EVALUATION OF DIFFERENT METHODS FOR THE DETERMINATION OF THE FREE ALKALI METAL CONTENT IN THE PORE SOLUTION OF CONCRETE

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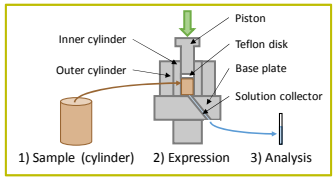
Content

- Review of different methods to measure the free alkali metal content
- Experimental comparison
- Recommendations
- Application of the selected method on a real structure: Votna I dam
- Conclusions

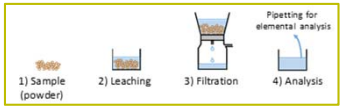
Review

- Pore Water Expression
  - Often used as reference method
  - ”direct” and quick measurement
  - Not applicable for:
    - Relatively dry samples
    - Dense samples
    - Samples with high aggregate content
  - **The representativity of the obtained solution is questioned: effect of the pressure?**
  - **Results in mol/l**



Review

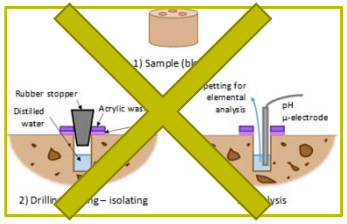
- Ex Situ Leaching methods → 3 variants
  - Hot water extraction
    - Particle size < 160 µm
    - L/S = 10
    - Leaching: boiling 10 min + stand overnight
  - Cold water extraction
    - Particle size < 80 µm
    - L/S = 1
    - Leaching: 5 min
  - Espresso
    - Particle size < 150 µm
    - Combination of steps 2 & 3: addition of boiling water while filtering



→ Release of alkalis from aggregates during the process?
→ Release of alkalis bound to hydrates?
→ Results in mol/g

Review

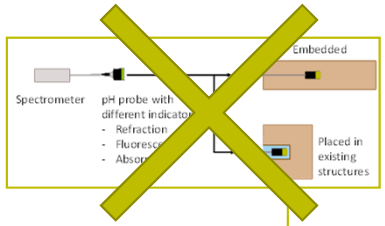
- In Situ Leaching
  - Long process:
    - Sample need to water-saturated → not always possible
    - Couple of weeks to reach equilibrium
  - Not accurate for samples with dense micro-structure



→ **Leaching and carbonation may occur**
→ **Local dissolution?**

Review

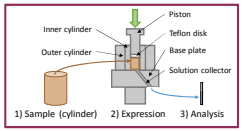
- Fibre optic sensors
  - Leaching of the indicator with time → drift of the signal
  - Higher leaching for pH ≥ 13,5

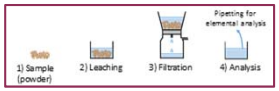


→ **Short lifetime in concrete**
→ **Same drawbacks as for In situ leaching**

Experimental comparison

- Materials
  - Mortars with CEM I and CEM II/B-V (30 % FA)
  - w/b = 0.5
  - s/b = 3, normsand, no release of alkalis
- Methods
  - Ex situ leaching
    - PWE (pore water expression) – mol/l
    - CWE (cold water extraction) – mol/g
    - HWE (hot water extraction) – mol/g
  - Expresso – mol/g
  - Free water content: oven-drying at 105 °C
  - Solutions analysed by ICP

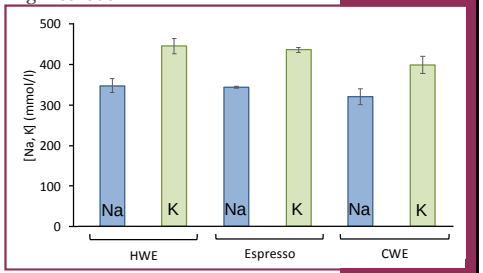




Experimental comparison

- CEM I – comparison between the ex situ leaching methods
  - CWE, HWE and Espresso give all similar results
  - Only the Na & K content can be determined: other element are influenced by phase dissolution. CWE seems to be the less destructive.

→ **CWE is selected**

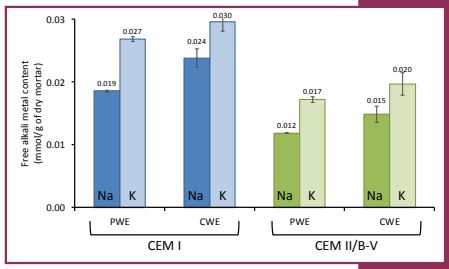


	Method	Ca, mmol/l	
		Average	Std dev
CEM I	HWE	250	102.3
	Espresso	4000	506.8
	CWE	100	1.6

Experimental comparison

- Comparison CWE / PWE
  - PWE gives slightly lower concentrations
    - Pressure effect?
      This study: up to 250 MPa.
      Literature: increase in concentration above 250 or 400 Mpa
    - Additional error induced by the measurement of the free water content
  - Both methods "see" the decrease of the alkali content due to the addition of FA.
  - Parallel investigation on the release of alkali bound by hydrates during CWE: no release

→ **CWE is validated**



Recommendations – choice of the method

- **Goal: Concentration, mol/l**
  - PWE is recommended as a simple and direct method…
    … but its applicability is questionable for field concrete and dense lab samples

    + pH can also be measured with an electrode
  - Among the various Ex situ leaching methods, CWE is recommended…
    … but the free water content needs to be known
    … determination only for Na & K

    + pH can be calculated
- **Goal: content, mol/g**
  - CWE is recommended.
  - PWE can be used…
    … if the free water content is known

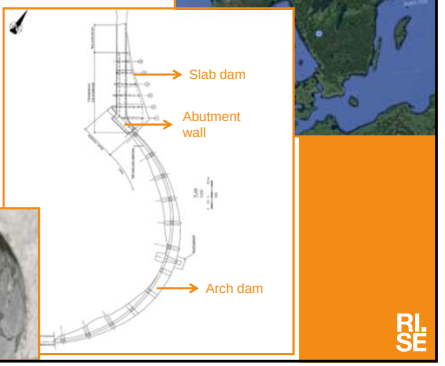
Recommendations – experimental

- **Ex situ leaching:**
  - The filter has to be checked for possible retention of ions
  - The release of alkalis by the aggregates has to be checked
    (Either their types are known or they have to be separated from the paste)
- **Analysis of the solution:**
  - Spectrometry techniques (e.g. ICP)
    → complicated calibration process: matrix reconstruction

Application on an ASR affected dam: Votna I

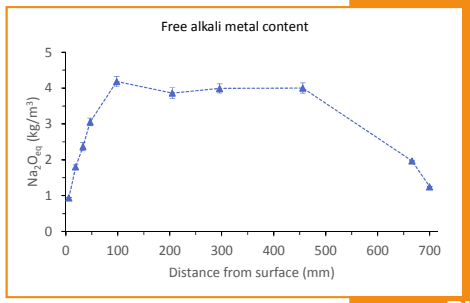
- Situated in South Western Norway
  - Built during 1965-1967
  - Arch dam with abutment wall + slab dam
  - Affected by ASR
  - Hight internal humidity

Photos: Plusquellec



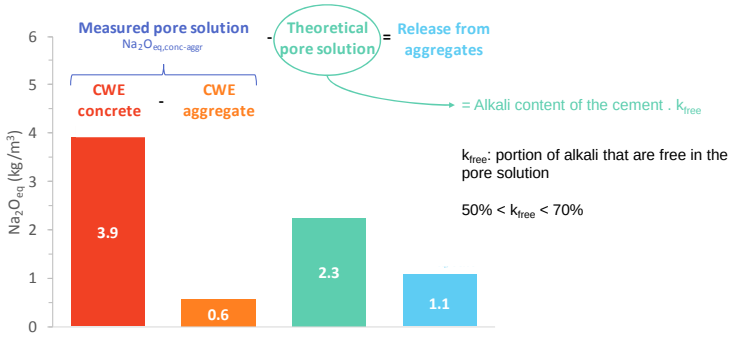
Application on an ASR affected dam: Votna I

- CWE
  - Core extracted from the bottom of the structure:
    "left side": immersed in water
    "right side": internal humidity of the dam
  - Clear leaching profile
    Leaching affects concrete up to 10 cm



Application on an ASR affected dam: Votna I

- Estimation of the alkalis release



Application on an ASR affected dam: Votna I

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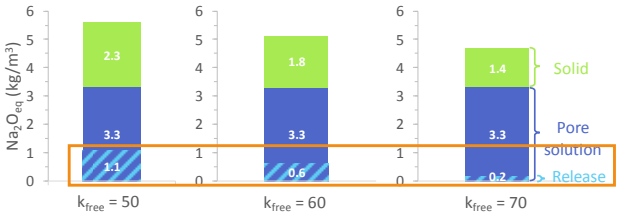

k_{free} is the most important parameter: release is ranging from 0.2 to 1.1 kg/m³ Na_2O_{eq}

Conclusions

- CWE & PWE give similar results
  - CWE is recommended if one wants to measure the free alkalis metal content, i.e. mol/g
    (no need to know the free water content)
  - PWE is recommended for the measurement of concentration, i.e. mol/l
- CWE allows the measurement of profiles
- The alkalis release can be estimated…
  … but more data on k_{free} are needed

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THANKS!!

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