PREDIS

WP6 Innovation in Solid Organic Waste treatment and conditioning

PUBLIC TECHNICAL WORKSHOP, 05.06.2024

TH. MENNECART & WP6 PARTNERS

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Overview of WP6 Objectives

- Perform a **gap analysis** during the first project year.
- **Demonstrate the reliability** of alkaline binders for conditioning of residues and secondary waste stemming from treatment of RSOW.
- **Verify the matrix performance** of conditioned final / ultimate waste according to a set of uniformed Waste Acceptance Criteria (WAC).
- **Improve understanding** of materials inventory before the thermal treatment and of the reconditioned waste once the conversion and immobilization has been achieved.
- Demonstrate **thermal treatment** methods leading to a significant **volume reduction** and to **safe reconditioned waste packages**.
- Deploy results for safe utilization by end users for **mathematical calculations** avoiding systematic experimental studies of the reconditioned waste.

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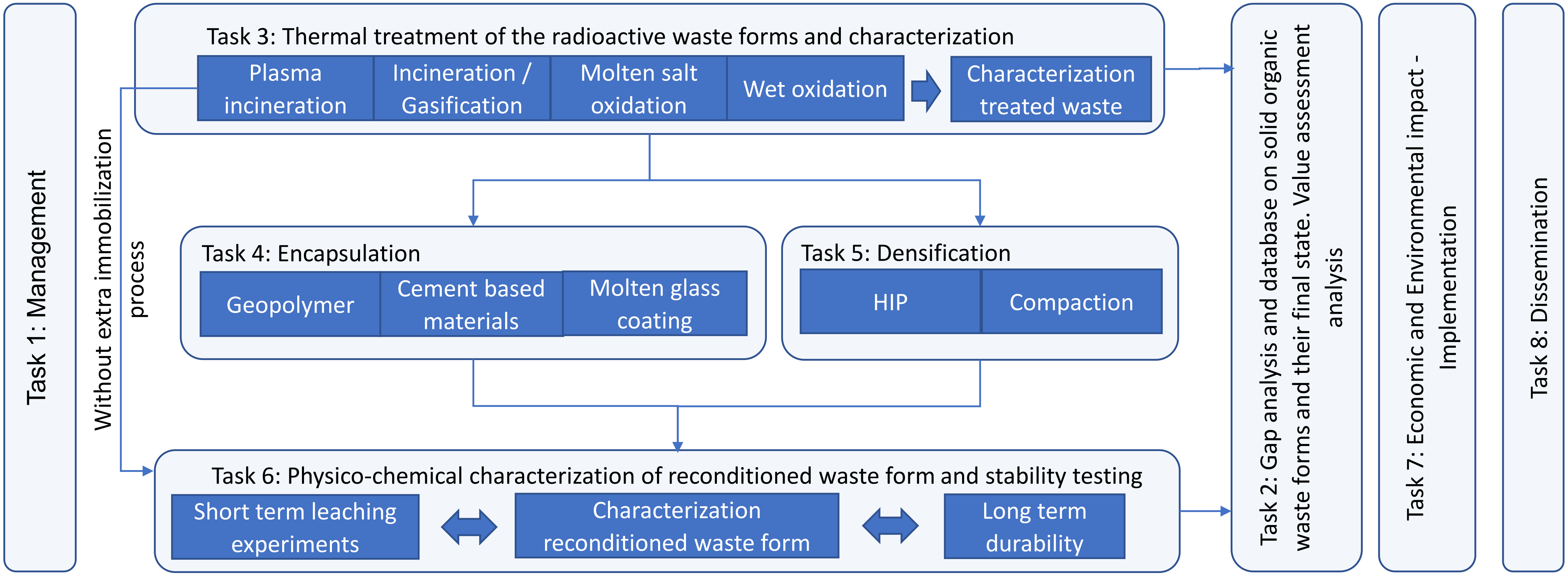
Work Package 6 Structure



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Partners of WP6

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WP Readiness & Actions Towards Completion

Achieved:

- Demonstration of the thermal treatment benefit (incineration, molten salt oxidation, wet oxidation) and characterisation of the treated materials
  Matrix reliability, Thermal treatment benefit & Materials understanding
- Feasibility of the encapsulation using geopolymer and cement-based materials with respect of the considered treated materials
  Matrix reliability & Matrix performance
- Feasibility of the molten glass coating for the immobilisation of ashes stemming from the incineration of surrogate technological waste of organic solids (plastic and cellulosic) + anionic IER
  Thermal treatment benefit
- Feasibility of the compaction and HIP processes for the immobilization of ashes
  Thermal treatment benefit

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WP Readiness & Actions Towards Completion

To be completed:

- Finalization of the stability and durability tests, including surface analyses to complete the physico-chemical characterization of the reconditioned waste form
  Matrix reliability & performance, Materials understanding

- Finalization / continuation of the upscale experiment and model development.
  Mathematical calculations

- Value assessment analysis
  Matrix reliability & performance

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Planning

D	*Deliverable = Intermediate report*
D-E	*Deliverable feeding to EURAD*
M	*Milestone = delivery of samples*
M-E	*Milestone feeding to EURAD*
	Active working months

			2020				2021												2022												2023												2024							
			september	october	november	december	january	february	march	april	may	june	july	august	september	october	november	december	january	february	march	april	may	june	july	august	september	october	november	december	january	february	march	april	may	june	july	august	september	october	november	december	january	february	march	april	may	june	july	august
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48
			Year 1												Year 2												Year 3												Year 4											
WP 6	**Innovations in solid organic waste treatment and conditioning**	**SCK•CEN**	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48
T6.1	**WP Management**	**SCK•CEN**																																										M						
T6.2	**Gap analysis and database on solid organic waste forms and their final state and value assessmen**	**GSL**																																															D	
T6.3	**Thermal treatment of the radioactive waste forms and characterisation of the treated / reconditio**	**CEA**																																				D												
6.3.1	Thermal treatment of the Radioactive Solid Organic Wastes	CEA																																		M														
6.3.2	Characterisation of the thermally treated / reconditioned wastes	CEA																																																
T6.4	**Immobilisation of the treat wastes by geopolymer or cement-based materials encapsulation or by**	**CVR**																																				D												
6.4.1	Geopolymer	CVR																					M																											
6.4.2	Cement based materials	CSIC																					M																											
6.4.3	Molten glass coating	CEA																																																
T6.5	**Densification**	**USFD**																							M																							D	
T6.6	**Physico-chemical characterisation of reconditioned waste form and stability testing**	**VTT**																																														D	
6.6.1	Characterisation of reconditioned waste form	CIEMAT																												M																			
6.6.2	Short term leaching experiments under different exposed conditions	CIEMAT											M																																	M		
6.6.3	Long-term durability of reconditioned waste form	POLIMI																																												M		
T6.7	**Economic and Environmental impact - Implementation**	GSL																							M																			D					D	
T6.8	**Dissemination**	SCK•CEN																																																D
6.8.1	Reporting and guidelines																																																	
6.8.2	Dissemination activities to the scientific community and public																																																	

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Milestones

- ✓ [**M12**], M6.1. Definition of the leaching procedure for the short-term experiments and the long-term durability experiments (6.2 & 6.3).
- ✓ [**M22**], M6.2. Delivery campaign of the goepolymer samples.
- ✓ [**M22**], M6.3. Delivery campaign of the cement-based materials samples.
- ✓ [**M24**], M6.4. Delivery campaign of wet oxidation samples for HIP tests.
- ✓ [**M24**], M6.5. Intermediate report on densification (HIP, compaction).
- ✓ [**M24**], M6.6. LCA Case Study Input to WP2.
- ✓ [**M29**], M6.7. Intermediate report on characterisation of the durability related properties of conditioned waste form.
- ✓ [**M34**], M6.8. Report on feasibility demonstration of the Wet oxidation and the Molten salt oxidation routes for the treatment of RSOW: Description of the processes and basic physico-chemical properties of the reconditioned waste form.
- ✓ [**M42**], M6.9. Value assessment workshop.
- ⌛ [**M46**], M6.10. Final report on short term leaching experiments.
- ⌛ [**M46**], M6.11. Final report on long term durability of conditioned waste form.

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Deliverables

- ✓ [**M36**], D6.1. Summary report: Description of the thermal processes used for the thermal treatment of the RSOW and the physical properties and chemical composition of the resulting treated wastes.
- ✓ [**M36**], D6.2. Conditioning of ashes from the thermal treatment of RSOW by geopolymer or cement based materials encapsulation or by molten glass coating.
- ⌛ [**M45**], D6.3. Economic, environmental and disposability impacts of novel treatment technologies for low-level and intermediate-level solid organic wastes.
- ⌛ [**M47**], D6.4. Implemented database: Matching the chemical composition of the investigated reconditioned wastes with the initial waste stream.
- ⌛ [**M47**], D6.5. Densification techniques test report. Description of the process, the type of treated waste immobilised and the physical properties and chemical composition of the resulting materials.
- ⌛ [**M47**], D6.6. Final report on the Physico – chemical characterisation of reconditioned waste form and stability testing.
- ⌛ [**M47**], D6.7. Modelling tool to predict the behaviour of the reconditioned wastes based on the results obtained within the project.
- ✓ [**M48**], D6.8. Submission in scientific journals of at least 5 papers about the RSOW stability and durability tests after reconditioning.

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WP Major Dissemination: Conferences / Workshops

17 (oral and poster presentations)

2021

- IAEA Conference on Radioactive Waste Management 2021-International Conference on Radioactive Waste Management: Solutions for a Sustainable Future

2022

- NUWCEM 2022, 4th International Symposium on Cement-Based Materials for Nuclear Wastes (**3 contributions**)
- FISA 2022 & EURADWASTE '22, 10th Euratom Conference on Reactor Safety & 10th Euratom Conference Radioactive Waste Management
- XVI CONGRESO NACIONAL DE MATERIALES, CNMAT 2022
- NENE2022, 1st International Conference Nuclear Energy for New Europe
- IGD-TP Symposium and Webinar: The role of optimization in radioactive waste geological disposal programmes
- NUMAT 2022, The Nuclear Materials Conference (**2 contributions**)
- Materials Research Symposium Fall Meeting 2022

2023

- Migration 2023
- 6th International Workshop on Mechanisms and Modelling of Waste / Cement Interactions & EURAD –WP CORI Final Workshop
- The 16th International Congress on the Chemistry of Cement 2023
- 3rd Summer School on nuclear and industrial glasses for energy transition (Sumglass 2023)

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WP Major Dissemination: Journal papers

1. “Fenton-like treatment for reduction of simulated carbon-14 spent resin”, M.A. Hafeez, J. Jeon, S. Hong, N. Hyatt, J. Heo, W. Um, Journal of Environmental Chemical Engineering (2021), 9-1, https://doi.org/10.1016/j.jece.2020.104740

2. “Stability of $SrCO_3$ within composite Portland-slag cement blends”, S.A. Walling, L.J. Gardner, D.P. Prentice, M.C. Dixon Wilkins, A.A. Hammad, W. Um, N.C. Hyatt, Cement and Concrete Composites, 135, 104823 (2023), https://doi.org/10.1016/j.cemconcomp.2022.104823

3. “Design of sustainable geopolymeric matrices for encapsulation of treated radioactive solid organic waste”, A. Santi, E. Mossini, G. Magugliani, F. Galluccio, E. Macerata, P. Lotti, G. D. Gatta, D. Vadivel, D. Dondi, D. Cori, H. Nonnet, M. Mariani, Frontiers in Materials (2022), https://doi.org/10.3389/fmats.2022.1005864

4. “Effect of the incorporation of a molten salt waste from nuclear power plants in the development of geopolymers and Portland cement systems”, P. Perez-Cortes, I. Garcia-Lodeiro, F. Puertas, M. Cruz Alonso, Cement and Concrete Composites, 142, 105210 (2023), https://doi.org/10.1016/j.cemconcomp.2023.105210

5. “Cementation of spent radioactive ion-exchange resin ashes using alkali-activated cements: physicochemical and structural changes”, Perez-Cortes, I. Garcia-Lodeiro, M. Cruz Alonso, F. Puertas, Physicochemical and Structural Changes. Cement and Concrete Composites, 149, 105517 (2024), https://doi.org/10.1016/j.cemconcomp.2024.105517

6 . “Direct conditioning of molten salt arising from the thermal treatment of solid organic waste”, A. Černá, V. Galek, P. Pražák, J. Hadrava, Proceedings of the International Conference Nuclear Energy for New Europe (2022), ISBN 978-961-6207-53-9

7. “Explorative scale-up of Fenton Oxidation and Geopolymer Encapsulation for the management of spent mixed bed ion exchange resins”, F. Galluccio, E. Mossini, A. Santi, G. Magugliani, M. Giola, E. Macerata, G. D. Gatta, P. Lotti, D. Cori, G. Bilancia, P. Peerani, and M. Mariani, *In preparation in “Nuclear Engineering and Technology Journal”*

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Level technology – Ambition

Task	Technology (2019)	Ambition after 4 years	Current situation
3.1	Plasma incineration. Treatment of IER, cement concentrate, others	Increase the SHIVA technology level. **TRL from 5 to 6**	✗
3.1	Incineration / Gasification. Treatment of IER	Feasibility demonstration of the technology by incineration of (inactive) bituminized waste. **TRL from 2 to 4**	✗
3.1	Molten Salt Oxidation used for the treatment of Radioactive Liquid Organic Waste	Transposition of the technology to the treatment of RSOW (IER). Trials with (inactive?) IER and conditioning of the salt using goepolymer or cement based materials. **TRL from 4 to 6**	✓
3.1	Wet Oxidation Route used for the destruction of dissolved organic contaminants	Development and optimization of the process for the destruction of IER leading to the complete recovery of the ^{14}C and associated radionuclide inventory into iron sludge. The sludge will be thermal treated for a complete immobilisation (task 5). **TRL from 2 to 3 - 4**.	✓
4.1	Goepolymer immobilisation	Determination of the best geopolymer formulation for a safe and long term immobilisation of treated wastes after thermal treatment (e.g. ashes, salt). **TRL from 2 - 3 to 5**	✓
4.2	Cement based materials immobilisation	Determination of the best cement based materials formulation for a safe and long term immobilisation of treated wastes after thermal treatment (e.g. ashes, salt). **TRL from 3 to 5**	✓
4.3	New technique: Molten glass coating	Feasibility demonstration of the glass coating for the immobilisation of ashes after incineration of IER at the lab scale. **TRL from 1 to 4.**	✓
5	HIP technology	Increase the technology level using radiotracers or radioactive samples. **TRL from 2 to 4**	≈
5	Compaction assisted by thermal treatment	Feasibility demonstration of densification of ashes coming from incineration process by compaction, eventually with adjuvants and temperature. **TRL from 1 to 3 - 4**	✓

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Scientific presentations – Flash talk

- Thermal treatment of the radioactive waste forms and characterization (Hélène Nonnet – CEA)
- Encapsulation (Vojtěch Galek – CVRez)
- Densification (Russel Hand – USFD)
- Physico-chemical characterization of reconditioned waste form and stability testing (Emmi Myllykylä – VTT)
- Economic and environmental impact (Callum Eldridge – GSL)

- Flash talk: Francesco Galluccio (Polimi)

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Thermal treatment of the radioactive waste forms and characterization

HELENE NONNET (CEA)

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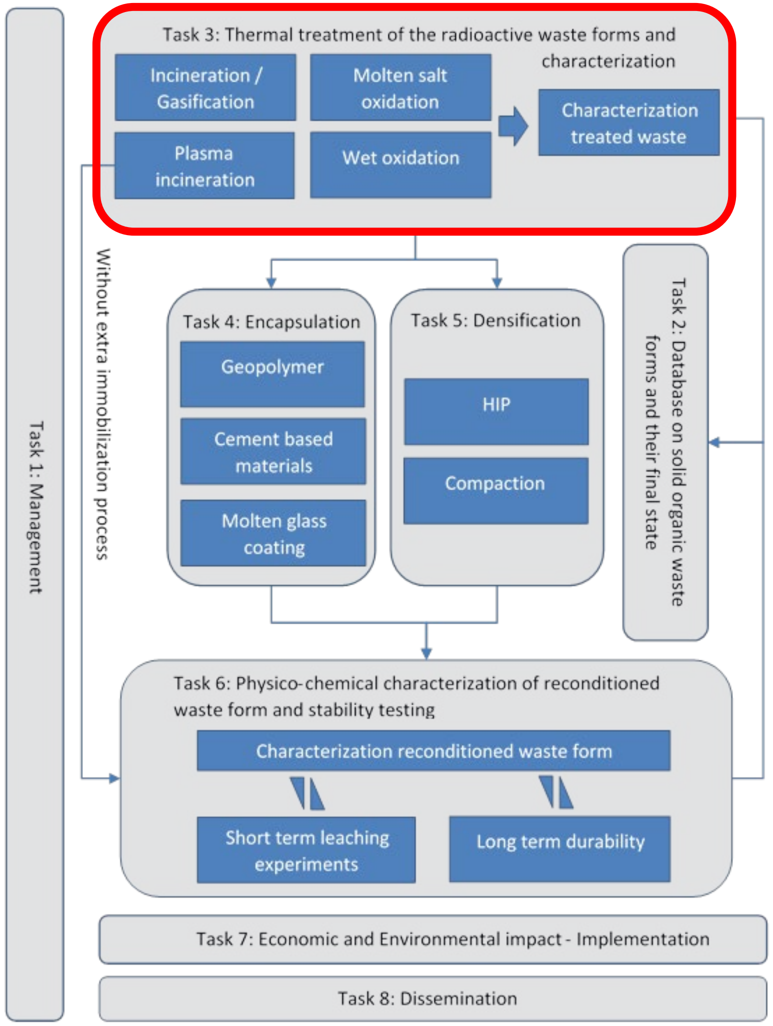

Process

- Thermal treatment → CIEMAT and CEA
- Oxidative pyrolysis and Fenton-like wet oxidation → POLIMI
- Thermal gasification → SIIEG and VTT
- Plasma vitrification/Wet Oxidation → USFD
- Molten Salt Oxidation (MSO) thermal treatment process → CVRez

Waste

- IER resins
- Mix IER and organic solids
- PCM material containing organic solids

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Deliverable 6.1
Summary report: Description of the thermal processes used for the thermal treatment of the RSOW and the physical properties and chemical composition of the resulting treated wastes

Date 23.8.2023 version Final

Dissemination level Public

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Project acronym PREDIS	Project title PRE-DISposal management of radioactive waste	Grant agreement No. 945098
Deliverable No. D6.1	Deliverable title Summary report: Description of the thermal processes used for the thermal treatment of the RSOW and the physical properties and chemical composition of the resulting treated wastes	Version 1.0
Type Report	Dissemination level Public	Due date M36
Lead beneficiary CEA		WP No. 6
Main author Hélène NONNET, CEA	Reviewed by Thierry MENNECART, WP6 leader, SCK CEN	Accepted by Maria Oksa, coordinator, VTT
Contributing author(s) E. Torres, A. Bahillo (CIEMAT) ; E. Mossini, F. Galluccio, A. Santi, E. Rizzi, G. Magugliani, E. Macerata, M. Giola, M. Mariani (POLIMI) ; Yu. Zabulonov, B. Zlobenko, A. Pugach, A. Rozko, Yu. Fedorenko (SIIEG) ; Veli-Matti Pulkkanen (VTT) ; Hélène Nonnet (CEA); Josh Radford (USFD), Claire Corkhill (USFD); Vojtěch Galek (CVRez), Anna Sears (CVRez), Petr Pražák (CVRez), Martin Vacek (CVRez), Jak Hadrava(CVRez)		Pages 53

Abstract

This report presents descriptions of the different organic solids treatment processes used and developed at CIEMAT, POLIMI, SIIEG, VTT, CEA, UFSD and CVRez.
It also describes the physico-chemical characteristics of the materials after these treatments.

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https://predis-h2020.eu/wp-content/uploads/2023/09/PREDIS_D6.1-Thermal-processes_vFinal-23.8.2023.pdf

Thermal treatment → CIEMAT

Surrogates

- Mix of **cationic** and **anionic IERs** (50:50) Amberlite IRN-77 and IRN-78
- **Doped** with **B** and traces of **Cs**, **Sr** and **activation products** (Co, Cr, Zn, Fe, Ag)

Conditioned waste

- Thermal treatment **450°C – 24 hours**
- Granulated product: **300 µm**-diameter
- Hygroscopic: **average water content 3%**
- **Acidic behaviour** in water and alkaline media

Physico-chemical characterization

- Elemental Analysis
- Laser Diffraction Particle Size Distribution Analysis
- XRD
- SEM-EDS
- FTIR
- BET - SSA

Behaviour in solution

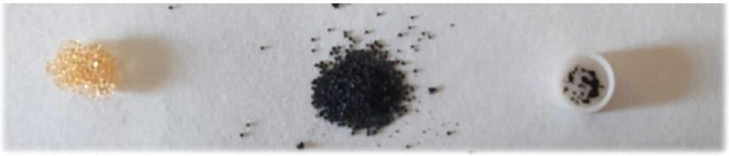
- Deionized water
- NaOH 1M

Leaching tests: after 1 and 4 months
- **pH** and **EC**
- **TOC analysis**
- Chemical Analysis (**ICP** and **IC**)

Cationic SIERs

IRN 77	IRN 77	IRN 77
Doped resin	SIERS after thermal treatment 450°	Ashes 1000°C

Anionic SIERs



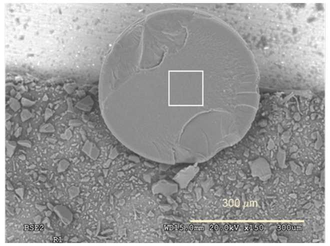
IRN78	IRN78	IRN 78
Doped resin	SIERS after thermal treatment 450°	Ashes 1000°C

Elemental analysis (C, N, H, S) of IERs surrogates and the thermally treated waste

	IERs surrogates	Thermally-treated waste
Moisture, %	59±3	4±1
Carbon, %	49.0±0.1	74±2
Hydrogen, %	7.1±0.1	5.3±0.2
Sulfur, %	6.81±0.04	7.1±0.5
Nitrogen, %	2.22±0.03	1.21±0.05
Ash, %	< 0,1	< 0,1
C/H	6,9	14,0

Boron and trace analysis of IERs surrogates and the thermally-treated waste

Element	IERs surrogates	Thermally treated waste
B	5%wt.	16.25%wt.
	mg element/Kg	mg element/Kg
Ca	4,1	17
Co	5	18
Cr	5	18
Cs	30	121
Cu	2	6
Fe	10	40
Mg	1	4
Si	13	48
Sr	4,5	23
Zn	12	48



Element	at.%
C	96,16
S	3,54
Al	0,11
Si	0,08

SEM observation and EDS analysis of a cross-section of an IRN-77 particle after thermal treatment.

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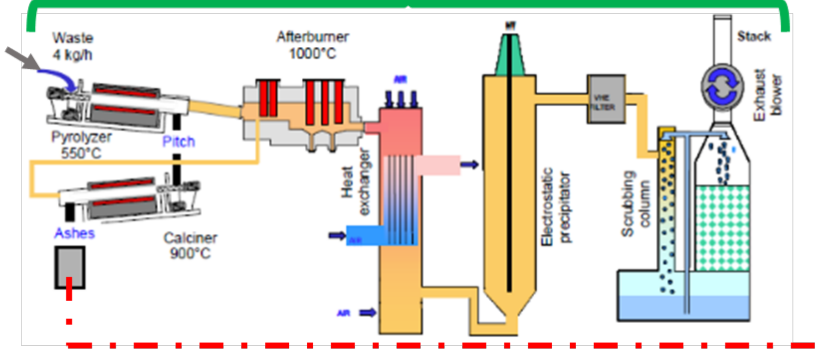
PREDIS Thermal treatment → CEA

RSOW

Component	wt%
PVC-1	19
PVC-2	12
Latex	17
Neoprene	17
EVA	25
Cotton	5
Kleenex®	5
Total	100

IRIS Process (CEA Marcoule) (pyrolysis/calcination)



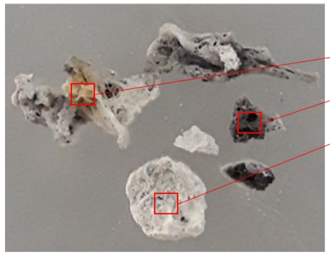
Secondary Waste : ashes

Mix of oxides
Carbon free

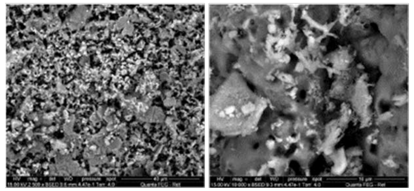
Volume reduction: /30

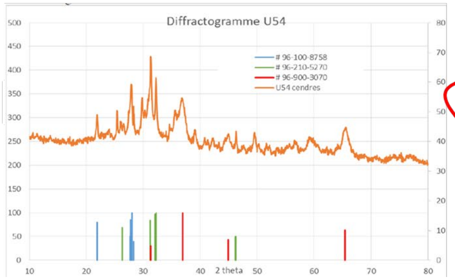
Non active waste
(surrogate similar to real waste)

IRIS ashes batch	
Component	%m
C	0,400
Cl	1,529
S	0,321
Ba	0,141
Cr	0,036
Al	16,037
Fe	0,462
Mg	2,651
K	2,960
Ti	0,390
P	2,125
Na	0,883
Ca	9,070
Si	13,205
Ni	0,377
Zn	5,135
Bi	0,092
Σ	55,81
O (difference)	44,189



	C	O	Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Fe	Ni	Zn	Ba	Re
S1	*3,2*	*36,0*	*0,8*	*2,8*	*10,4*	*11,1*	*2,5*	*0,2*	*1,4*	*3,0*	*13,3*	*4,3*	*0,7*	*0,7*	*4,2*	*0,3*	*5,1*
S13	*5,3*	*34,7*	*0,4*	*2,7*	*17,5*	*4,4*	*1,5*	*0,2*	*4,5*	*0,5*	*17,9*	*0,1*	*0,2*	*0,1*	*8,9*	*1,1*	*0,0*
S12	*8,4*	*34,6*	*0,8*	*1,2*	*13,0*	*12,5*	*1,8*	*0,2*	*1,1*	*3,1*	*13,1*	*1,7*	*0,4*	*0,5*	*7,3*	*0,3*	*0,0*





Major phases:

96-210-5270	51	2105269	Ca20.00 O48.00 P12.00 Cl4.00
96-900-3070	46	Ringwoodite	Si8.00 Mg15.97 O32.00
96-100-8758	39	Anorhtite sodian	Na1.92 Ca2.08 Si10.00 Al6.00 O32.00

Chlorapatite $Ca_5(PO_4)_3Cl$

Decomposition beyond 1100°C

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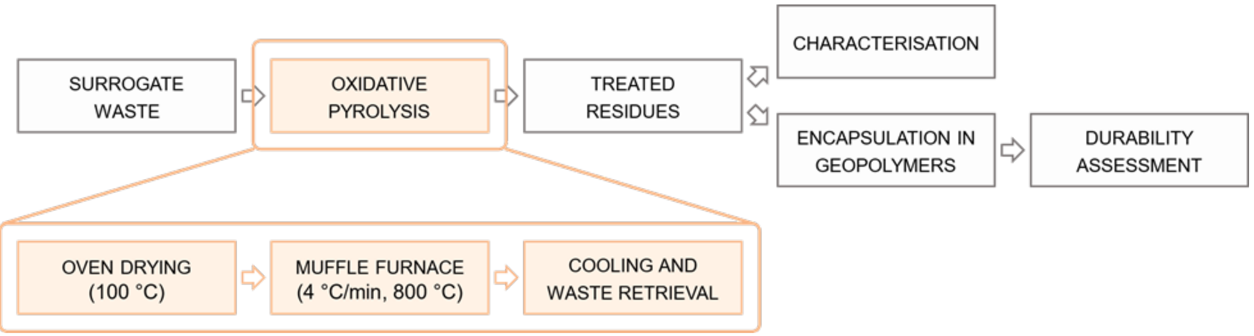
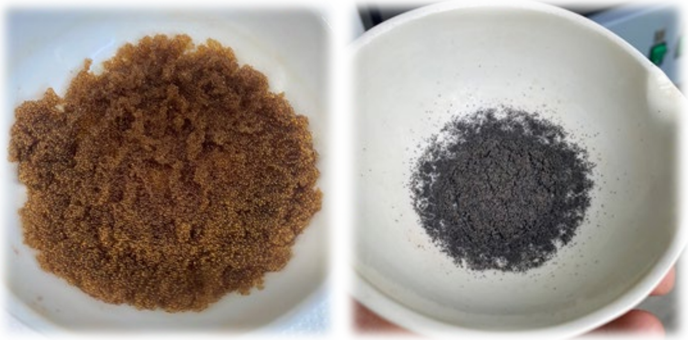
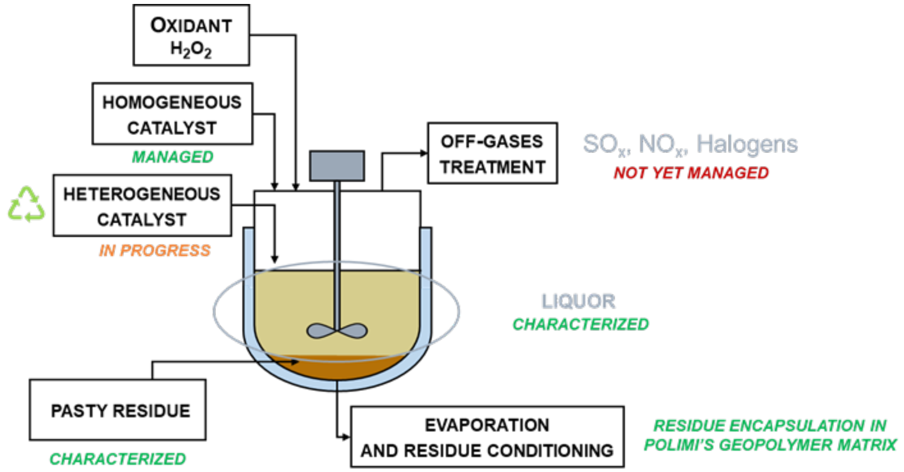
Oxidative pyrolysis and Fenton-like wet oxidation of ion-exchange resins → POLIMI



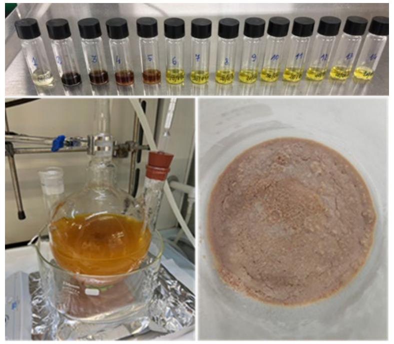
Scheme of the oxidative pyrolysis process



Spent cation-exchange resins: (left) before treatment; (right) after treatment



Schematic overview of wet oxidation process and tasks accomplished by POLIMI



Colour shift of the mixture over time (top), final liquor and obtained residue (bottom).

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Thermal gasification and plasma technology → SIIEG

Figure 1. System for thermal treatment based on gasification technology.

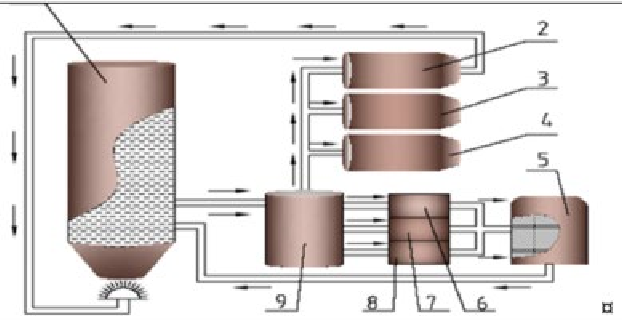

Figure 2. The multiloop circulation gasifier system.

1 – reactor, 2 – output circuit gaseous compounds, 3 – closed circuit solid compounds, 4 – output circuit for the solid residue, 5 – a unit of catalyst, 6,7,8 – sectional contours, 9 – Multiloop coolant distribution unit

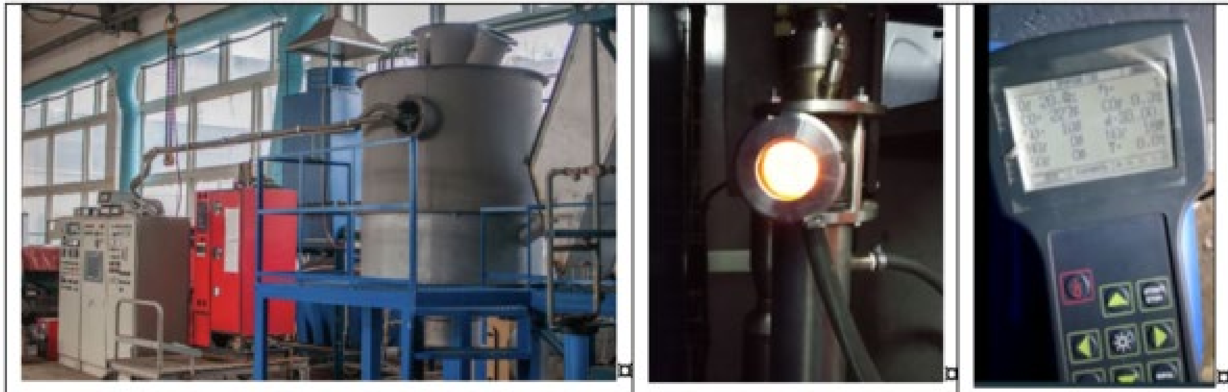

Figure 7. System for thermal treatment of the radioactive wastes based on gasification technology with a plasma reactor.

Figure 3. Cation-exchange resins KU-2-8

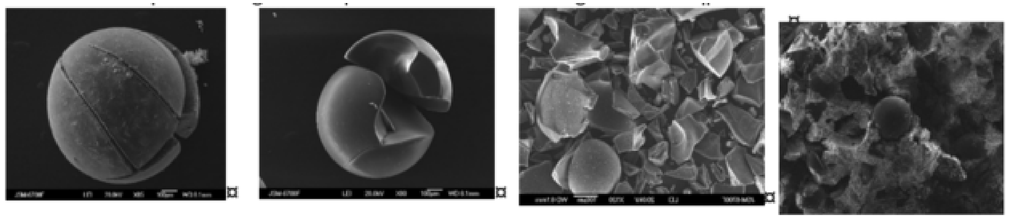

Figure 4. Destruction of IER under gasification.

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

PREDIS Thermal gasification → VTT

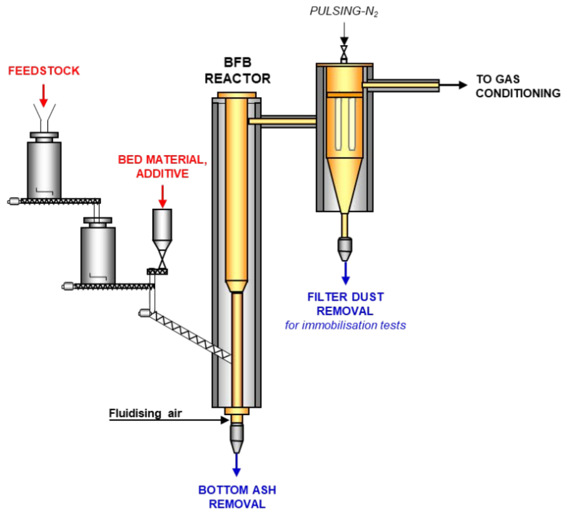

Figure 1. Bench-scale atmospheric-pressure gasification test rig (BFB100).

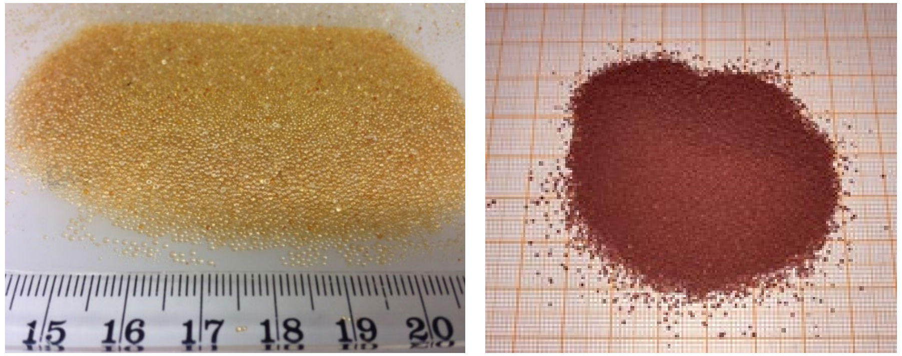

Figure 1. IXR for test run PR-3 (left) and for test run PR-4 (right).

Table 1. Measured concentrations. Concentrations below the limit of detection are highlighted in light red and the sample specific limit of detection is stated.

Element/ isotope	Method	Sample / OES wavel. (nm)	PRE1 Al2O3	PRE2 Resin 1	PRE3 Resin 2	PRE4 BFB/PR-4 Remaining bed	PRE5 BFB/PR-4 Remaining bed	PRE6 BFB/PR Cleaning run	PRE7 Resin+ Fe2O3	PRE8 Resin+ Fe2O3	PRE9 BFB/PR-3 Filter dust	PRE10 BFB/PR-4 Filter dust	PRE11 BFB/PR-2 Cleaning run filter dust	PRE12 Coal Fly Ash
Al	OES	237,312	4677,47	<9,37	8,96	3730,81	4498,98	6446,67	15,51	<12,08	5044,84	4434,52	8268,81	52031,18
Ba-137	MS		1,52	<0,25	0,86	2,09	2,26	13,96	0,60	0,45	39,14	16,47	53,96	662,74
As-75	MS		<0,91	<0,83	<0,80	<1,04	<0,75	<0,98	<1,02	<1,03	22,92	12,10	15,59	134,07
Ca	OES	317,933	<207,19	<180,08	<176,65	<234,77	<168,87	1269,11	<231,28	<232,12	2958,85	1303,43	5741,39	15005,60
Cd-111	MS		<0,10	<0,09	0,12	<0,11	<0,08	<0,11	<0,11	<0,11	1,60	0,61	4,01	0,78
Ce-140	MS		<0,06	8,49	7,78	17,35	5,11	0,52	8,38	8,54	65,83	113,87	37,83	67,83
Co-59	MS		<0,67	<0,60	<0,58	2,95	1,55	4,66	<0,74	<0,75	5,93	19,02	49,97	46,47
Cr	OES	267,716	<4,39	<3,82	<3,75	361,08	124,13	243,96	15,15	11,60	1038,26	1249,48	4779,90	206,18
Cr-52	MS		<3,45	<3,12	<3,01	323,72	108,92	218,95	14,32	9,45	966,22	1140,68	4547,71	184,28
Cs-133	MS		<0,06	9,75	9,66	1,32	1,53	0,08	8,82	10,99	292,87	250,37	103,74	8,37
Cu	OES	217,895	<13,88	<12,06	<11,83	38,71	31,39	<14,97	<15,49	<15,55	175,48	148,41	348,18	139,78
Cu-63	MS		2,62	<1,76	2,28	34,88	20,01	12,15	<2,17	<2,18	171,77	133,91	321,50	109,20
Eu-153	MS		<0,07	5,60	4,66	13,00	5,37	0,45	7,77	9,96	37,98	73,38	22,89	0,75
Fe	OES	239,563	43,22	<5,83	33,42	3021,33	10568,38	5514,49	31766,96	27573,29	4527,28	363803,01	350093,89	66943,19
Fe-56	MS		<59,61	<54,05	<52,04	2819,46	9582,94	5101,59	28955,70	25775,47	4224,87	382955,04	366441,76	60813,15
K	OES	769,897	<3,82	<3,32	<3,26	<4,33	<3,11	<4,12	<4,26	<4,28	<4,43	<4,41	291,93	5637,08
Mn	OES	261,02	<4,16	14,42	<3,55	<4,72	<3,39	83,54	<4,65	8,21	17,64	678,34	1161,32	67,45
Mo	OES	268,799	<11,45	<9,95	<9,76	<12,98	9,19	<12,34	<12,78	<12,83	39,20	175,50	369,10	1154,55
Na	OES	589,592	217,52	<4,82	6,87	100,41	151,02	234,70	8,99	9,03	308,02	232,76	613,06	997,71
Ni-60	MS		<4,20	<3,81	<3,67	1309,60	392,26	577,74	<4,70	<4,72	940,60	2626,43	7369,38	121,75
P	OES	177,434	<79,95	<69,49	<68,16	<90,59	<65,16	<86,26	<89,24	<89,57	<92,63	<92,39	<72,01	2375,95
Pb-208	MS		0,19	0,09	0,31	0,03	0,07	0,09	0,15	0,13	61,38	40,04	125,03	72,52
S	OES	181,972	<89,09	70589,27	70629,24	1146,55	165,66	124,48	66813,26	70179,98	48331,04	118026,88	16428,15	2189,88
S-32	MS		<101,40	75017,11	68436,12	1131,99	194,70	<109,32	65891,85	67983,98	48641,95	114700,37	15694,21	1939,36
Si	OES	221,667	1383,89	1184,34	1660,39	941,07	1187,72	155,77	<36,42	502,36	1713,33	1010,07	3170,32	220426,91
Sr-88	MS		0,60	0,11	0,46	0,48	0,49	6,12	0,52	0,06	6,80	3,10	19,50	960,21
Ti	OES	337,28	<0,51	<0,45	<0,44	3,06	1,54	0,30	0,76	<0,57	154,80	50,32	68,85	7771,73
V-51	MS		<0,55	<0,50	<0,48	<0,62	<0,45	<0,59	<0,61	<0,61	3,57	11,04	20,38	276,89
Zn-66	MS		6,60	1,20	2,36	40,37	16,90	22,96	<0,98	<0,99	585,49	300,24	1466,83	221,24
Unit			mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Plasma vitrification/Wet oxydation → USFD

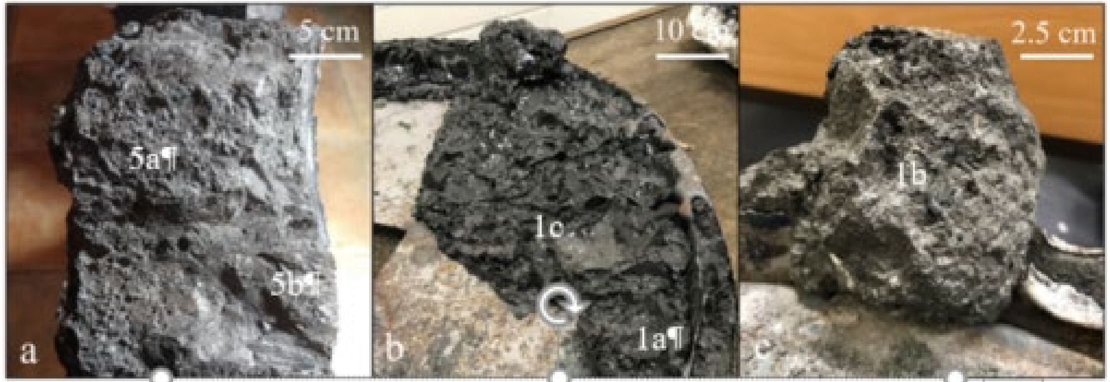

Figure 1: Example images of the vitrified PCM wasteforms, a) Material from Trial 5, b) Trial one, Sample 1a and 1c, c) Trial 1, Sample 1b

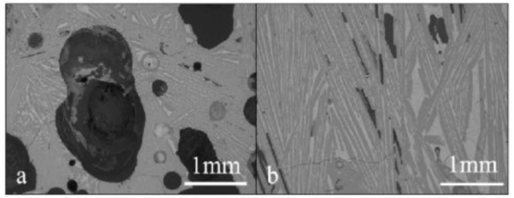

Figure 5: Comparison of Sample 5a (right) and Sample 5b (left)

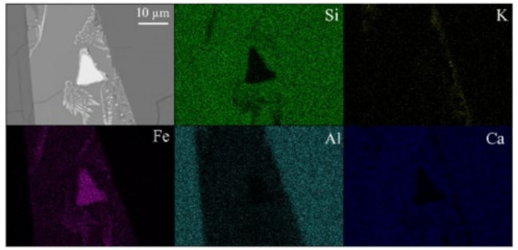

Figure 6: SEM image of sample 5a and corresponding EDX maps

- Wastes: Filtered precipitate or fully evaporated
- Low output, stockpile of material building up from many runs
  - 0.35g output per 15g resin treated (filtered)
  - ~3g if evaporated

Table 1: Simulant PCM Drum Compositions [3]

Constituent	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Container	17.4	17.42	17.36	17.4	17.4
PVC Liner	1.52	1.52	1.86	1.52	1.52
Steel Drum Liner	8.74	8.82		8.78	8.84
Bricks	18.04				
Concrete	17.14				
Mild Steel Pipe		5.58			
Galvanised Sheet Steel		1.30			
Cast Iron		2.00			
Stainless Steel Pipe		1.06			
1 Beam		10.26			
Steel Scaffolding		9.84			
Aluminium Scaffolding		0.98			
Steel Plate		5.20			
Copper Pipe		0.28			
Hand Tools and Equipment			20.16		
Rubber Hose			1.78		
Wellingtons			1.86		
PVC Suits and Storage Bags			7.24		
Paper			0.13		
Scaffolding Board			1.88		29.4
Lab Glassware			0.56		
Floor Sweepings			0.54		
Vermiculate and Water			0.25		
Strippable Coating (Decongelsure)			0.32		
Polybottles			0.27		
Alumina Furnace Bricks			0.57		
Electrical Cabling			0.55		
Rubber Gloves			0.25		
PVC Suits			12.9		
Total	62.84	64.29	55.57	40.60	57.16

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Molten Salt Oxidation (MSO) thermal treatment process → CVRez

- Device for large pilot experiments:
  - Reactor vessels are made from Inconel 600
  - Reactors volume is 80 l (40 maximum volume of the molten salt)
  - Reactor vessel is protected from overpressure
  - Concentration of CO is limited to 100 mg/m^3 for dry flue gas
  - Traces of SO_2 and NO_x
  - Oxidizing agent – air
  - Surplus of oxidizing agent λ=2
  - Temperature during process 400 – 1000 °C

- Support:
- XRD analysis molten salt structure
- Chemical step by FTIR and Raman
- determination of waste gas composition
- online determination of CO, CO2 and SO2
- offline determination of unburned proportion
- modularization to solid types of waste

Table 1. The semiquantitative analysis of waste salt determined by w. %

Ident		R430	R431	R432	R433	R434
Al_2O_3	(%)	7.48	6.23	7.29	3.8	6.38
CaO	(%)	0.137	0.161	0.142	0.134	0.148
Cl^-	(%)	0.066	0.072	0.064	0.073	0.076
Co_3O_4	(%)	0.029	0.038	0.031	0.026	0.036
Cr_2O_3	(%)	0.079	0.082	0.077	0.094	0.073
CuO	(%)	0.009	0.007			
Fe_2O_3	(%)	0.26	0.19	0.22	0.22	0.24
K_2O	(%)	1.647	1.67	1.663	1.744	1.585
MgO	(%)	0.223	0.269	0.255	0.261	0.252
MnO	(%)		0.01			
Na_2O	(%)	81.927	84.662	82.448	89.283	84.185
NiO	(%)	0.243	0.219	0.233	0.246	0.222
P_2O_5	(%)	0.009	0.009	0.013	0.009	0.01
SO_3	(%)	0.164	0.263	0.176	0.193	0.157
SeO_2	(%)		0.003			
SiO_2	(%)	7.494	5.959	7.193	3.763	6.434
SrO	(%)	0.034	0.034	0.034	0.035	0.031
TiO_2	(%)	0.15	0.082	0.12	0.077	0.13
ZnO	(%)	0.043	0.041	0.04	0.041	0.045

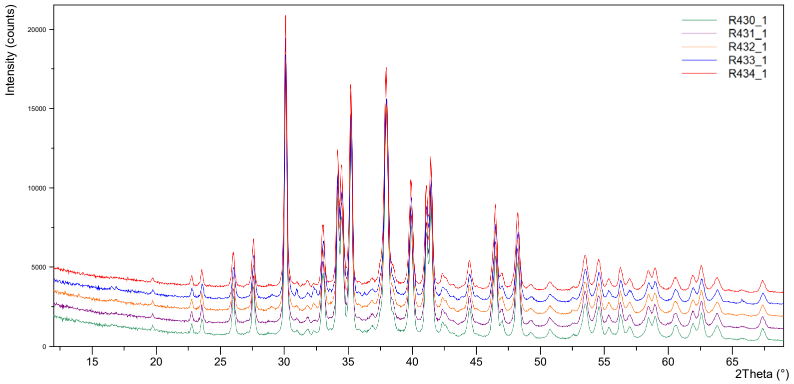

Figure 1. The XRD analysis of MSO waste salt samples.

Table 1. The phase composition generated from XRD analysis of each sample by (w.%)

Phase		R430	R431	R432	R433	R434
Natrite	Na_2CO_3	96.41	96.52	95.07	94.12	96.25
Essenite	$CaFeAlSiO_6$	3.38	3.48	3.32	3.14	3.71
Thermonatrite	$Na_2CO_3 \cdot H_2O$	0.21	-	0.79	1.79	0.04
Trona	$Na_3H(CO_3)_2 \cdot 2H_2O$	-	-	0.81	0.95	-

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PREDIS

Encapsulation

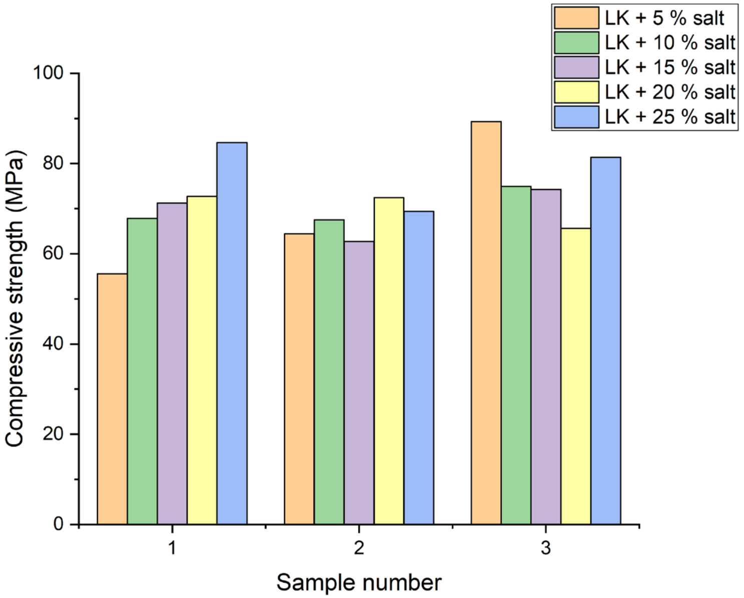
VOJTĚCH GALEK – CVREZ

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

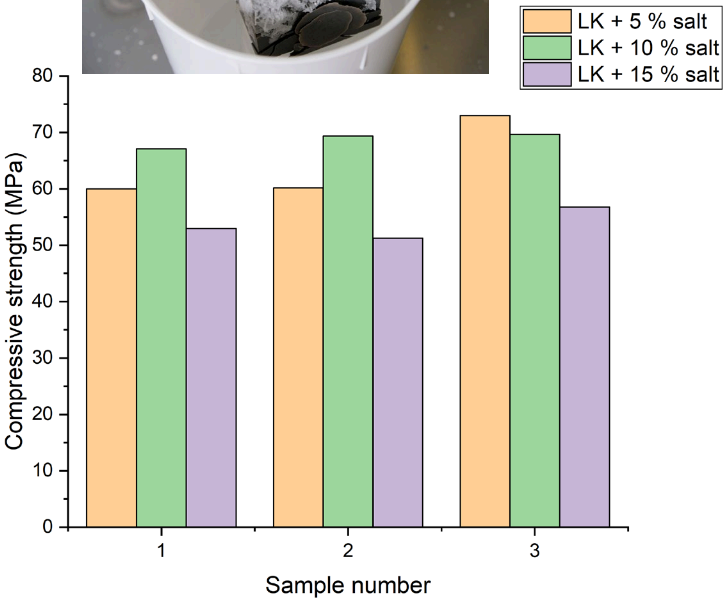
T6.4 – CVRez Overview: Encapsulation of MSO salt waste

CVŘ Research Centre Řež

- The recipe from Czech MK supplier upgraded – LK10



Air dried sampels



Wet environment

- Scale-up experiment
  - 15 wt.% MSO waste
  - 200 kg in total
  - 100 l drum

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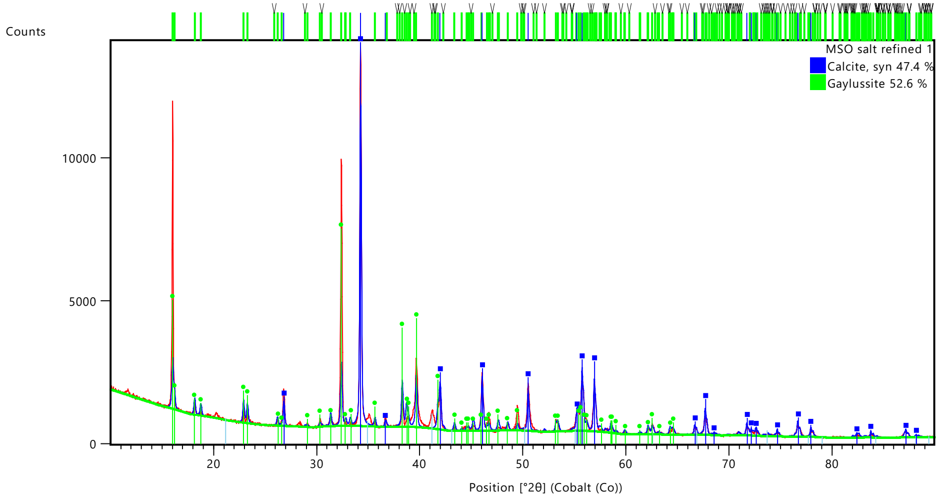
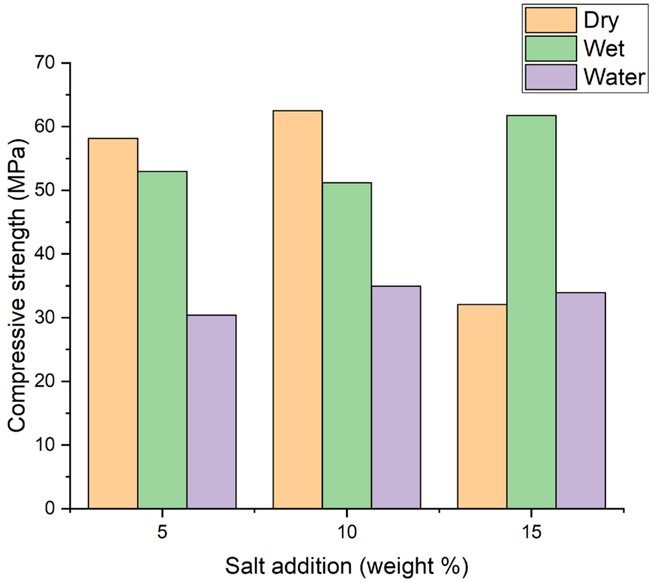
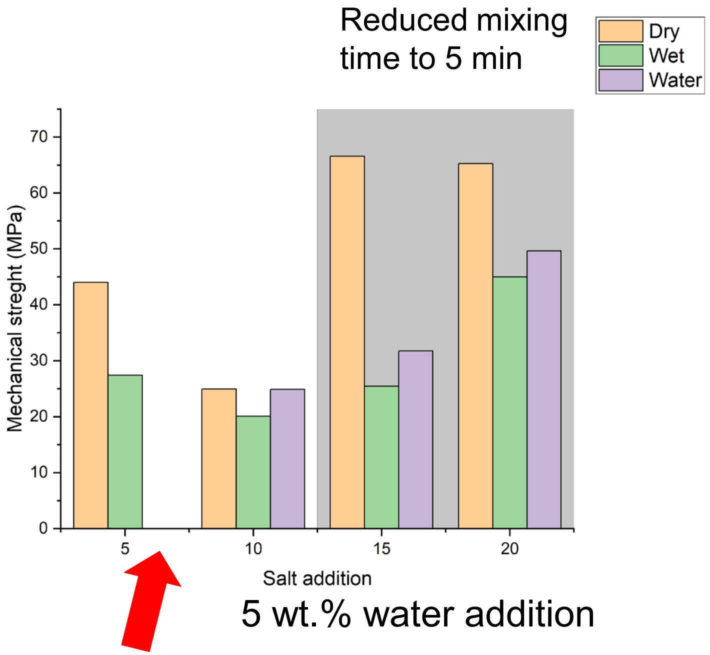
T6.4 – CVRez Overview: Encapsulation of MSO salt waste

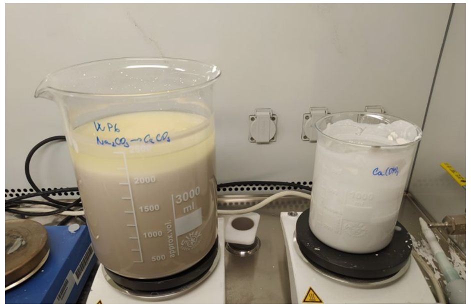
CVŘ Research Centre Řež

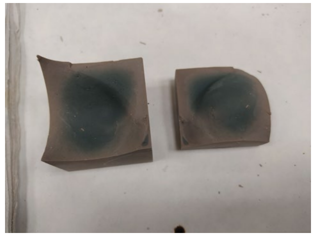
- Enhanced salt – chemically improved MSO waste
- Better stability in wet environment
- Bad workability, fast setting time











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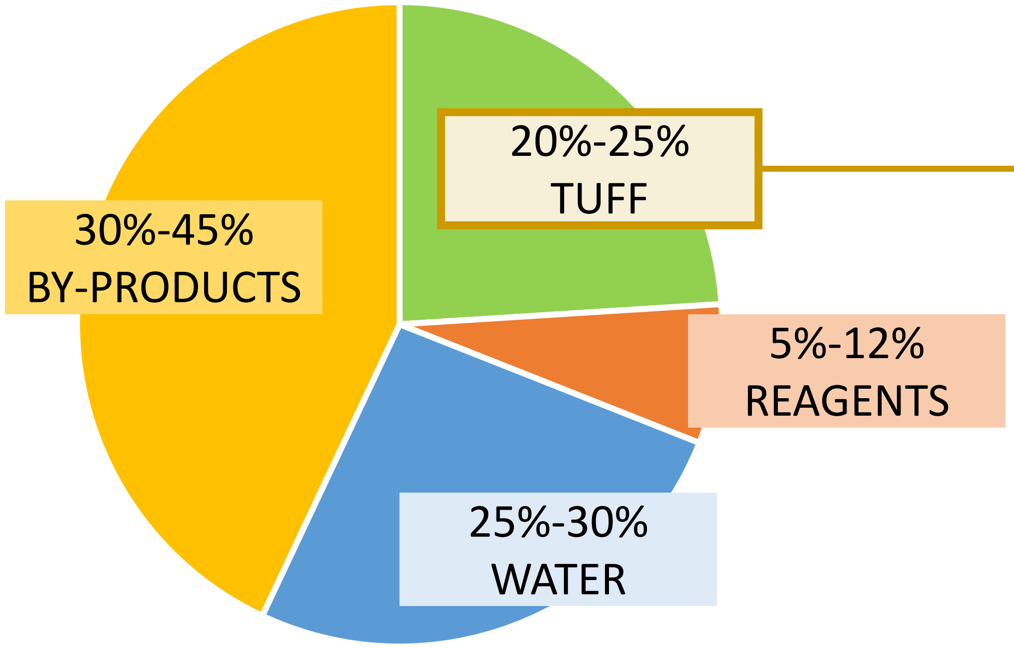
T6.4 – POLIMI: Tuff-based matrix

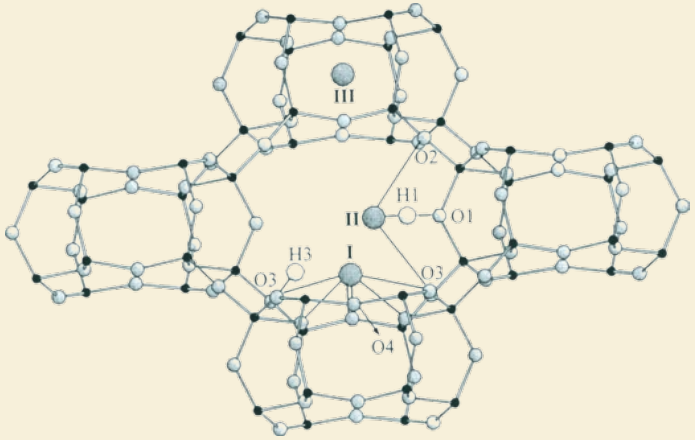
POLITECNICO MILANO 1863

LOW ENVIRONMENTAL IMPACT
SATISFACTORY PROPERTIES



VOLCANIC TUFF

- worldwide availability
- chelation of radionuclides with zeolites
- improved retention of contaminants



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T6.4 – POLIMI: Ongoing work

POLITECNICO MILANO 1863

OXIDATIVE PYROLYSIS

loading of ashes greater than 15 wt.%

satisfactory properties (WAC compliance)

$1.0\ m^3 \rightarrow 2.5\ m^3$

FENTON-LIKE WET OXIDATION

loading of residues greater up to 12 wt.%

satisfactory properties (WAC compliance)

$1.0\ m^3 \rightarrow 1.5\ m^3$

MOLTEN SALT OXIDATION

loading of residues up to 15 wt.%

promising durability (1.5 years immersion)

scarce mechanical properties

IRIS INCINERATION

loading of ashes greater than 20 wt.%

satisfactory properties (WAC compliance)

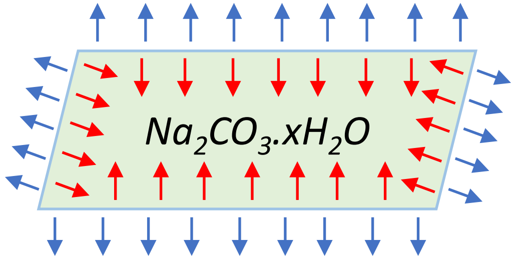
chosen as case study for LCA

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

T6.4 – SCK-CEN Overview: Encapsulation of MSO salt waste

sck cen

MSO salt residue



Strongly hygroscopic, causing volumetric changes

Pretreatment

+ $Ca(OH)_2$

- Calcite ($CaCO_3$)
- Gaylussite ($Na_2Ca(CO_3)_2$)
- NaOH

Blended cement

- CEM I/BFS/Silica fume/lime/limestone
- Waste loading 10-14 wt.%
- F_c 15-18 MPa

Alkali-activated slag

- BFS precursor
- Activated by $Na_2O.2SiO_2$ and NaOH (from salt)
- Waste loading 10-20 wt.%
- F_c 37-42 MPa

PREDIS

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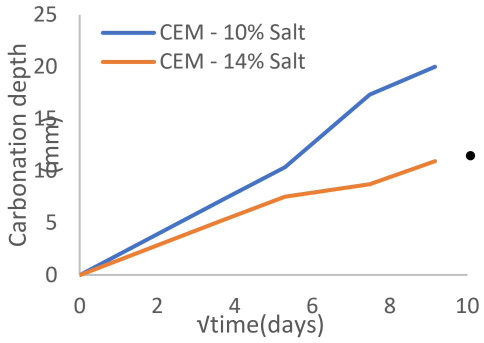
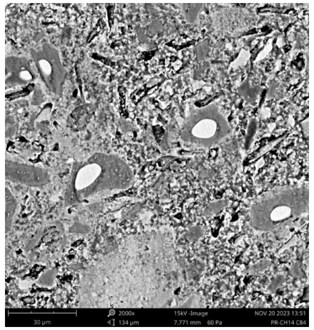
T6.4 – SCK-CEN Overview

Blended cement

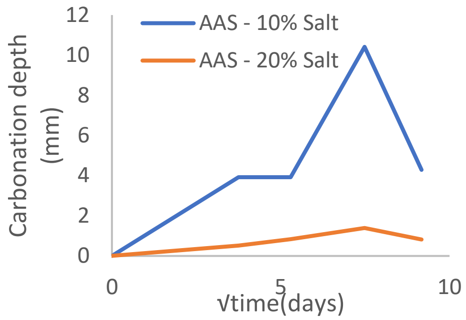
Carbonation



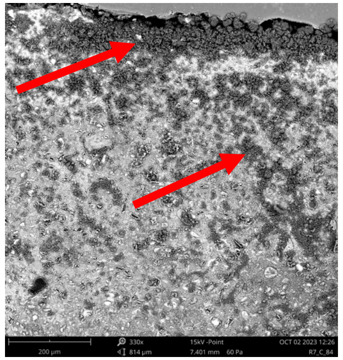
- Higher degree of carbonation compared to AAS
- Gaylussite and unreacted slag react further to form matrix



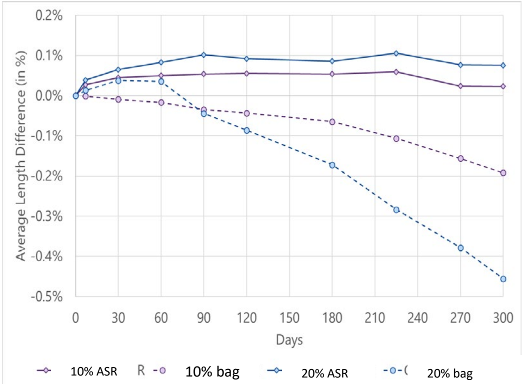
Alkali-activated slag



- High waste loading inhibits carbonation (↑ $Ca(OH)_2$)
- Na_2CO_3 crystals precipitate in pores of carbonated zone

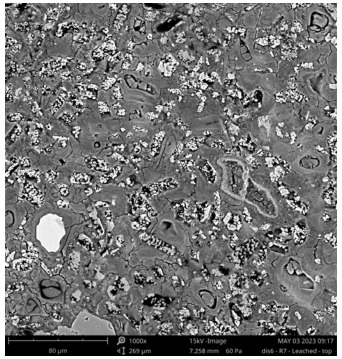


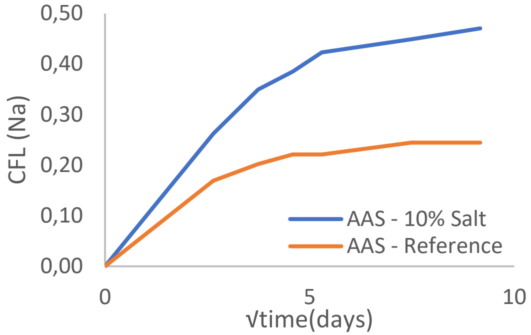
ASR



- Expansion of samples exposed to ASR conditions (40°C, 95% RH)
- Shrinkage of samples aged in a sealed bag

Leaching





- High CFL mostly related to initial runoff (samples not presaturated)
- Negligible effect of leaching on microstructure

PREDIS

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T6.4 – CSIC Overview: Main achievements

CSIC
CONSEJO SUPERIOR DE INVESTIGACIONES CIENTÍFICAS

- The activity of CSIC was focused on developing a procedure for the smart designing of matrixes that better adapt to the type of RSOW.
- Binder matrixes of one-part Geopolymer (*use MK+BFS and powder activators Na_2SiO_3 and NaOH*) and Portland Cement-based systems (CEM I and CEM III) for Ion Exchange Resins (IER) surrogates from different type of treatments: 1) Untreated IER, 2) thermally treated IER (450 ºC) and 3) molten salts (IER oxidation). RSOW doses: 0,10,20 and 30 wt%

Untreated IER

GPO matrices can confine up to 30wt% of UIER, the amount of this RSOW should be limited to 10wt% in Portland cement systems to avoid instabilities in volume

Thermally treated IER

GPO matrices can confine up to 30wt% of IER. In Portland cement systems, the IER loading is limited to 20wt% to meet the waste acceptance criteria (WAC) of compressive strengths of at least 10MPa after 28 days

Molten salt

For GPO and Portland cement-based matrices MS content is limited to 10wt% to obtain suitable mechanical and microstructural properties

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

T6.4 – CSIC Overview:Future practical recommendations

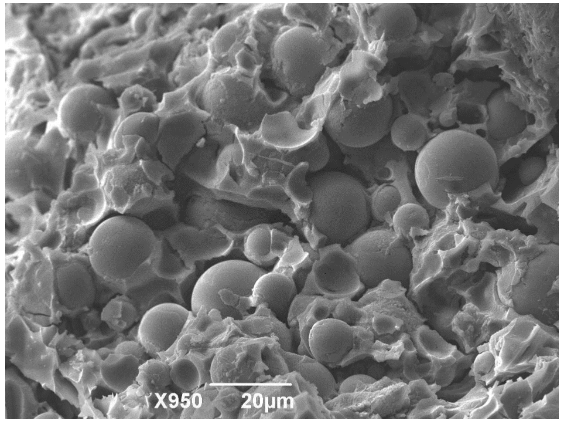
CONSEJO SUPERIOR DE INVESTIGACIONES CIENTÍFICAS

- CSIC contribution highlights that each RSOW requires an specific confining cementitious matrix for an adequate and safe immobilization.
- Designing and developing any cementitious systems is going to be highly depended on the typology of the RSOW to immobilize, being necessary a case-by-case assessment.
- The customization of the cementitious matrix, highly depend on the type of RSOW, will therefore be necessary to obtain a robust system.
- The future validation of the performance of the waste-form matrices at higher tailor-readiness level (scale-up) is fundamental.

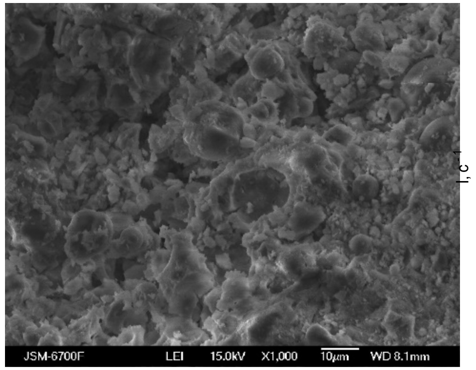
This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

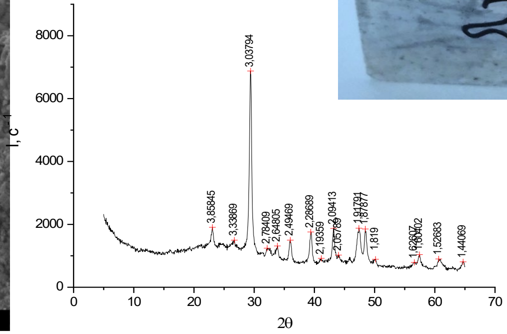
T6.4 – SIIEG NASU Overview

- SIIEG NASU has developed matrices (cement-based and geopolymer) for ash waste after treating IER.
- The direct encapsulation method, involving mixing slag-MK-based GP and using sodium hydroxide + sodium silicate solution as activators, has proven highly effective in our research.
- The samples were thoroughly analysed using various techniques, including XRD, DTA, SEM, etc., ensuring the robustness and reliability of our findings.



The SEM geopolymer matrices





The SEM and XRD cement-based matrices

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

T6.4 – VTT Overview: MK based geopolymers

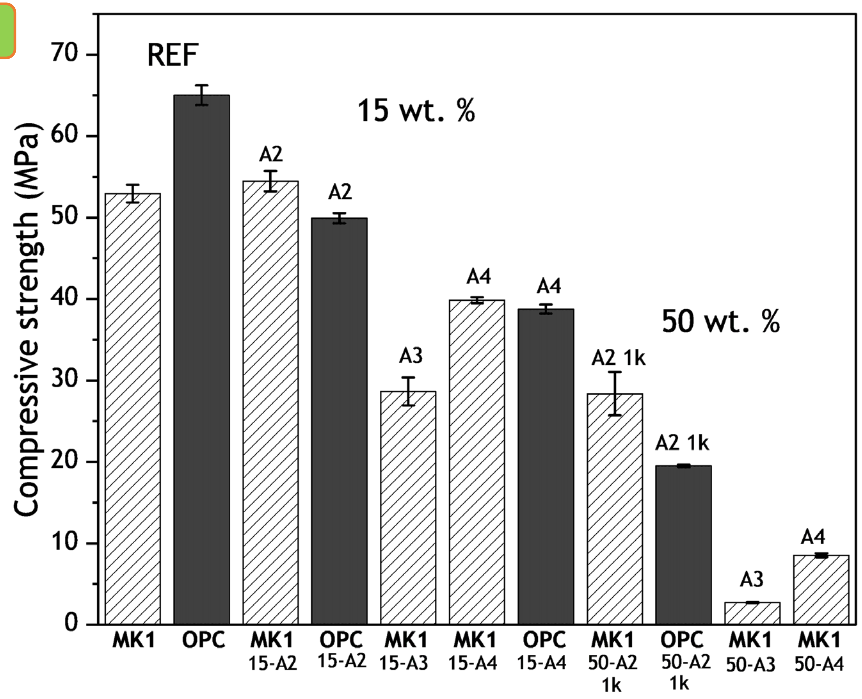
- Different gasified IXRs and metakaolin types used in pruduction of geopolymers and reference Portland cement (OPC) waste matrices

Metakaolin (MK) + ash + Na/K silicate solution → geopolymer matrix

Sample	Ash (wt. %)	Ash type	Cs, Eu, Ce (ppm)	Fe_2O_3 (wt. %)
MK1	-	-	-	-
OPC	-	-	-	-
MK0 1-A1	1	A1	Cs [200]	
MK1 15-A2	15	A2	-	-
MK1 50-A2	50	A2	-	-
MK1 50-A2 1k	50	A2	1000 in liquid	-
MK2 50-A2 1k	50	A2	1000 in liquid	-
OPC 50-A2 1k	50	A2	1000 in liquid	-
MK1 15-A3	15	A3	300, 50, 50	-
MK1 50-A3	50	A3	300, 50, 50	-
MK1 15-A4	15	A4	250, 50, 75	8
OPC 15-A4	15	A4	250, 50, 75	8
MK1 50-A4	50	A4	250, 50, 75	8
OPC 50-A4	50	A4	250, 50, 75	8

MK0 – metakaolin Metastar
MK1 – metakaolin Argical
MK2 – metakaolin MK40
OPC – Ordinary Portland cement

Ash type	Comp	Cs, Eu, Ce
A2	High Ca, Mg, no S	1k – in liquid
A3	Amorphous sulfates	300, 50, 50
A4	Pyrite + magnetite	250, 50, 75 + Fe_2O_3



- Fe_2O_3 in S-rich ashes restrain the initial damage to geopolymer structures due to the formation of pyrite (more stable than amorphous S).
- Geopolymer microstructure was found to be less porous and more durable for the waste ash containing added iron (vs. without added Fe)
  → performance benefit from pre-treatment of iron-containing resins
- Mechanical performance of geopolymer matrices formed wherein tracers have been added in liquid, "separately from the ash", at equivalent loadings suggests minor reductions in compressive strength

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

T6.4 – VTT Overview: Conclusions

VTT

- Overall volume reduction of combined gasification and immobilization of wasteforms: 98% for geopolymers, and 94% for cements (by weight)

- Geopolymers and OPC based binders have similar limitations of mechanical for loading of **un-gasified IXRs (15%)** but geopolymers have more tolerance than OPC for higher loading of **gasified IXRs** (geopolymers up to **50%** vs. **30%** for cement).

- Geopolymer waste forms can exhibit higher compressive strength to cement-based waste forms at the same waste loading

- Composition of the ash and other additives (e.g. nuclides, elements) showed notable effects on characteristics of the geopolymer waste form → Need for studies with more detailed optimization of waste form initial composition and studies with real waste

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T6.4 – CEA Overview

- **Molten glass coating**

- ✓ Low T° melting
- ✓ Morphology
  - flakes
  - powder

- ✓ Morphology
  - raw
  - crushed

Mix ratio ashes/glass from 20/80 to 80/20

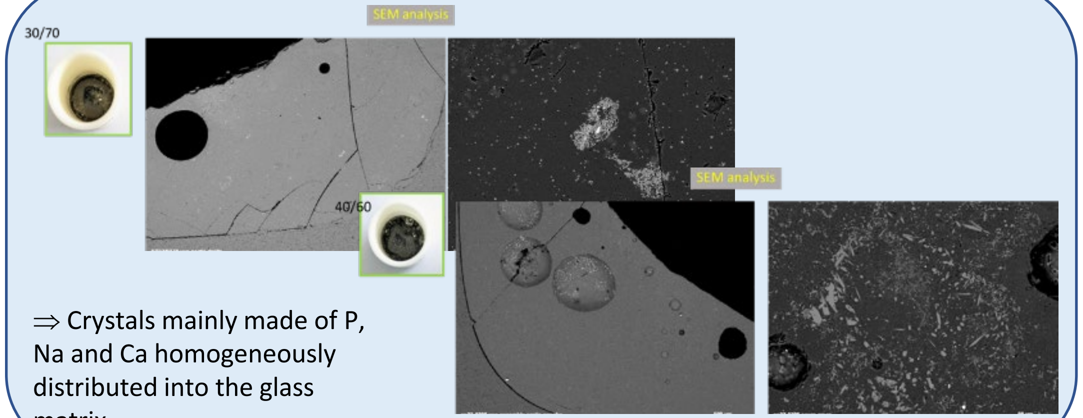
- ✓ Soft process
- ✓ Avoid radionuclide volatilization
- ✓ Coating of the waste
- ✓ No pressing, under air condition
- ✓ Obtaining a monolithic form

- **Screening tests: best ratio waste/glass, material morphology, T° treatment to obtain an homogeneous monolithic bock**

20/80	30/70	40/60	50/50	60/40

ratio waste/glass

⇒ Complete incorporation of waste up to load rates of 40%



⇒ Crystals mainly made of P, Na and Ca homogeneously distributed into the glass matrix

- **Stability testing (Short term leaching experiments) in cementitious water**

Alteration speed (g/m²/j) Average 91 d	Molten glass
Si	0,39
B	0,49
Al	0,36
Zn	0,50
Barycentric 91 d	0,42

⇒ Highly satisfactory results compared with SON68 nuclear glass

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WP6 T6.4. Overview

Thank you

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PREDIS

Densification

RUSSEL HAND / LUCY MOTTRAM (USFD)

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

PREDIS

Work package 6.5 overview

- Densification of materials to form solid wasteforms:
  - Direct reconditioning (e.g. polymers)
  - Conditioning of thermally treated materials from WP 6.3
- Utilising various presses/compaction equipment with or without additives
- Production of material for characterisation / stability testing in WP 6.6

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

PREDIS

Work package 6.5 overview

- Aims of the WP:
  - Demonstrate densification using Hot Isostatic Pressing (HIP) technology for wet oxidation sludges, ashes, and polymeric material
  - Demonstrate simple compaction-based densification of thermally treated wastes (e.g. ashes)
  - Increase HIP TRL by addition of radiotracers or radioactive samples
  - Optimise process variables (temp, pressure, time, matrix, etc.)

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WP 6.5 HIP Technology

HP 630: Research HIP at USFD
Process canister volumes up to ~300 cm^3

- Hot Isostatic Pressing (HIP) – application of temperature and pressure simultaneously using pressurised inert gas
- Isostatic pressure – uniform work piece consolidation, not just uniaxial pressure, allowing more uniform densification
- Sealed containers: minimising secondary wastes
- Sealed containers: volatile element retention
- No volatile off gas during treatment

Inactive canister processed at 1250 °C/ 103 MPa/ 4 hrs

PREDIS
This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

WP 6.5 HIP Technology

HP 630: Research HIP at USFD
Process canister volumes up to ~300 cm^3

- Batch process: flexibility and accountability
- Up to 100% waste loading possible (e.g. inorganic zeolites)
- Range of processing conditions
- Can be tailored to each waste
- Volume reduction
- USFD capability to process radioactive materials using an AFIC (Active furnace isolation chamber)

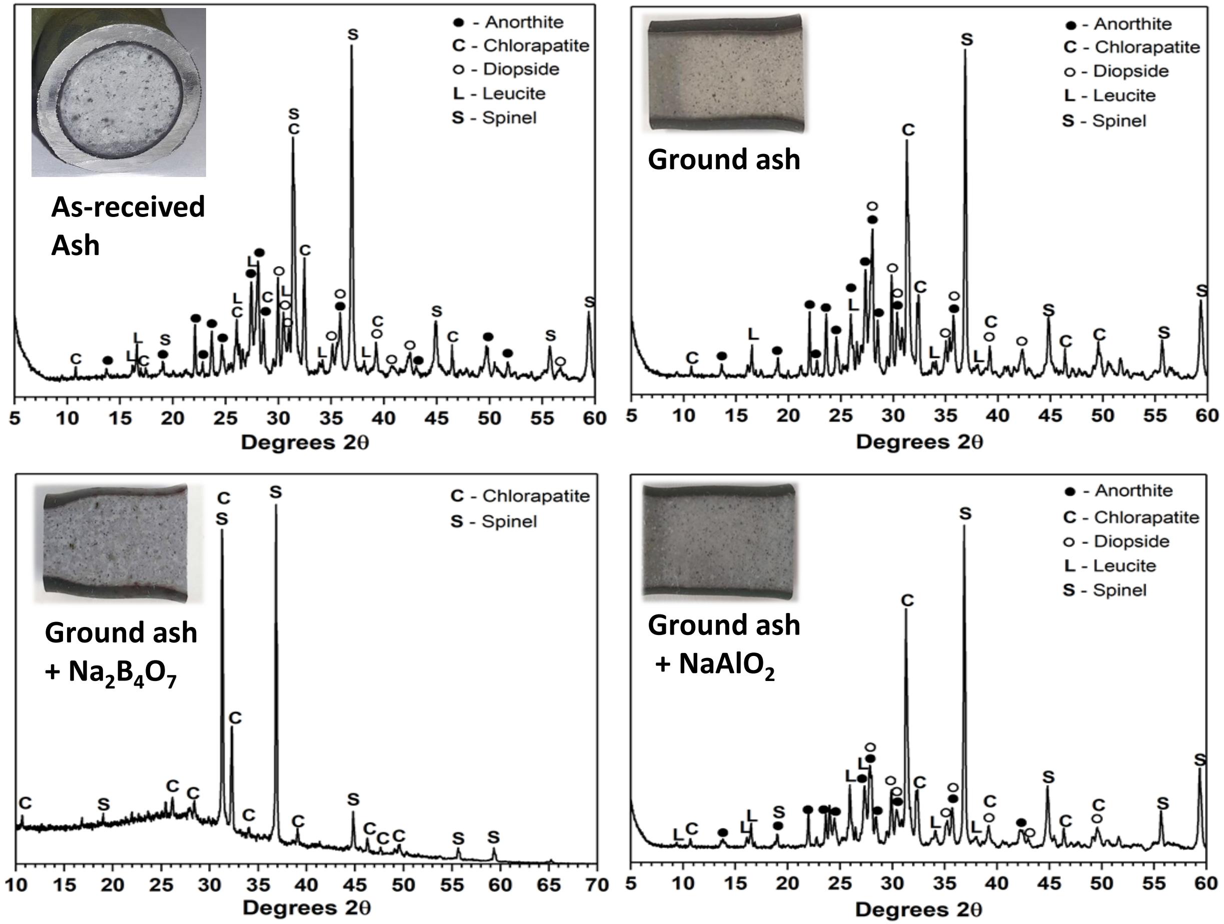
Inactive canister processed at 1250 °C/ 103 MPa/ 4 hrs

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

WP 6.5 IRIS Ashes

IRIS Ashes

- Supplied by CEA (WP 6.3)
- Four compositions attempted
  - Ashes
  - Ground Ashes
  - Ashes + 5 wt.% $Na_2B_4O_7$
  - Ashes + 5 wt.% $NaAlO_2$
- 1250 °C and 100 MPa for 2 hours
- HIPping of all compositions successful
- $Na_2B_4O_7$ most successful additive, most of ash melted, no crystalline silicates present.



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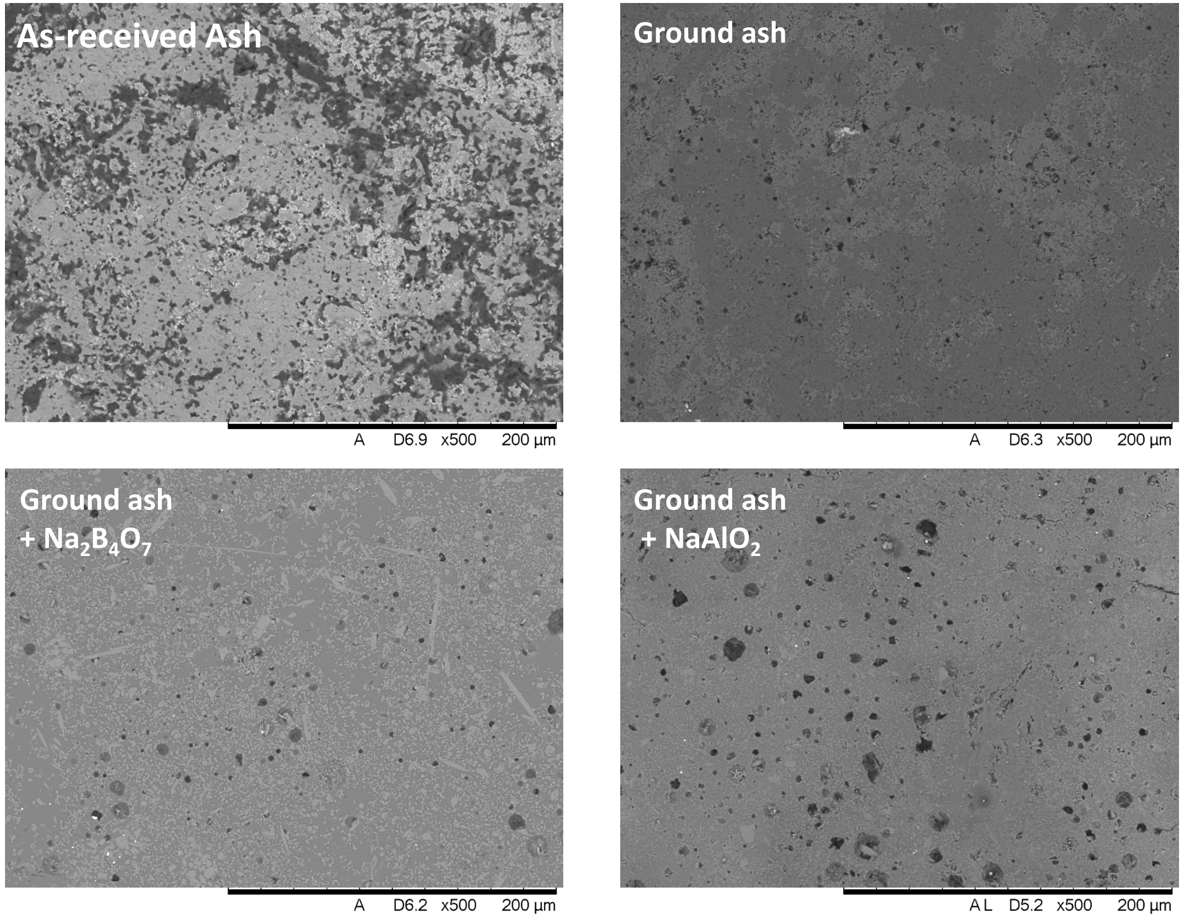
WP 6.5 IRIS Ashes

- Grinding improved homogeneity
- Ground ash + $Na_2B_4O_7$ also had least aggressive wateform-HIP can interactions
- Samples of ground ash + $Na_2B_4O_7$ were produced for long-term dissolution trials and sent to SCK-CEN for (WP 6.6)
- Long term dissolution trials also undertaken at USFD (WP 6.6)



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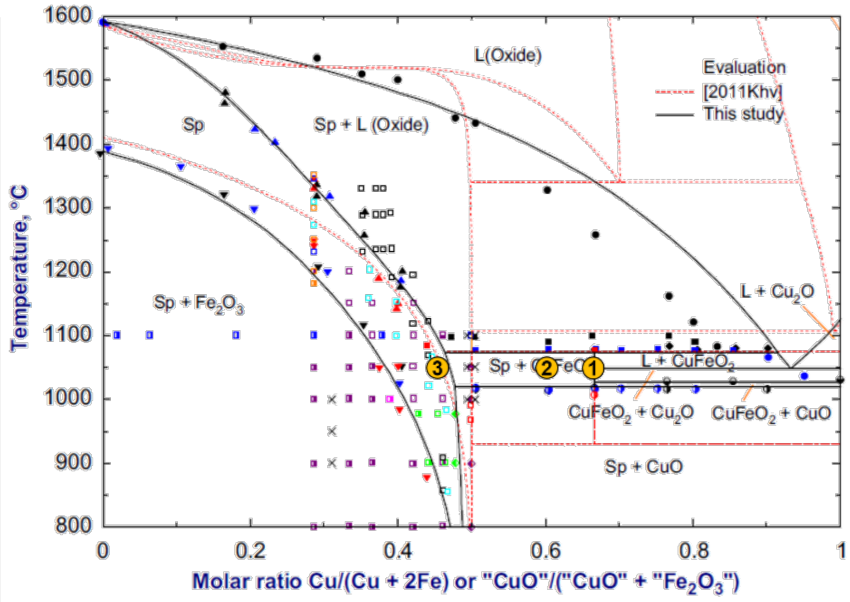
WP 6.5 Wet oxidation sludge

- Wet oxidation sludges – composition from WP6.3
- Preliminary trial based on three points selected along the CuO-Fe_2O_3 binary phase diagram
- Wet oxidation composition should form delafossite ($CuFeO_2$) **(1)**
- Addition of extra Fe_2O_3 should form spinel (e.g. $CuFe_2O_4$) **(2)(3)**.
  - Could form $NiFe_2O_4$, $CoFe_2O_4$, $MnFe_2O_4$, etc. in 'real' waste
- Phase diagram suggests 1080 °C is maximum processing temperature before melting occurs
  - CPS used to check if single-phase products can be formed at this temperature before HIPping trials



Composition	Fe_2O_3:CuO	CuO/(CuO+Fe_2O_3)	Target phase
1(WetOx)	0.5	0.67	delafossite
2	0.67	0.60	spinel
3	1.21	0.45	spinel

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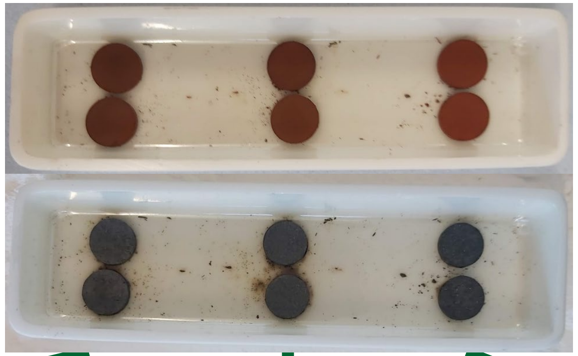
WP 6.5 Wet oxidation sludge

CPS trial

- Milled, dried, pressed, heated to 1050 °C – 4hr dwell
- WetOx (0.5) composition gave:
  - Delafossite ($CuFeO_2$)
  - Tenorite (CuO)
  - A small amount of spinel ($CuFe_2O_4$)

Fe_2O_3:CuO

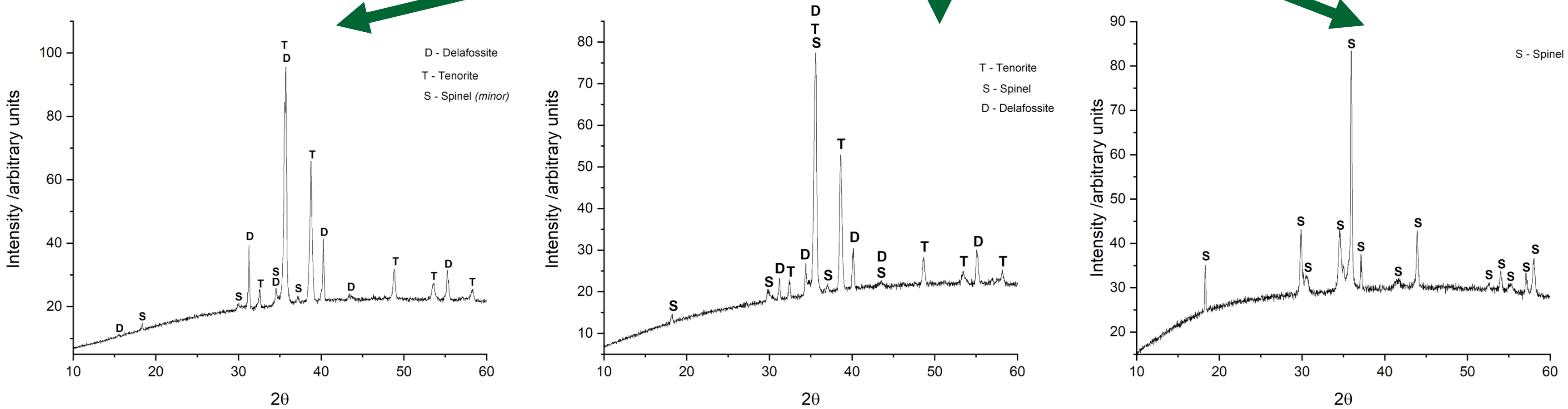
0.50 0.67 1.21



Pre thermal treatment

Post thermal treatment

- Formation of spinel increases with Fe
- Spinel only phase detected in the highest Fe content composition.



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WP 6.5 Wet oxidation sludge

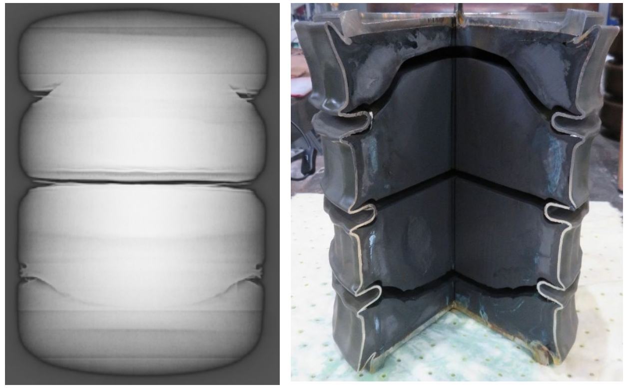
HIP trials

- HIPping delayed at USFD due HIP being offline for many months
  - Same compositions as CPS trials have now undergone HIPping.
  - Characterisation of HIPped samples ongoing

- NNL conducted larger scale trial before data supplied
  - One can baked out imperfectly – abandoned at this stage
  - Second one contained excess nitrogen which resulted in cracking
  - Still trying to ascertain cause of failure

USFD WetOx HIP cans pre and post HIPping

HIP at NNL Workington facility



HIP can from second attempt

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This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

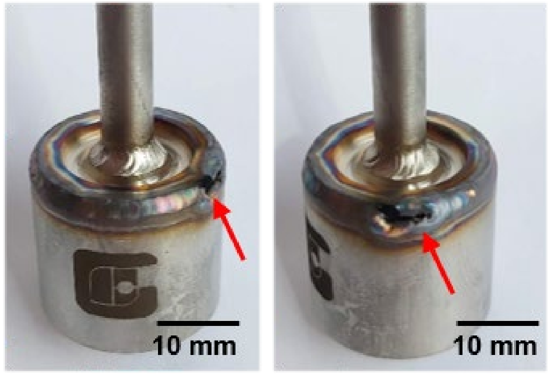
WP 6.5 Polymer HIP

Amberlite IRN-150

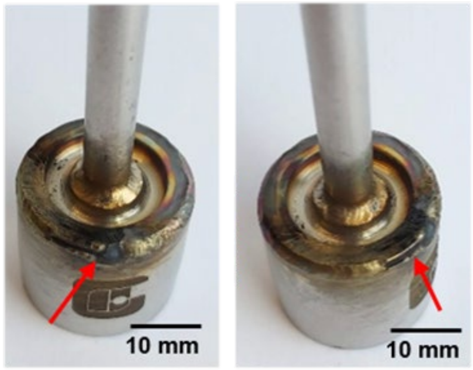
- A mixed bed cation-anion nuclear grade exchange resin
- The same resin that was treated via Fenton wet oxidation within WP 6.3.

HIP attempts

- Amberlite IRN-150 and Amberlite IRN-150 + low density polyethylene (LDPE) (1:1 wt.)
- Attempts to weld the HIP cans caused heated, pungent liquid to escape from the yet unwelded area of the HIP can.
- Attempts to complete welds faster or at a lower power setting were unsuccessful.
- As HIP canisters could not be welded, and welding heat caused melting or thermal degradation of waste attempts were ceased.



Attempt 1: Amberlite IRN-150



Attempt 2: Amberlite IRN-150 + LDPE

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WP 6.5 Conclusions

HIP summary

- Polymers – unsuccessful
- IRIS ashes – successful
- Wet oxidation sludge – successful

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

WP 6.5 Acknowledgements

- Sam Walling, Josh Radford, Sarah Pepper, Neil Hyatt and Claire Corkhill
- Thank you PREDIS WP6 colleagues!

HADES

midas

HENRY ROYCE INSTITUTE

NucleUS
Immobilisation Science Laboratory

University of Sheffield

NATIONAL NUCLEAR LABORATORY

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PREDIS

Physico-chemical characterisation of reconditioned waste form and stability testing

EMMI MYLLYKYLÄ, ELENA TORRES ALVAREZ, EROS MOSSINI

(CSIC, CEA, CIEMAT, POLIMI, SIEEG, SCK CEN, UAM, UH, USFD, VTT) (VTT)

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Objective

- To study the **chemical and mechanical durability** of different waste forms produced from low and intermediate level waste in previous tasks (6.3, 6.4 & 6.5)
- Characterization of the produced waste forms (initial form)
- Leaching experiments
  - Release of the elements from waste matrix (degradation / durability)
  - Release of elements originating from the waste (immobilization efficiency)
- Characterization of changes in materials after the leaching periods from 3 to 24 months

→ **Expected mechanical and chemical behaviour of the waste forms during disposal**

←→ Scientific understanding behind the observations

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

PREDIS

Common efforts for comparability of the results

- **Reference leaching protocol** created together in the beginning of the project
  - Instructions for preparation of synthetic cementitious water (SCW)

  + Additional protocols in agreement with national standards or conditions

- **Shared data sheets** for collection and calculation of **final results**
  - All samples indexed with same rules
  - Normalized mass losses
  - Leaching indices
  - Pre and post characterization observations

PREDIS Reference protocol		**Additional information**
Leachant	**Synthetic cementitious water** Composition for the leachant from EURAD. "CEM I + silica fume" synthetic water (without silica)	**pH ~12.7** For 1 L: - 1.8858 g **K_2SO_4** - 0.0774 g **$CaSO_4$, 2 H_2O** - 50 ml **1 M KOH** - 950 mL Milli-Q® water
Type	**Semi-dynamic** (each step refreshing the complete volume of leaching solution)	Changing frequency: **1st year** 7 days, 14, 21, 28 d, and monthly there after **2nd year:** 14, 16, 18 months and 2 years
Sampling intervals	Modified from ISO 6961-1982; Long-term leaching testing of solidified radioactive waste forms	**1st year**: 7 days, 14, 21, 28 d, and monthly there after **2nd year**: 14, 16, 18 months and 2 years
Temperature	**22 ± 2 ° C**	Some partners will use also 40, 70 or 90 °C
Duration	At least **90 days** (or until leaching rate has become constant) for short term studies AND **2 years** for long-term studies	
Leachant/Specimen	Volume of the leachant / exposed **"geometric" surface area of specimen"** **Ratio** 0.10 ± 0.02 m (= 10 cm) between leachant volume and specimen external surface area.	Ratio means e.g. 10 cm^3 of solution per 1 cm^2 of sample surface area. Ratio kept same, specimen geometry and size can vary

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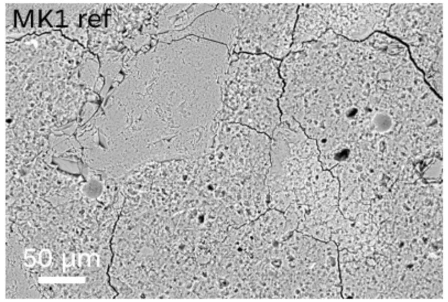
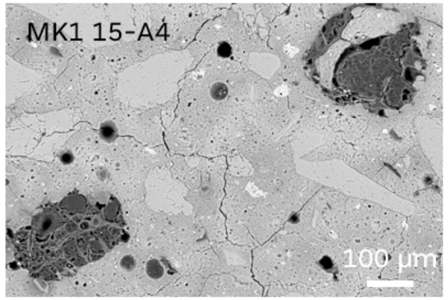
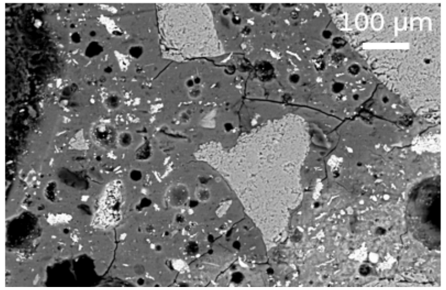
Geopolymer samples for leaching

Institute	Matrix composition	Type of waste	Waste loads tried (%w/w) in T. 6.6.	Challenges found	Homogeneous distribution of waste	Compatibility waste-matrix
Geopolymer						
VTT - UH	Metakaolin-based (2 types)	Ashes	15 & 50	Mechanical strength decreases with waste load (but > 10MPa)	Yes	Good
SCK·CEN	$MK+BFS+Na_2Si_2O_5$	Molten salts	10 & 20	Unable to mix high viscosity salt slurries	Yes	Good
CSIC/UAM /CIEMAT	$MK+BFS+Na_2SiO_3$	IERs ashes	Up to 30	Waste reduces compressive strength	Yes	Acceptable, but porosity increased
POLIMI	Volcanic tuff, BFS, FA, NaOH	IRIS ashes	Up to 50	Delayed setting	yes, by visual inspection	Good but setting time increases
		Molten salts	Up to 20		yes, by visual inspection	Good
		Ashes (dryox)	Up to 40	Not yet assessed	yes, by visual inspection	Good
		Ashes (wetox)	Up to 35	Poor mechanical properties	yes, by visual inspection	Good





MK1 50-A4



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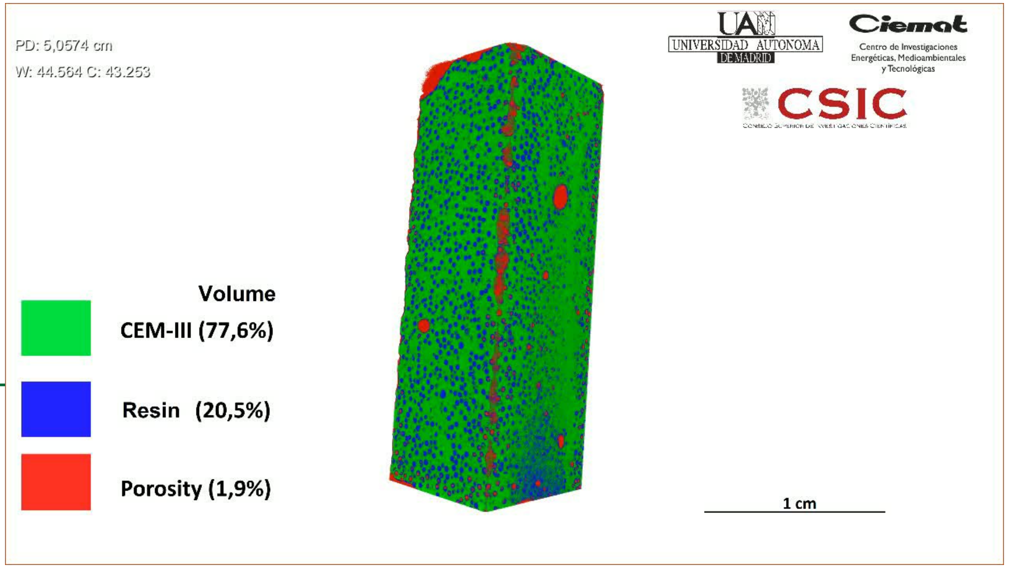
Cement samples for leaching



Institute	Matrix composition	Type of waste	Waste loads tried (%w/w) in T. 6.6.	Challenges found	Homogeneous distribution of waste	Compatibility waste-matrix
Cement						
VTT-UH	CEM I	Ashes	15 & 50	Mechanical strength decreases with waste load	Yes	Good
SCK·CEN	CEM I (BFS, limestone filler and sand, lime & silica fume)	Molten salts	10 & 14	Unable to mix: high viscosity salt slurries	Yes	Good
CSIC/UAM/ CIEMAT	CEM I/42.5 SR	IERs ashes	Up to 30	The waste substantially delayed setting and reduced the mechanical properties	Yes (µ-CT)	Low compatibility: poor adhesion waste/matrix and the pososity increased
	CEM III/B32.5	IERs ashes	Up to 30	The waste delayed setting and reduced the mechanical properties	Yes (µ-CT)	Acceptable compatibility, but the pososity increased

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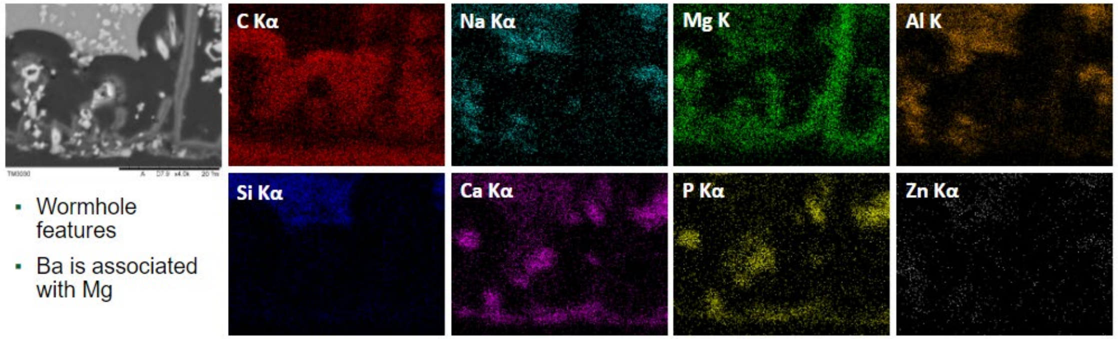
Glass/ceramic samples for leaching

Institute	Matrix composition	Type of waste	Waste loads tried (%w/w) in T. 6.6.	Challenges found	Homogeneous distribution of waste	Compatibility waste-matrix
Glass/ceramic						
USFD	Glass	IRIS ashes	95	------		
	Glass-ceramic	IRIS ashes	95 & 100	------		
SCK·CEN	Glass-ceramic	IRIS ashes	95	------		
CEA	Borosilicate & ashes (SiO_2, Al_2O_3, Na_2O, CaO, B_2O_3)	IRIS ashes	30	------	No, some crystalline phases	Good, some crystals and increased porosity
	Densified thermally-treated ashes (SiO_2, Al_2O_3, CaO, ZnO)	IRIS ashes	100	------	Yes, sintered pellets	

Month 3 - Ground Ash + $Na_2B_4O_7$



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Characterization methods

Analysis of leachates	WP6 Leaching Protocol	Additional
Monitoring:		
pH	✓	
Electrical conductivity	✓	
ORP		✓
Chemical Analysis:		
Main elements (Si, Al, Ca, B,….)	✓	
Traces (Cs, Sr, Co, Cr, Fe, Zn, Co, Ni, I, Lanthanides)		✓
Main anions		✓
TOC		✓
Colloidal Size particle		✓

Characterization of solids *(pre-leaching & post-mortem)*	WP6 Leaching Protocol	Additional
Mechanical properties *(Compressive Str.)*	✓	
Microstructural characterization		
XRD	✓	
SEM-EDS	✓	
MIP /BET		✓
FTIR		✓
TG/DTA		✓
NMR		✓
Micro-tomography		✓
EXAFS		✓

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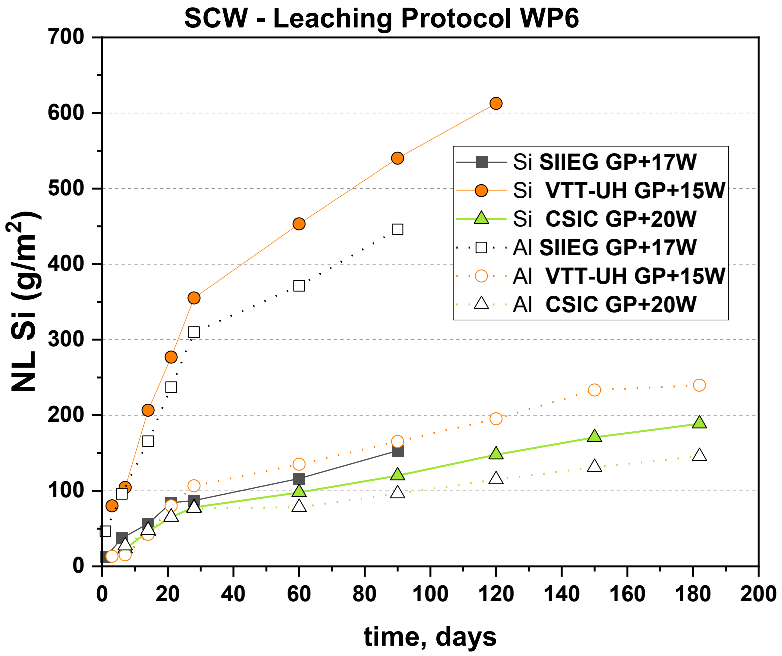
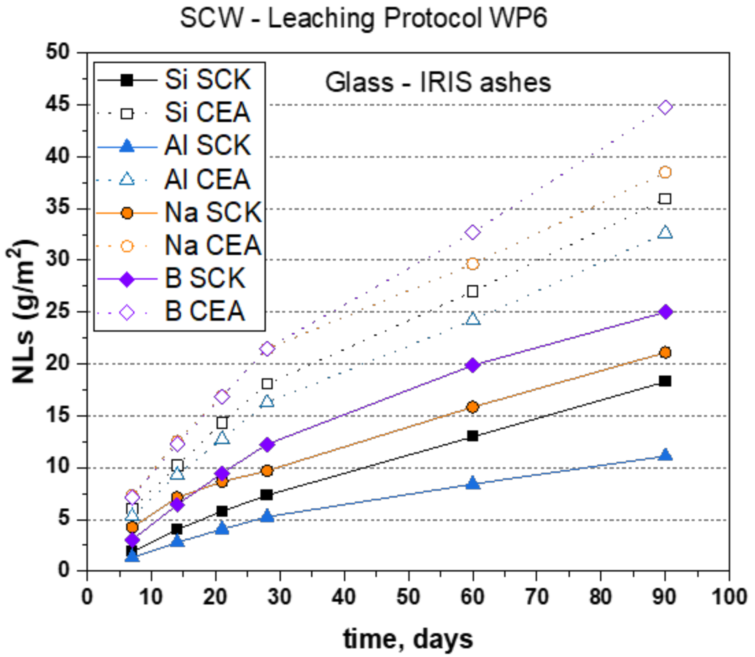
Leaching – matrix degradation

- In general higher release rate during during first 30 to 90 days leaching



GPO's:
Different samples - same leaching method

- Differences in behaviour of Al vs. Si



Molten glass vs. HIPed IRIS ashes ($Na_2B_4O_7$ based)

- B> Na> Si>Al

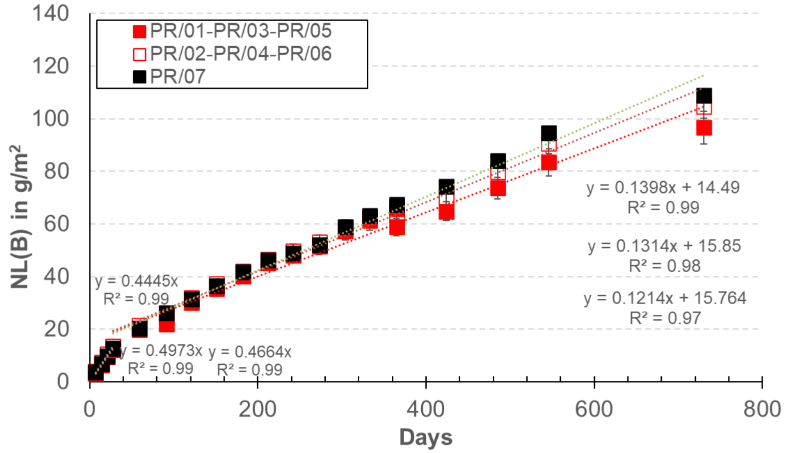
This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

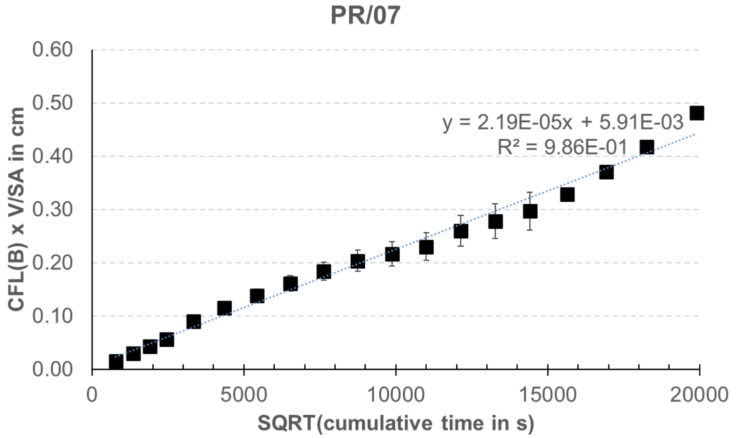
Results from solution data HIPed IRIS ashes (95 wt.% + 5 wt.% $Na_2B_4O_7$

sck cen

- Higher normalized mass loss for B than for the main elements constituting the ashes
  - Average maximum **dissolution rate** between 0 and 28 days: 0.47 ± 0.05 g/m^2
    - Maximum rates for borosilicate glasses in KOH at pH 12.5 and 30 °C: 0.1 – 0.2 g/m^2d
  - Slight rate decrease after 28 days: 0.13 ± 0.01 g/m^2d
- Dissolution rates from the sample mass losses ~ rates from NL(B)
- According to ANSI/ANS-16.1-2019
  - Average effective **diffusion coefficients**:

  3.87E-12 (Zn) < D_e < 3.77E-10 cm^2/s (B)
- Good repeatability of the results





- De = (PI)/4 * $(2.19e-05)^2$ = 3.77E-10 cm^2/s
- LI = log (1/ 3.77E-10) = 9.42

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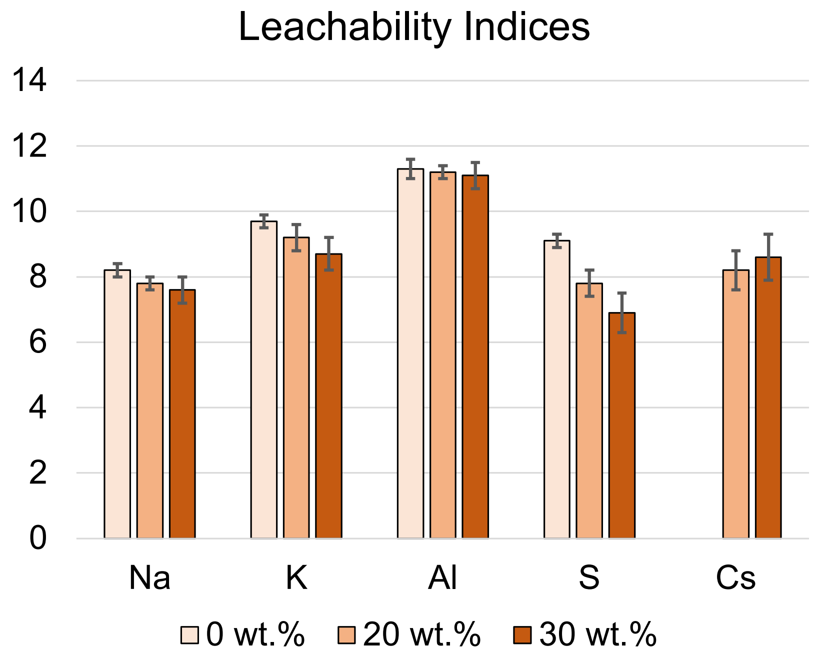
Leachability index according ANSI/ANS

Gained leachabillity indices **above the treshold limit > 6** for all waste matrixes studied

- **Release of immobilized elements e.g. Sr, Cs**

*Deionized Water *Synthetic Cement Water (WP6)

6 months	Cs	Sr
GPO+20RW	**12,3* / 8,1***	**13,1 / 11,4**
CEM III+20RW	11,9 / 12,4	7,7 / **10,1**
CEM I+20RW	10,3 / 10,5	7,4 / **11,4**
18 months		
GPO+20RW	**12,4**	**13,1**
CEM III+20RW	12,2	7,7
CEM I+20RW	10,6	7,4

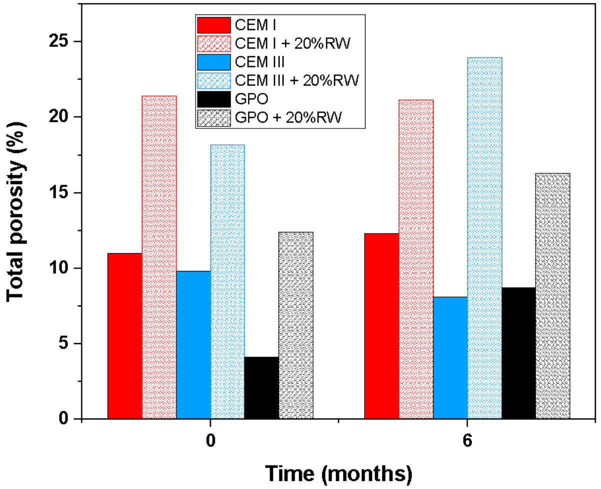


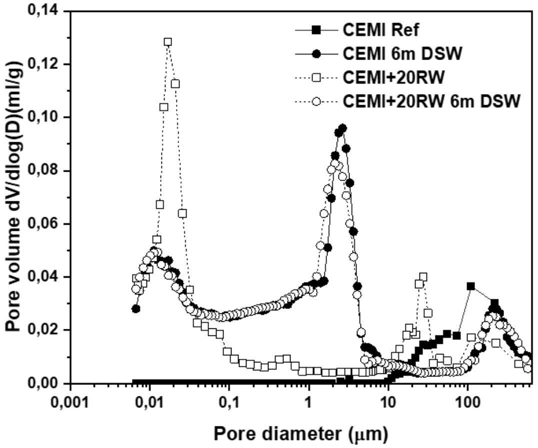
This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

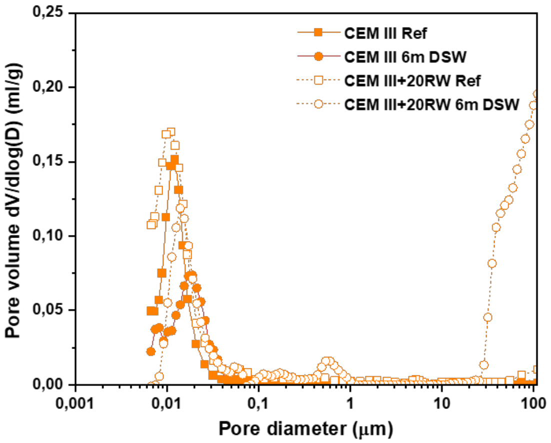
Post-mortem characterization – changes in Porosity

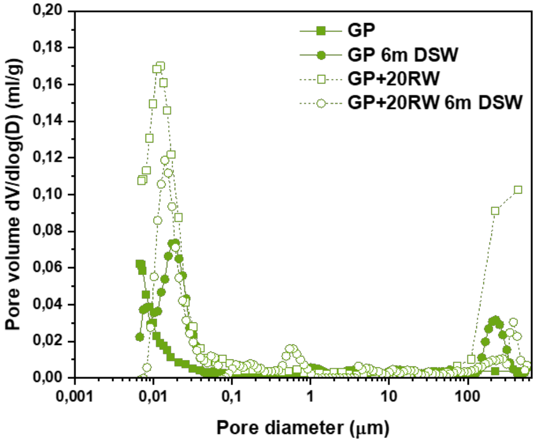
Initial vs 6 months

- Increase of total porosity but not significantly
- Evolution of pore size distribution towards smaller pore sizes









- In CEM III total porosity remains almost same
- Slight increase in CEM I and GPO

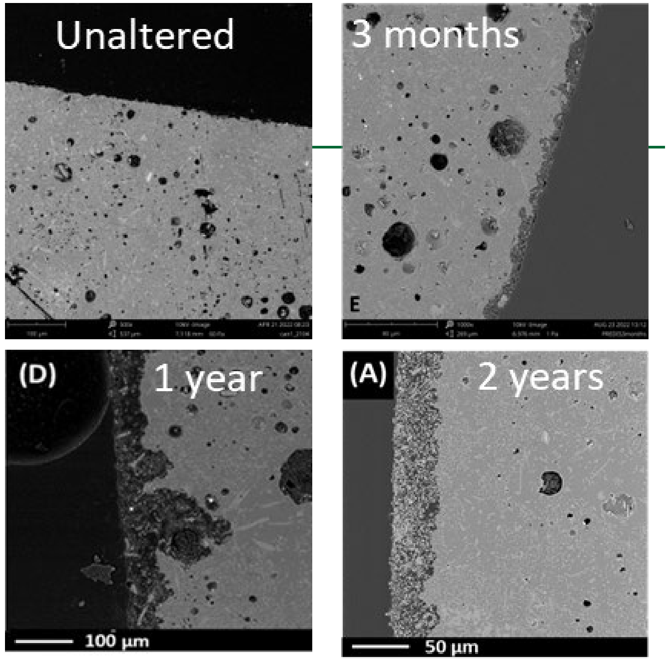
CSIC Consejo Superior de Investigaciones Científicas

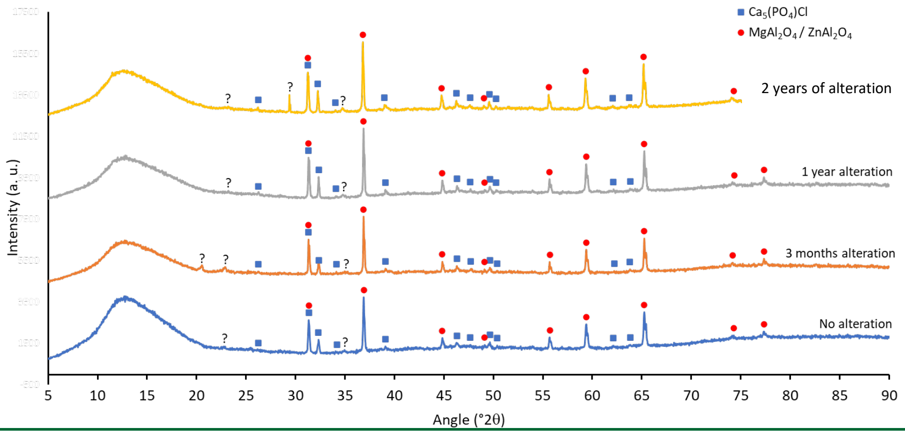
UAM Universidad Autónoma de Madrid

Ciemat Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Characterization of HIPed ashes





SEM –EDX analysis (cross section of the embedded altered sample)

- Increase of the mean thickness alteration layer with time: from ~25 µm after 1 year to ~45 µm after 2 years
  - Higher thicknesses in some areas due to the presence of pores at the surface of the original sample
- Alteration layer made of an amorphous matrix with crystals, which are similar to those present in the original sample; wide range of compositions for both the amorphous matrix and the crystals; penetration of the resin
  - Dissolution of the amorphous matrix of the original sample
    - Better chemical durability of the crystalline phases wrt. amorphous phases

XRD analysis

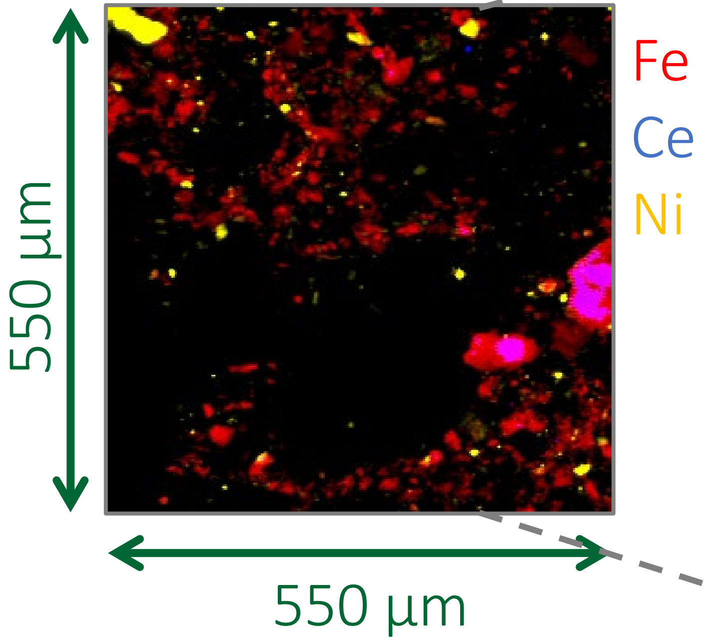
- Main crystalline phases in the original sample and in the alteration layer: calcium chloride phosphate and magnesium and zinc aluminum oxides
- A few diffraction peaks observed but no identification of the phases

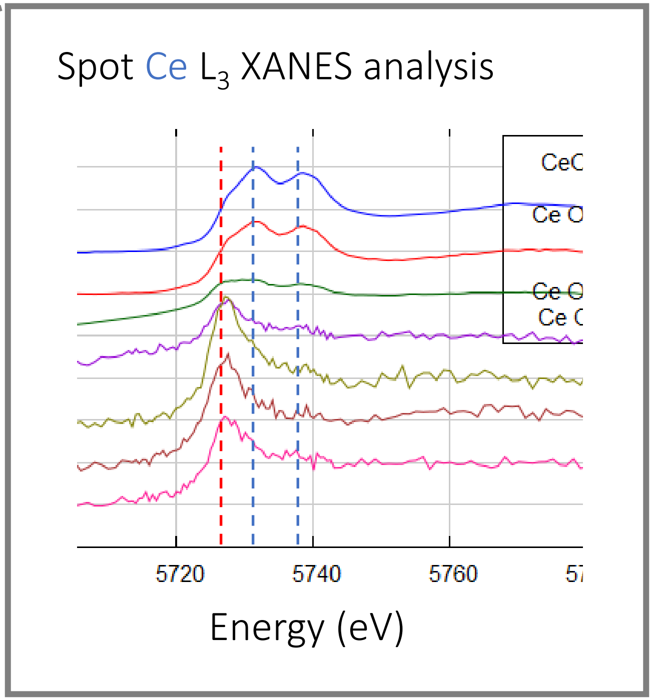
This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Post-mortem characterization
µ-XRF, -XRD and -XANES

- Synchrotron based **µ-XRF**, **-XRD** and **-XANES** provide spatially resolved information on the contaminants (Cs, Ce, Eu, Ni), their bonding environments, its evolution and leaching mechanisms.
- Early analysis of the data suggests
  - Following leaching in the OPC we can see clear changes in Ce speciation & oxidation state following leaching.
  - Eu and Ce are co-located, and generally with Fe.

15% OPC Leached





This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Conclusions - Durability

- **Matrix degradation was not significant after 1-1.5 years**
- **Good dimensional stability, no unusual cracking**
- **Good mechanical stability**
  - **Slight decrease in mechanical strength observed with increasing leaching time >> WAC**
- **Slight increase of porosity**

Observed processes (CEM, GPO**)**

- **Enrichment in Ca** of wasteforms **in SCW**
- **Decalcification of OPC-matrices in DW**
- Release of Si and Al: more relevant at high pH in SCW – significant observation for cement-based EBS

No significant microstructural changes

  - Precipitation of crystalline phases: **portlandite, ettringite** (CEM I and CEM III) and **calcite**

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Conclusions - Durability

Alkali-activated materials

- Carbonation includes microstructural changes of the CASH matrix, but it's effect are inhibited by the presence of $Ca(OH)_2$
- Initial leaching rate of Na is high due to high pore water concentrations
- Leaching of the aluminosilicate framework is significant possibly due to the fast hydration of BFS

Cementitious materials

- Carbonation combined effect of calcite crystallisation and gaylussite dissolution
- Expansion under humid conditions is limited after 1 year
- Similar leaching mechanism to AAMs

Molten glass vs. compressed pellet

- Perceptible alteration despite low temperature (SCW)
- Similar results for both samples (SCW)
- saturation appears to occur rapidly, probably due to a retroactive effect (UPW)
- Pellet appears slightly less altered than molten glass (UPW)

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Conclusions – Immobilization

- Gained leachability indices **above the treshold limit > 6** for all waste matrices studied
- Radionuclides (Cs, Ce, Sr) leaching rates differ depending of the leaching solution → lower rates for geopolymers in 0,1 M NaOH than in SCW water, **Higher alkalinity → Increased leaching rates**
- For **CEM waste** forms **DI water** is the most aggressive leachant
- The initial rate of tracer analogue release is most significant during the first 30 days, suggesting multiple mechanisms for radionuclide retention, linked to matrix dissolution
- The rate of radionuclide leaching does not increase linearly with ash loading (1%, 15%, 50%), suggesting that the added ash % may be optimized

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Thank you!

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

PREDIS

WP6 - Economic and Environmental Impact

CALLUM ELDRIDGE (GSL)

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Value Assessment

- Definition of 'Value' (Theramin D2.5): *"the realisable benefit in safety, monetary and environmental outcomes that will arise from implementing this technology at a specified time".*
- See *Project Impacts* session on Thursday for more detail on methodology

Attribute	Data Category	Assessment considerations					
		Construction	Pre-treatment	Treatment operations	Post-treatment	Storage and Disposal	Decommissioning
Operational and Transport Safety	Facility construction and decommissioning						
	Waste pre-treatment requirements (conventional and radiological safety implications)						
	Waste post-treatment requirements (conventional and radiological safety implications)						
	Waste operational safety issues (e.g., ease of providing shielding during operation)						
	Transport safety issues						
Environmental Impact	Material requirements						
	Energy requirements						
	Secondary waste and gaseous/liquid discharges generated						
	Nuisance						
Impact on disposability / long-term safety	Ability to meet waste acceptance criteria						
	Disposability of secondary waste						
Implementation	Indicative lifetime feed						
	Ease of achieving required throughput for process (full-scale facility) (m^3/year)						
	Potential to treat a wide range of waste groups (flexibility) including problematic and orphan wastes						
	Impact on waste management strategy						
Timescale	Design, construction and active commissioning timescale						
	Lifetime operating timescale						
	Decommissioning timescale						
Technical Readiness	Maturity of technology						
Strategic Cost Impact	Costs of construction, operation and decommissioning						
	Impact on disposal costs (total packaged waste volume, disposal route, and required storage and disposal capacity)						

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

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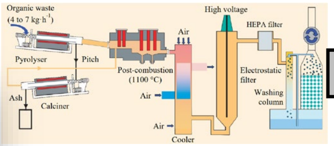
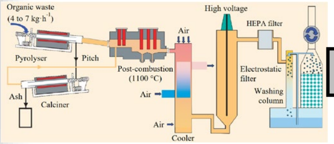
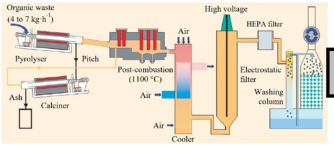
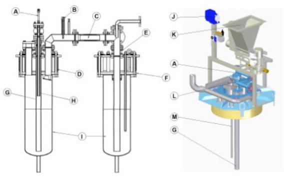
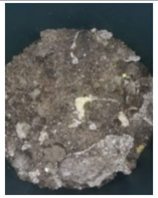
Value Assessment Scenarios

Scenario ID	Raw waste	Treatment baseline or novel technology	Treatment output
6.1	Mixed organics and IERs	Thermal treatment with **IRIS process** and then **HIP of ashes** (USFD, NNL)	Cement encapsulated HIP cans in a 200 L drum.
6.2	Mixed organics and IERs	Thermal treatment with **IRIS process** and then **compaction of ashes** (CEA)	Cement encapsulated compacted pellets in a 200 L drum.
6.3	Mixed organics and IERs	Thermal treatment with **IRIS process** and then **encapsulation in Tuff-based geopolymer** (POLIMI)	Waste conditioned in a geopolymer matrix in a 200 L drum.
6.4	IERs	Thermal treatment by **Molten Salt Oxidation** then **encapsulation in geopolymer** (CVRez)	Waste conditioned in a geopolymer matrix in a 200 L drum.
6.0.A - Baseline	IERs	Direct cementation	Waste conditioned in a 200 L drum
6.0.B - Baseline	Mixed organics	Compaction and cementation	Waste conditioned in a 200 L drum

programme 2019-2020 under grant agreement No 945098.

PREDIS

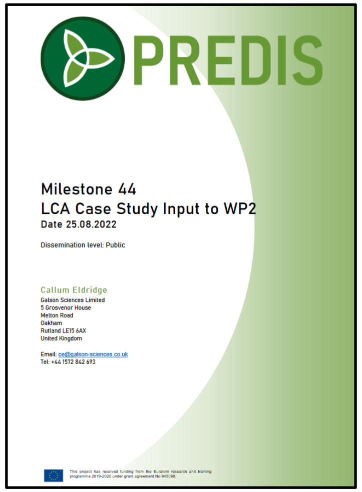
Value Assessment Scenarios

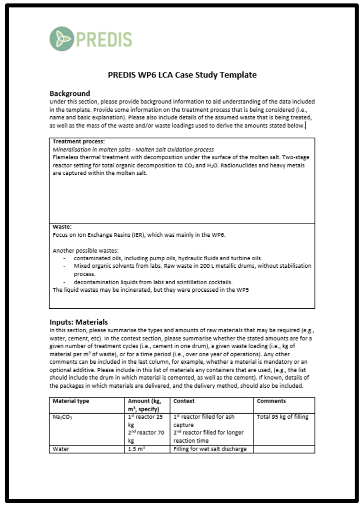
Scenario ID	Raw waste	Treatment baseline or novel technology	Treatment output
6.1	  Anionic IER Organic solids	   IRIS & HIP	200 L Drum 
6.2		   IRIS & Compaction	
6.3		   IRIS & Geopolymer	
6.4	Anionic IER 	   MSO & Geopolymer	

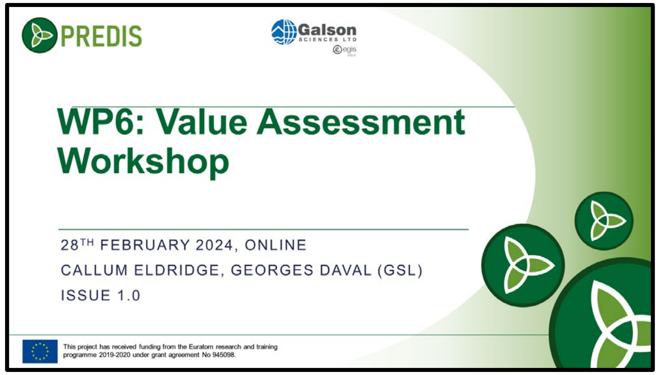
Value Assessment inputs

Inputs to VA process:

- Input from PREDIS Partners involved in technical development of the technology.
- Data from the LCA/LCC assessment within WP2 (D2.9).
- Literature.
- Value assessment workshop with all WP6 partners.

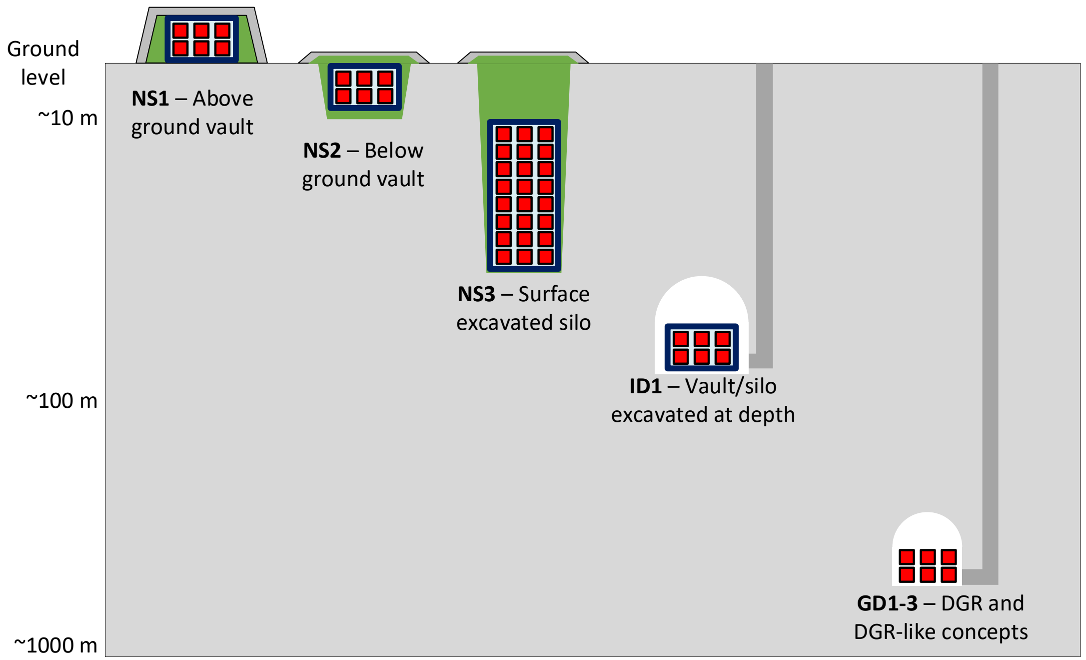






This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Disposability considerations



Scenario 6.1.B – Supercompaction of mixed organics	
Assessment area	**Identified disposability risks**
Physical form	**Concept dependent disposability risk (NS1-2):** Potential negative impact on disposability to shallow near surface as compacted drum or contents may be considered a discrete object within cemented waste package. **Otherwise, no risk:** Cemented drum will be solid.
Mechanical stability	**No risk identified:** Drum and cement have adequate mechanical stability.
Homogeneity	**General disposability risk:** Compacted wasteform is not homogeneous, although this may not be required for this type of wasteform is some disposal concepts.
Dose-rate	**No risk identified:** Annular grouted wasteform provides some shielding. Activity concentration will be comparable to initial waste.

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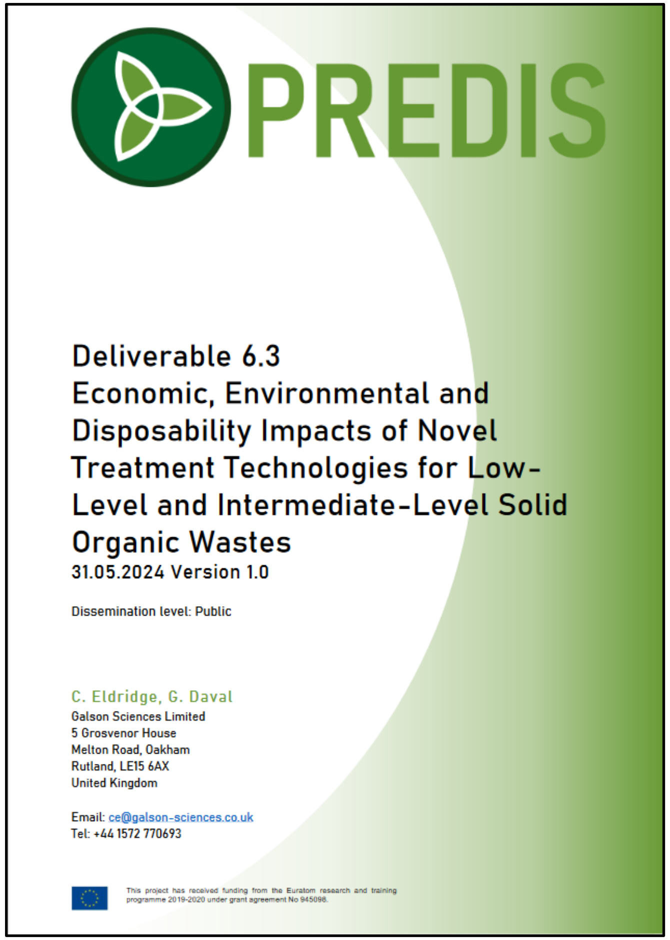
D6.3 – Economic and Environmental Impact of Novel Treatments

Main findings:

- The novel treatment technologies considered typically provide benefits in terms of material environmental impact, package disposability and the disposal and storage costs for the product drums.
- The disbenefits are typically related to the safety and cost impacts of the additional facilities, the uncertainties associated with a novel technology and the environment impact of the energy consumption.
- Further development of the new technologies to the point where operating TRL 9 versions of these treatment facilities are available would remove or lessen many of the negatives or uncertainties, in which case they could in future become more sustainable, less costly alternatives.


PREDIS

Deliverable 6.3
Economic, Environmental and Disposability Impacts of Novel Treatment Technologies for Low-Level and Intermediate-Level Solid Organic Wastes
31.05.2024 Version 1.0

Dissemination level: Public

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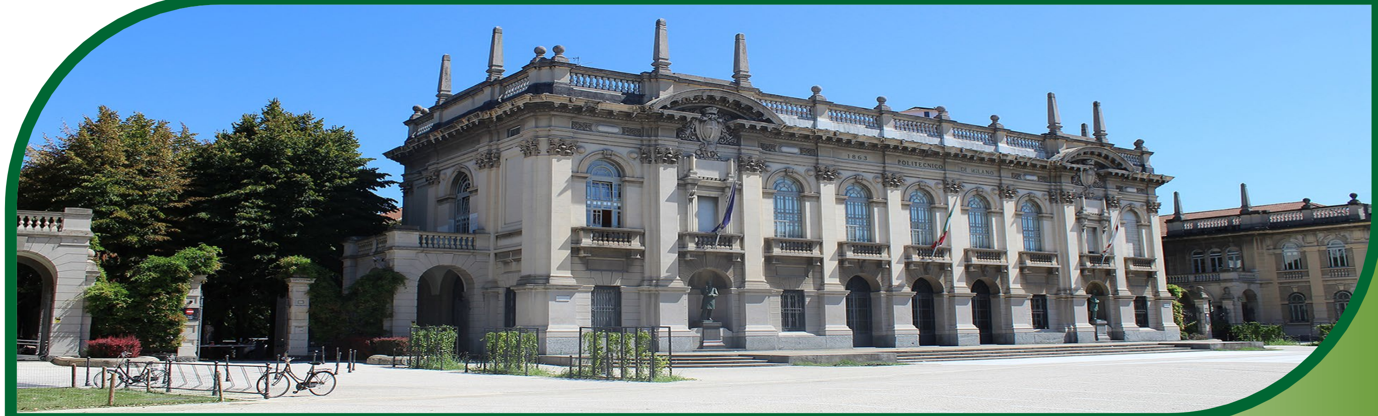
This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Thank you

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 945098.

PREDIS



A drum full of beads

FRANCESCO GALLUCCIO

POLITECNICO DI MILANO

WORK PACKAGE 6

5 JUNE 2024, AVIGNON (FRANCE)

POLITECNICO MILANO 1863

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

About Myself

- Master of Science in *Nuclear Engineering* at Politecnico di Milano
- PhD in *Energy and Nuclear Science and Technology* at Politecnico di Milano in cooperation with European Commission's Joint Research Centre – Ispra site
- Temporary Research Fellowship in *Nuclear Decommissioning and Waste Management* at Politecnico di Milano

EXPERTISE AREAS

- Recycling of Spent Nuclear Fuel
- Treatment and conditioning of nuclear waste
- Radiological characterization

HOBBIES

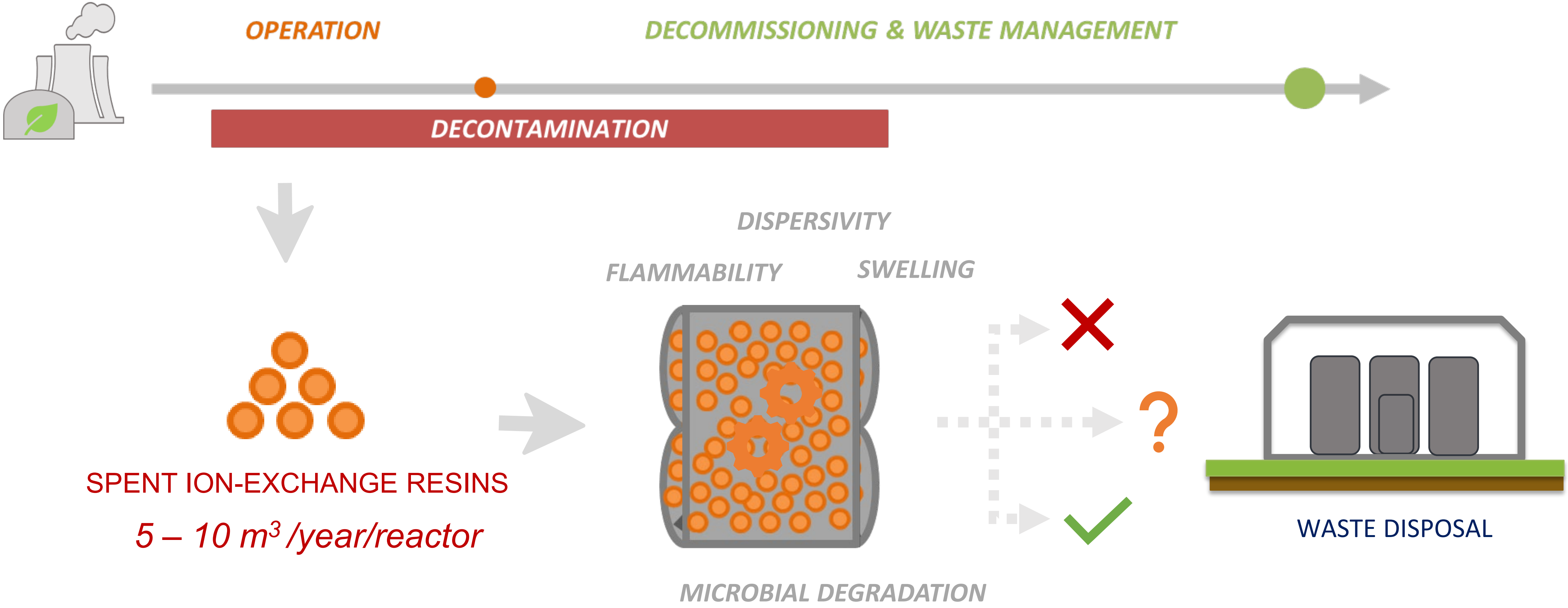
- Science and Arts
- Nature walking
- Swimming and fishing
- Baking

Tropea, Calabria, Italy

"L'ingegno è vedere possibilità dove gli altri non ne vedono" (Enrico Mattei)

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

My contribution to PREDIS (Tasks 6.3, 6.4 and 6.6)



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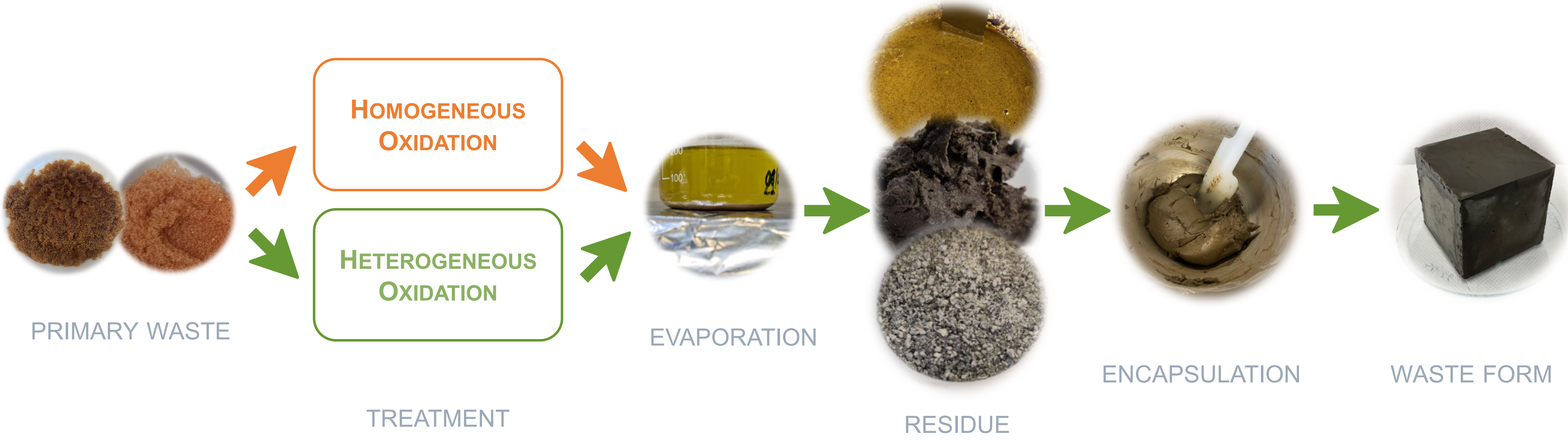
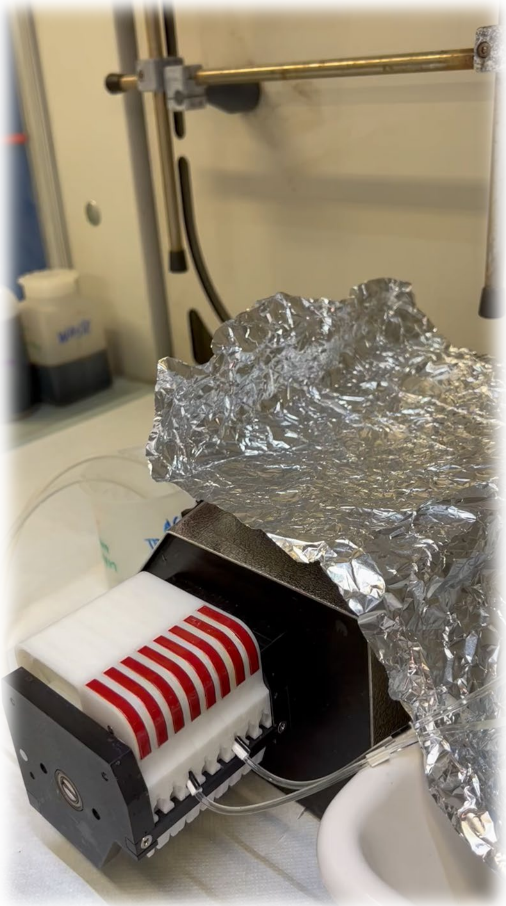
Methods

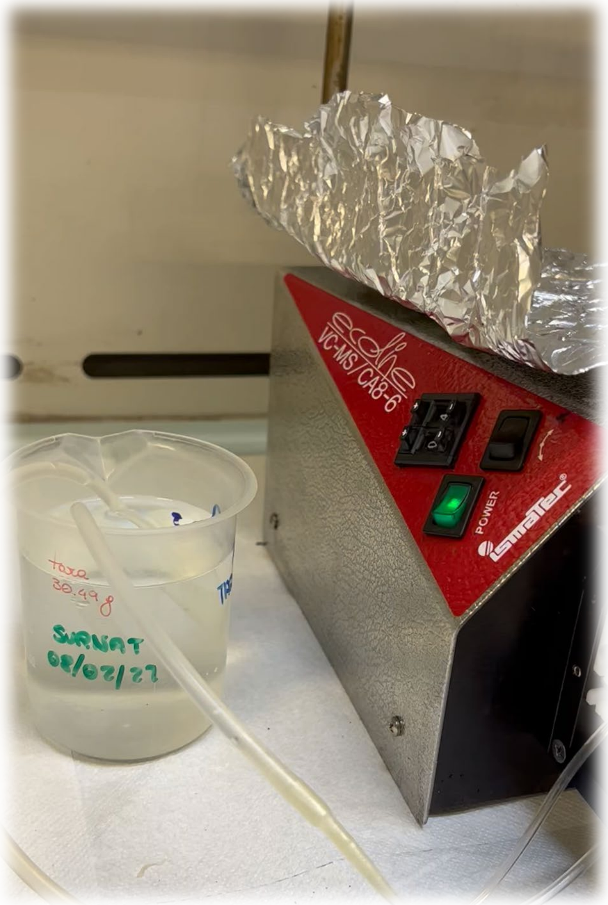
- Mineralisation of spent ion-exchange resins by *Fenton wet oxidation*
- Encapsulation of the residues in *geopolymer* matrices



This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Hands on the wheel…

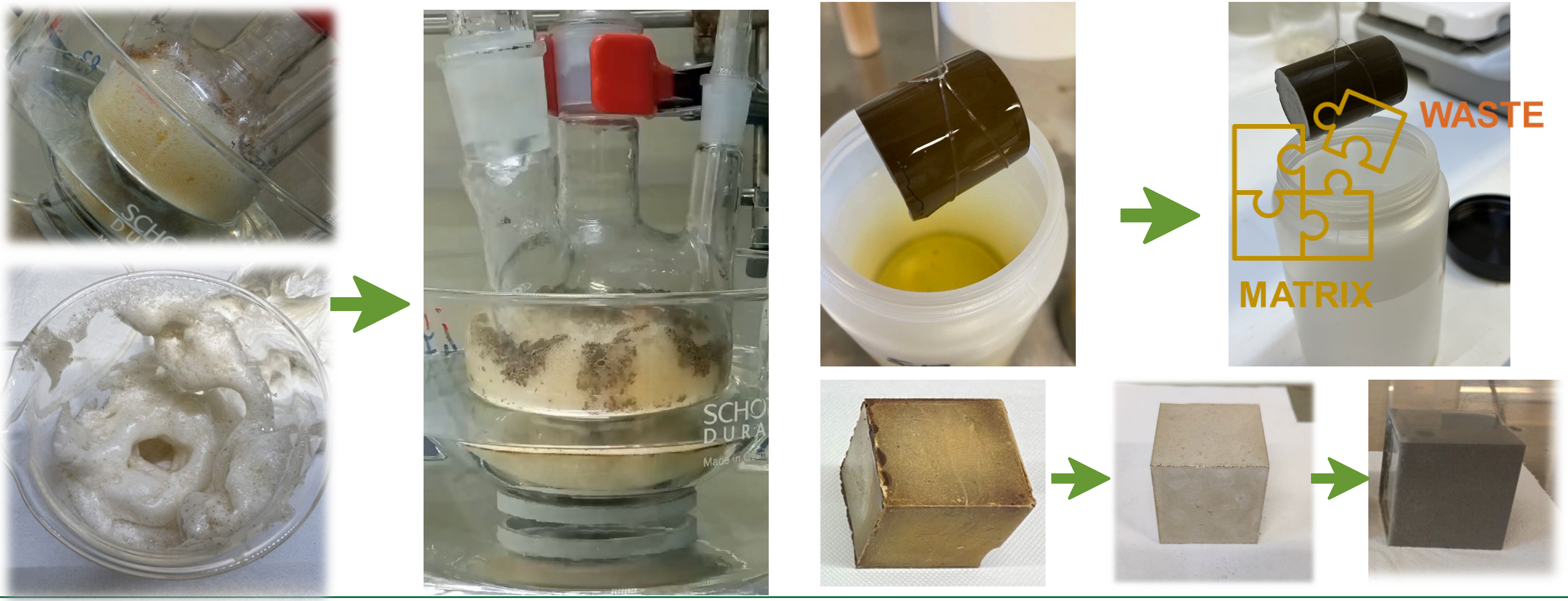




- CATALYST
- OXIDANT
- TEMPERATURE
- TIME
- ENCAPSULATION
- CHARACTERIZATION
- QUALIFICATION

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Challenges



This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

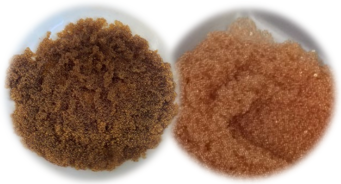
Results

INTEGRATED WASTE MANAGEMENT

A NEW LIFE FOR SOME INDUSTRIAL BY-PRODUCTS

Catalyst

Precursor



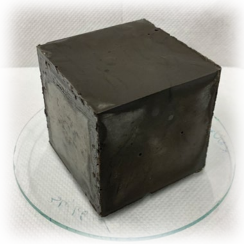
PRIMARY ORGANIC WASTE

FENTON-LIKE WET OXIDATION

TREATMENT

GEOPOLYMER MATRIX

ENCAPSULATION



WASTE FORM

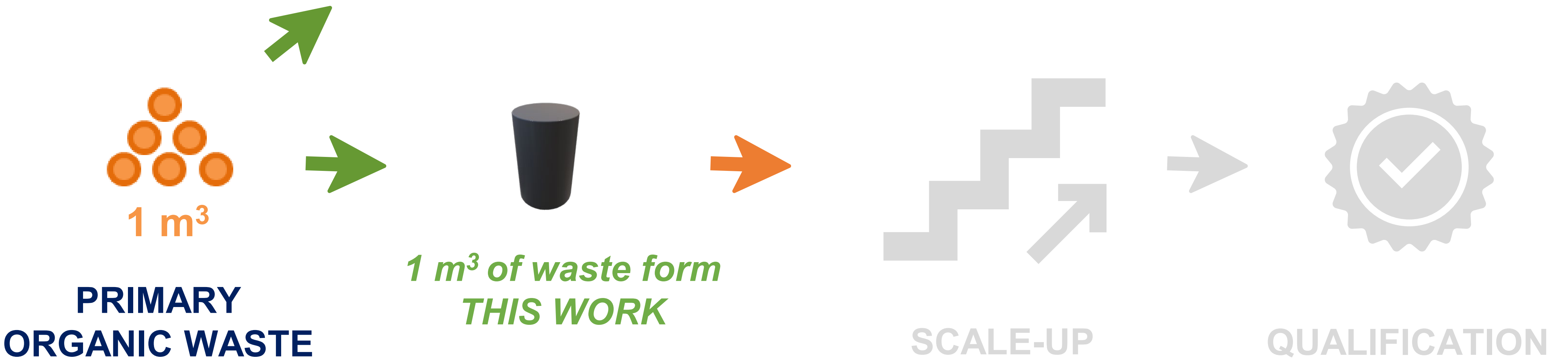
WAC

PREDIS

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

Conclusions

- Decomposition of organic matter
- High retention of nuclides in the residues
- Great waste volume reduction
- Easy scalability of this sustainable technology



This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

One way or another!

F. Galluccio, E. Mossini, A. Santi, M. Mariani
Integrated process to manage solid organic matrices
(Italian Patent Application, N° 102023000002082, 8th February 2023)

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

PREDIS and Scientific Dissemination

IAEA
International Atomic Energy Agency

Co-chair of session

Poster presentation at IAEA International Conference on Radioactive Waste Management: Solutions for a Sustainable Future (***1 – 5 November 2021, Vienna, Austria***).

WM SYMPOSIA 2024 50 1974 2024 YEARS

Application for a PREDIS mobility

Oral presentation at Waste Management Symposia 2024 Conference Planning for the Future: Innovation, Transformation, Sustainability.
(***9 – 14 March 2024, Phoenix, Arizona, USA***).

Oral presentation at 4th International Symposium on Cement-Based Materials for Nuclear Wastes – NUWCEM 2022 (***4 – 6 May 2022, Avignon, France***).

FISA 2022
EURADWASTE'22

MSc/PhD competition award

Oral and poster presentation at 10th edition of Euratom research and training conferences on fission safety of reactor systems [FISA 2022] and radioactive waste management [EURADWASTE '22] (***May 30 – June 3 2022, Lyon, France***).

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

No I in Team!

ANDREA SANTI

PhD Candidate in Energy and Nuclear Science and Technology

Cycle XXXVIII

EDOARDO RIZZI

Master’s Thesis in Nuclear Engineering

AY 2022/2023

MARCO MONTI

Master’s Thesis in Nuclear Engineering

AY 2023/2024

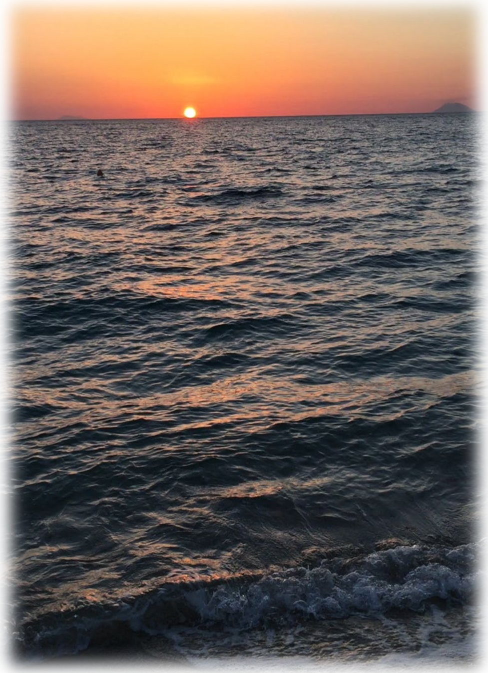
This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

My next step


ADVANCED RADIOCHEMICAL METHODS AND GREEN PROCESSES TO ADDRESS CHALLENGING WASTE IN NUCLEAR DECOMMISSIONING

Cum Laude

Politecnico di Milano, 16/10/2023



*Research and Valorization of **Fenton-like wet oxidation** and **geopolymer technology** for the management of challenging organic waste!*

PREDIS

This project has received funding from the Euratom research and training programme 2019-2020 under grant agreement No 945098.

PREDIS

POLITECNICO MILANO 1863

thank you

francesco.galluccio@polimi.it

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