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PREDIS Final Conference
WP 5 Innovations in liquid organic waste treatment and conditioning

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And all **great Partners**!

05 JUNE 2024

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WP Partners
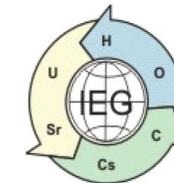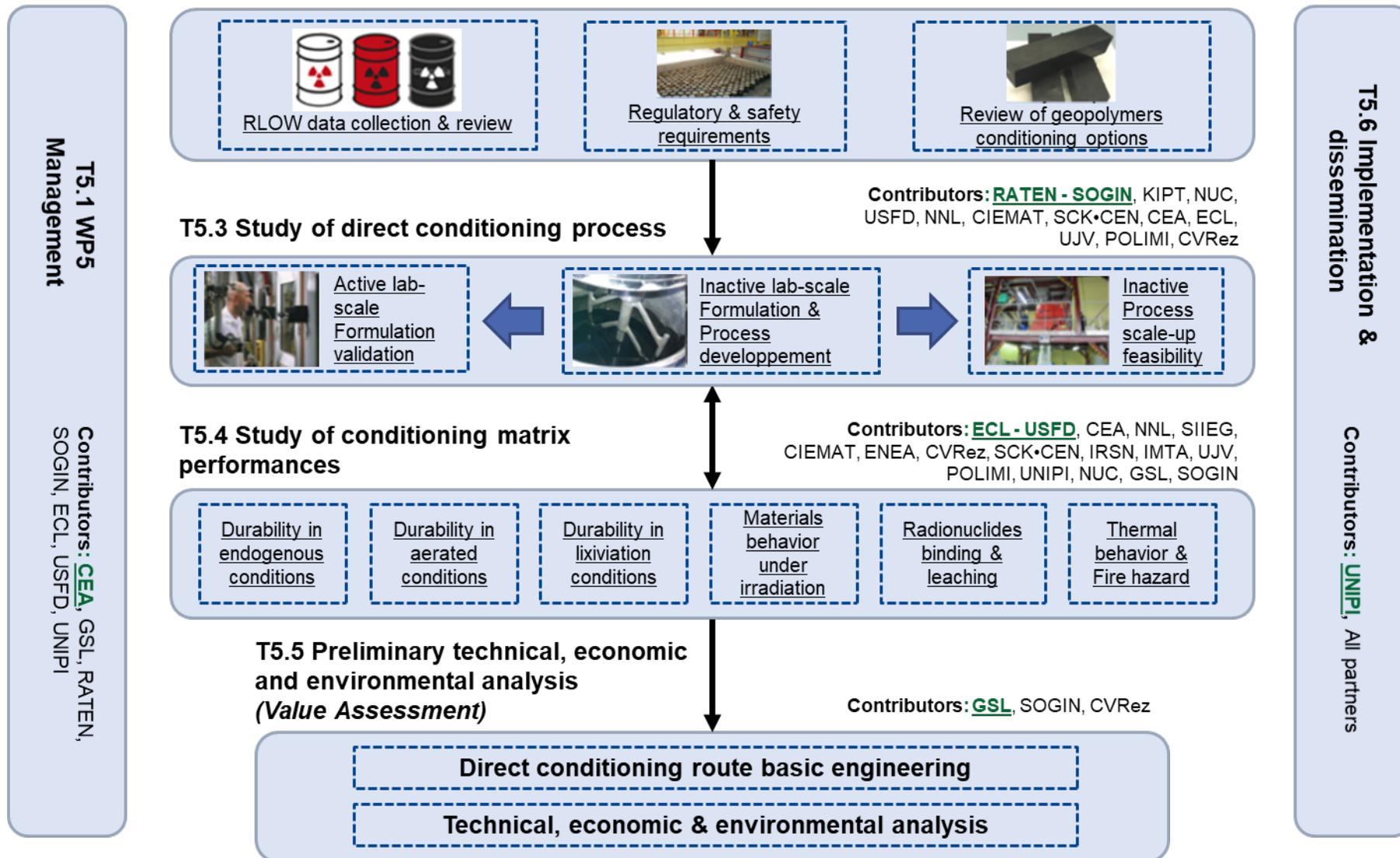
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Outline

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WP Objectives

- Implementing **geopolymers** and related **alkali-activated materials** as mineral binders.

- Fulfilling **technical and economic** requirements related to RLOW:

  - robustness regarding **waste variability**,
  - easiness of **implementation** and **operation** (e.g., mobile units),
  - capacity ranging from **low to large volumes** and limitation of secondary waste,
  - reduction of **disposal costs** by minimisation of volumes (pre-treatment and waste loading).

- **Leading to a final waste showing properties and performances compatible with safety and technical requirements** related to disposal but also prolonged storage and transport.

  - Disposability assessment and demonstration is a key issue and challenge of WP5. In particular, applicability of the direct conditioning route according to RLOW radiological categories (VLLW, LLW/ILW-SL, ILW-LL) and according to disposal facilities features (near-surface and/or intermediate-depth and/or geological) has to be investigated and analyzed.

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WP Expected Impacts

- **<u>Availability of a treatment and conditioning process for RLOW up to TRL 6</u>** including validation tests (real waste) and feasibility scale-up tests.

- **<u>Disposability assessment related to Waste Acceptance Criteria (WAC)</u>** : Availability of a set of experimental and technical data demonstrating the performances of the final waste materials in long-term storage and disposal conditions allowing to assess disposability

    o Leading to final wasteform showing **properties and performances compatible with safety and technical requirements** related to disposal.

- **<u>A shared view of pre-disposal solutions for RLOW</u>** translated into management strategies applied at national or European scales.

- **<u>A fully cooperative working program</u>**, targeting a common goal (development of direct conditioning route for RLOW as common pre-disposal solution), with strong partners interactions where each partner generates a part of the required information and data to be merged together.

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WP5 Structure

Task leader

T5.1 WP5 Management

Contributors: **CEA**, GSL, RATEN, SOGIN, ECL, USFD, UNIPI

T5.2 Collection & review of waste, regulatory, scientific & technical data

Contributors: **GSL**, CEA, KIPT, SCK•CEN, USFD, NNL, RATEN, UJV, SOGIN, CIEMAT

RLOW data collection & review

Regulatory & safety requirements

Review of geopolymers conditioning options

Contributors: **RATEN - SOGIN**, KIPT, NUC, USFD, NNL, CIEMAT, SCK•CEN, CEA, ECL, UJV, POLIMI, CVRez

T5.3 Study of direct conditioning process

Active lab-scale Formulation validation

Inactive lab-scale Formulation & Process developpement

Inactive Process scale-up feasibility

T5.4 Study of conditioning matrix performances

Contributors: **ECL - USFD**, CEA, NNL, SIIEG, CIEMAT, ENEA, CVRez, SCK•CEN, IRSN, IMTA, UJV, POLIMI, UNIPI, NUC, GSL, SOGIN

Durability in endogenous conditions

Durability in aerated conditions

Durability in lixiviation conditions

Materials behavior under irradiation

Radionuclides binding & leaching

Thermal behavior & Fire hazard

T5.5 Preliminary technical, economic and environmental analysis *(Value Assessment)*

Contributors: **GSL**, SOGIN, CVRez

Direct conditioning route basic engineering

Technical, economic & environmental analysis

T5.6 Implementation & dissemination

Contributors: **UNIPI**, All partners

WP Structure

WP Major Achievements
Task 5.2 : Collection and review of data: reference waste

- **Description of RLOW inventories at European level** (owners, radiological and chemical compositions, volumes, storage conditions…) using questionnaires completed by WP5 partners and EUG members.
  - Collation of data and identification of main RLOW materials groups

- Used RLOW inventories to identify **reference wastes**, a combination of:
  Oils – Solvents – Scintillation cocktails
  (Cleaning and decontamination liquids – Organic effluents)

  - **common materials:** give continuity and permit **direct comparison**

    Oils: viscosity-controlled simple oils – Solvents: TBP, Dodecane – scintillation cocktails

  - **partners' own materials:** directly applicable to national context

- A selection of raw materials and additives to formulate matrices:
  Locally-sourced **Metakaolin** and **Blast furnace slag** – **"Mixes (MK + Slag + Fly Ash)"**

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WP Major Achievements
Task 5.3 : Study of direct conditioning process

- The partners involved in this task first investigated basic formulations and processes for direct conditioning of radioactive liquid organic waste (RLOW) in geopolymer and related alkali-activated materials.

- All the promising conditioning formulations were further studied and grouped into **three formulation families, based on: metakaolin (MK), blast furnace slag (BFS), and a mixture of Fly Ash, BFS and MK (MIX)**.

- The **optimisation and robustness of these formulations** were studied with surrogate RLOW, and the optimized reference formulations were further investigated with real RLOW and upscaled.

- In parallel the possibility of an **alternative route** (a two-step pre-impregnation methodology) has also been investigated.

WP Major Achievements
Task 5.3 : Study of direct conditioning process

- ## Identification of 3 reference formulations families

1) NNL formulation – MK based

➢ **Row material:**
- Metamax® - RC: $Al_2O_3 = 43.99\%$, $SiO_2 = 51.48\%$

➢ **Activator:**
- K silicate (K120): $K_2O = 21.3$wt%, $SiO_2 = 30.38$ wt%, $H_2O = 48.32$ wt%

➢ **Optimized formulation:**
- $SiO_2:K_2O = 1.2$
- $K_2O:Al_2O_3 = 1.2$
- $H_2O:K_2O = 13$

➢ **RLOW:**
- Nevastane Oil (20% vol.)

2) SCK CEN formulation – BFS based

➢ **Row materials:**
- BFS = 46.5 wt% ($Al_2O_3 = 11.10\%$, $SiO_2 = 32.40\%$)
- Sand = 28 wt%

➢ **Activator:**
- $Na_2O.2SiO_2.xH_2O$ - 1.5 wt.%
- NaOH (10M) - 5.5 wt.%
- Additional water -18.4 wt.%

➢ **RLOW:**
- Ionic liquid (Aliquat 336) - 9.9 wt. % [1]
- TBP - 19.1 wt. %

(1) Tween 80 surfactant used : 0.5 % and 0.95 % relative to the waste volume

3) KIPT formulation – MIX based

➢ **Row materials:**
- FA = 34 wt.% ($Al_2O_3 = 18$ %, $SiO_2 = 46.12$ %)
- BFS = 20 wt.% ($Al_2O_3 = 6.02$ %, $SiO_2 = 40.6\%$)
- MK = 14 wt.% ($Al_2O_3 = 35.50$ %, $SiO_2 = 51$ %)

➢ **Activator:**
- $K_2SiO_3 = 11$ wt.%
- KOH - 9 wt.%
- Water -12 wt.%

➢ **RLOW:**
- ShellSpirax: from 10% to 40% vol [2]

(2) Castament FW 10 (solid Polyethylene glycol-based additive) used to improve several propertie: 0.5 %

50 vol% loading of Nevastane EP 100 in MetaMax geopolymer (left) and Argicem geopolymer (right).

Geopolymer samples (curing 24h): comp. No.5 (left), and the same with pump oil content of 30 %wt (comp. No.8) of pump oil (right).

Robustness (at least 2 partners for each formulation) to test:
- RLOW variability
- Raw materials variability
- Process variability

- **MK-based :**
  - Oil
  - solvent (TBP-dodecane)
  - Scintillation cocktail
- **BFS-based :** oil
- **MIX-based :** oil

WP Major Achievements
Task 5.3 : Study of direct conditioning process

Figure 1 Samples extracted from the top, centre and bottom of the drum (left) and a close-up photo of the pores in the top sample (right)

Tests with Real RLOW

- No significant differences
- Good leaching resistance
- Decrease in compressive strength with RLOW

	Oil $^{137}Cs + ^{60}Co$	TODGA (solvent) $^{241}Am + ^{244}Cm + ^{152}Eu$	Ionic solution $^{63}Ni + ^{14}C$	Scintillation cocktail $^{63}Ni + ^{14}C$
MK-Based	~30 % vol.	~30 % vol.	~15 % vol.	~15 % vol. ~30 % vol.
BFS-based	~20 % vol.	/	~20 % vol.	~20 % vol.
MIX-based	~30 % vol. No incorporation of real RLOW	/	/	/

MK-based specimen

Leaching test
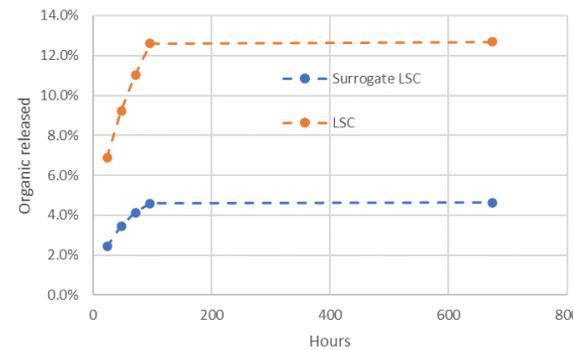
Figure 1 Cumulative percentage organic release for conditioned LSC real waste (orange) and surrogate waste (blue).

Process scale-up

	Nevastane oil 20 and 30 % vol.
MK-Based	50L – 100L Drum (20-30 % vol. pilot-scale) TRL = 6
BFS-based	50L Drum (20 % vol. pilot-scale) TRL = 5 Need formulation optimization
MIX-based	/

Figure 1 Geopolymer composition during mixing (left), after curing (middle) and cut bottom of the drum after curing (right)

WP Major Achievements
Task 5.4 : Study of conditioning matrix performance

Objectives in terms of durability

- Capacity of the developed matrixes (in WP5.3) to avoid RLOW release when immerged in a leaching solution :
  - Analysis of the leaching solution (global measurement)
  - Analysis of the matrix strength evolution (global measurement)
  - Analysis of local matrix damage (permeability)
- Capacity of the developed matrices to resist high temperature
- Capacity of the developed matrices to resist irradiation
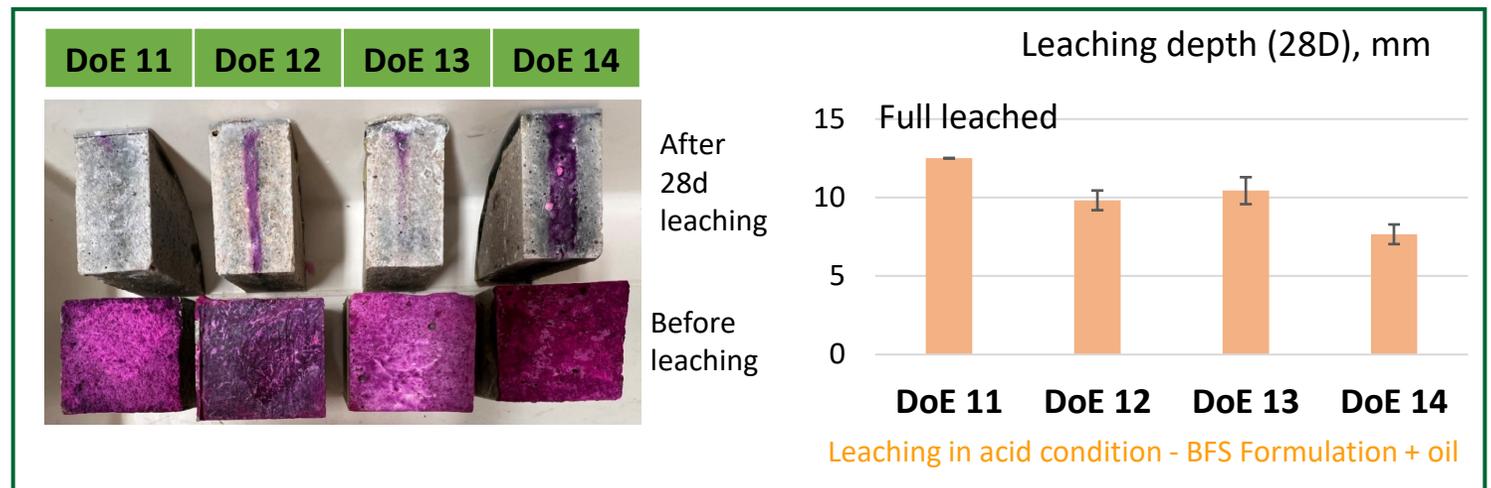
WP Major Achievements
Task 5.4 : Study of conditioning matrix performances

- **Leaching behavior of three formulation families** (see Task 5.3): MK-based, BFS-based and MIX-based formulations were studied

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- For the three formulations (MK-based, BFS-based and MIX-based) : **Storage conditions must be in endogenous and not in aerated conditions (cracks)**

Cracks

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- For the three formulations (MK-based, BFS-based and MIX-based) : **Leaching and waste release** (surrogate or real radionuclides) **in basic and neutral solutions** remains low

Oil release during BFS leaching experiments
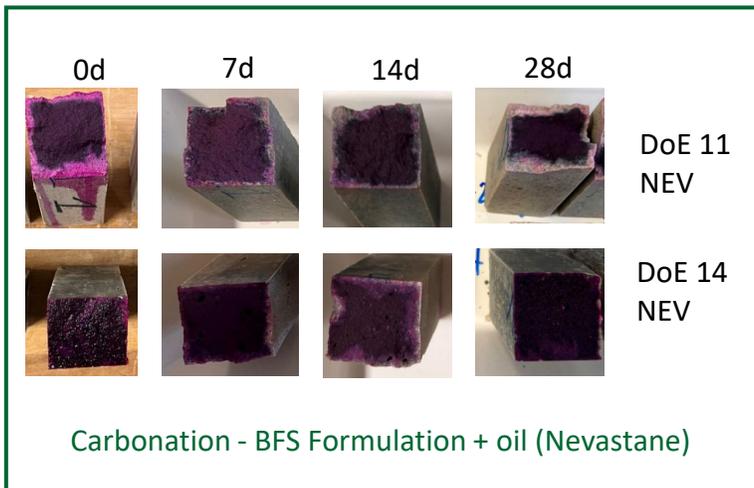
Oil release during MK leaching experiments

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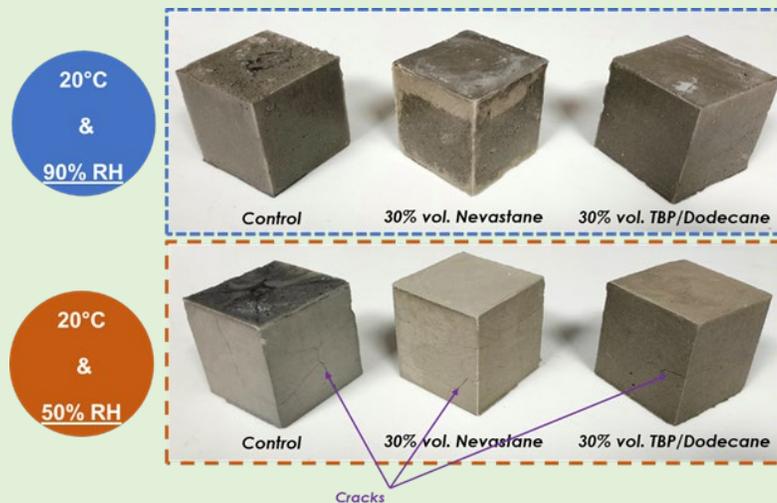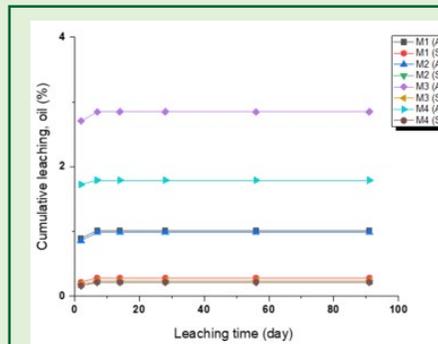
WP Major Achievements
Task 5.4 : Study of conditioning matrix performances

- **Leaching behavior of three formulation families** (see Task 5.3): MK-based, BFS-based and MIX-based formulations were studied
    - For BFS-based and MK-based formulation : **Carbonation** occurs without deleterious effect
    - For MK-based formulation : **Leaching in acid conditions** (HCl pH =1) highly damages the matrix

0d   7d   14d   28d

DoE 11 NEV

DoE 14 NEV

Carbonation - BFS Formulation + oil (Nevastane)

| DoE 11 | DoE 12 | DoE 13 | DoE 14 |

After 28d leaching

Before leaching

Leaching depth (28D), mm

Full leached

Leaching in acid condition - BFS Formulation + oil

WP Major Achievements
Task 5.4 : Study of conditioning matrix performances

- **Stability to irradiation** (^{60}Co and ^{137}Cs sources, up to 200kGy) : No significant impact on leaching
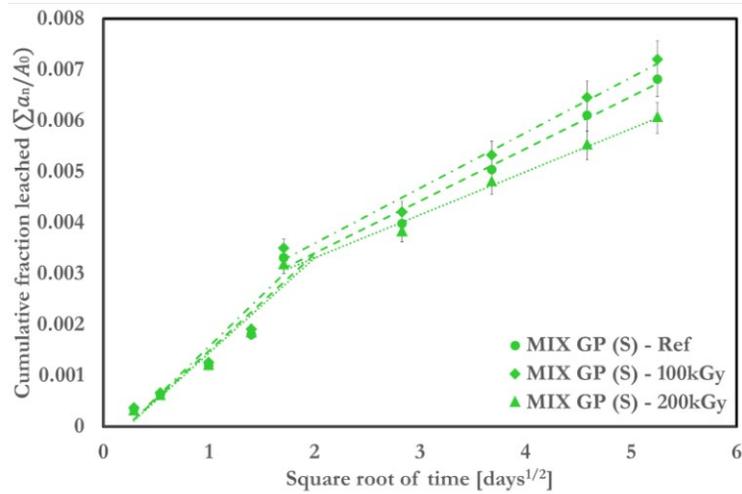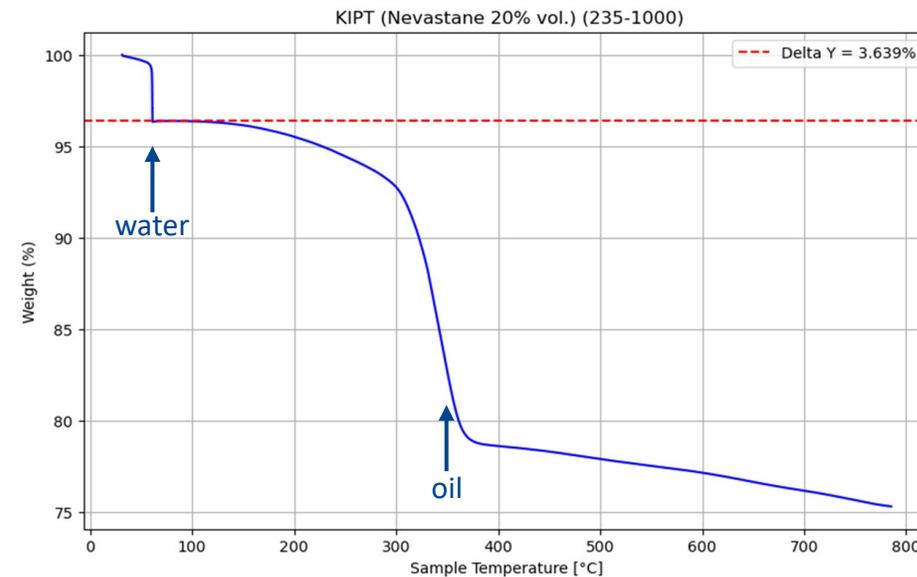
- **Thermal behavior of the three formulations**

Figure 7: Cumulative leached fraction of Si from MIX GP (S).

Irradiation (up to 200 kGy)

TGA Analysis

Fire tests

WP Major Achievements
Task 5.4 : Study of conditioning matrix performances
Disposability assessment (T5.4.9) of RLOW conditioned with various geopolymer formulations

- Evaluation of the RLOW geopolymer waste form characteristics and properties in terms of acceptability for disposal at near-surface and geological disposal facilities

- Comparison of geopolymers with different RLOW with the typical current waste management approach, called **the baseline scenario**

	Oil	Solvent	Liquid scintillation cocktails
Geopolymers	MK / BFS / MIX	MK	MK
Baseline for comparison	Absorption on Experlit or Nochar + cementation	Incineration with IRIS process + cementation	Absorption on Experlit or Nochar + cementation

- Criteria studied: physical form, mechanical stability, homogeneity, void space, free liquids, chelating/complexing agents, leaching

Rating	Risk to disposability
Green	No risk to disposability
Grey	No, limited or partially applicable experimental data
Amber	Limited risk to disposability
Red	Significant risk to disposability

Assessment area	Oil - MK	Oil - MK	Oil - Mix	Oil - BFS	Solvent - MK	Solvent - MK	Solvent - MK	CS - MK	CS - MK	CS - MK
	CIEMAT	NNL	KIPT	CVRez	NNL	POLIMI	UJV	POLIMI	UJV	CIEMAT
Physical form										
Mechanical stability										
Homogeneity										
Void space										
Free liquids										
Chelating agents										
Leaching										

- Disposability assessment is country-dependent as the criteria and their limits differ from country to country.
- Major experiments at a laboratory scale → Many criteria cannot be assessed, because they are waste package and/or radioactivity dependant
- The analysis shows promising results for the encapsulation of oils, while the use of surfactant for the solvent is potentially problematic in some formulations (need of formulation optimization).

WP Major Achievements

Task 5.5 : Preliminary technical, economic and environmental analysis

- **Information and data exchange with WP2**: LCA/LCCA case studies have been provided, comparing cement conditioning against geopolymer conditioning matrices developed in WP5 ➜ *MS37 in Oct 22*

- **Value Assessment**

Used to perform a **strategic analysis of the performance of alternative waste management options** for RLOW studied under WP5

For **WP5**, we identified waste type and treatment / conditioning technology combinations (called **variant scenarios**) for comparison with the typical current waste management approach, called **the baseline scenario**
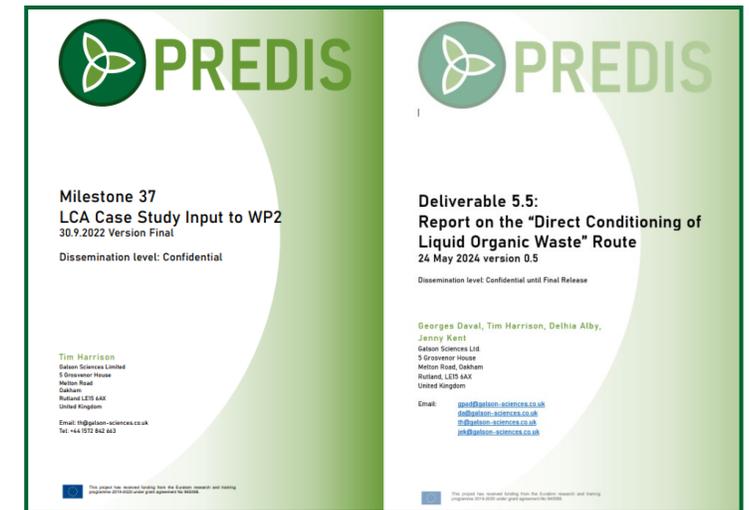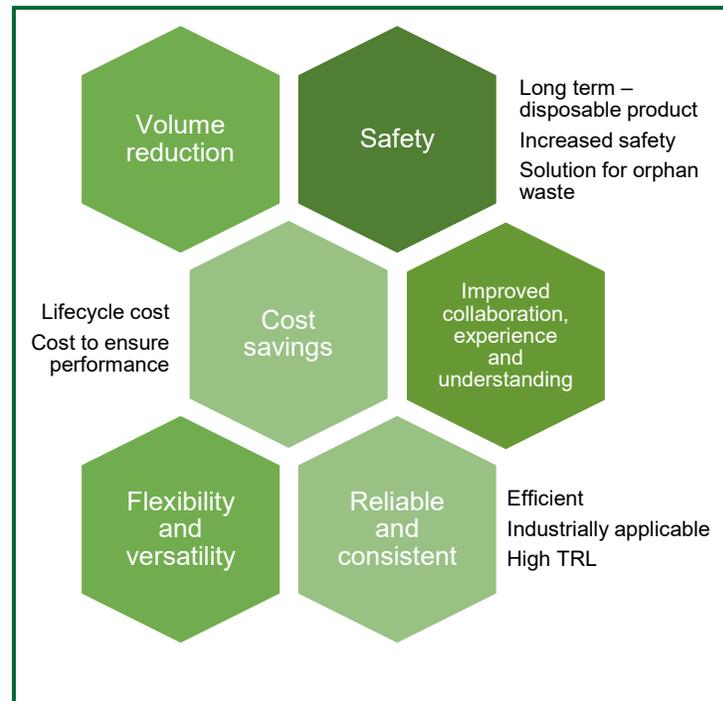
Task 5.5 : Preliminary technical, economic and environmental analysis Value Assessment Workshops

- **Consistent methodology and approach was developed across all PREDIS WPs (4, 5, 6, 7)**

- **WP5 workshop (15 and 16 February 2024)**
  - Consideration of geopolymer encapsulation variants compared with different baseline treatment for oils, solvents, scintillation cocktails
  - Comparison against key attributes done live with online record
  - +2, +1, 0, -1, -2 scoring scale with ratings supported by facilitated workshop discussions
  - Quantitative inputs where possible, including waste loading

- **The higher RLOW waste loading achievable with geopolymers in comparison with absorption/ cementation (oils and scintillation cocktails) leads to benefits in terms of safety, materials use and cost**
  - Benefits not as significant for solvents owing to IRIS process (chosen as the baseline for solvents) leading to large volume reduction
  - Further R&D needed to bring the process towards a TRL 9 is acknowledged and is reflected in the EURAD-2 proposals.

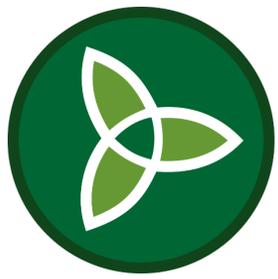PREDIS

WP Overall Summary and Reflections

- **A fully cooperative working program** – After 4 years, in WP5 we have seen amazing teamwork across all partners. Almost all the objectives have been achieved. Information and data sharing have allowed to implement geopolymers and related alkali-activated materials as mineral binders, the development of a direct conditioning route for RLOW as a common pre-disposal solution.

- **A shared view of pre-disposal solutions for RLOW** – All partners have worked together in tasks of WP5, which has help to increase knowledge about pre-disposal solutions for RLOW across different countries, which could be transferred into management strategies applied at national or European scales.

- **Availability of a treatment and conditioning process for RLOW up to TRL 6** – Validation tests with real waste and feasibility scale-up tests have been done. Tests have been done at different drum scale up to 100L for the MK-based formulation leading to a TRL 6. The TRL has been increased during these years also for the other developed Formulations (TRL 4 for the MIX-based and TRL 5 for the BFS-based), but further efforts are required to increase it to TRL 6.

- **Disposability assessment related to Waste Acceptance Criteria (WAC)** - Experimental and technical data demonstrating the performance of the final waste materials in long-term storage and disposal conditions allowing to assess disposability were finished a short time ago. Disposability assessment is country-dependent as the criteria and their limits differ from country to country. Qualitative disposability assessment shows promising results for the encapsulation of oils. Further R&D needed to bring the process towards a TRL of 9 is acknowledged and is reflected in the EURAD-2 proposals, to go further in disposability assessment and to contribute to the description of WAC across the different countries.

Thank you for your attention!

- 11.00-12.15 **WP5 Innovations in liquid organic waste treatment and conditioning**
  - Summary by WP leader, Isabelle Giboire, CEA (20 min)
  - Scientific presentations (40 min)
    - Study of direct conditioning processes:
      - Investigation of reference formulation for real waste Real Waste (POLIMI + UJV)
      - Investigation of direct conditioning Process scale-up (CVRez)
    - Study of conditioning matrix performances (SCK+ ECL)
  - Student presentations (10 min)
    - Sara Koubeissy (ECL)
    - Gabriele Magugliani (POLIMI)

Aim of the work

- **Aim:** study of reference formulations with real waste

  - Are the properties of the final matrix affected by a real RLOW?

  - Possible effects of radionuclides and/or organic by-products content?

  - Possible effects of different physico-chemical properties with respect to surrogate RLOW?

- **General approach:**

  - Characterization of the available real RLOW (e.g. chemical and radiological composition);

  - Conditioning of the real RLOW in the best matrix specifically developed and optimized;

  - Characterization and validation of the waste-form according to WAC

Materials
Real wastes and conditioning matrix

POLIMI materials

- <u>Real RLOW:</u>
  - **Solvent**: ligand in kerosene mixture containing ^{241}Am, ^{244}Cm, and ^{152}Eu
  - **LSC**: spent LSC cocktail (Ultima Gold) containing ^{63}Ni

- <u>Matrix</u>: **MK-based formulation (NNL)**
  - metakaolin (METAMAX *32.5%*)
  - activator (KOH *10%*, BETOL *40%*, water *17%*)
  - **RLOW LF = 30 %v.**
  - **Surfactant:** Tween-80

- <u>Real RLOW characterization</u>
  - **Solvent**: radionuclides at **5 kBq/L each** + stable lanthanides Eu, Nd, Ce (1 g/L)
  - **LSC**: ^{63}Ni at **20 Bq/L** + stable Ni (100 mg/L) and Cs (5 mg/L)
  - → **Activity in leachates likely < DL**
  - → ICP-MS on stable elements (similar behaviour)

  - Real LSC waste already **contains water** as it includes 20%v. 1 M HNO_3
  - → **Water to be compensated** in the activator

Materials
Real wastes and conditioning matrix

UJV materials

- ### Real RLOW:
  - **Scintillation Cocktail** containing **^{14}C, ^{63}Ni**; *ROTISZINT R Cocktail, ROTH Company, Karlsruhe, Germany*

    Activity: $^{14}C \sim 0.56$ Bq/ml, $^{63}Ni \sim 0.84$ Bq/ml
  - **Ionic Solution** containing containing **^{14}C, ^{63}Ni**, uranium content below detection level; *Sigma-Aldrich Company, USA*

    Activity: $^{14}C \sim 12.05$ Bq/ml, $^{63}Ni \sim 11.04$ Bq/ml

- ### Matrix:
  - **Blast Furnace Slag** (BFS); *Moravia Steel, Trinec, Czech Republic*
  - **Geopolymer** (GP) = **Metakaolin & Activator**; *Mefisto L05, Czech Shale Plants Company, Nove Straseci, Czech Republic*
  - + alkali activator $Na_2.3SiO_2$, 10M NaOH + sand + water

Methods
Definition of protocols

POLIMI samples preparation according to NNL protocols

Pre-emulsification

Solvent waste: LF = 30 %v.

TODGA in K/O (95:5) + surfactant → Silverson 3 min → Add activator solution → Silverson 3 min → Add MK → Silverson 13 min → Manufact

LSC waste: LF = 30 %v.

MK + Activator solution → Silverson 3 min → Add LSC → Silverson 13 min → Manufact
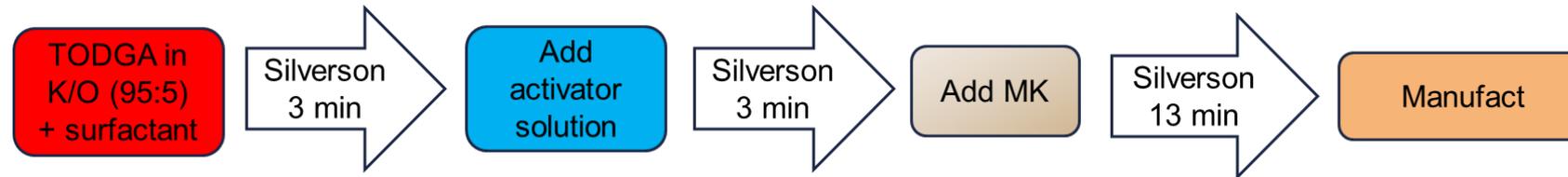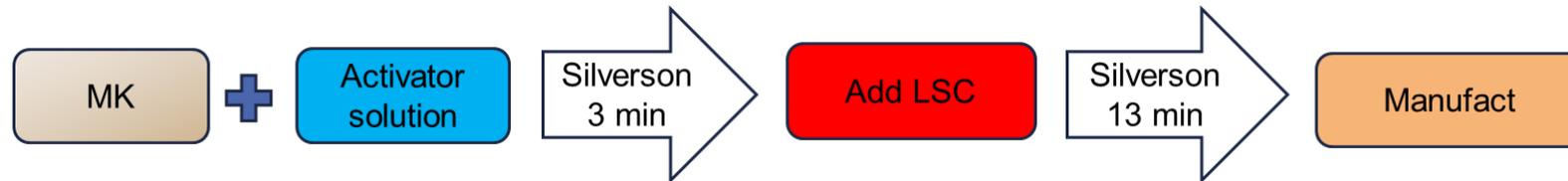
- cylinders for **leaching** test (Ø = 2.5 cm)
- cylinders for **compression** tests (Ø = 4.6 cm) w/ "surrogate real" waste (inactive)

Samples for compressions
Samples for leaching
Samples for characterizations
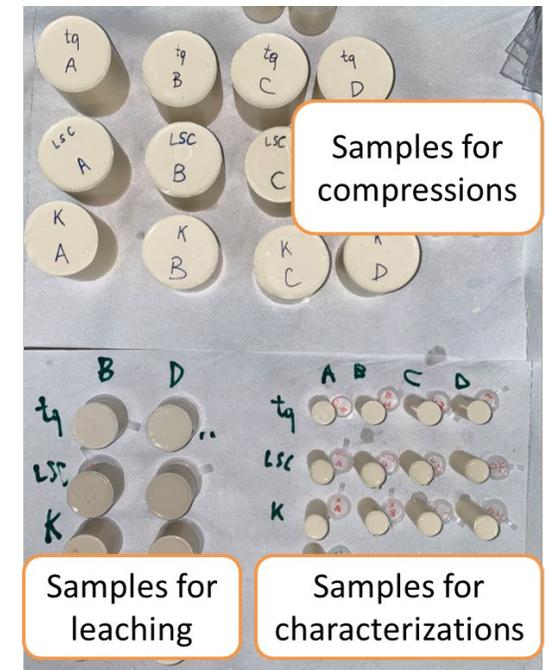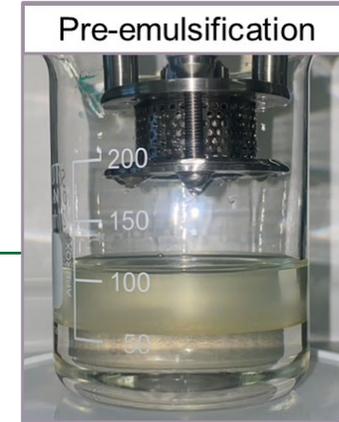
Methods
Definition of protocols

UJV samples preparation

- **Specific weights of the components**

Matrix	g	MK g	Activator g	RLOW (addition of 1.5 ml $^{63}Ni/^{14}C$)	ml	g	Na$_2$O.3SiO$_2$ g	10M NaOH ml	g	Sand g	Add. water g	wt% of RLOW fulfillment
BFS	465.44			scintilliation cocktail	100.0	86.3	15.15	46.87	62.33	279.93	50.0	9.9
	465.44			ionic liquid	100.0	118.0	15.15	46.87	62.33	279.93	50.0	13.5
GP		600.0	400.0	scintillation cocktail	100.0	86.3						8.6
		600.0	400.0	ionic liquid	100.0	118.0						11.8

Curing of samples

Samples after curing

- **Samples**
  - Cubes 50x50x50 mm
  - Cylinders V = 20 ml
  - Curing of samples: 28 days under controlled wet conditions in a closed box
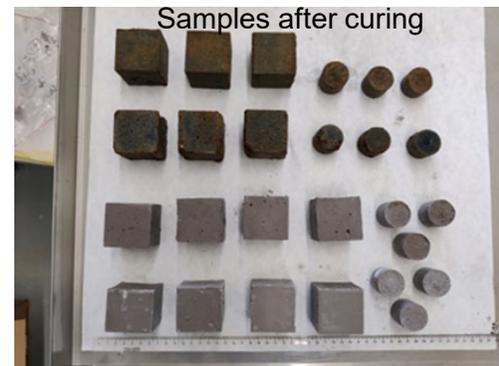
Methods
Definition of protocols for characterization

- **Leaching tests: ANSI/ANS-16.1.2019**

  - Leachant: ultrapure water (22 °C)

  - Duration: <u>1 week</u> (extended to 1 month)

  Daily renewal of the leachant for the first week, then weekly

  Characterization of the leachates (L_1, …):

  - Measure of <u>pH</u> and <u>conductivity</u>

  - Measure of released <u>matrix constituents</u>, <u>contaminants</u>, and <u>organic liquid</u> (COD) for calculation of <u>leachability indices</u>

- **Porosity: water saturation method**

- **Compression tests** on surrogate and real RLOW samples, after 28d of curing.

1 sample n leaching intervals (Δt_1 to Δt_n)

Monolith (all faces exposed)

Δt_1 Δt_2 Δt_n

A_1 A_2 … A_n

leaching solution exchange between leaching intervals

L_1 L_2 L_n

n analytical solutions

MEGA 11-300 DM1-S Presser
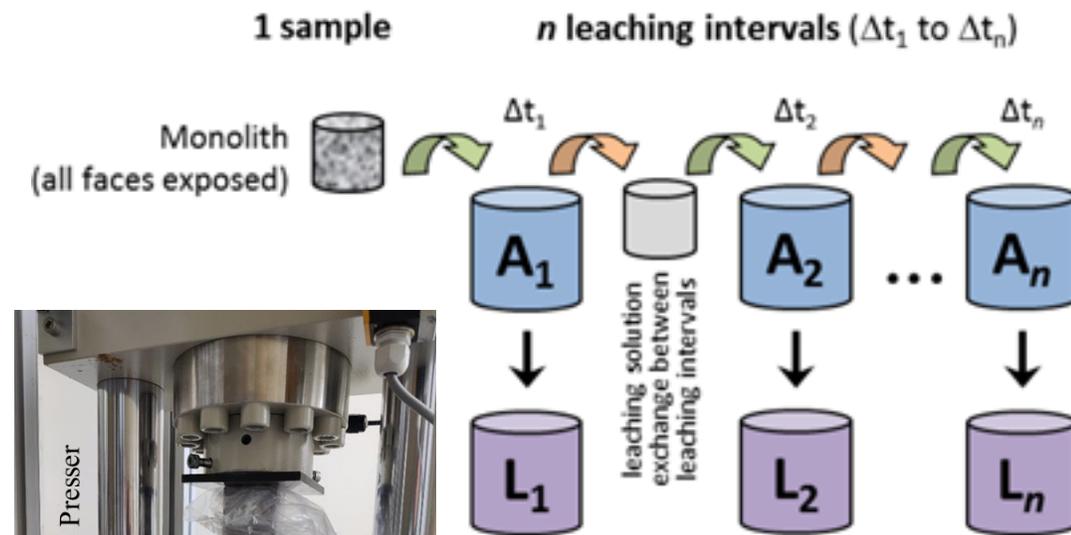
Results
POLIMI – Real RLOW in MK-based geopolymer (part 1/2)

- **Workability**: excellent

- **Bleeding**: approx. 1-2% (all samples)

- **Setting time**: 5-8 h (all samples)

- **Compression resistance (R_c) and porosity**
  - → RLOW loading causes Rc degradation
  - → RLOW loading causes slight porosity increase
  - → Water immersion generally causes Rc degradation
  - → Negligible difference between real and surrogate RLOW

- **Phase composition (XRD – not reported)**
  - → The material is almost completely amorphous
  - → No differences due to ageing conditions (immersion)
  - → No differences between samples with/without waste

	SAMPLES	R_c (MPa)	Porosity %
	only matrix	18.6 ± 0.9	32.8 ± 1.6
	only matrix - immersed	16.1 ± 0.8	n.a.
LF = 30 %v.	LSC	8.9 ± 0.9	36.6 ± 1.8
	LSC - immersed	7.1 ± 0.7	n.a.
	real LSC	**9.6 ± 0.9**	**36.1 ± 1.8**
	real LSC - immersed	**11.1 ± 0.5**	n.a.
	TBP/ker	10.1 ± 0.5	34.6 ± 1.7
	TBP/ker - immersed	6.3 ± 0.6	n.a.
	real solvent	**12.1 ± 0.6**	**37.6 ± 1.9**
	real solvent - immersed	**9.3 ± 0.9**	n.a.

Results
POLIMI – Real RLOW in MK-based geopolymer (part 2/2)

- **Leaching resistance**: Leachability indices (L_i)

	Matrix constituents			Main contaminants		
SAMPLES	K	Si	Al	Cs	Ni	Ce
only matrix	8.7 ± 0.3	11.4 ± 0.2	13.3 ± 0.5	9.7 ± 0.2	11.3 ± 0.2	14.4 ± 0.4
LSC	8.6 ± 0.1	11.7 ± 0.2	13.5 ± 0.5	9.6 ± 0.2	11.7 ± 0.2	13.2 ± 0.4
real LSC	8.6 ± 0.3	11.8 ± 0.2	12.5 ± 0.5	10.3 ± 0.3	11.8 ± 0.2	/
TBP/ker	8.1 ± 0.2	11.0 ± 0.2	12.5 ± 0.5	9.6 ± 0.2	9.0 ± 0.3	13.4 ± 0.4
real solvent	8.6 ± 0.2	11.3 ± 0.2	12.6 ± 0.5	/	/	14.5 ± 0.5

LF = 30 %v.

Lower L_i → higher release (WAC$_{lt}$: $L_i > 6$)

→ K and Cs are the most leached (higher mobility)

→ Also for Co, Eu, Nd, U, Th, the $L_i > 10$

→ **Negligible difference due to RLOW loading**

→ **Negligible difference between real and surrogate RLOW**

Results
UJV – Compression tests result

Compressive Strength [MPa]			addition of ^{63}Ni	addition of ^{14}C
inactive BFS matrix	20.64	BFS + scintillation cocktail	19.88	13.28
		BFS + ionic liquid	21.37	21.59
inactive GP matrix	21.11	GP + scintillation cocktail	25.71	18.6
		GP + ionic liquid	21.93	16.51

- **Strenghts in the range of 13.28 – 25.71 MPa**

- **Max. strenght: GP + scintillation cocktali (25.71 MPa)**

- **No significant differences of values between the inactive and radioactive samples**

Results
UJV – Leaching tests results

Leaching tests
Dependence of the Li on the number of days for Ni-63

- BFS - scintillation cocktail
- BFS - ionic solution
- GP - scintillation cocktail
- GP - ionic solution

Leaching tests
Dependence of the Li on the number of days for C-14

- BFS - scintillation cocktail
- BFS - ionic solution
- GP - scintillation cocktail
- GP - ionic solution
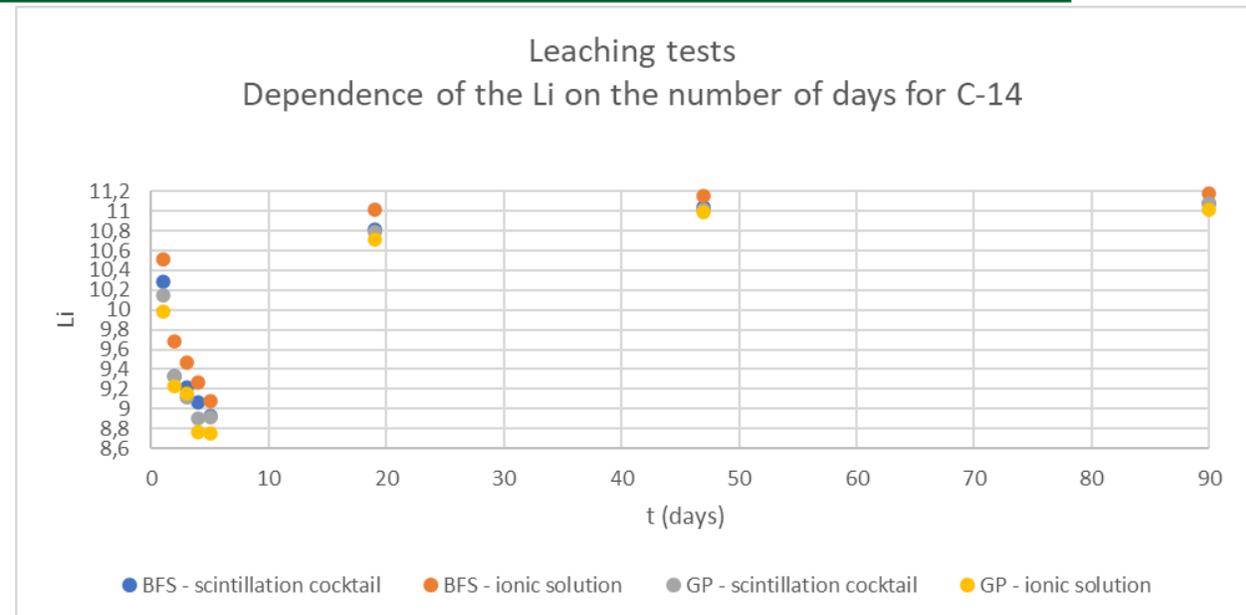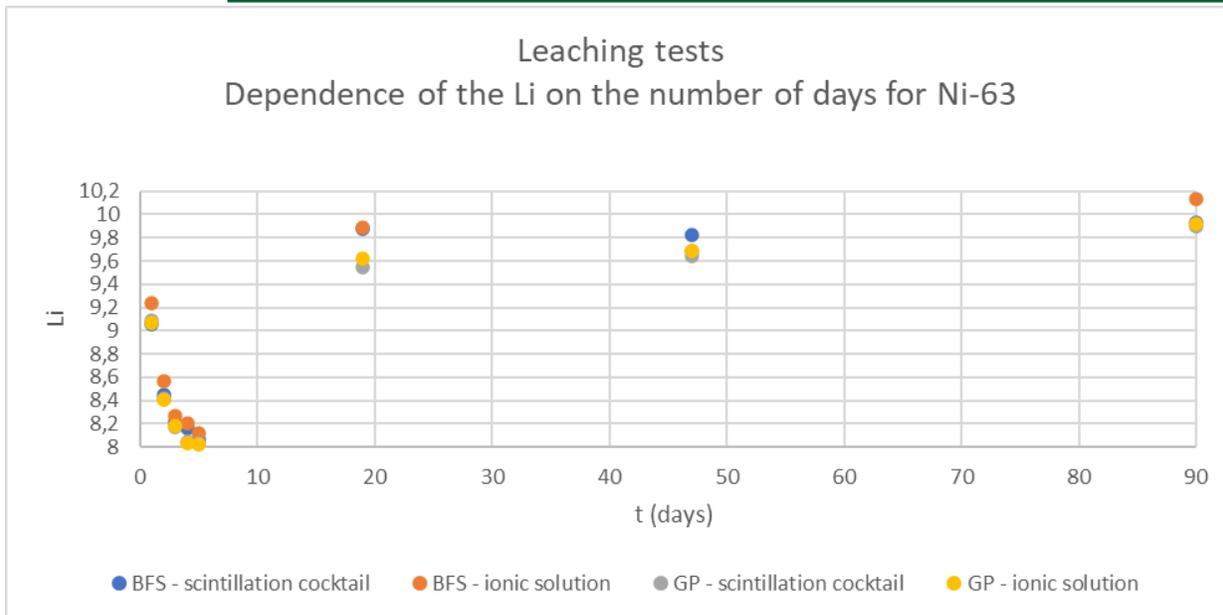
- The **required minimum value** of L_i (> 8 for CZ ~ lower than 3.10^9 Bq/m^3) was attained for all tested samples

- There is **negligible difference between different RLOW and different matrices**

- The max. value of L_i was reached at 90d of leaching for both radionuclides (BFS matrix, ionic solution RLOW)

Main conclusions

Different real RLOW were tested, characterized and conditioned in geopolymeric matrices.

- **Are the properties of the final matrix affected by a real RLOW? Possible effects of radionuclides and/or organic by-products content?**

  - **acceptable workability, setting time and bleeding** with/without RLOW;

  - **reduced R_c (still acceptable)** and increased porosity for samples containing the RLOW;

  - **no phase composition differences** (XRD) for aged samples with/without RLOW.

- **Possible effects of different physico-chemical properties with respect to surrogate RLOW?**

  - **no changes in leaching resistance** for samples loaded with real/surrogate RLOW;

  - the required minimum value of the leachability index (L_i) was attained for all tested samples.

PREDIS

PUBLIC Technical Workshop – WP5

Investigation of direct conditioning process scale-up

ANNA SEARS

VOJTĚCH GALEK, PETR PRAŽÁK, MARTIN VACEK

AVIGNON 5TH JUNE 2024

The objective of work and key activities

- Advance the understanding and application of geopolymers on a larger scale.
- Test selected geopolymer formulations for direct conditioning of liquid organic RW surrogate.

- Scale-Up Experiments: Test feasibility and effectiveness on a larger scale.

- Temperature Measurement: Provide temperature data to our project partner SCK-CEN for modelling to understand thermal behaviour.

- Homogeneity Evaluation: Wet coring and samples analysis to ensure a uniform distribution of materials within the geopolymer matrix.

- Compressive Strength Tests: Evaluate the mechanical strength of the resulting geopolymer with incorporated liquid organic RW surrogate.

Equipment and setup

Solidification Device

- top mixer with horizontal and vertical movement
- independent drum rotator
- compatible with 50 L and 100 L drums

Mixing setup

- speed up to 1000 rpm
- mixing time 35 - 45 min
- raw material added in parts during the mixing process

Equipment and setup – thermocouple layout

- 50L Drum

  - five inside thermocouples - each contained three sensors
  - five surface thermocouples

- 100 L Drum

  - six inside thermocouples - each contained three sensors
  - six surface thermocouples

- Drum Specifications:

  - 50 L drum: 704 mm high, 303 mm diameter
  - 100 L drum: 820 mm high, 482 mm diameter
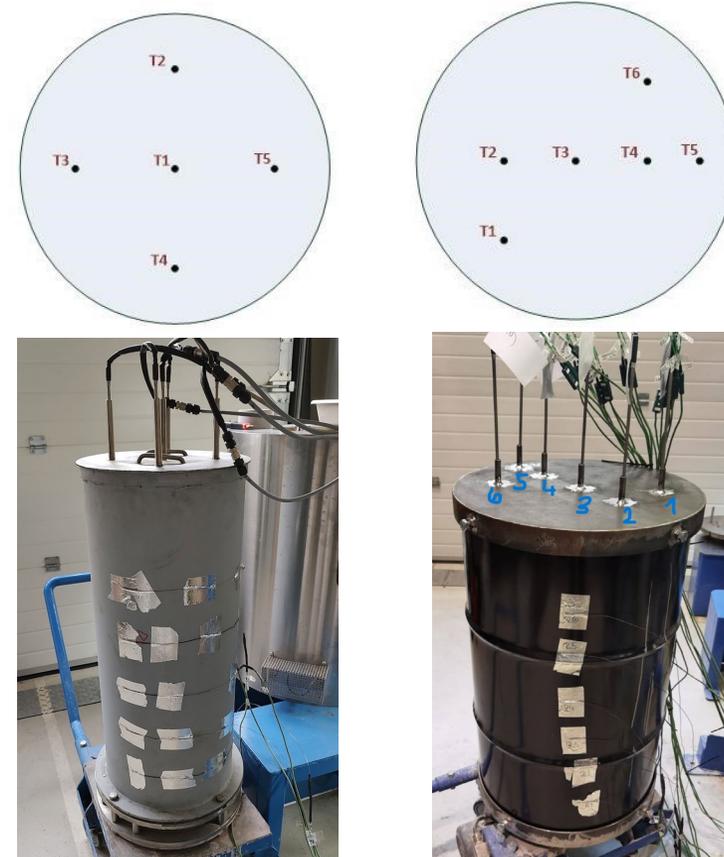
Raw materials - MK

- metakaolin Mefisto L05
- commercially available from ČLUZ a.s.
- premix alkali activator potassium silicate solution (35 wt.%)

Mefisto L05

Physical properties	Typical value	Guaranteed value
Loss during annealing (%)	2.20	0.3 - 0.4
Moisture (%)	0.50	-
K_2O Specific surface (m^2/g)	12.69	-
Pozzolanic activity (°C)	4.30	min. 4.0
Degree of whiteness	60.00	-

Mefisto L05

Al_2O_3	40.10 wt.%
SiO_2	54.10 wt.%
K_2O	0.80 wt.%
Fe_2O_3	1.10 wt.%
TiO_2	1.80 wt.%
MgO	0.18 wt.%
CaO	0.13 wt.%

Raw materials - BFS

- finely ground (< 100 µm) granulated BFS

- from Třinec Iron and Steel Works CZ

- locally obtained quartz sand as the added component

- BFS preparation protocol same as for the laboratory scale (SCK-CEN)

- added surfactant Tween® 80

05-0586 (*) – Calcite, syn – $CaCO_3$
35-592 (*) – Akermanite, syn – $Ca_2MgSi_2O_7$
35-0591 (*) – Merwinite, syn – $Ca_3Mg(SiO_4)_2$

Intensity / a.u.

2 theta / deg. (CoKalpha)
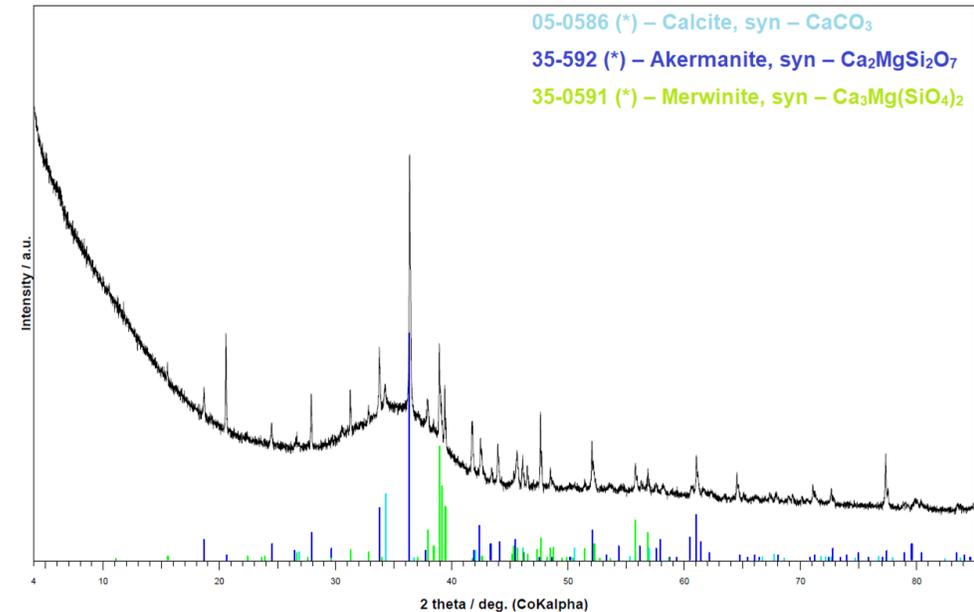
Experiment overview

- **MK-based**
  - 50 L, no oil
  - 50 L, 10 wt.% Nevastane oil
  - 100 L, 10 wt.% Nevastane oil -> wet coring
  - 100 L, 20 wt.% Nevastane oil -> wet coring

- **BFS-based**
  - 50 L, 10 wt.% Nevastane oil, with added surfactant Tween®80
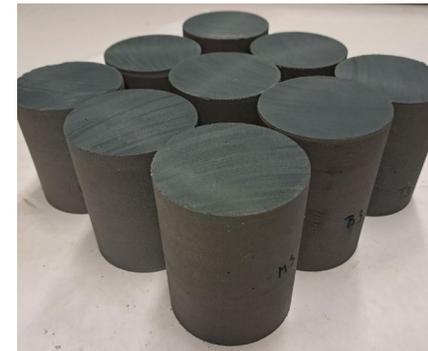
MK-Based Geopolymer - 50 L Scale-Up

Formulation	No oil	10 wt. %
MK	36 kg	36 kg
Alkali activator	24 kg	24 kg
Nevastane	-	6 kg
Surfactant	-	-
Highest recorded temperature during curing		
Inside	32 °C	75 °C
Timescale	9 hrs	4 hrs
Drum surface	23 °C	-

- cutting the drum base for homogeneity assessment

MK-Based Geopolymer - 100 L Scale-Up

Formulation	10 wt. %	20 wt. %
MK	109 kg	102 kg
Alkali activator	73 kg	68 kg
Nevastane	18 kg	34 kg
Surfactant	-	-
Highest recorded temperature during curing		
Inside	75 °C	59 °C
Timescale	9 hrs	11 hrs
Drum surface	45 °C	30 °C

- wet coring
- compressive strength analysis

Wet coring and sample analysis – 10 wt.% oil, 100 L

- Wet coring after 20 days of curing

  - HILTI DD 350-CA diamond drilling system
  - three cylindrical samples, diameter 100 mm

- Compressive strength analysis

  - MTS 300 Exceed®
  - according to the Czech National Standard CSN EN 12390-3
  - **21 ± 0.3 MPa**

- Mercury porosimetry

  - AutoPore IV 9500
  - pores of 0.01 µm predominant within the sample matrix
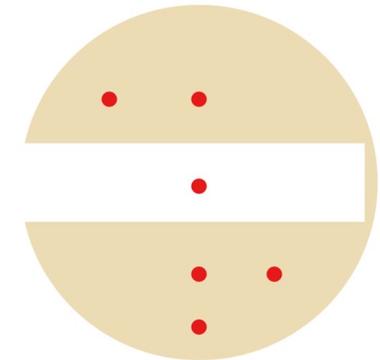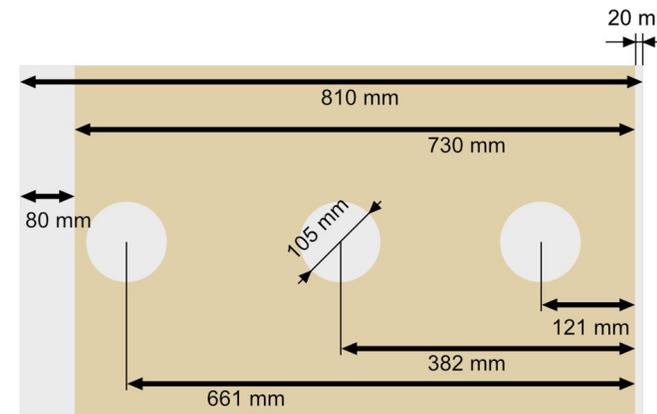
Wet coring and sample analysis – 20 wt.% oil, 100 L

- Wet coring after 6 days of curing

  - HILTI DD 350-CA diamond drilling system
  - three cylindrical samples, diameter 100 mm

- Compressive strength analysis

  - MTS 300 Exceed®
  - according to the Czech National Standard CSN EN 12390-3
  - **15 ± 1 MPa**

- Mercury porosimetry

  - AutoPore IV 9500
  - pores of 0.01 µm predominant within the sample matrix
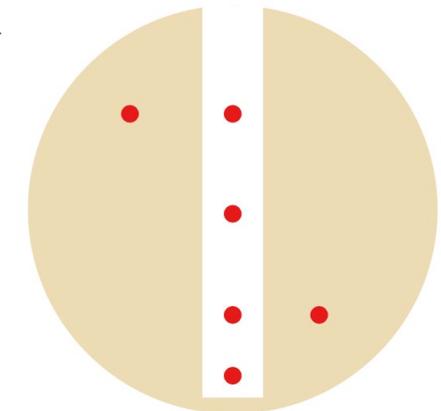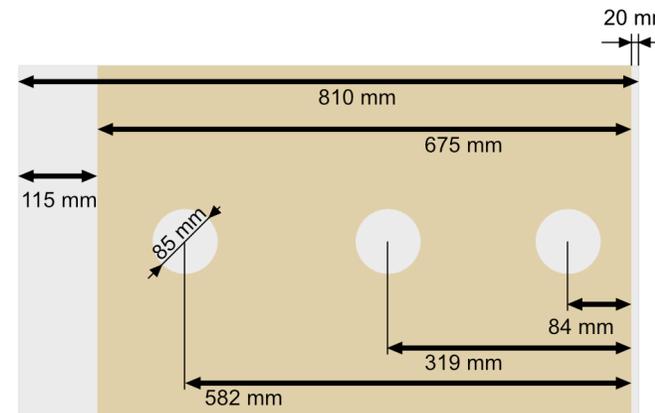
BFS-Based Geopolymer - 50 L Scale-Up

Formulation	10 wt. % oil
BFS	23 kg
Sodium silicate	0.8 kg
Sodium solution	3 kg
Additional water	9 kg
Quartz sand	14 kg
Nevastane	5 kg
Surfactant	0.8 kg

Highest recorded temperature during curing	
Inside	23 °C
Timescale	23 mins
Drum surface	21 °C

- suboptimal texture and softness

Temperature profile - inside the drum
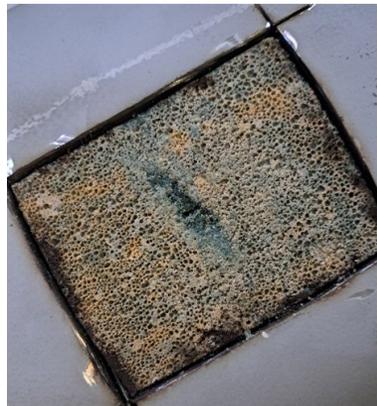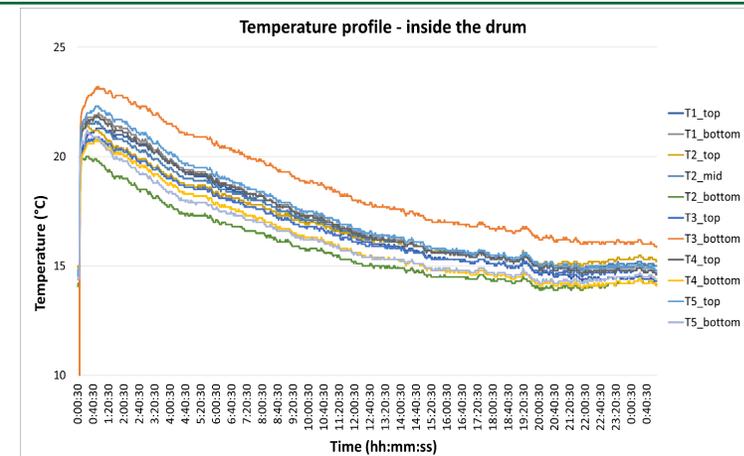
Key findings

- <u>MK-based</u>

  - Both 50 L and 100 L scale-ups were successful with Nevastane incorporation.
  - Similar temperature profiles, higher temperature with added oil compared to no oil (50 L).
  - Sufficient compressive strength of samples.
  - All MK samples were homogeneous, with no visible cracking.

- <u>BFS-based</u>

  - Lower maximum temperature during curing compared with MK (23 °C).
  - Suboptimal texture, softness, and possible separation of solidified phases.

Conclusion

- Insights into the behaviour of geopolymers on a larger scale.

- Successful scale-up experiments with MK-based geopolymers in 50 L and 100 L drums.

- Further optimisation and exploration of BFS formulation is needed to enhance performance.

Thank you for your attention

Objectives

- **Performance Evaluation:** Long-term performance of conditioning matrices for radioactive liquid organic waste:
  - Durability in endogenous and aerated conditions
  - Durability in acid & liquid lixiviation conditions
  - Irradiation
  - Radionuclide binding and leaching
  - Thermal behaviour and fire hazard

Joint effort of 11 partners
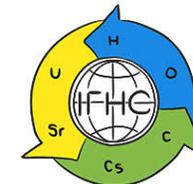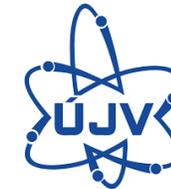
Reference geopolymers and waste types

- **Reference formulations**

NNL formulation – MK based

➢ **Row material:**
- Metamax® - RC: $Al_2O_3 = 43.99\%$, $SiO_2 = 51.48\%$

➢ **Alkaline activator:**
- K silicate (K120): $K_2O = 21.3$wt% $SiO_2 = 30.38$ wt% $H_2O = 48.32$ wt%

➢ **Optimized formulation:**
- $SiO_2{:}K_2O = 1.2$
- $K_2O{:}Al_2O_3 = 1.2$
- $H_2O{:}K_2O = 13$

➢ **RLOW:**
- Nevastane Oil (20% vol.)

SCK CEN formulation – BFS based

➢ **Row materials:**
- BFS = 46.5 wt% ($Al_2O_3 = 11.10\%$, $SiO_2 = 32.40\%$)
- Sand = 28 wt%

➢ **Alkaline activator :**
- $Na_2O.2SiO_2.xH_2O$ - 1.5 wt.%
- NaOH (10M) - 5.5 wt.%
- Additional water -18.4 wt.%

➢ **RLOW:**
- Ionic liquid (Aliquat 336) - 9.9 wt. % [1]
- TBP - 19.1 wt. %

(1) Tween 80 surfactant used : 0.5 % and 0.95 % relative to the waste volume

KIPT formulation – MIX based

➢ **Row materials:**
- FA = 34 wt.% ($Al_2O_3 = 18$ %, $SiO_2 = 46.12$ %
- BFS = 20 wt.% ($Al_2O_3 = 6.02$ %, $SiO_2 = 40.6$%)
- MK = 14 wt.% ($Al_2O_3 = 35.50$ %, $SiO_2 = 51$ %)

➢ **Alkaline activator :**
- $K_2SiO_3 = 11$ wt.%
- KOH - 9 wt.%
- Water -12 wt.%

➢ **RLOW:**
- ShellSpirax: from 10% to 40% vol [2]

(2) Castament FW 10 (solid Polyethylene glycol-based additive) used to improve several propertie: 0.5 %

- **Waste types**

  - TBP-Dodecane (up to 30%)

  - Scintillation cocktails (up to 10%)

  - Lubricating oils (up to 40%)

PREDIS

Carbonation

- **Accelerated conditions (1% CO_2, 60% RH)**
  - Waste types affect the carbonation rate
  - Higher waste loading and w/b ratio → less carbonation resistance
  - Wasteform > reference
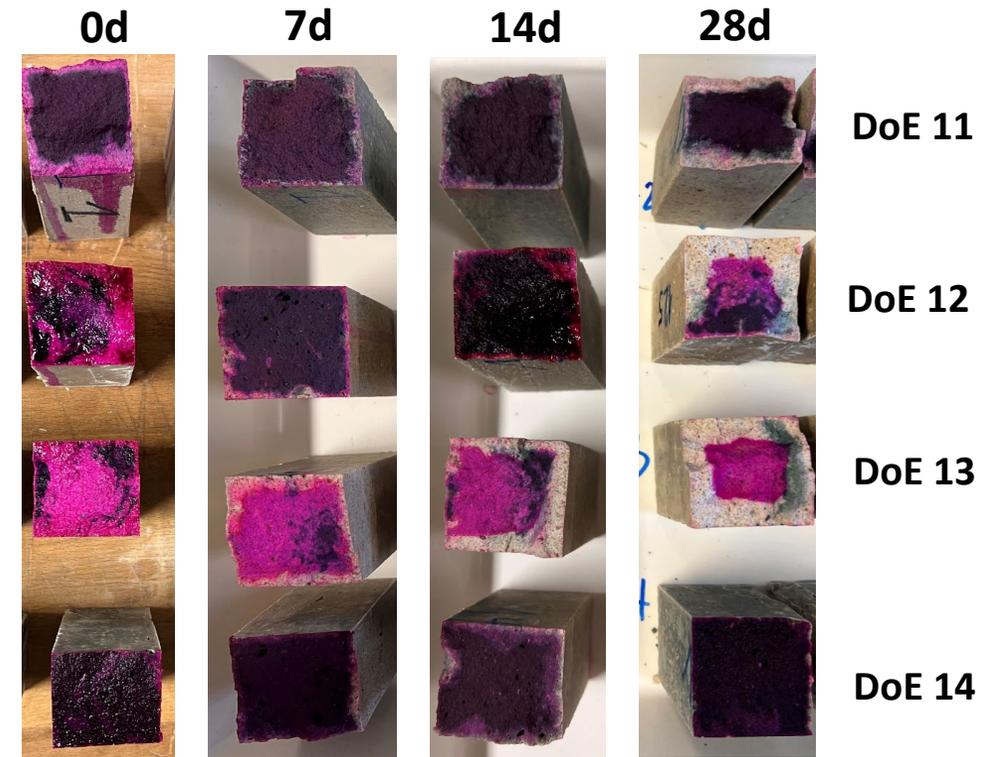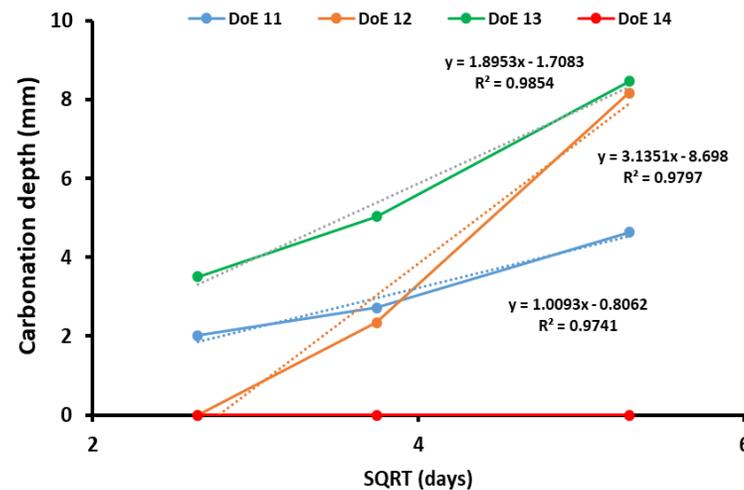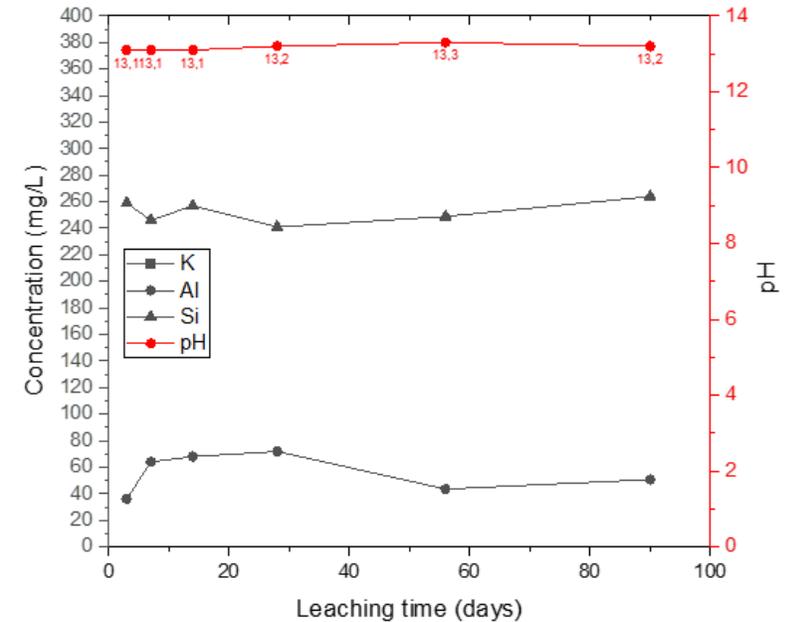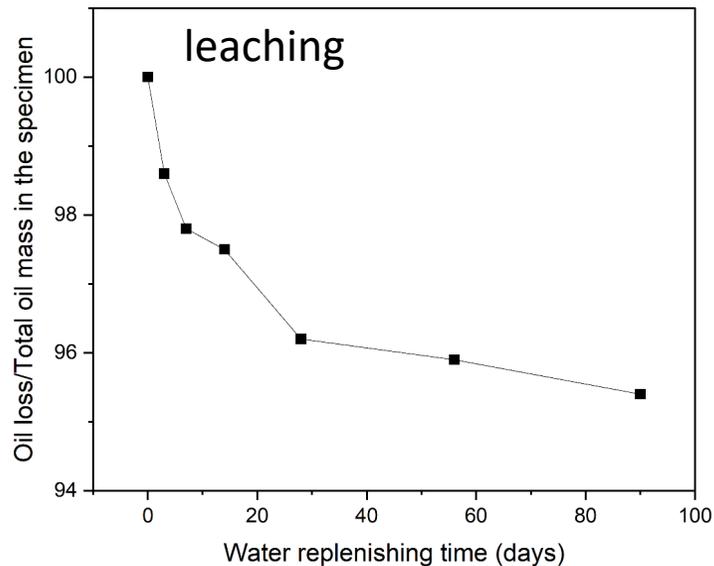
Carbonation
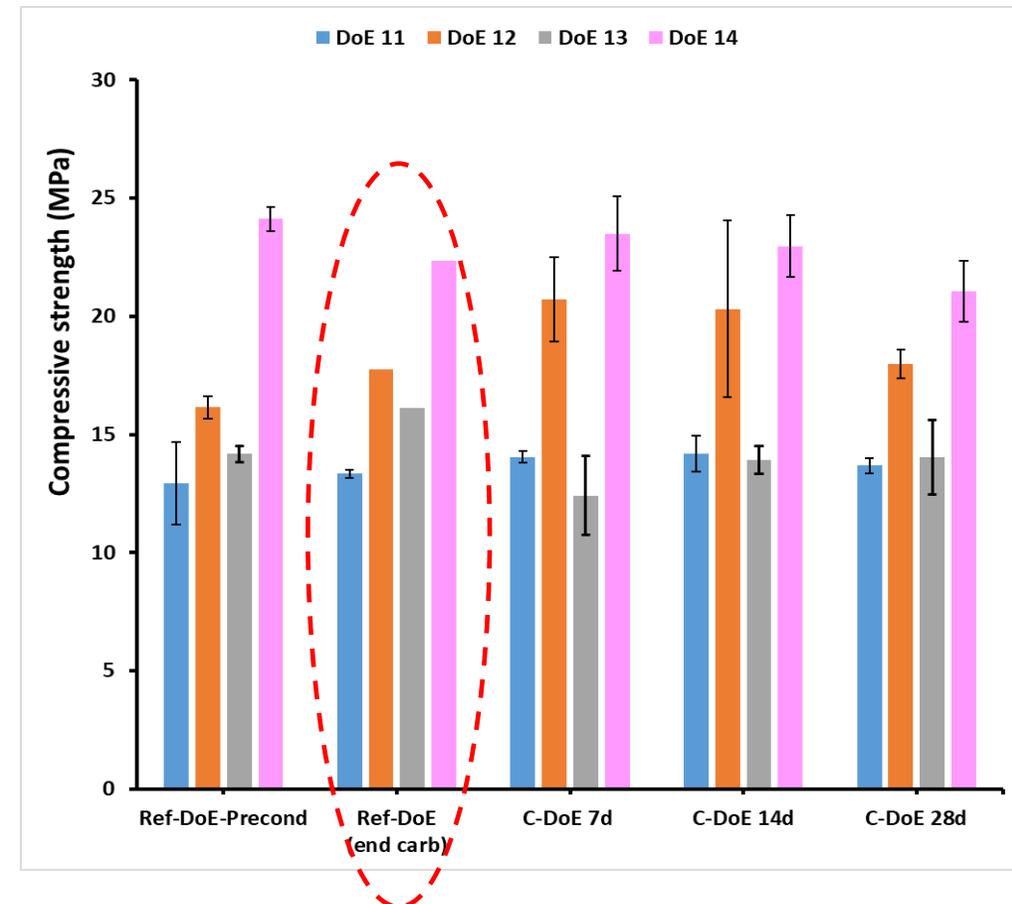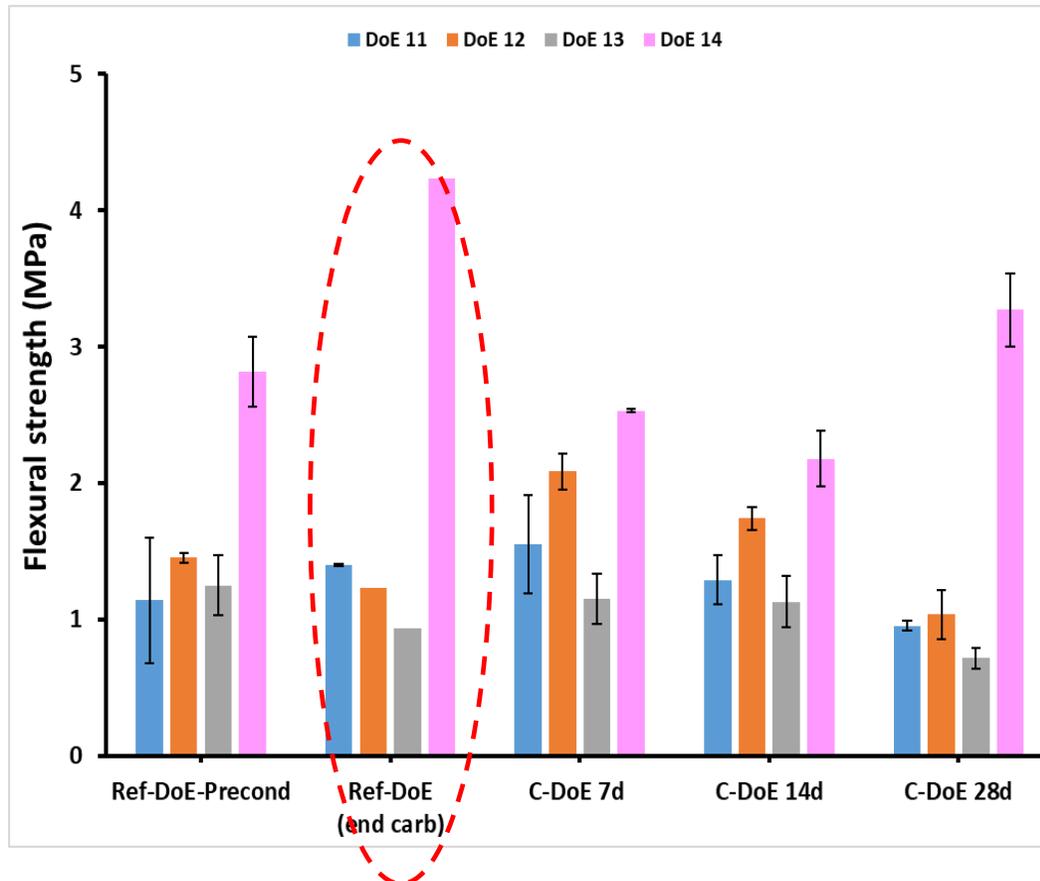
Leaching - Alkaline– MK recipes

- **Organic waste leaching:**
  - Oil leaching rate is considerably high
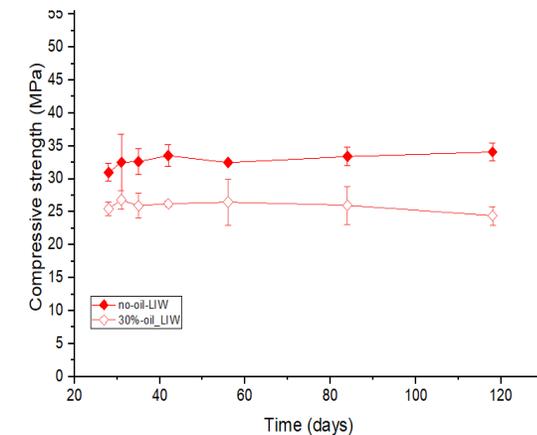 - Insignificant change in permeability
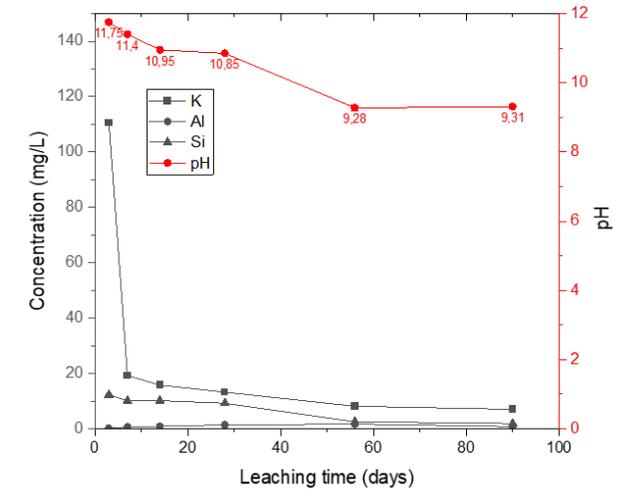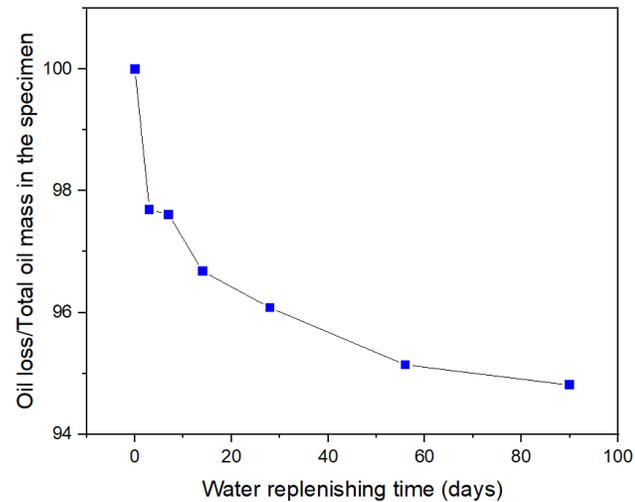
Permeability evolution

1/P (1/Pa)	No-oil-14d	No-oil-28d	30%-oil-14d	30%-oil-28d
2e-6	6.28e-16	6.15e-16	7.14e-17	6.64e-17
1e-6	1.86e-16	2.16-16	1.85e-17	1.38e-17
4e-7	4.34-17	4.5e-17	4.56e-18	4.07e-18

- **Organic waste leaching:**
  - **Waste type dependence:**
    - Oil << Scintillation liquid
    - But still high!

Leaching – Deionized water – BFS recipes

- **Oil leaching up to 3%**

- **Main elements leached out: Si, Ca**

- **Strengths increase after leaching**

	Waste (wt.%)	Waste type	Surfactant type	Surfactant (wt.%)
M1	5	Mogul oil	SDS	0.45
M2	5	Mogul oil	SDS	0.75
M3	5	Mogul oil	Tween® 80	0.45
M4	5	Mogul oil	Tween® 80	0.75
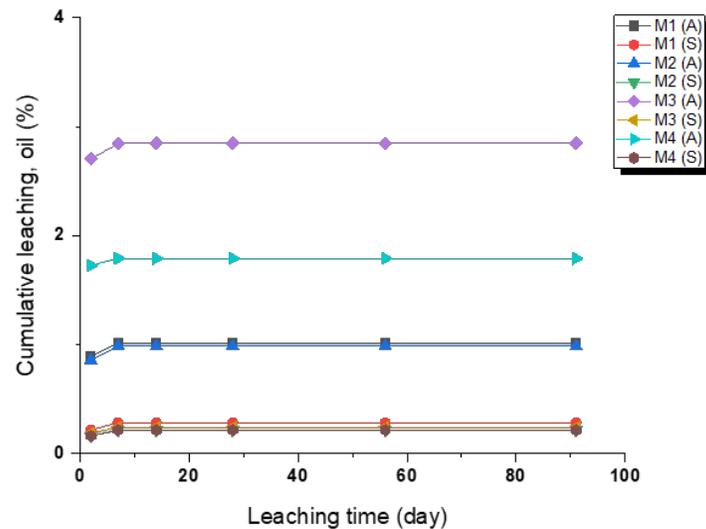
Sample	Surfactant	Surfactant addition (wt. %)	Compressive strength before leaching (MPa)	Compressive strength after leaching (MPa)
M1 (A)	SDS	0.45	26.41	34.55
M1 (S)		0.45	29.73	29.97
M2 (A)		0.75	23.32	29.37
M2 (S)		0.75	22.30	27.56
M3 (A)	Tween® 80	0.45	17.03	31.37
M3 (S)		0.45	19.72	28.59
M4 (A)		0.75	20.42	29.75
M4 (S)		0.75	20.71	24.96

- **Structure for waste-form**
  - C=C, C-H → oil is leached out
  - Si-O-T shifts to higher wavenumber → C-A-S-H gel becomes siliceous after leaching

| DoE 11 | DoE 12 | DoE 13 | DoE 14 |
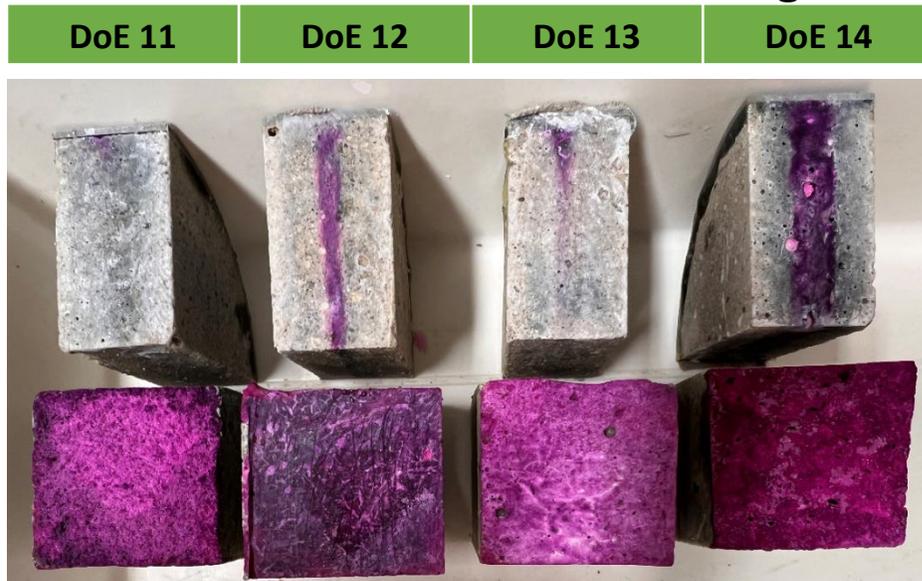
After 28d leaching

Before leaching
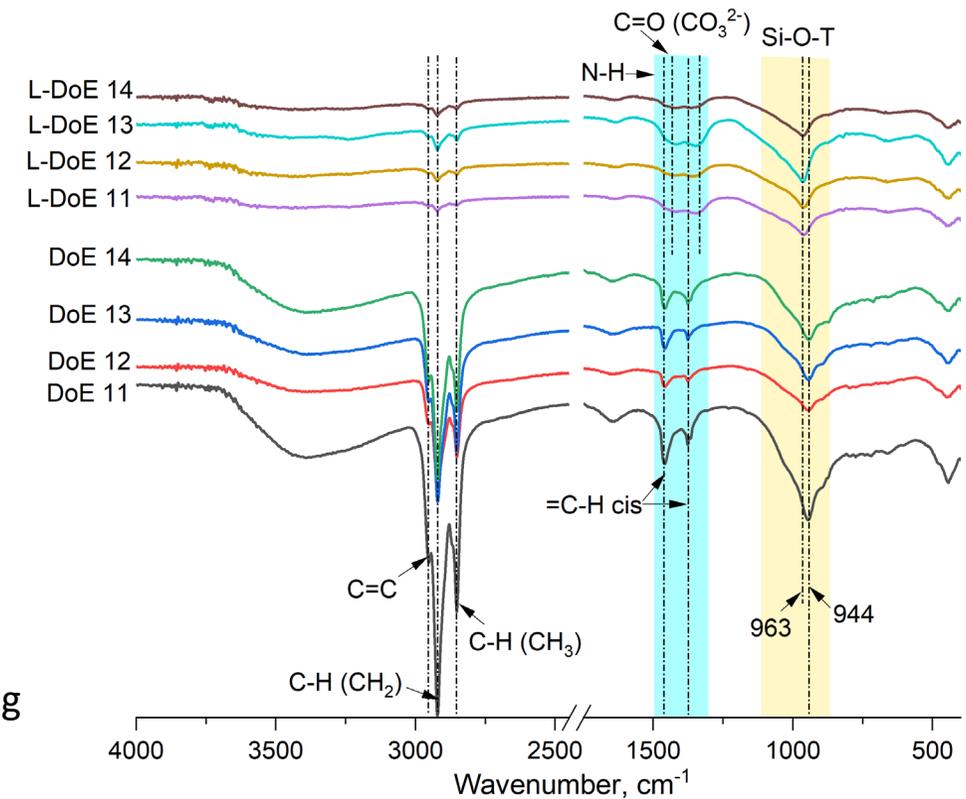
Leaching – NH$_4$NO$_3$ – BFS recipes

- **Main leaching elements: Ca, Na, K**
- **Slightly damage microstructure → permeability increase**

Sample	Concentration in leachate solution (mg/L)						
	Ca	Na	K	Mg	Fe	Si	Al
L-DoE 11	15000 ± 380	4900 ± 700	480 ± 270	<100	<80	<60	<50
L-DoE 12	12750 ± 320	4000 ± 700	400 ± 270	<100	<80	<60	<50
L-DoE 13	12650 ± 320	4200 ± 700	400 ± 270	<100	<80	<60	<50
L-DoE 14	12140 ± 310	4400 ± 700	390 ± 270	<100	<80	<60	<50
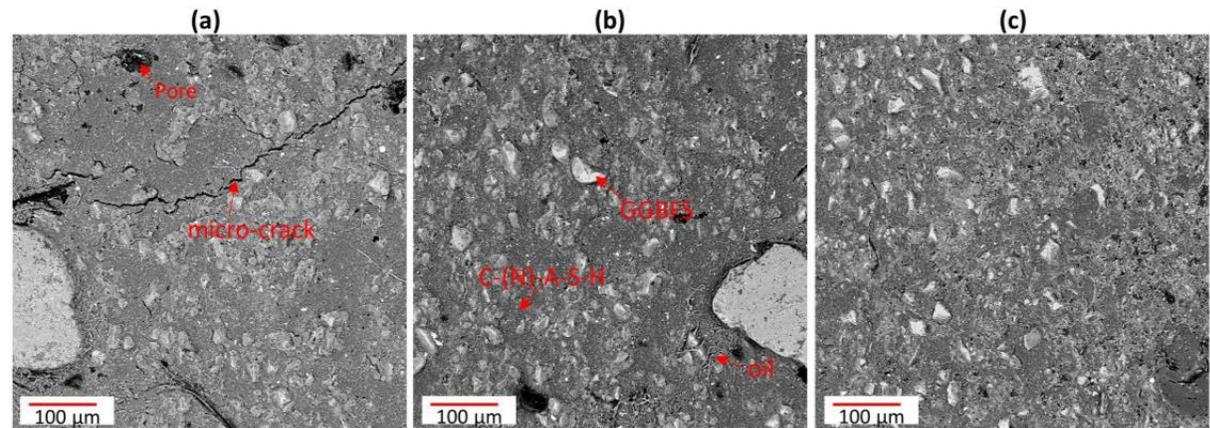
SEM images of depth layers 0-3 mm (a), 3-6 mm (b), and 6-9 mm (c)

Gamma irradiation – **All recipes**
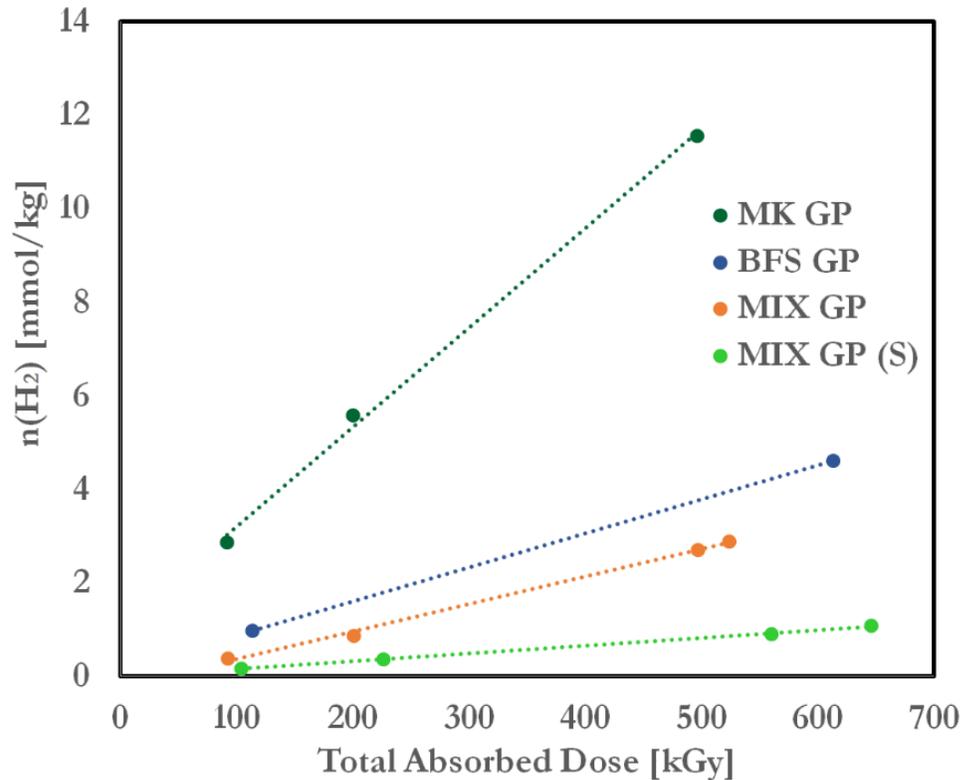
Sample	Average D_a [cm²/s] (x10⁻¹²)		Leachability Index	
	1st domain	2nd domain	1st domain	2nd domain
MK GP – Ref	54.6	1.6	10.6 (0.6)	11.9 (0.3)
MK GP – 100 kGy	68.3	5.9	10.4 (0.5)	11.6 (0.7)
MK GP – 200 kGy	150.5	4.8	10.1 (0.6)	11.7 (0.7)
MIX GP – Ref	4.8	1.8	11.7 (0.8)	11.9 (0.4)
MIX GP – 100 kGy	5.6	2.4	11.6 (0.7)	11.7 (0.3)
MIX GP – 200 kGy	4.1	1.9	11.7 (0.7)	11.8 (0.3)
MIX GP (S) – Ref	3.2	1.0	11.7 (0.5)	12.1 (0.4)
MIX GP (S) – 100 kGy	3.5	1.1	11.7 (0.5)	12.1 (0.4)
MIX GP (S) – 200 kGy	2.9	0.8	11.8 (0.5)	12.3 (0.5)

Gamma irradiation – **MK recipes**

Sample	Irradiation	Immersion	Compressive strength (MPa)
No waste	No	No	18.6 ± 0.9
	No	Yes	16.1 ± 0.9
	Yes	No	7.9 ± 0.6
	Yes	Yes	NA
LSC	No	No	8.9 ± 0.4
	No	Yes	7.1 ± 0.5
	Yes	No	4.9 ± 0.4
	Yes	Yes	NA
TBP/dodecane	No	No	10.0 ± 0.5
	No	Yes	6.3 ± 0.6
	Yes	No	NA
	Yes	Yes	NA
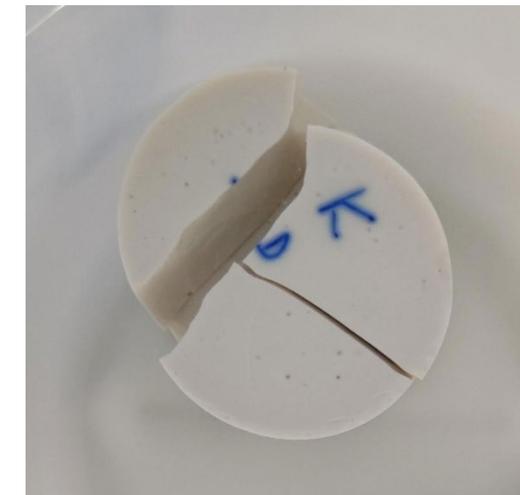
Irradiated sample showing cracking under immersion

Leaching of RNs – MK recipes

- Waste presence → Insignificant effect on leachability
- Minor effect of irradiation
- Similar observation for BFS and MIX recipes

Sample	Irradiation	Ce	Co	Cs	Eu	Nd	Ni	Sr	Th	U
No waste	No	11.1 ± 0.2	11.3 ± 0.3	9.5 ± 0.2	11.2 ± 0.3	10.9 ± 0.2	10.1 ± 0.3	11.4 ± 0.5	10.0 ± 0.2	11.0 ± 0.2
	200 kGy	10.8 ± 0.1	12.4 ± 0.4	9.6 ± 0.4	11.7 ± 0.3	10.4 ± 0.1	10.4 ± 0.4	9.1 ± 0.2	10.1 ± 0.5	9.0 ± 0.7
LSC	No	10.2 ± 1.1	9.8 ± 0.1	9.5 ± 0.1	12.5 ± 0.1	9.5 ± 1.1	10.7 ± 0.2	8.8 ± 0.2	10.4 ± 0.2	11.6 ± 0.6
	200 kGy	11.1 ± 0.3	10.4 ± 0.2	9.6 ± 0.2	12.7 ± 0.3	11.2 ± 0.2	9.2 ± 0.8	9.3 ± 0.1	10.5 ± 0.3	10.8 ± 0.5
TBP/ dodecane	No	10.7 ± 0.6	10.0 ± 0.1	9.5 ± 0.1	12.3 ± 0.3	11.0 ± 0.3	8.2 ± 0.8	8.8 ± 0.2	10.1 ± 0.3	11.7 ± 0.3
	200 kGy	11.4 ± 0.3	11.0 ± 0.1	9.6 ± 0.2	12.4 ± 0.3	11.1 ± 0.3	10.5 ± 0.3	8.9 ± 0.7	10.2 ± 0.3	10.5 ± 0.5

Thermal cycling resistance

- **Cycling -20°C and +40°C, 15 times, 100h**
  - Visible micro cracks
  - Loss of strength: MK (22%) > Mix formulation (9%)
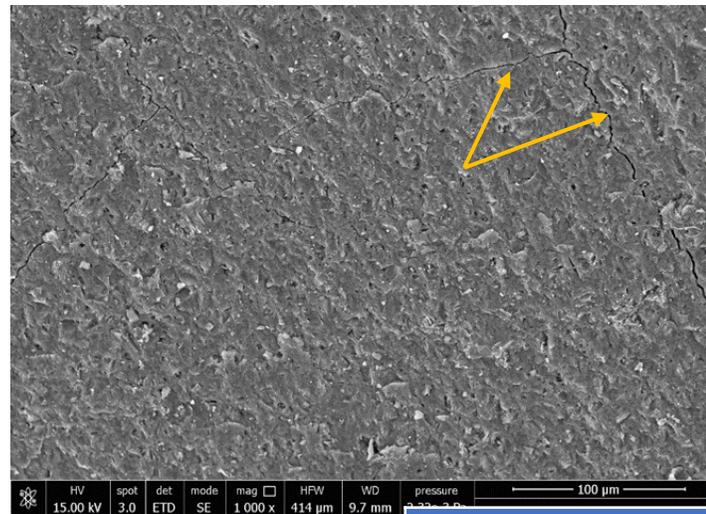
MK Based   MIX formulation

Fire hazard

- **Up to 800 ºC**

- **Strength loss: MK > BFS > Mix**

| MK Based | MIX formulation | BFS Based |

Summary

- **Physical encapsulation:** waste encapsulation in the geopolymer matrices is primarily a physical process. Metakaolin-based geopolymers a greater waste incorporation
- **Challenging** to incorporate certain liquid wastes (LSC and TPB/kerosene) into BFS-based formulations, and the use of surfactants, although helpful, was not always effective.
- **Aging and Durability:** Aging tests under various conditions did not significantly affect the strength or transport properties of the matrices. The matrices maintained their strength and exhibited low oil release rates.
- **MK-based** formulations were found to be sensitive to drying/wetting cycles, leading to significant cracking and increased waste release.

Thank you for your attention!

PREDIS

Public Technical Workshop – WP5

Durability evaluation of the MK formulation

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Objectives

- Water infiltration
- Structural material
- Package material
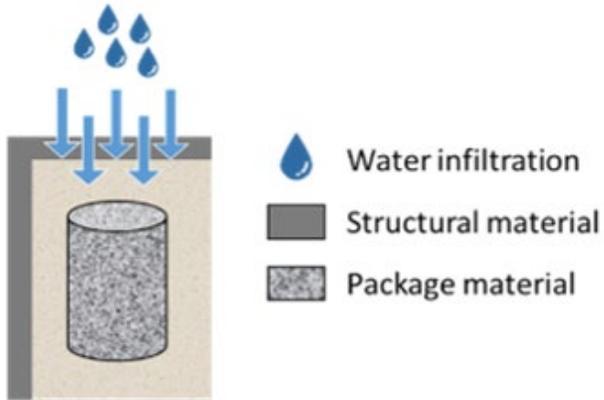
Aim of the study:
To evaluate the durability of alkali-activated materials in the event of rewatering of the nuclear waste storage site, in order to guarantee the safety of the waste containment barrier

Selection of the study environments:

Type of water / Condition	pH	Environmental conditions
Endogenous (100% RH)	-	Reference (stoed in airtight bag)
CEM V	**13,3**	**Representative of cem V water**
Acidic solution	3	Representative of acid attack environments
Carbonation		CO_2 penetration when exposed to air

Precursor:
Metakaolin powder

+

Alkaline solution:
Potassium alkaline solution
(K silicate + KOH)

Nevastane is directly incorporated
in the reactive slurry of
geopolymer

+
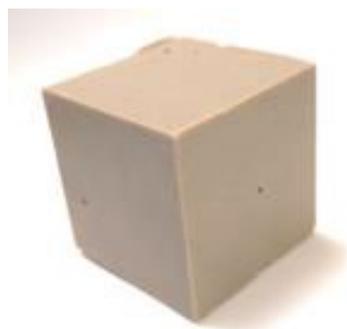
Geopolymer:
K-based
formulation

RLOW surrogate:
Nevastane EP 100

Davy,
2018
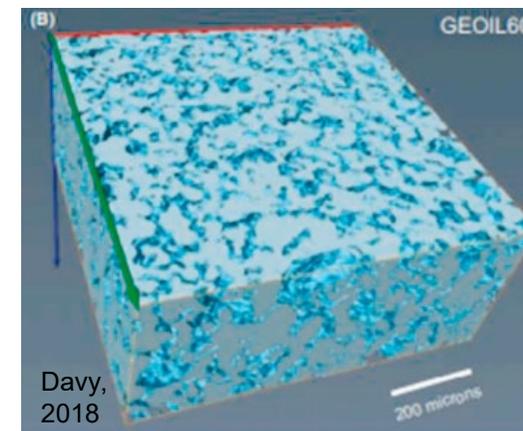
GEOIL
GP + Oil

INTRO: Waste loading factor determination

> **Optimized formulation** (Result from WP5.3 NNL):
- $SiO_2 : K_2O$ = 1.2
- $K_2O : Al_2O_3$ = 1.2
- $H_2O : K_2O$ = 13

Formulation	Compressive strength (MPa)	Flexural strength (MPa)
REF GP	32.5	3.5
20%. vol GEOIL	27,9	2.4
30%. vol GEOIL	25.5	1.7
40%. vol GEOIL	18.0	0.8

Compressive and flexural results of GP/GEOIL after 28 days of curing

- A target threshold of 8 MPa must be achieved post durability testing

REF

20%

30%

40%
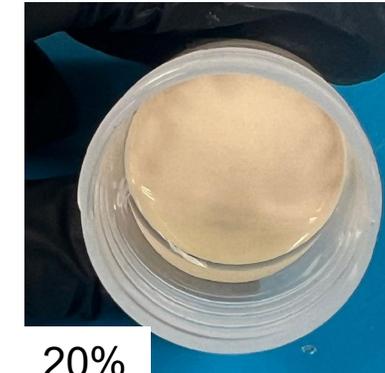
Red dye was added to the oil before mixing.

When 40% oil is added, red-colored water is observed.

Methodology Overview

Preparation of GP samples → Sample characterization **before durability tests** → Leaching + Carbonation tests → Characterization of samples **after exposure to the durability tests**
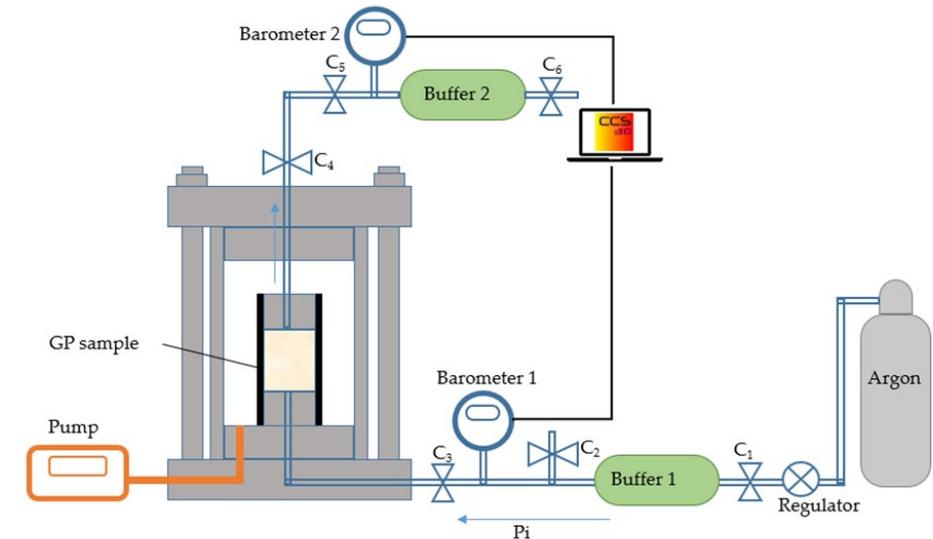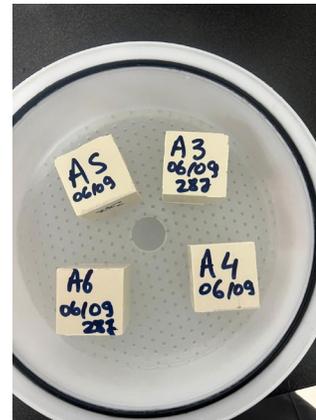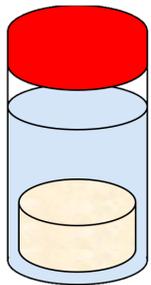
Methodology Overview
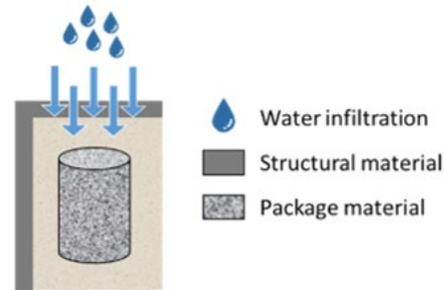
From Andra specification → Good lixiviation with:

$$\frac{V_L}{S} \geq 0, 1\ m;$$

V_L Leaching volume (m³),

S surface area of the leached specimen (m²)

Natural Carbonation tested to K-GP

➤ Carbonation is assessed in carbonation chamber with the following parameters
20°C, 65%RH, 0.1%CO2

- Water infiltration
- Structural material
- Package material

➤ Renewing the leaching solution + testing at: 3,7,14,28,56 and 90 days

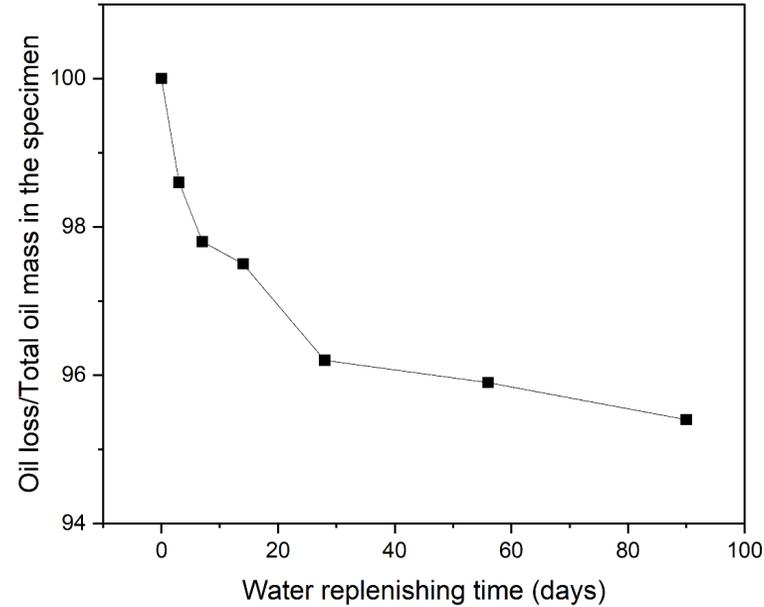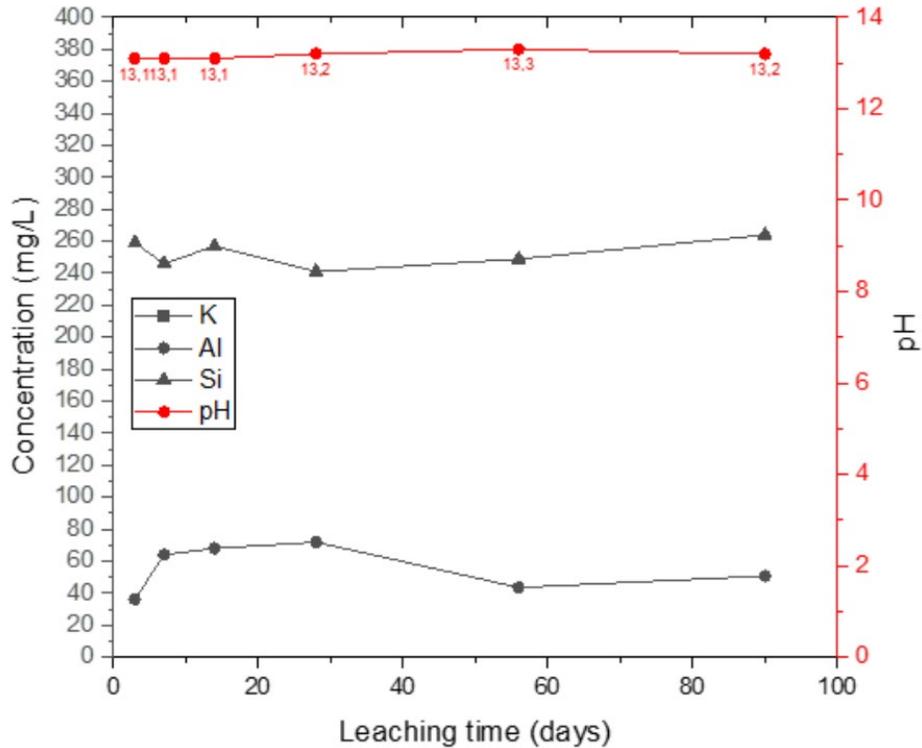➤ Alkaline solution: representative of a CEM V pore water / carbonated pore solution **(pH 13)**

After 14 days of natural carbonation, samples are splitted into halves using a vise protected with jaws

Leaching results

K is saturated in the ICP measurement, since the alkaline solution (Ph 13) is fabricated by mixing KOH + NaOH.
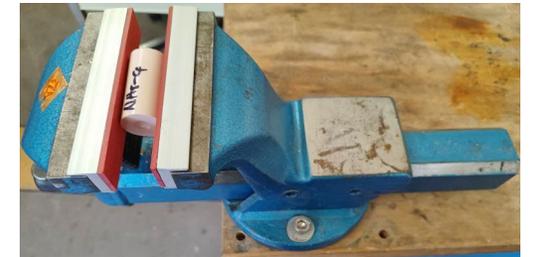
Oil percentage is leaching is calculated relatively to the initial oil content.

Confinement pressure (Mpa)	30%. Vol oil 14 days leaching	30%. Vol oil 28 days leaching
5	4,5 E-17	4,07 E-18

Carbonation results

NO OIL	30%. vol OIL

| Indicator solutions	Transition pH and colors					
Phenolphtalein	colourless	pH < 9	x	x	pink	pH > 9
Alizarine yellow	Yellow	Ph < 10,2	Orange	10,2 < pH < 12	Red	pH > 12
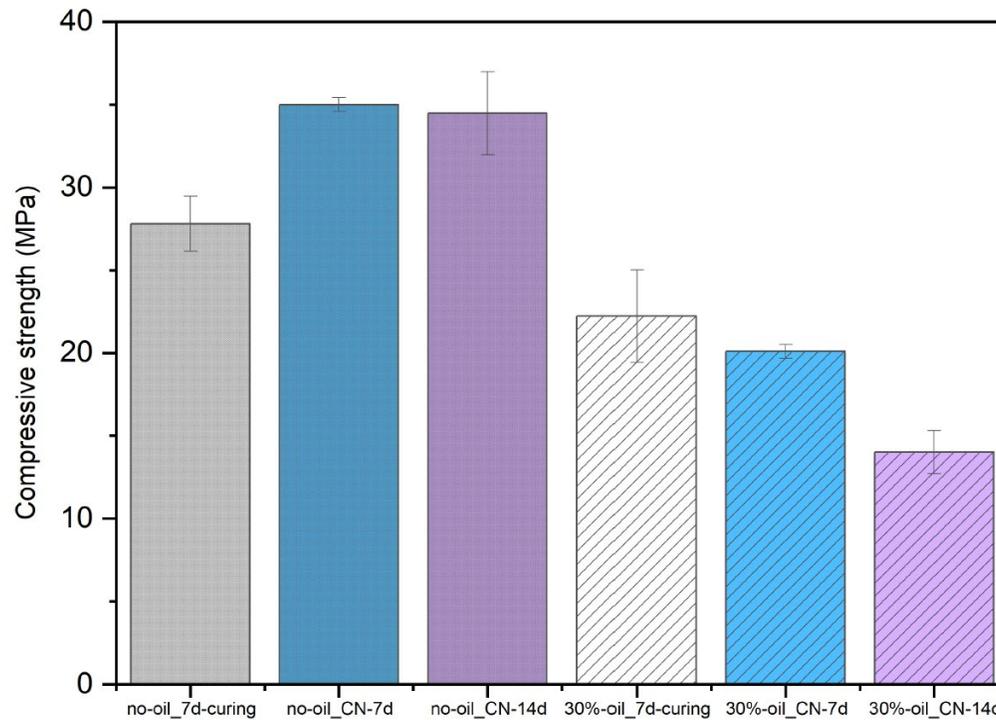
The decline in compressive strength, particularly evident in samples containing oil, stems from the matrix's susceptibility to drying and desaturation.

Conversely, samples stored in high humidity environments don't exhibit this issue.

13/06/2024   8

Conclusions

✓ **During leaching in an alkaline solution the pH remains stable, as the GP are within their safe range of alkalinity.**

✓ **Permeability of GP matrix is enhanced by 10 after leaching in pH 13.**

✓ **Samples containing oil exhibit reduced susceptibility to carbonation. This association may stem from oil effectively blocking pores, thereby aiding in maintaining a higher pH level.**

✓ **These results show that GEOIL materials seem to be stable material in different durability environments.**

PREDIS

Thank you !
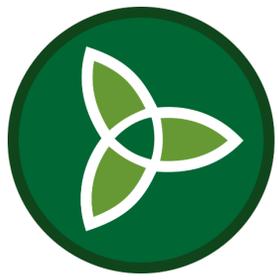
Public Technical Workshop – WP5

RLOW pre-impregnation

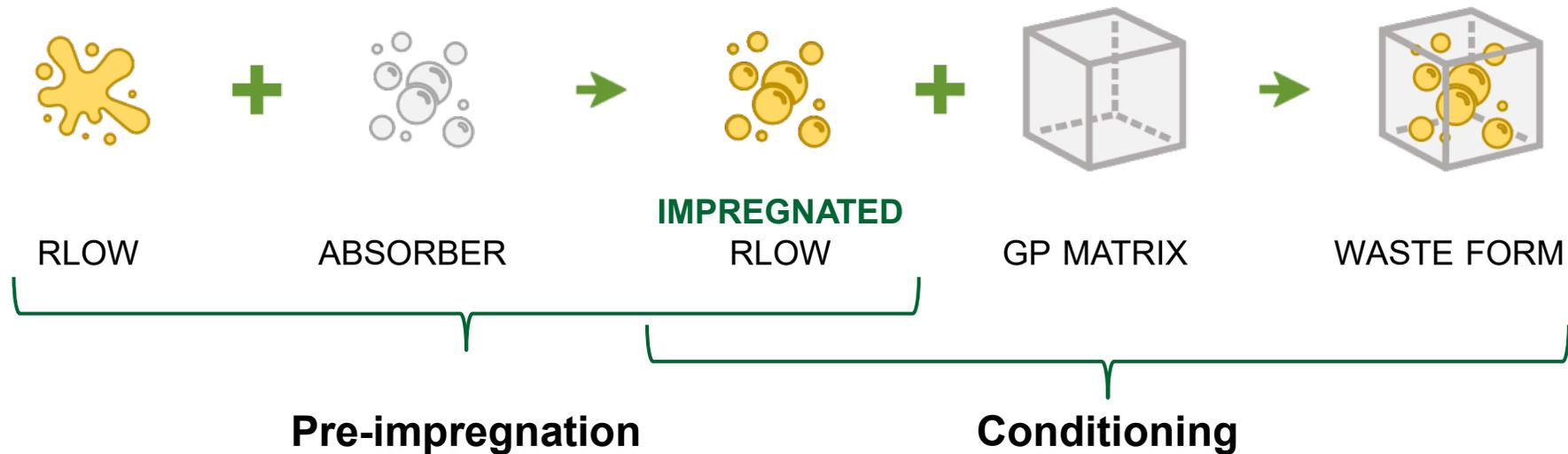PREDIS FINAL CONFERENCE, 3 – 7 JUNE 2024, AVIGNON

Gabriele Magugliani - POLIMI

Eros Mossini, Andrea Santi, Francesco Galluccio, Elena Macerata, Marco Giola, Mario Mariani

gabriele.magugliani@polimi.it

INTRO: Pre-impregnation approach

| RLOW | ABSORBER | **IMPREGNATED** RLOW | GP MATRIX | WASTE FORM |

Pre-impregnation — Conditioning

Parameters:

- Absorber type
- Matrix type

- RLOW/absorber ratio and loading factor
- Impregnation-encapsulation order

Materials and operating conditions

Materials

- Waste: liquid scintillation cocktail (LSC) and tributyl-phosphate/kerosene (TPB-K 30-70) mixture

- Absorber: recycled polyurethane (rPU)

- Matrix: tuff-based geopolymer

rPU absorber

TBP-k / LSC RLOW

Operating conditions

- Waste-to-absorber: variable ratio

- Waste loading factor: up to 20% wt.

- Procedure: pre-impregnation followed by addition to fresh grout

Matrix precursors

- Blast furnace slag
- Fly ash
- Volcanic tuff

RESULTS: feasibility of pre-impregnation on rPU

✓ LSC and TBP-k are **successfully absorbed** on rPU

✗ Encapsulation: **rPU degradation**

→ cavities & bleeding

Mechanical strength
25 MPa → <5 MPa

Preservation of structural integrity but
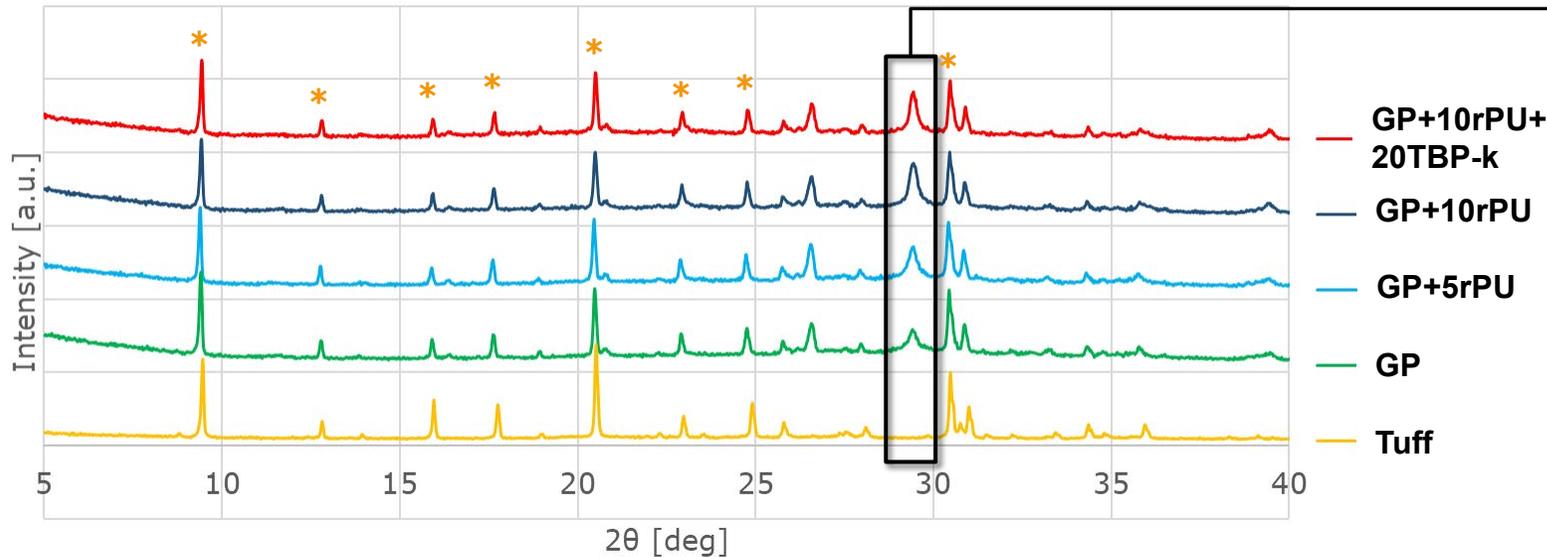RLOW release upon water immersion

TBP-k loaded samples

LSC loaded samples

| rPU | 5% wt. | 10% wt. | 10% wt. |
| RLOW | 10% wt. | 10% wt. | 20% wt. |

RESULTS: rPU degradation

GP+10rPU+20TBP-k
GP+10rPU
GP+5rPU
GP
Tuff

Increase of carbonate peak is consistent with the **rPU hydrolysis** in alkaline media, to produce CO_2 which undergoes **carbonation**.

This is coherent with the observed **rPU degradation**.

✓ Preservation of **parental chabazite zeolite** (*) upon geopolymerization

✓ Preservation of the geopolymer mineralogic structure even in presence of rPU and RLOW

X Matrix-absorber incompatibility

NEW materials and operating conditions

Materials

- Waste: liquid scintillation cocktail (LSC) and tributyl-phosphate/kerosene (TPB-K) mixture
- Absorber: NOCHAR N910
- Matrix: tuff-based geopolymer
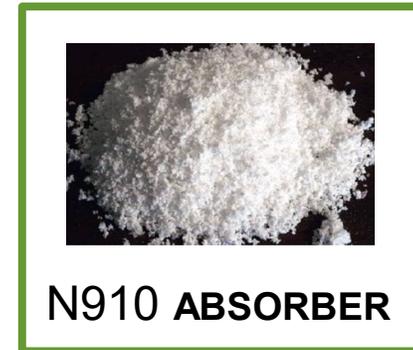
N910 **ABSORBER**

TBP-k / LSC RLOW

Operating conditions

- Waste-to-absorber: variable ratio
- Waste loading factor: up to 20% wt.
- Procedure: pre-impregnation followed by addition to fresh grout

Matrix precursors

- Blast furnace slag
- Fly ash
- Volcanic tuff

RESULTS: N910 working conditions

N910 swelling upon mixing with TBP-k

N910 alone

RLOW added

after 10 min

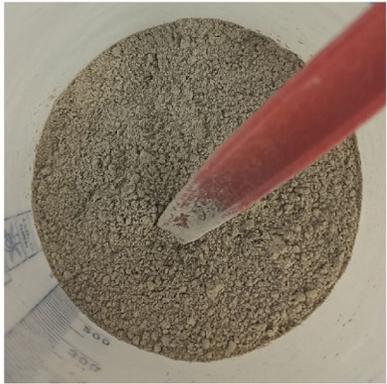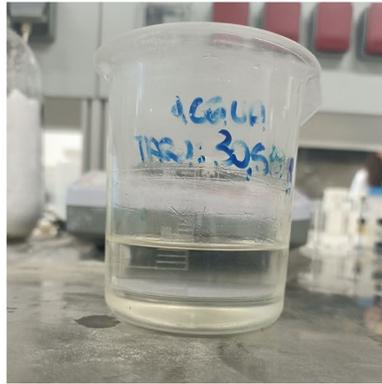| N910	8% wt.	6% wt.	5% wt.	4% wt.
RLOW /N910	3:1	4:1	5:1	6:1

- High viscosity → **heterogeneities**
- Higher RLOW/absorber ratios favour **bleeding**
- Working range limited to low loading factors

RESULTS: N910 optimization

Different orders of addition have been tested to avoid **heterogeneities** in the final wasteform.
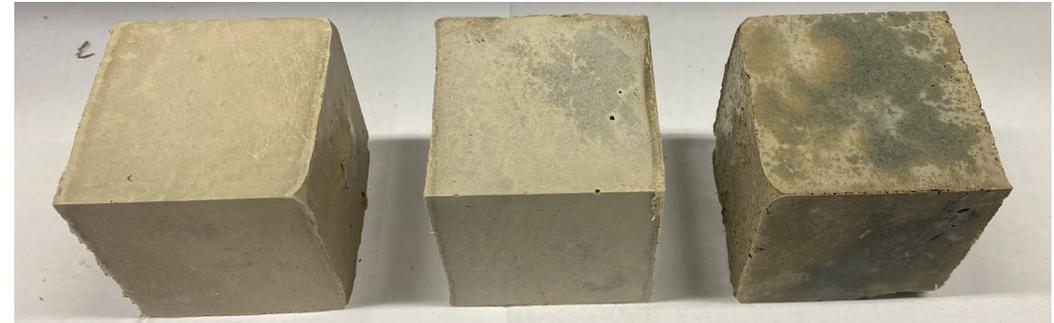The most promising (direct encapsulation):

Precursors + N910 powder

Activation solution + RLOW

Powders + liquids

N910	5% wt.	5% wt.	5% wt.
RLOW	0% wt.	10% wt. TBP-k	10% wt. LSC

	Tuff-GP [%wt.]	N910 [%wt.]	TBP-k [%wt.]	R_c [MPa]
	100	0	0	23.7 ± 1.9
Old	85	5	10	5.9 ± 0.7
New	85	5	10	8.1 ± 1.4

Conclusions

✓ N910 is a better RLOW absorber as it is **compatible** with alkaline matrices

X The **heterogeneity** of wasteforms reduces strenght and durability

X Only **low loading** factors (10% wt.) could be achieved

? Future tests with other matrices developed in PREDIS

Thank you!