Cumulative Release as a Function of Leaching Time
Equilibrium Leaching Test
- Parallel batch as function of pH

Test Specifications
- 8 specified target pH values plus natural conditions
- Size-reduced material
- L/S = 10 mL/g-dry
- Dilute HNO₃ or NaOH

Reported Data
- Equivalents of acid/base added
- Eluate pH and conductivity
- Eluate constituent concentrations

Titration Curve and Liquid-solid Partitioning (LSP)
- Curve as Function of Eluate pH
### Development of Standards and Materials Covered

<table>
<thead>
<tr>
<th>Test</th>
<th>Matrix</th>
<th>Soil, sediments, compost and sludge</th>
<th>Waste</th>
<th>Mining waste</th>
<th>Construction products</th>
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<tbody>
<tr>
<td>pH dependence test</td>
<td>ISO/TS21268-4</td>
<td>PrEN14429</td>
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<td>Vienna</td>
<td>EN15875</td>
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<td>Reactive surfaces</td>
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<td>Vienna Agreement</td>
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* EPA methods included in SW846  & WI of CEN/TC292  # Not yet adopted in CEN/TC 351 (very relevant for CPR)

**Same basic testing approach in different fields**

*In yellow work ongoing in Working group 1 of CEN/TC351*
Integration of Characterisation Leaching Tests

**GRANULAR MATERIALS**
- pH DEPENDENCE TEST: BATCH MODE
  - ANC, CEN/TS 14429, or EPA Draft method 1313
  - or, COMPUTER CONTROLLED CEN/TS 14997
- PERCOLATION LEACHING TEST
  - CEN TS 14405 or EPA Draft method 1314

**MONOLITHIC MATERIALS**
- Same as granular

**Chemical speciation aspects**
**Time dependent aspects of release**

Test set covers almost any practical condition for any material

Standardisation:
- CEN/TC292, ISO/TC190
- CEN/TC345, CEN/TC351
- SW846 (US EPA)

[Images and diagrams showing laboratory setups for testing.]
RELEVANT pH DOMAINS FOR DIFFERENT FIELDS

pH dependence test covers all relevant environmental pH conditions
Eluate analysis in WG5 of CEN/TC351

Eluate analysis

Analysis of eluates for **inorganic** substances (WI 00351016):
- ICP – OES (62 substances major, minor and trace)
- ICP – MS (62 substances major, minor and trace)
- CV AAS (Hg)
- HG AAS (As, Sb, Se)
- IC (Cl, Br, F)
- GF-AAS (Cd)

Analysis of eluates for **organic** substances still in preparation
- Expected to cover phenols, PAH, PCB, phthalates, PBDE, organotin, dioxin and furans, biocides and pesticides
Aqua regia digestion for content of **inorganic** substances (WI 00351017)

Analysis of aqua regia digests by:
- ICP – OES (62 substances major, minor and trace; WI 00351018)
- ICP – MS (62 substances major, minor and trace; WI 00351019)
- CV AAS (Hg)
- HG AAS (As, Sb, Se)
- GF-AAS (Cd)

Extraction, clean-up and analysis of **organic** substances still in preparation
- Expected to cover phenols, PAH, PCB, phthalates, PBDE, organotin, dioxin and furans, biocides and pesticides
This is the status today:

- CEN/TC351 Robustness work completed (TS-2 and TS-3). Preparation for intercomparison validation. Eluate and content analysis will run in parallel.

- US EPA Intercomparison validation finalized (pH dependence, percolation, monolith, CGLT)

- CEN/TC292 in the process of adopting EPA validation results to upgrade TS to EN’s

From: CEN Guide on validation tasks in the process of standardisation of environmental test methods, April 2008, ENV TC 215rev, supported by SABE Resolution 06/2008 - Validation policy
Range of release behaviour (example vanadium) as function of pH and time for standard mortar bars covering a wide range of OPC and blended cements.
Cement mortar and Recycled Concrete Aggregate

Carbonation will lead to substantial changes in release behaviour of some substances.

Roman volcanic ash mortar fully carbonated after 2000 yrs.

Field exposed recycled concrete aggregate substantially carbonated after 4 yrs.

Carbonation key factor for finishing mortars also.
- Different users of the release information have different needs. Both regulators and industry need information with sufficient detail to allow proper judgment of the materials in their intended use and recycling scenario.

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<th>Tiered approach in testing</th>
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<td><strong>Level of detail</strong></td>
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<td><strong>Frequency of testing</strong></td>
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<td>&quot;compliance&quot; testing</td>
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<td>&quot;characterisation&quot; testing</td>
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- Once the release characteristics of a product type or class are established much simpler conformity testing, if needed, will suffice for potentially critical parameters only at a frequency consistent with the risk of approaching/ exceeding set limit values by notified regulations.

Initial type testing/ Characterisation

Factory production control

WFT Dossier

Hans van der Sloot Consultancy

ECN
Statistics applied to consistent data sets for quality control purposes

Benchmark - pH dependence

Concentration (mg/L)

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<th>pH</th>
<th>Cr</th>
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Single data point comparison

Full test comparison

Upper 90% confidence interval

Mean value for product type

Lower 90% confidence interval
Regulatory context

European Landfill Directive (EU LFD)
End of Waste regulation (EU EoW)
Waste Catalogue (EU WC)
Hazardous Waste Directive (EU HW)
REACH Regulation
Soil Quality Regulation – Fertilizer use
Groundwater Directive

With multiple regulations: preferably not multiple testing and multiple impact judgment approaches for the same material or product
Mortar Leaching Research

Leaching studies currently ongoing at Fraunhofer Institute, IWM and ECN.

Aspects covered:

- Data collection from earlier work (including field data)
- Importing data in LeachXS database
- Monolith leach test on 11 mortars
- pH dependence test on 11 mortars
- Establish relationship between lab and field work

- Information forms the basis for drafting a WFT Dossier for mortars
Conclusions

- The harmonised leaching methods standardised in CEN/TC351 (and USA) are adequate to assess release from construction products such as mortars.
- The pH dependence will be necessary to cope with the requirements from the CPD.
- Bringing data together in a database helps to understand relationships between lab tests and field data, thus providing a benchmark for the sector.
- Such information is highly relevant for a WFT Dossier.
- Geochemical modelling will provide background information on processes controlling release and allow conclusions on long term release behaviour.
- In the case of mortars the role of carbonation on release will be crucial.
Thank you for your attention
Comparison of leaching data on cement mortar with modelling results for cement paste and concrete

Geochemical modelling points at very similar release behaviour for concrete and standard cement mortar bars (same solubility controlling phases), while cement paste features slightly deviating results (to be confirmed experimentally)
Partitioning between solubility controlling mineral phases obtained from modelling.

Partitioning liquid-solid, Cr

Partitioning liquid-solid, V

Partitioning liquid-solid, Cu

Partitioning liquid-solid, Pb