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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

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Determination of the pH and the free alkali metal content in the pore solution of concrete: Review and experimental comparison
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Determining the free alkali metal content in concrete – Case study of an ASR-affected dam
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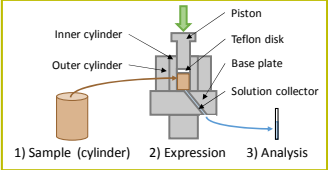
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Review

- 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**



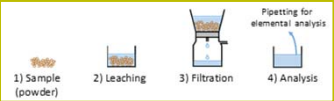
1) Sample (cylinder) 2) Expression 3) Analysis

Labels: Inner cylinder, Outer cylinder, Piston, Teflon disk, Base plate, Solution collector

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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



1) Sample (powder) 2) Leaching 3) Filtration 4) Analysis

Pipetting for elemental analysis

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

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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?

1) Sample (bl...)
2) Drilling... - isolating...
3) Analysis

Labels: Rubber stopper, Distilled water, Acrylic wafer, pH μ-electrode, Setting for elemental analysis.

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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

Labels: Spectrometer, pH probe with different indicators (Refraction, Fluorescence, Absorption), Embedded, Placed in existing structures.

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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
 - 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

Ex situ leaching

Labels: Piston, Teflon disk, Base plate, Solution collector, Inner cylinder, Outer cylinder.

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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

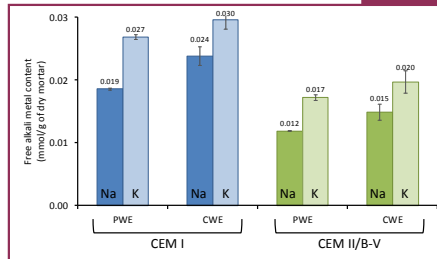
Method	Na, mmol/l	K, mmol/l
HWE	~350	~450
Espresso	~350	~450
CWE	~330	~400

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

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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

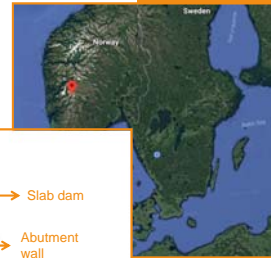
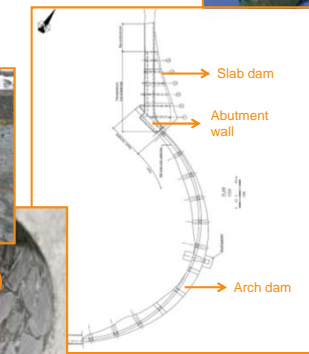
- Ex situ leaching:**
 - The filter has to be checked for possible retention of ions
 - The release of alkalis by the aggregates has to be checked (Either their types are known or they have to be separated from the paste)
- Analysis of the solution:**
 - Spectrometry techniques (e.g. ICP)
→ complicated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

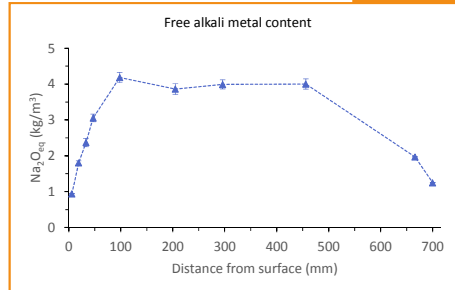


Photos: Plusquellec



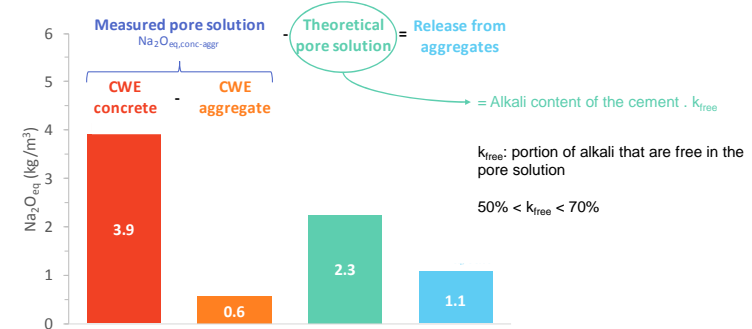
Application on an ASR affected dam: Votna I

- 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



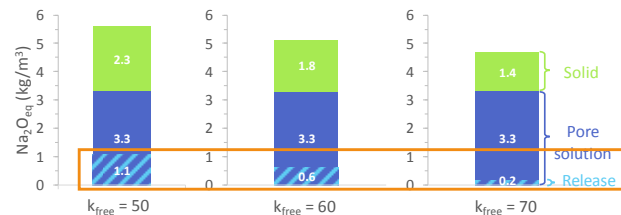
Application on an ASR affected dam: Votna I

- Estimation of the alkalis release



Application on an ASR affected dam: Votna I

- Estimation of the alkalis release



k_{free} is the most important parameter: release is ranging from 0.2 to 1.1 kg/m³ Na₂O_{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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A collage of four images arranged in a 2x2 grid. The top-left image shows a modern, multi-story building with a glass facade and a street scene with bicycles. The top-right image shows a hand wearing a bright blue nitrile glove holding a metal cup filled with a grey, granular material, likely concrete or soil. The bottom-left image shows a close-up of a person in a white lab coat holding a glass test tube. The bottom-right image shows a wall covered in colorful graffiti, with some text visible, including 'Sjunde Skolan' and '1972'.