

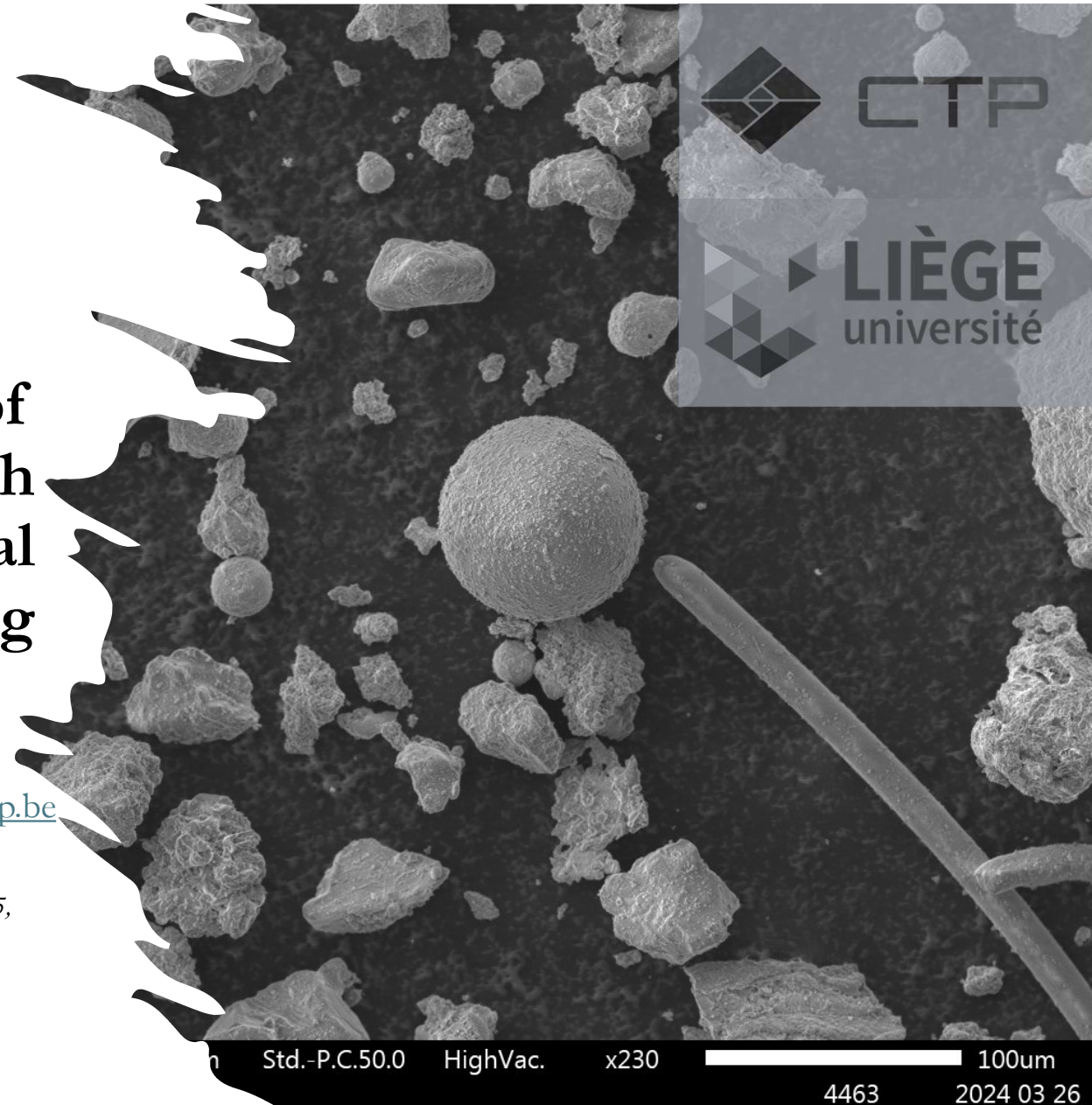


Improving the Properties of Woody Biomass Fly Ash through Mechanochemical Activation and Water Washing Treatments

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Opportunities

- Active search for secondary materials in the cement industry and the construction sector : economic, logistical, and environmental incentives.
- Disposal costs and associated environmental risks
→ BFA: readily available secondary resource

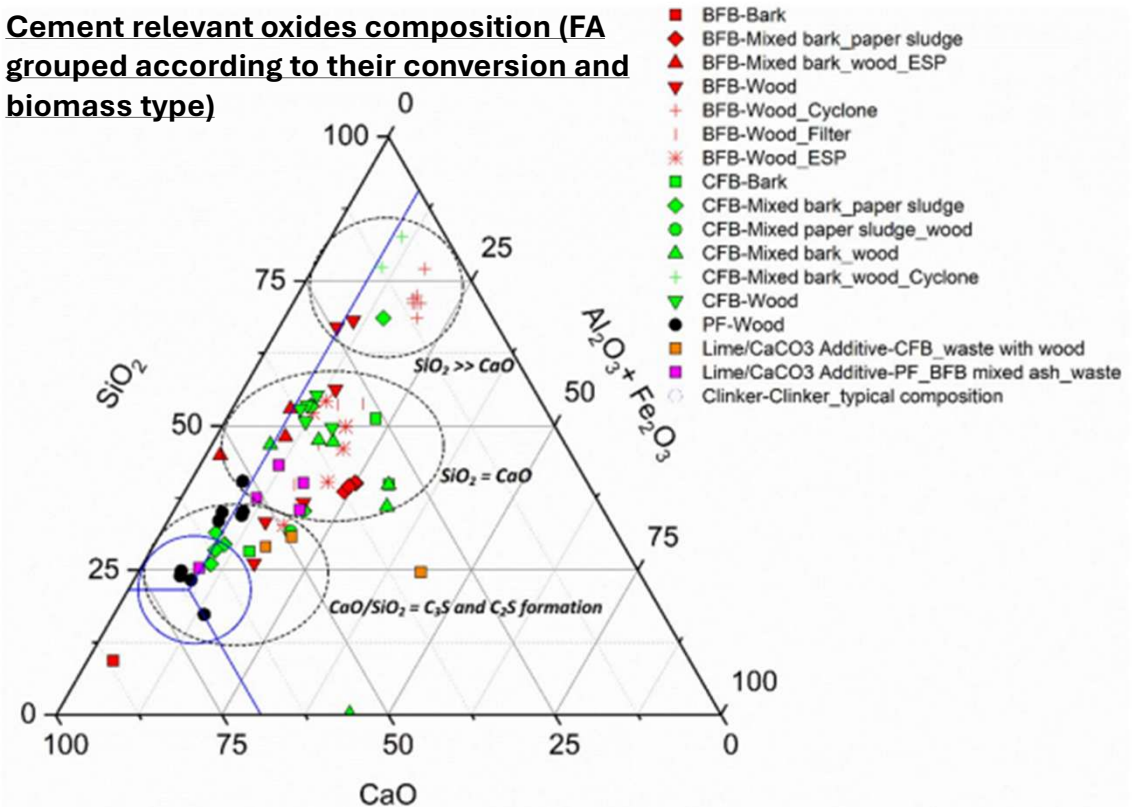
Challenges

- Variability (physical, chemical and mineralogical)
 - Solubles compounds and alkalis
 - Content of pollutant elements (heavy metals)
 - Reactivity (lime, reactive silica)

→ Various treatment approaches:

- Mecano-chemical activation;
- Water/reactive washing treatment
- Carbonation

Cement relevant oxides composition (FA grouped according to their conversion and biomass type)



Tosti, L., Van Zomeren, A., Pels, J. R., & Comans, R. N. J. (2021). Evaluating Biomass Ash Properties as Influenced by Feedstock and Thermal Conversion Technology towards Cement Clinker Production with a Lower Carbon Footprint. *Waste and Biomass Valorization*, 12(8), 4703–4719.

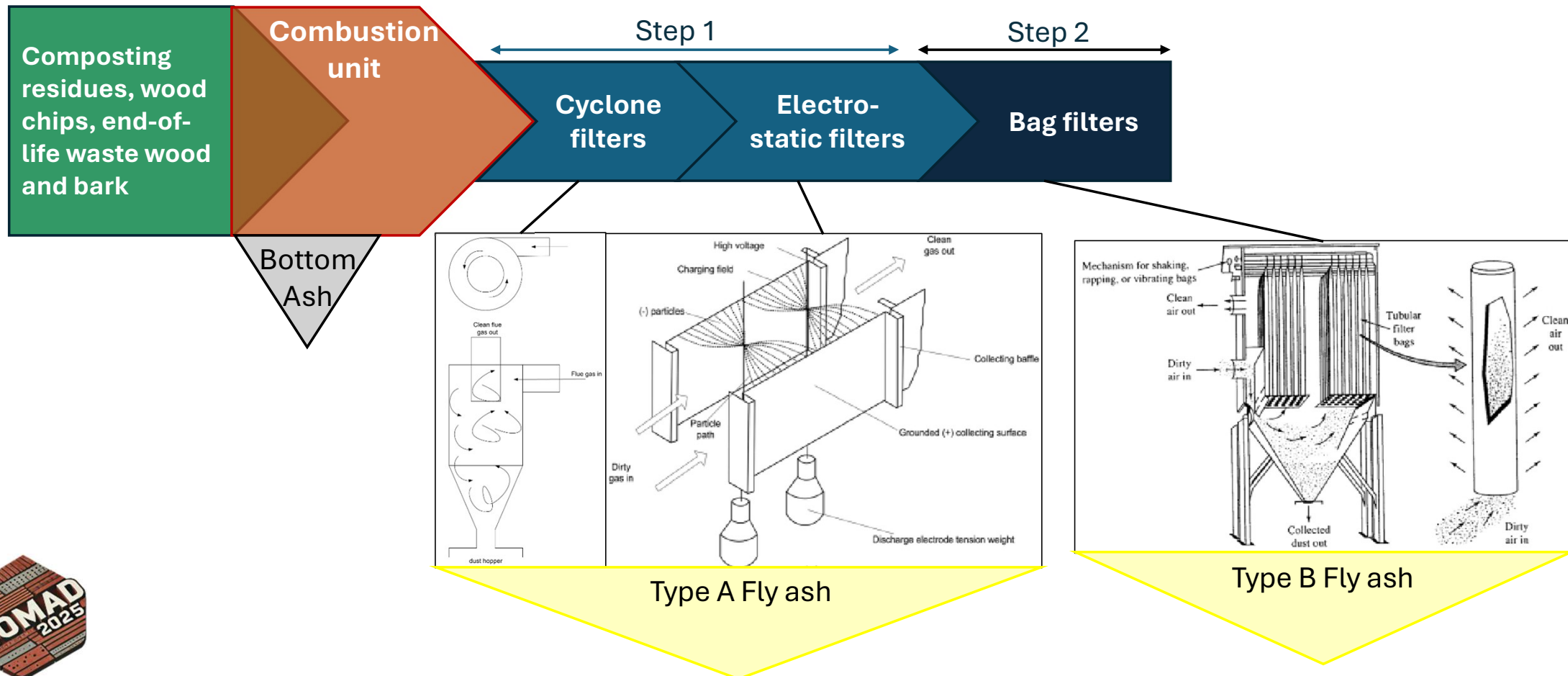
BFB : bubbling fluidized bed, CFB: circulating fluidized bed and PF : pulverized fuel combustors

BFA as a SCM*



*Supplementary Cementitious Material

Same power plant (PP1): 2 flue gas treatment steps → 2 types of fly ash: A and B

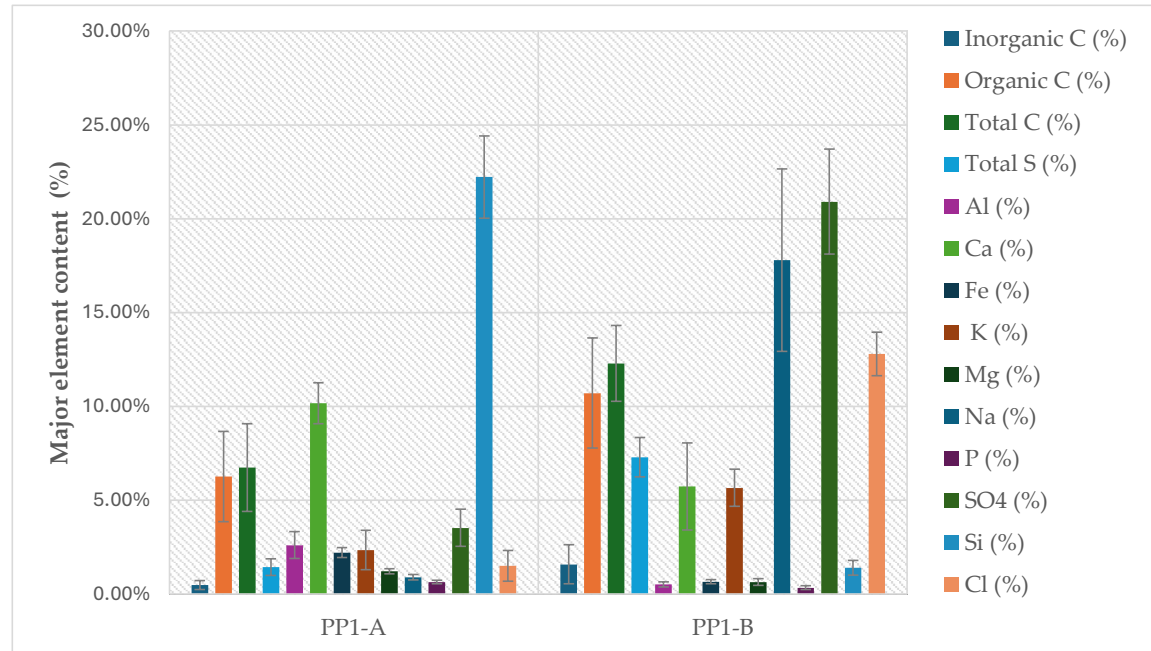


BFA as a SCM



ID	Sampling step in the flue gas treatment process	d50 (μm)	Density (kg/m³)	BET specific surface (m²/g)
PP1-A	First step (A)	51.27	2629.45	16.29
PP1-B	Second step (B)	14.69	2189	9.42

Mineralogical phases (Rietveld with internal standard S/Q)	PP1-A		PP1-B	
	1	2	1	2
Amorphous	68%	50%	69%	69%
Quartz (SiO ₂)	23%	40%	-	-
Anhydrite (CaSO ₄)	3%	3%	2%	2%
Pseudowollastonite (CaSiO ₃)	Traces	3%	2%	2%
Calcite (CaCO ₃)	Traces	Traces	Traces	Traces
Albite (Na(AlSi ₃ O ₈))	4%	2%	Traces	Traces
Chaux (CaO)	2%	2%	-	-
Halite (NaCl)	Traces	-	12%	15%
Natrite (Na ₂ CO ₃)	-	-	6%	Traces
Thenardite (Na ₂ SO ₄)	-	-	4%	6%
Aphthitalite (NaK ₃ (SO ₄) ₂)	-	-	5%	6%
Sylvite (KCl)	Traces	-	-	-
Akermanite (Ca ₂ Mg(Si ₂ O ₇))	Traces	Traces	-	-
Gehlenite (Ca ₂ AlSiO ₇)	-	Traces	-	-
Hematite (Fe ₂ O ₃)	Traces	Traces	-	-
Rutile (TiO ₂)	Traces	Traces	Traces	Traces



“Type A” fly ash:

- 25% silicium (quartz), 10% Ca
- 5 to 10 % organic carbon (unburned)
- Low proportion in soluble phases (Na, Cl, K)

“Type B” fly ash:

- Low silicium content
- High proportion in soluble phases containing Na, Cl, SO₄
- 8 to 13% organic carbon



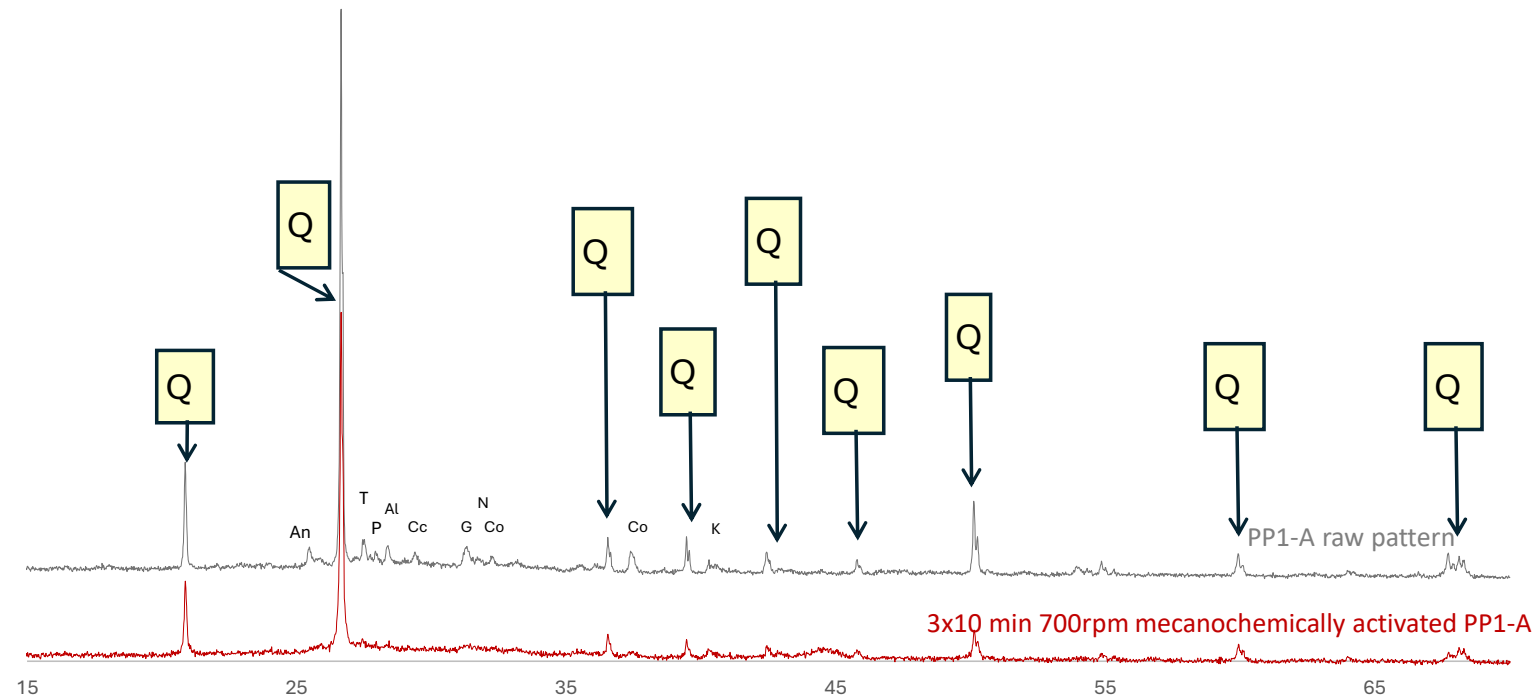
« Type A » Fly ash: mecanochemical activation



Principle: High-energy grinding

→ Structural changes: deformation and partial amorphisation of crystal lattices → XRD pattern: progressive decreasing (or disappearing) of crystalline phase (=increase of amorphous content): Quartz → reactive SiO₂ → CSH

Parameters	Medium scale tests
Fixed parameters	
Max batch	2 kg
Ball-to-material ratio	10
Variable parameters	
Grinding time	1x10 min, 2x10 min, 3x10 min, 2x10 + 1x20 min
Power	350, 425, 550, 625 et 700 rpm
Efficiency check	Visual XRD pattern amorphisation, reactive silica analyses, activity index

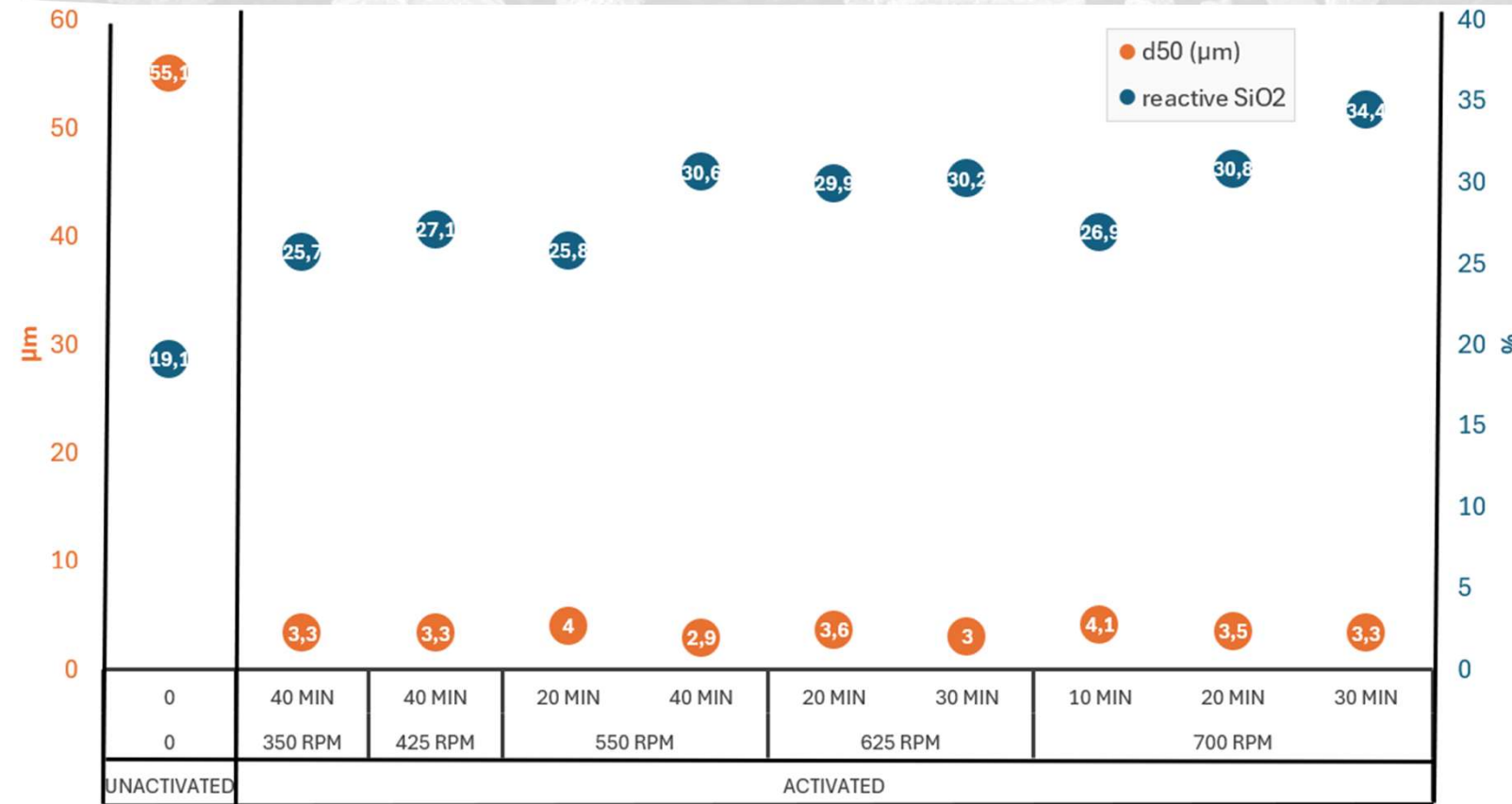


2 theta (Coupled Two Theta/Theta) WL=1,54060

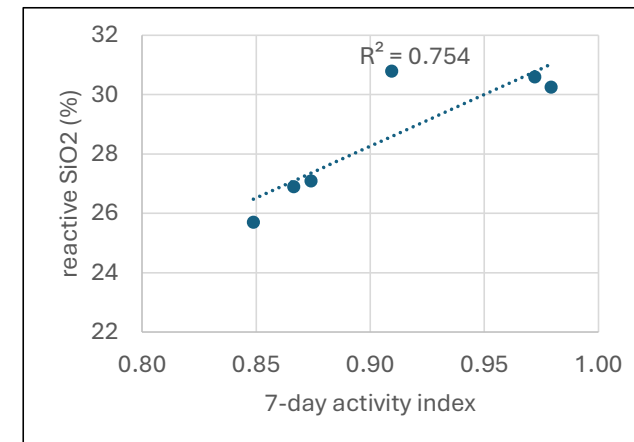
Q=Quartz, An=Anhydrite, Al=Albite, P=Pseudowollastonite, T=Rutile, Co=Calcium oxide, K=sylvite, G=Gehlenite, N=halite.



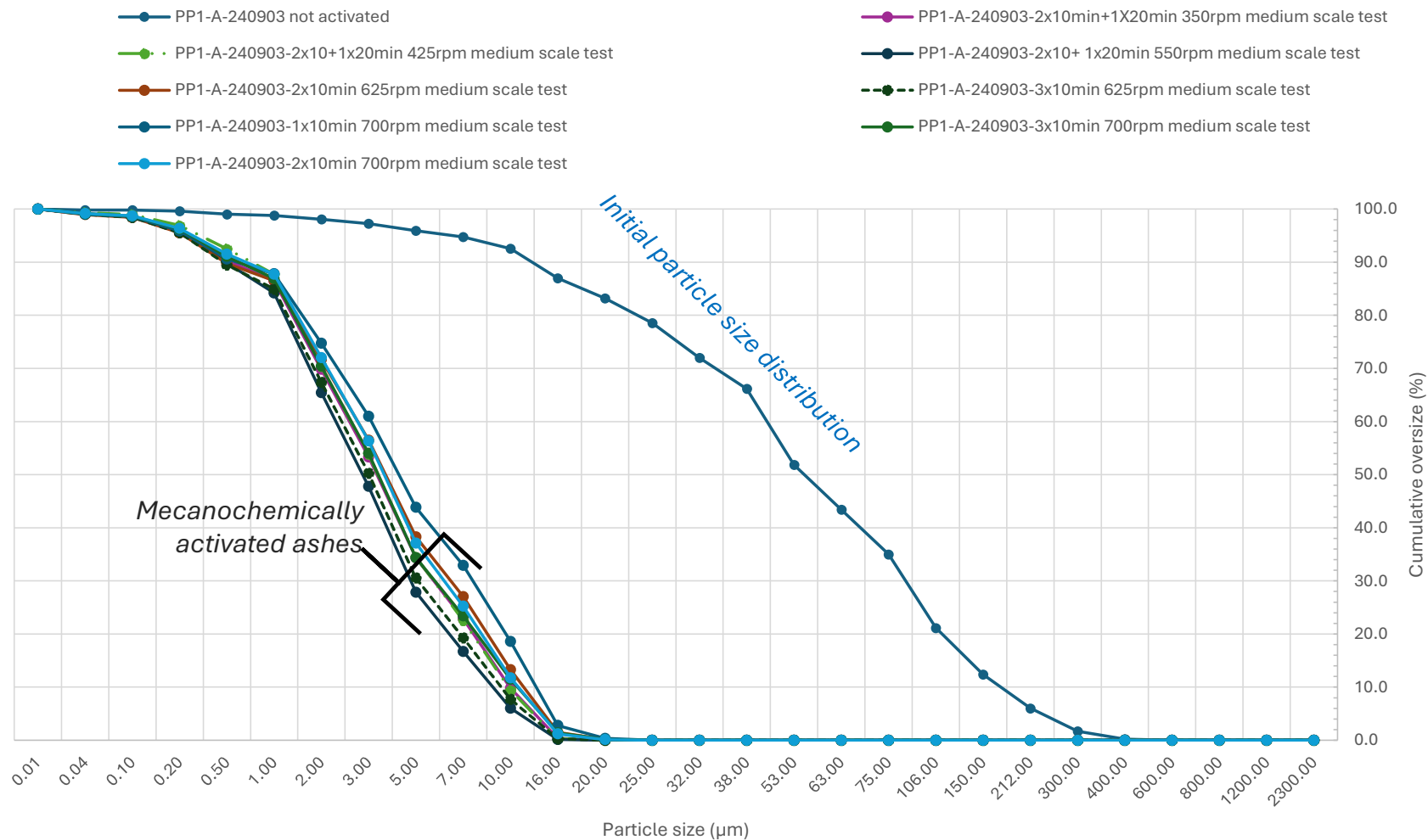
« Type A » Fly ash: mecanochemical activation



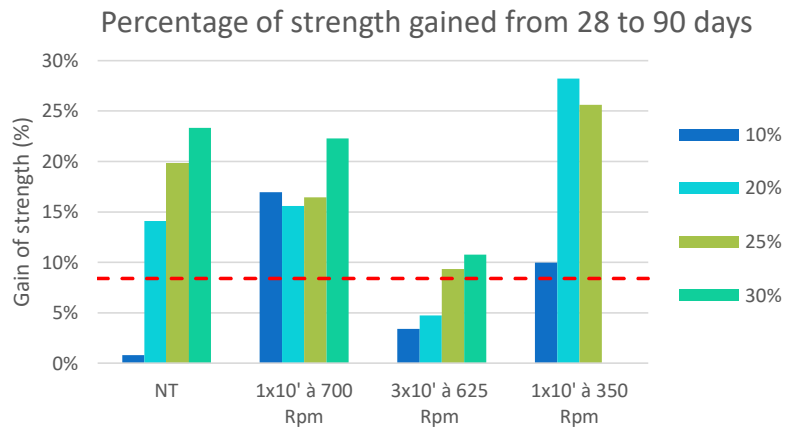
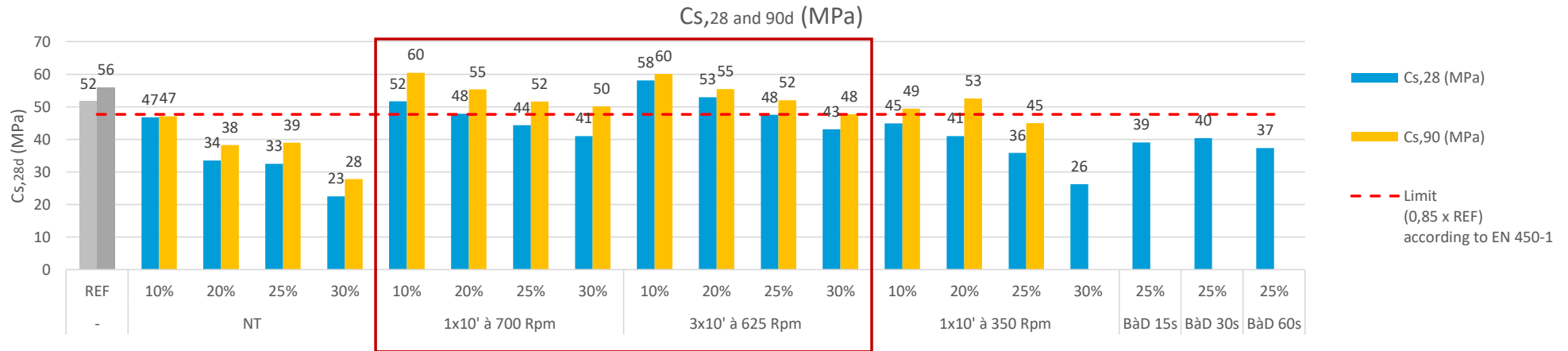
Increasing power and time :
 No more major changes in particle size while reactive SiO₂ continue to rise
 → in line with the increase in 7 day-compressive strength at 25% cement replacement (activity index up to 0,98 depending on the activation)



« Type A » Fly ash: mecanochemical activation



« Type A » Fly ash: mecanochemical activation



- For 2 mecanochemically activated ash, the activity index remain > 90% for cement replacement levels of up to 25% (activity index of 1.1 at 10% of cement replacement)
- Mechanochemical activation appears to accelerate strength development in mortars; for example, when comparing 3 × 10 min at 625 rpm to 1 × 10 min at 700 rpm, strength develops more rapidly, although not to a greater extent.



« Type A » Fly ash : Additional Treatment Strategies



Grinding

Improves packing and water demand control
May induce chemical activation by breaking Si-O-Si bonds
Complementary to MCA; less energy intensive, limited chemical effect

Sieving

Physical removal of coarse and inert particles
Increases surface area and fineness without milling
Simple, low-cost pre-treatment improving reactivity and stability

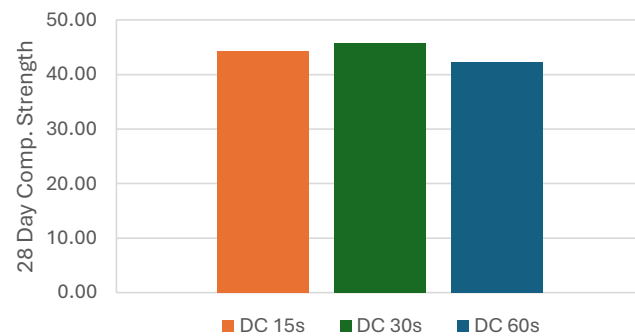
Carbonation

CO₂ uptake and ash stabilization
Reduces leachability and pH of ashes
Slightly refines porosity and ash stabilization

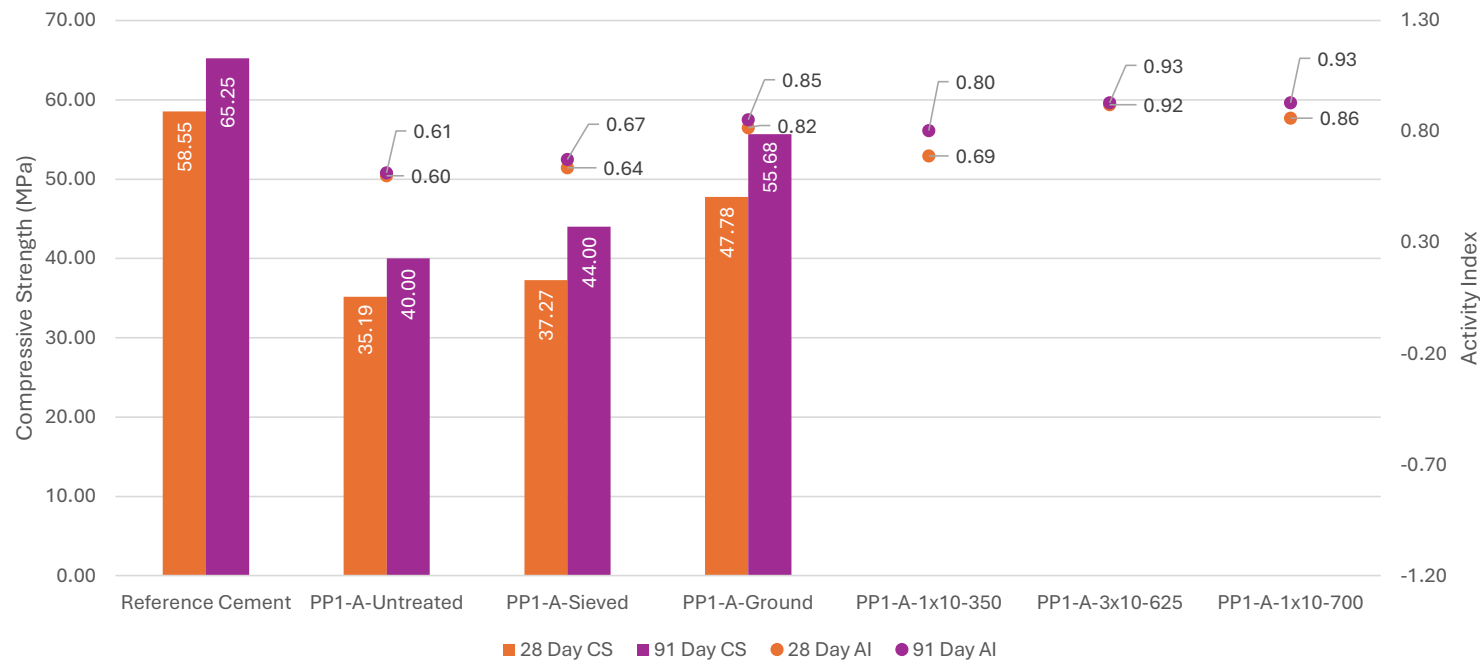


« Type A » Fly ash: Grinding & Sieving

- Grinding:
 - Disc crusher used as grinding operator, treatment durations chosen between 15s, 30s and 60s to avoid overheating.
- Sieving:
 - Electromagnetic sieve shaker used as sieving operator, 250 μm sieve chosen due to particle size of unburned carbon particles



« Type A » Fly ash: Grinding & Sieving



- Sieving improves strength slightly by removing coarse fractions, while grinding provided better packing and filler effects, giving the best performance among non-activated ashes.

« Type A » Fly ash: Accelerated Carbonation

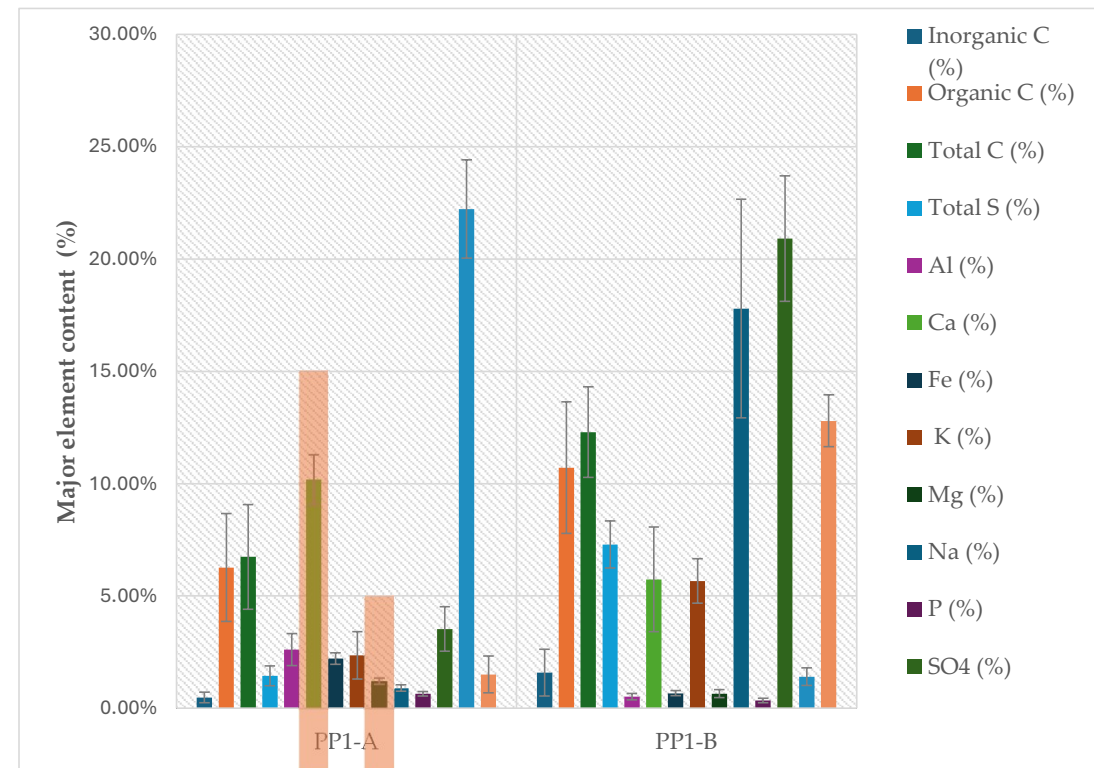
PP1-A ashes have limited capacity to capture CO₂ due to low Ca and Mg content.

Goals:

- Microstructure and performance
- Chemical stabilization
- Compatibility and usability

Co-benefits:

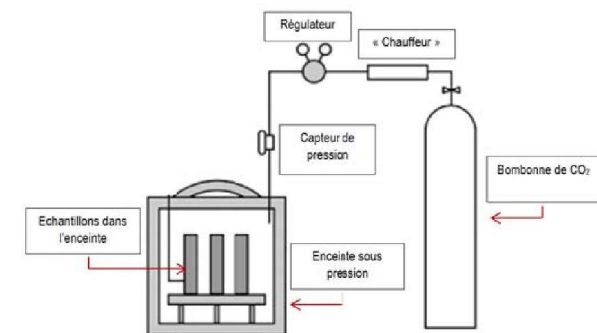
- Limited yet quantifiable environmental benefit due to permanent mineralization of CO₂



Ca and Mg contents

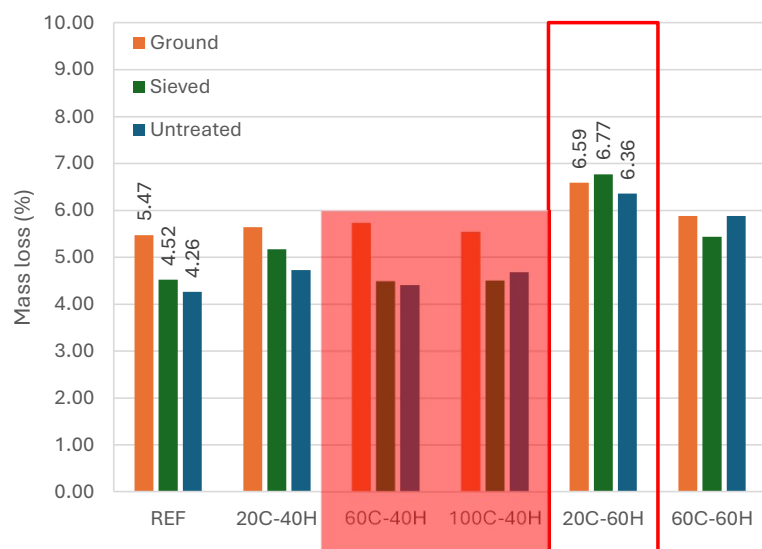
« Type A » Fly ash: Accelerated Carbonation

- CO₂ incubator:
 - CO₂ content: 20%, 60%, 100%
 - Relative Humidity: 40%, 60%
 - Duration: 120 h
 - Temperature: 25 °C
 - Fixed CO₂, static conditions
- 20% CO₂ treatments conducted in Binder incubator
- 60 and 100% CO₂ treatments conducted in modified column incubator



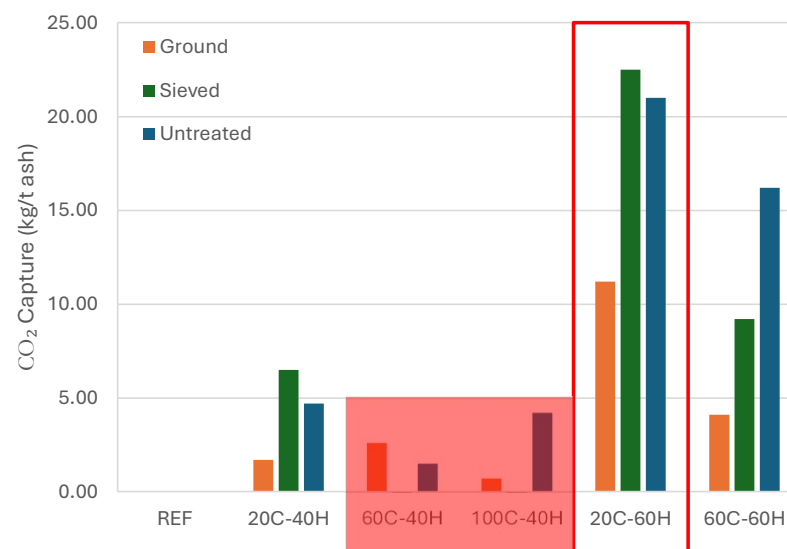
« Type A » Fly ash: Accelerated Carbonation

Mass loss in the decarbonation range (CaCO_3 decomposition)



The increase of about 2 wt% compared to the reference confirms CO_2 uptake after carbonation, most pronounced under the most favourable condition (20% CO_2 - 60% RH). In contrast, high- CO_2 /low-humidity conditions were the least favourable for carbonation.

Calculated CO_2 uptake derived from TGA data

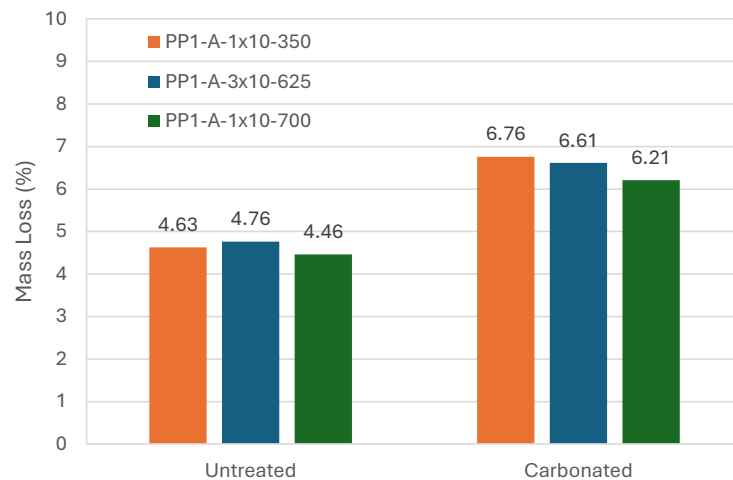


C: CO_2 content (%),
H: Relative humidity

The highest capture (~ 10 - $20 \text{ kg CO}_2/\text{t ash}$) occurred under 20 % CO_2 - 60% RH (most favourable), while 60C - 40H and 100C - 40H showed minimal uptake, indicating the least favourable conditions for mineral carbonation.

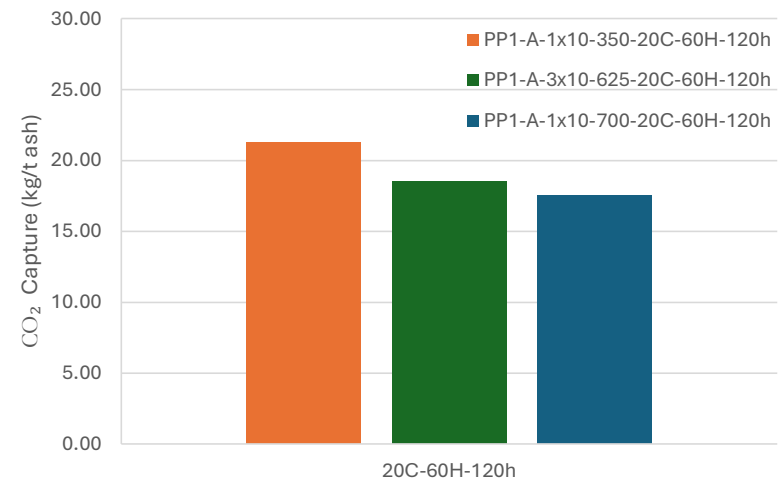
« Type A » Fly ash: Accelerated Carbonation

Mass loss in the CaCO_3 decomposition range for



All mechanochemically activated samples (PP1-A series) showed an increase of about 2 wt% after carbonation, confirming CO_2 uptake and the formation of additional carbonate phases.

CO_2 uptake calculated from TGA results under the most favourable carbonation condition

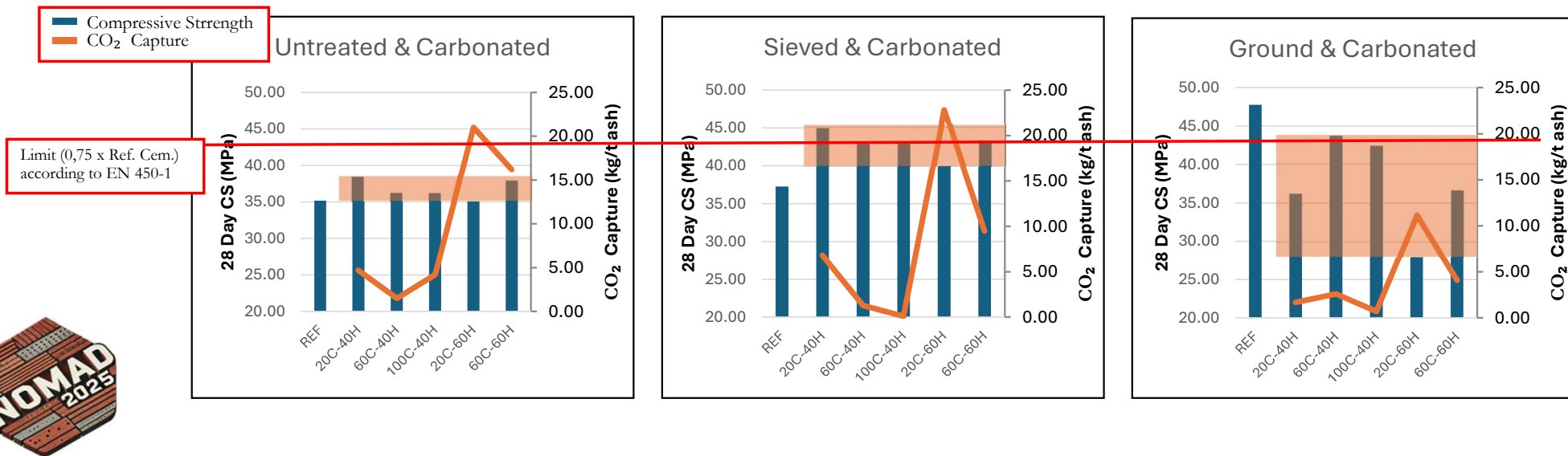


The mechanochemically activated ashes captured 17-22 kg CO_2 per ton, indicating measurable mineral carbonation despite their limited Ca content.

« Type A » Fly ash: Accelerated Carbonation

- Moderate carbonation (20C-40H, 60C-40H, 100C-40H, 60C-60H) improved strength in untreated and sieved ashes; high efficiency carbonation (20C-60H) led to higher CO₂ uptake but lower strength due to **surface passivation**.
- In ground ashes, carbonation reduced strength under all intensities; moderate conditions caused smaller losses, while intensive carbonation led to severe strength loss.
- Sieved ashes performed most consistently thanks to the removal of coarse inert particles, improving carbonation homogeneity.

Further optimization is needed to optimize CO₂ capture ratio/strength gain balance for sieved ashes and to clarify microstructural stabilization mechanisms.



« Type A » Fly ash: Accelerated Carbonation



Mechanochemically activated (MCA) PP1-A ashes achieved high compressive strength while maintaining a balanced CO₂ uptake under optimal carbonation conditions (20% CO₂ - 60% RH). The MCA treatment increased the availability of reactive Ca and silicate phases, enabling efficient carbonation without surface blockage.

This synergy resulted in ashes that are both mechanically stronger and more CO₂-responsive.

Hence, controlled carbonation of MCA-treated biomass ashes offers a dual benefit: improved SCM performance and measurable, though limited, carbon sequestration.

Further optimization is needed to optimize CO₂ capture ratio/strength gain balance for activated ashes and to clarify microstructural stabilization mechanisms.

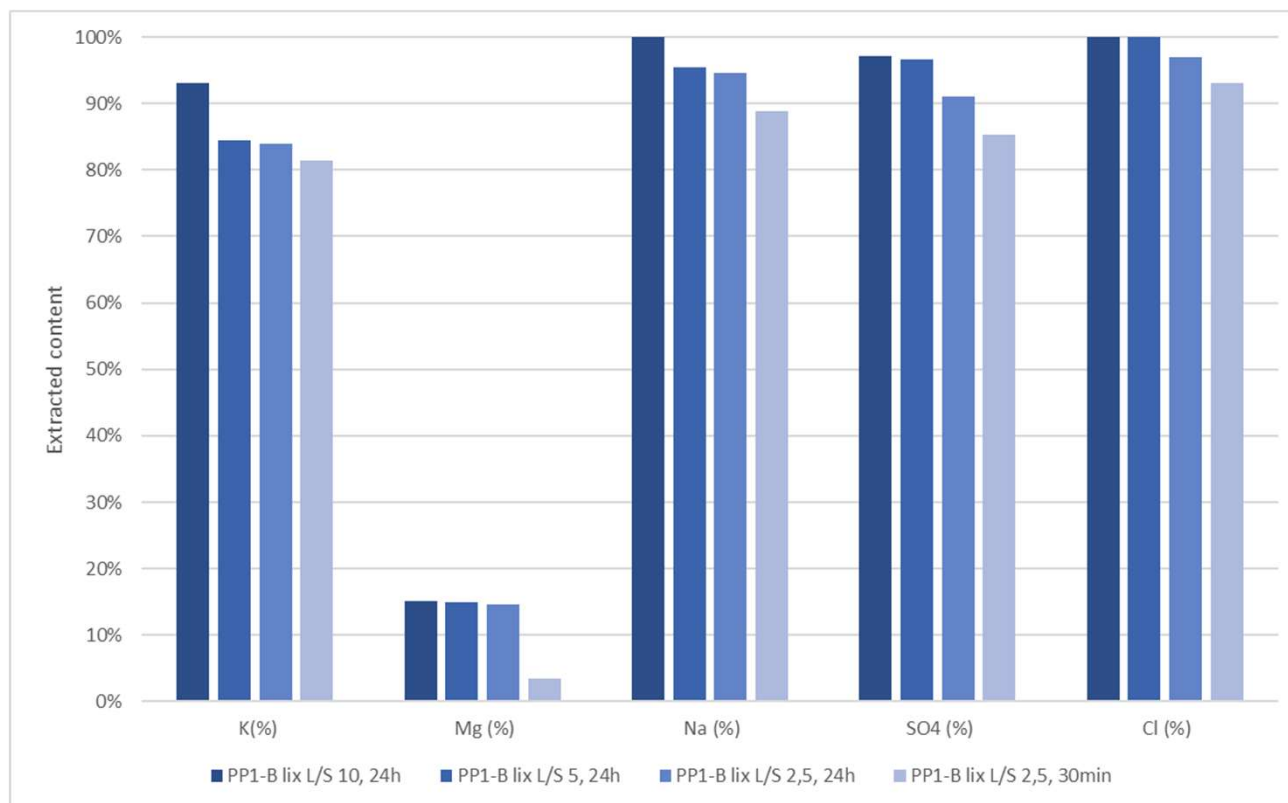
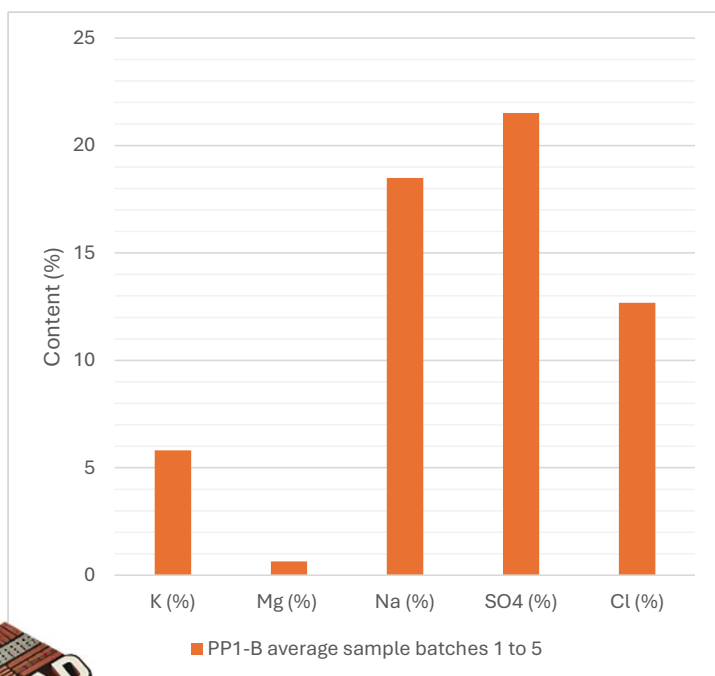
« Type B » Fly ash: Water Washing



« Type B » fly ash

Preliminary washing tests: L/S: 10→2,5; time: 24 h→ 30 min

➔ K, Na, Cl, SO₄ extraction globally above or close to 90%



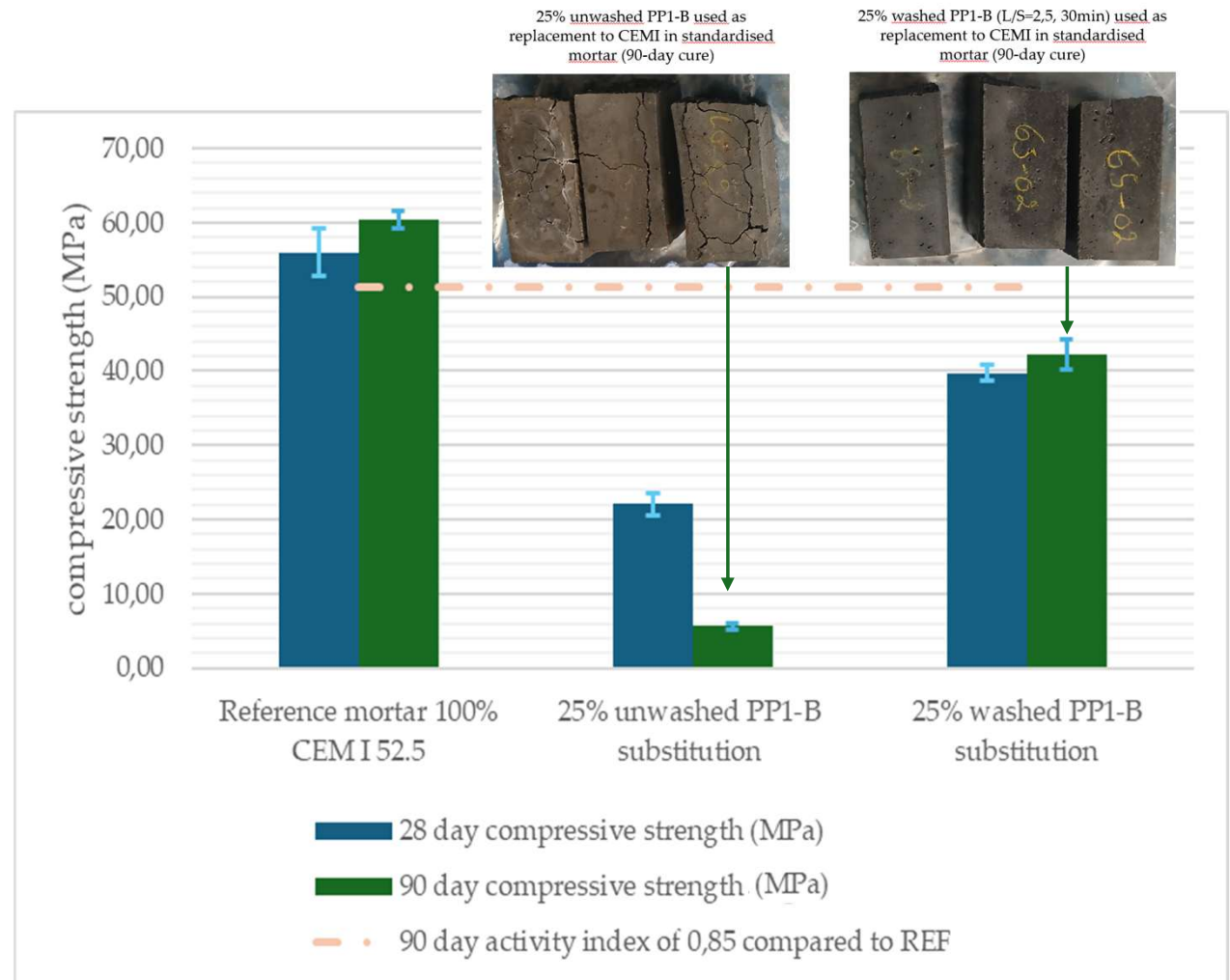
« Type B » Fly ash: Water Washing



« Type B » fly ash

Water washing successfully mitigated the deleterious effect observed at 90-day cure due to the use of 25% fly ash as cement substitution in mortars

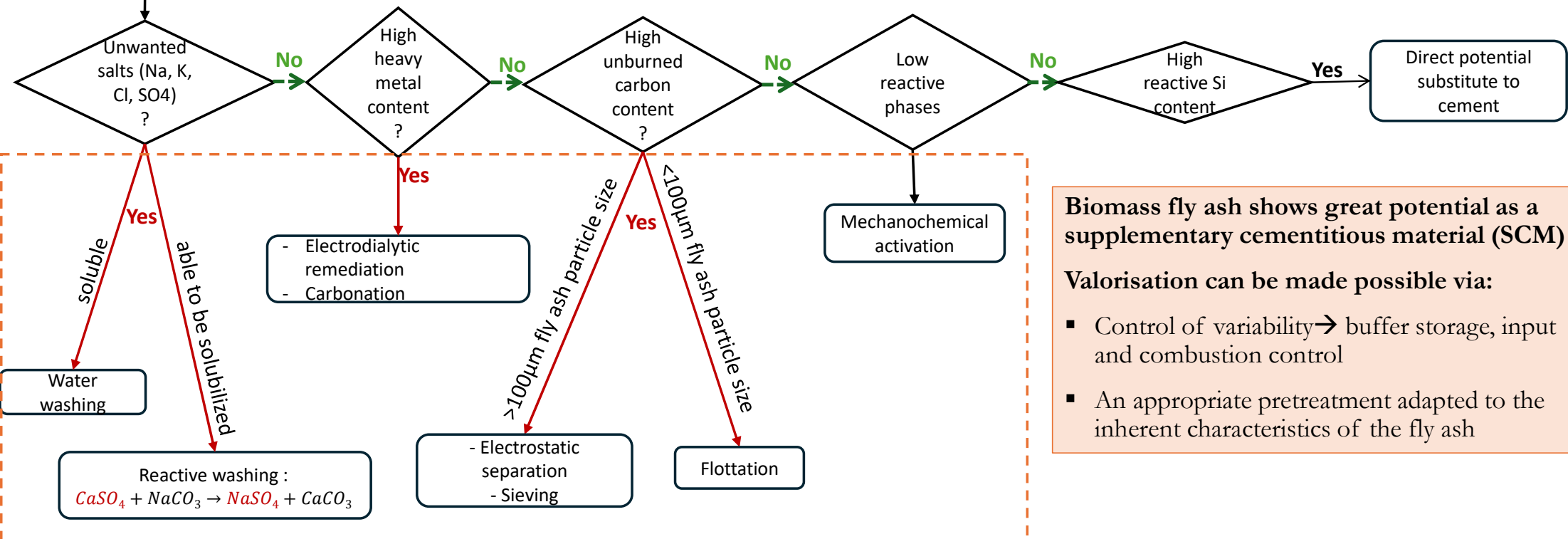
- 28-day activity index increased from 39% to 71% using water-washed fly ash as 25% cement substitution
- Activity index target of 0,75 and 0,85 at 28 day and 90 day respectively (EN-450) still not reached, but significant improvement.



Conclusion



BFA characterisation



Possibility to combine treatments

Biomass fly ash shows great potential as a supplementary cementitious material (SCM)

Valorisation can be made possible via:

- Control of variability → buffer storage, input and combustion control
- An appropriate pretreatment adapted to the inherent characteristics of the fly ash



Thank you for your
attention!

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