MatCHMaker
Materials Characterisation & Modelling

ML for phase assemblage analysis of low carbon cements

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EMMC Joint workshop on advances in characterization methods and computational modelling

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Outline

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- Industrial needs
  - CO_2 / limited resources / digitalization
  - New generation of low-carbon binders have different phase assemblages
- Presentation of the image analysis method
  - Problem setting
  - Clustering algorithms
  - Limitations and next steps
- Conclusions and collaborations potentials

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Heidelberg Materials is one of the world’s largest building materials companies

51,000
employees
on 5 continents

3,000
locations
worldwide

Leading positions in cement, aggregates, and ready-mixed concrete

The global building materials industry faces major challenges

Climate change

In order to achieve the goals of the Paris Agreement for climate protection, the building materials industry needs to focus on decarbonisation.

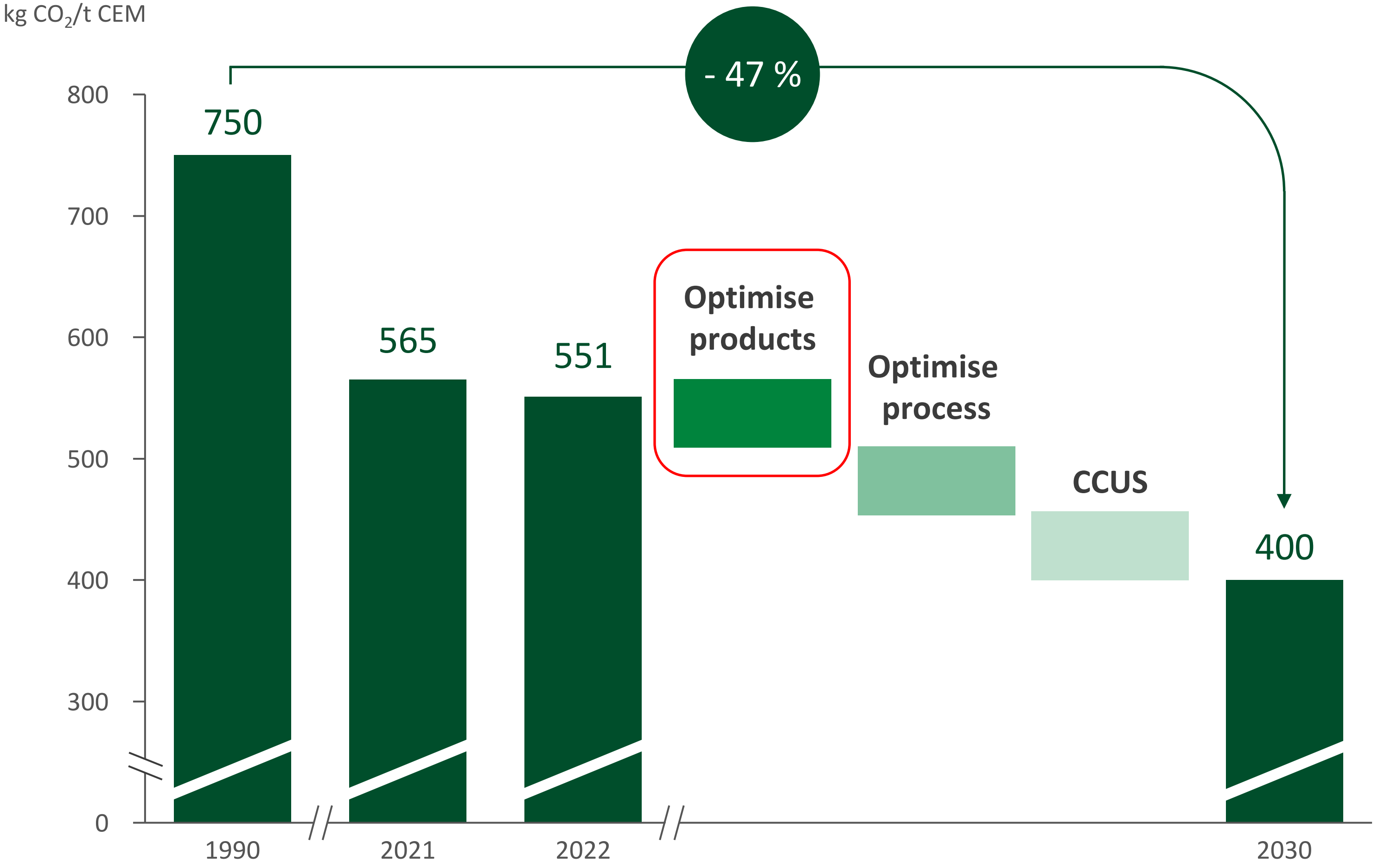
Limited natural resources

For a more sustainable use of natural resources, industry must use fewer primary raw materials and rely more on recycling, for example.

Digitalisation

In production and on construction sites, digital solutions are needed that make processes simpler, faster, safer, more sustainable and efficient.

By 2030, we want to reduce our CO_2 emissions by almost 50% vs. 1990



Levers to reach our 2030 targets

Products

Clinker incorporation <68% Drive circularity

Process

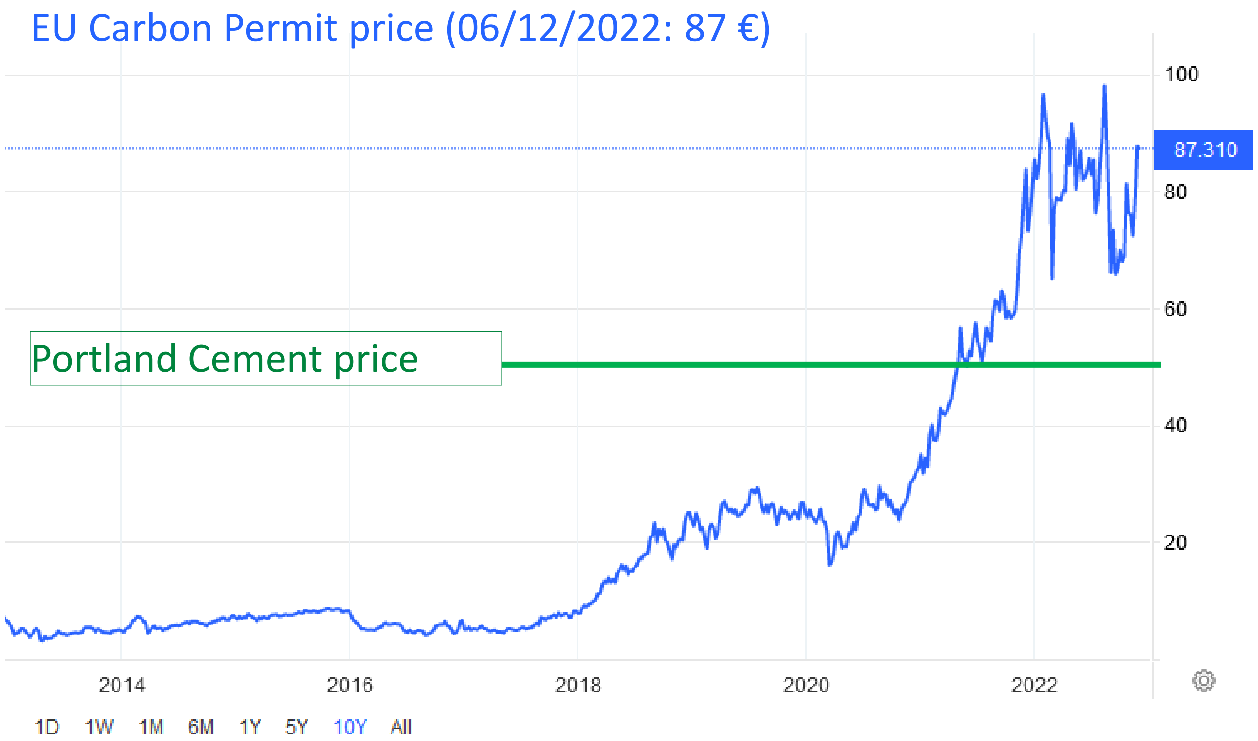
45% Alternative fuels rate

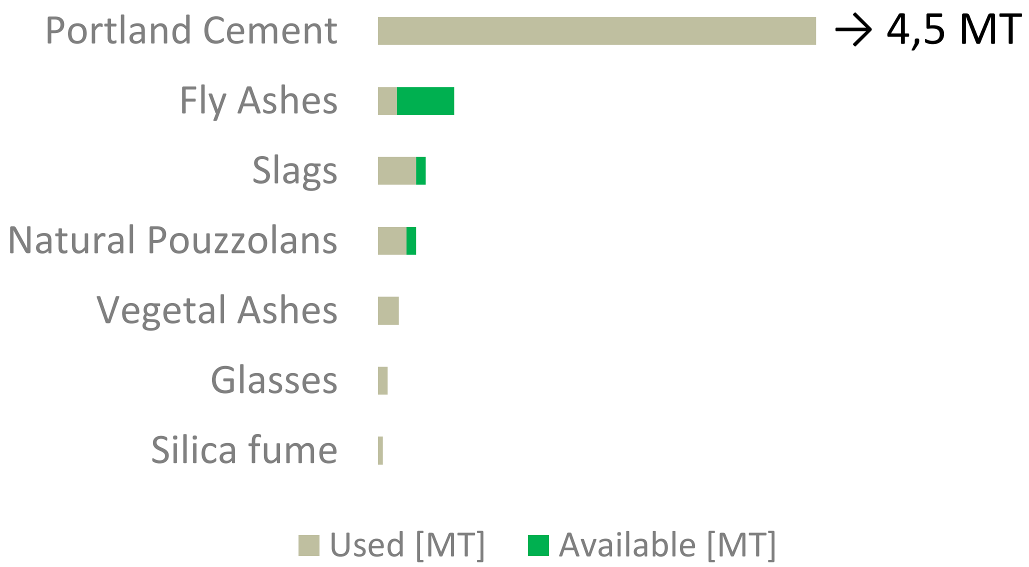
20% Biomass fuels rate

CCUS

10 mt CO_2 captured by 2030 (cumulative)

Context: Rising CO2 prices and decreasing SCM





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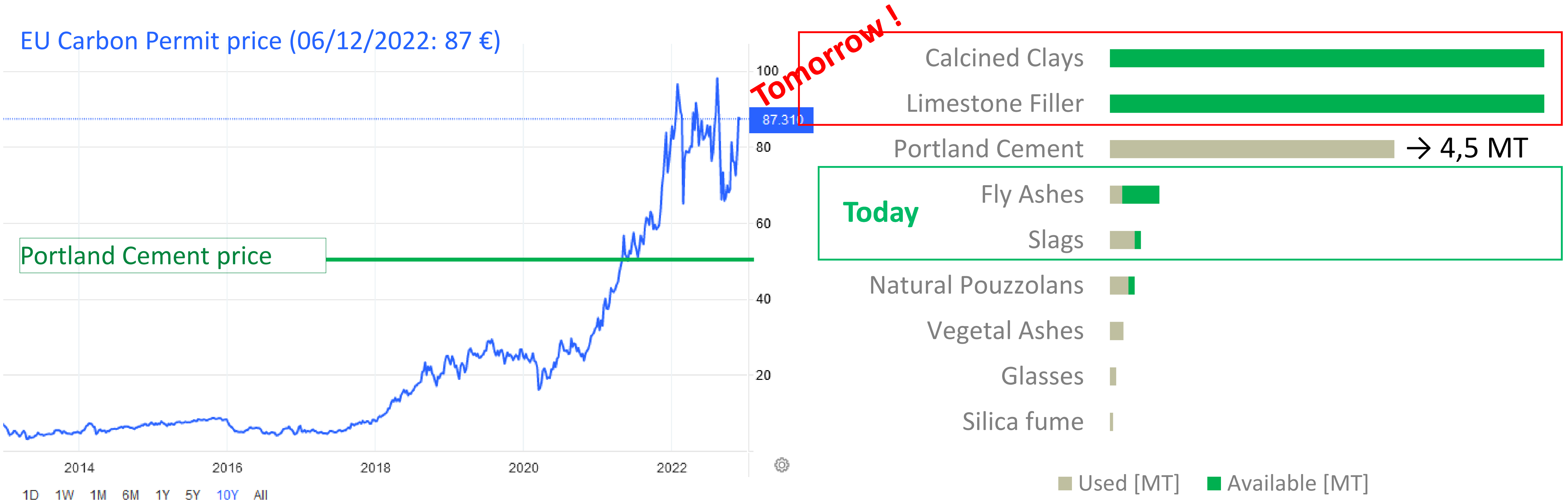
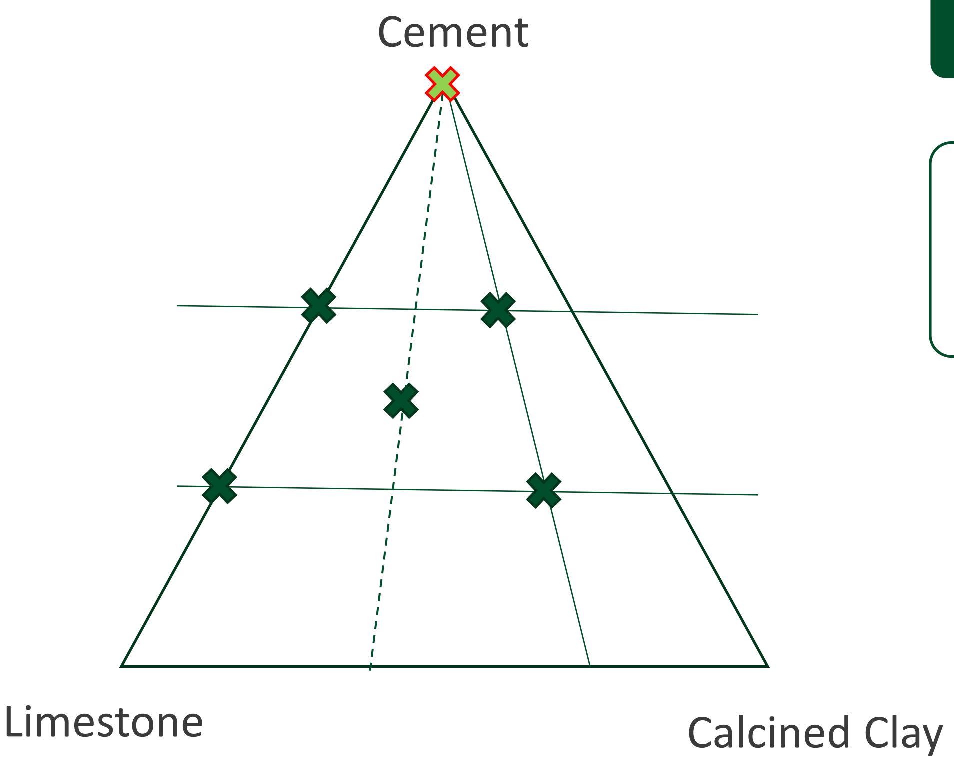
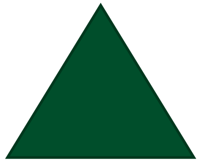


Approach: the Material Science paradigm and Design of experiments



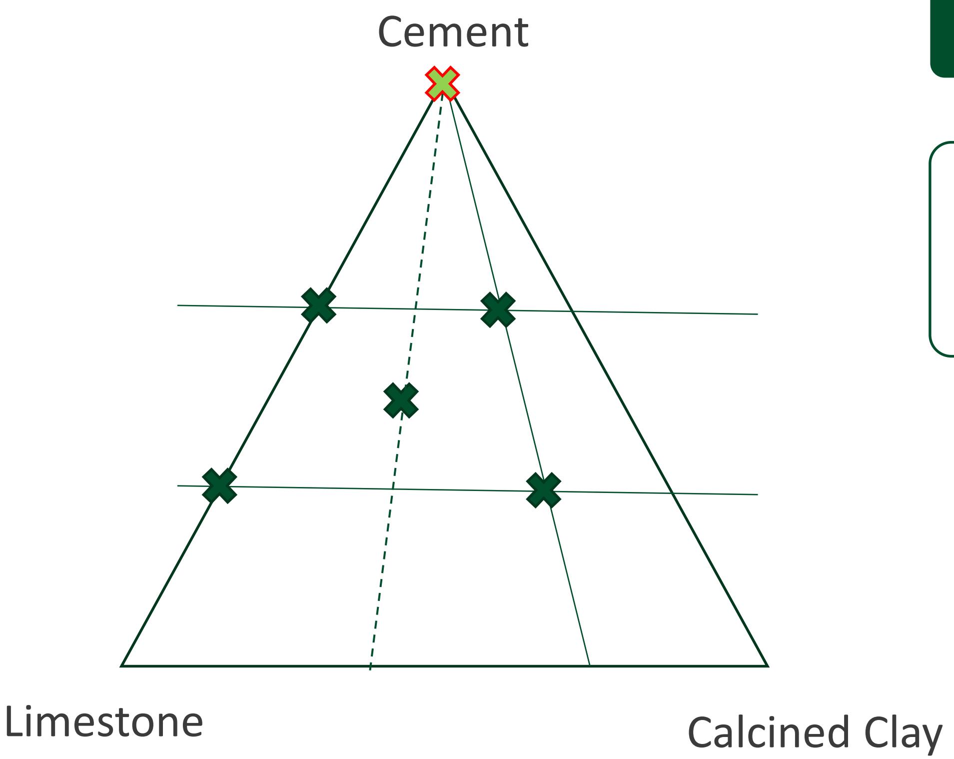
Mix design	Microstructural features	Macroscopic properties
	**Microstructure**: - Phase assemblage - Pore network	**Macroscopic properties:** - Strength (2,7 & 28d) - Durability

Goals:

- The key *applied* goals are the optimization of:
  - The mechanical (2,7 and 28 days) strength
  - The durability (chloride binding, carbonation)
- The key *fundamental* goals are to understand:
  - What phases are forming? Are they strong and durable?
  - What limits the reaction rate?
  - What limits the pore refinement

Scope: Limestone – Calcined Clay - Cement

Approach: the Material Science paradigm and Design of experiments



Mix design → **Microstructural features** → **Macroscopic properties**

Microstructure:
- Phase assemblage
- Pore network

Macroscopic properties:
- Strength (2,7 & 28d)
- Durability

Limitations of current methods:

- XRD only
  - measures crystalline phases
  - Is not able to be precise when the phase amount is below 5% …. Which is often the case in new low carbon cements (they have a diversity of phases but in smaller vol. % than in current cements.**Limitations of current methods:**

Limitations of current methods: How industry can profit from such method?

- Finer and faster characterization methods lead to faster innovations

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Novelty of the method

Technical problem: Phases identification on SEM/TEM images

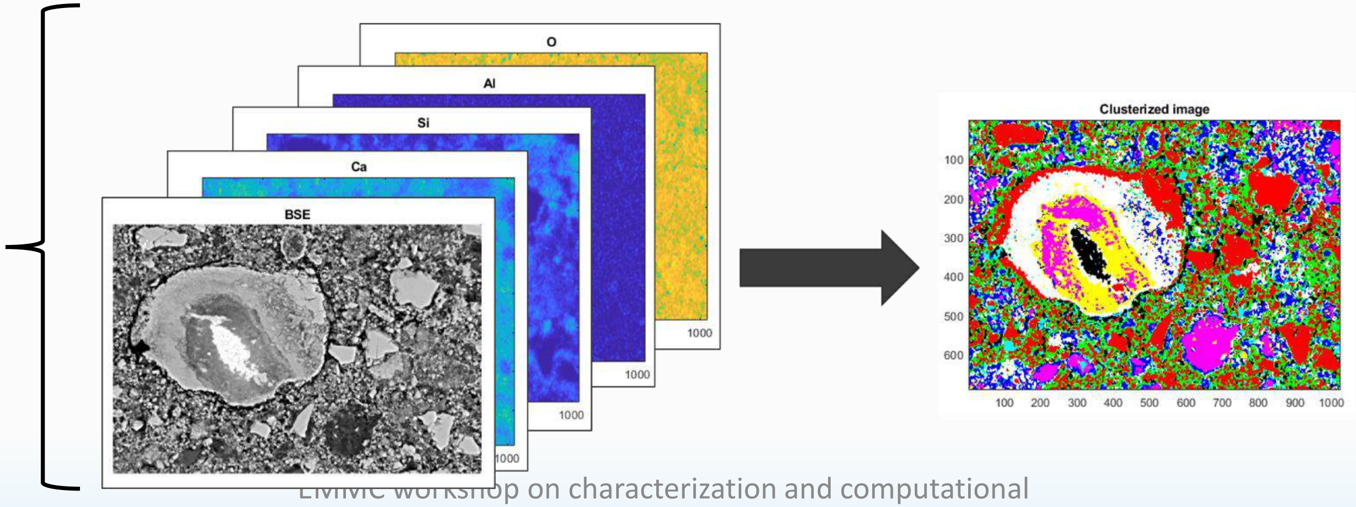
Microstructure characterization → relations between the process properties and material performances

Characterization through the identification of the different phases in the microstructure

Phase: areas of the microstructure with similar element composition

Element composition obtained through Energy-Dispersive X-ray Spectroscopy (EDX)

Backscattered Electrons +
EDX information



Novelty of the method

Materials

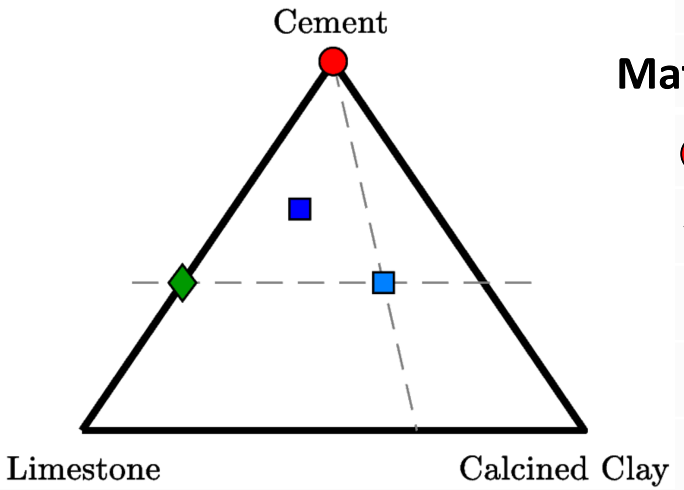
Methods: 2d and 28d compressive strength

Material definition for mortar binders

Combinations

- 2 cements
- 3 *w/b*-ratios
- 4 binders + sand

24 mortars



Material	f_cement	f_limestone	f_calClay
● (OPC)	100.00%	0.00%	0.00%
◆ (LPC)	40.00%	60.00%	0.00%
■ (LC3)	40.00%	20.20%	39.80%
■ (LC3 middle)	60.00%	26.73%	13.27%

● OPC ◆ LPC ■ LC3 ■ LC3 middle

12 most relevant mortars were further characterized by XRD and SEM

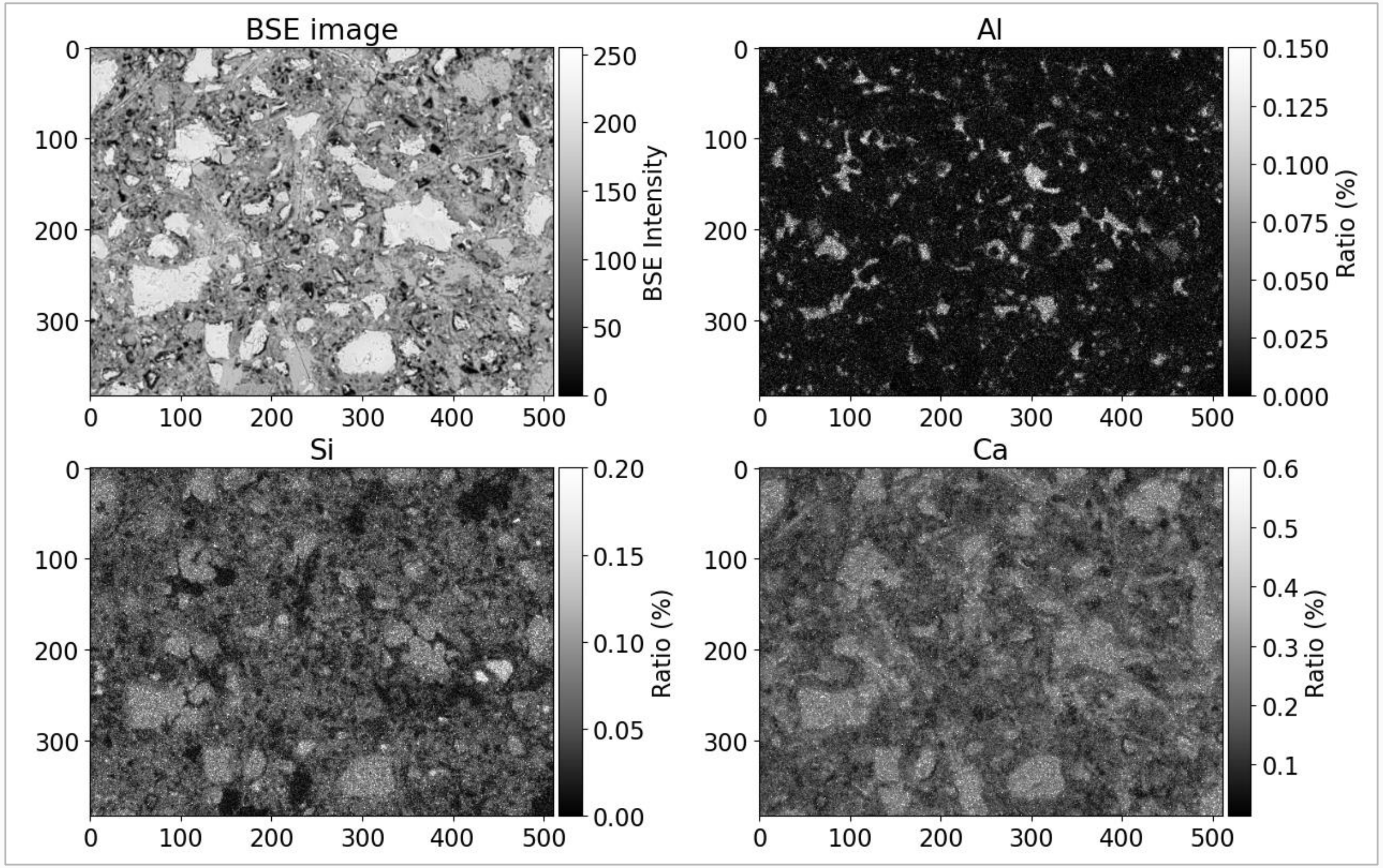
Novelty of the method

Materials

12 samples prepared:
- Portland Cement (PC)
- Portland Cement blended with Limestone (PLS)
- Limestone Calcined Clay Cements (LC3)
- Cast and cured for 2 days and 28 days

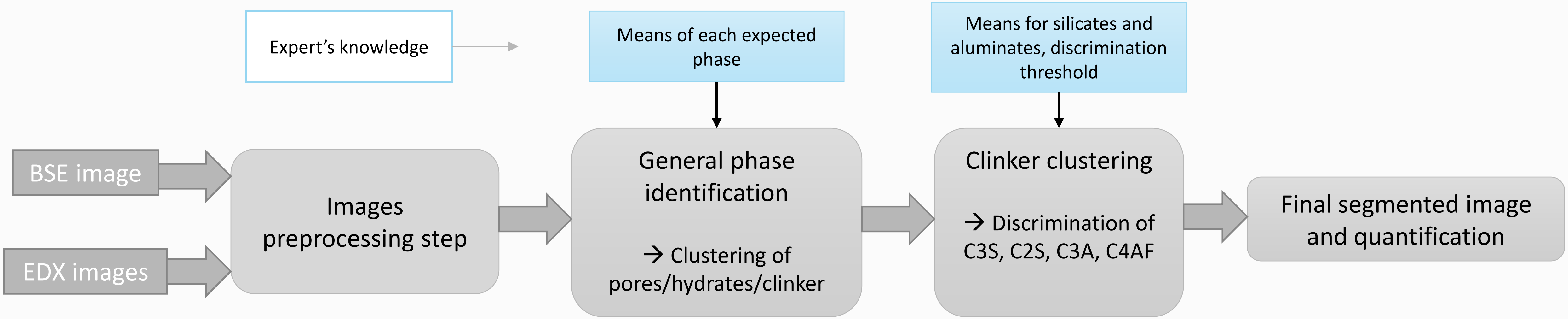
Measurements with SEM and EDX

On a specific area:
- Backscattered electron image
- EDX image for different elements:
  - **Al**, C, **Ca**, Cl, **Fe**, K, Mg, Na, O, S, **Si**, Ti



Novelty of the method

General Workflow



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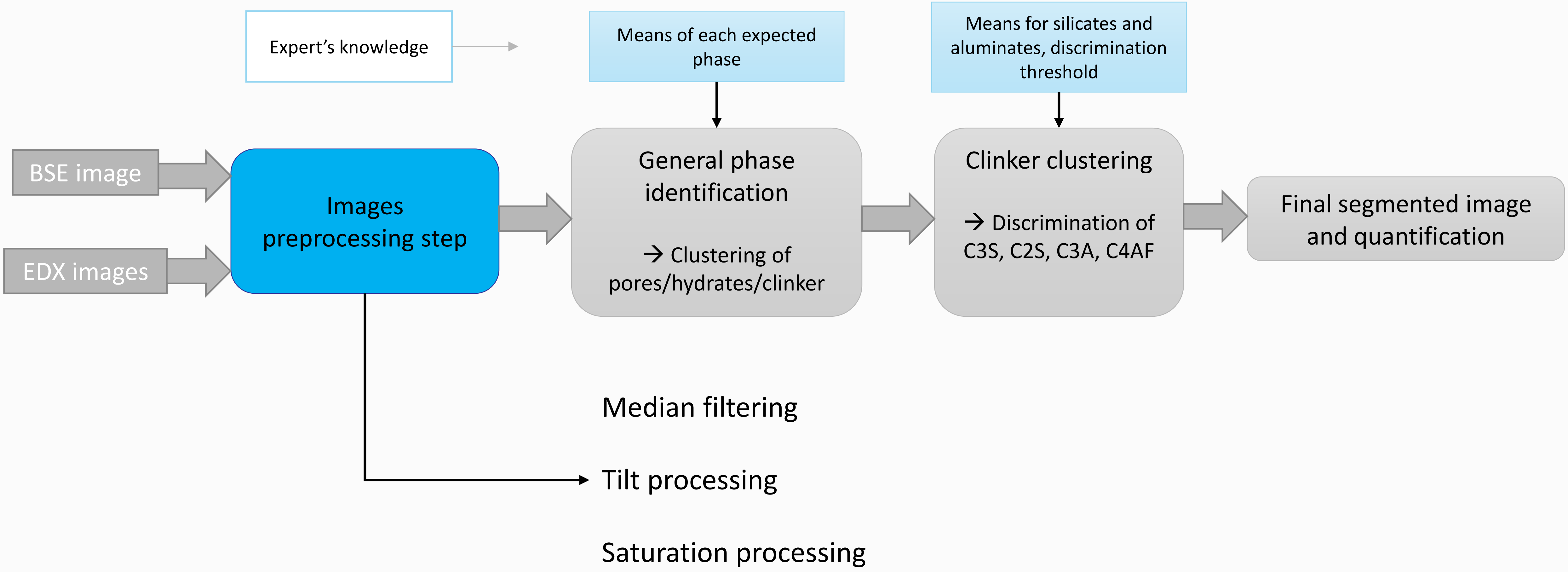

Novelty of the method

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General Workflow

Expert's knowledge

Means of each expected phase

Means for silicates and aluminates, discrimination threshold

BSE image

EDX images

Images preprocessing step

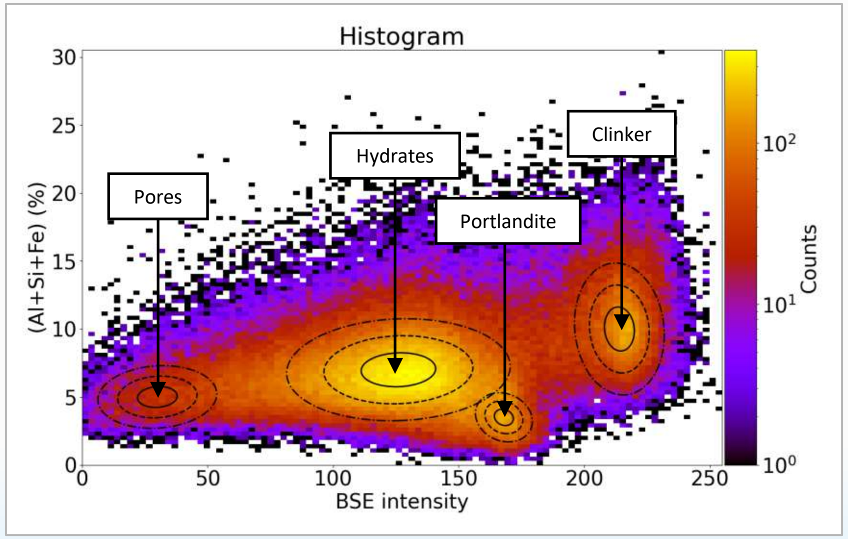
General phase identification

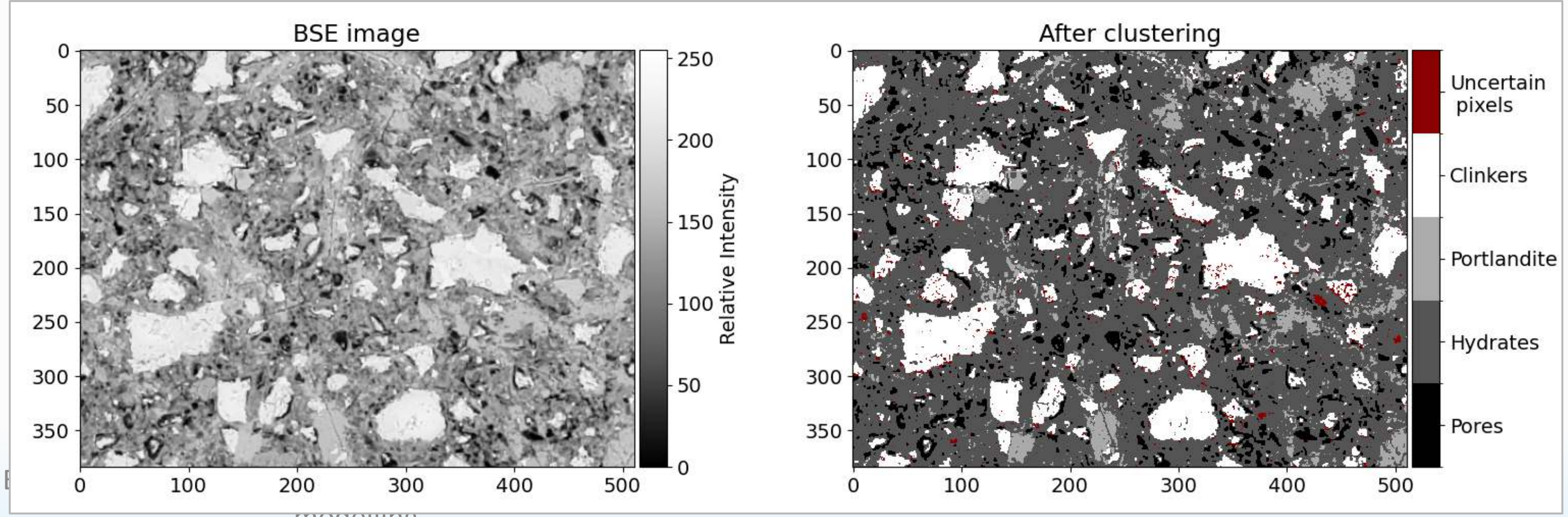
→ Clustering of pores/hydrates/clinker

Clinker clustering

→ Discrimination of C3S, C2S, C3A, C4AF

Final segmented image and quantification





modelling

Novelty of the method

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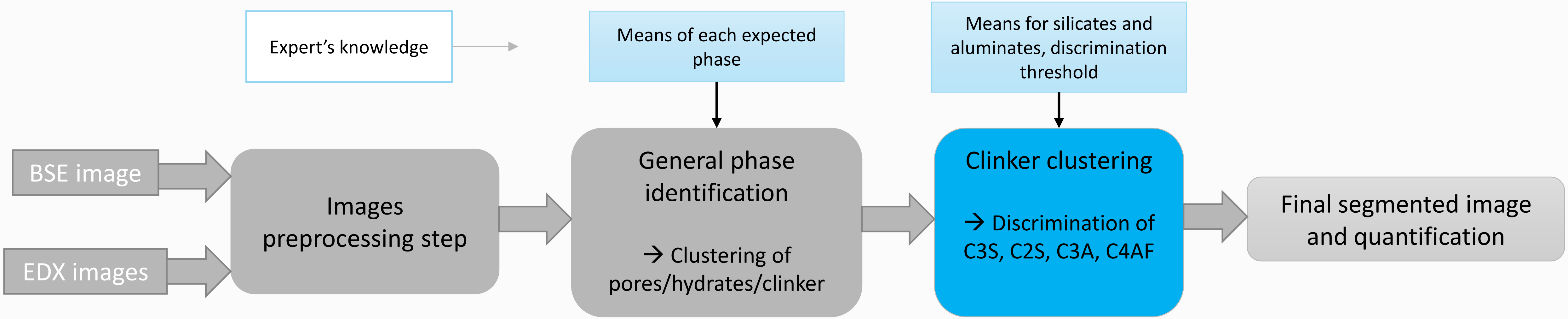
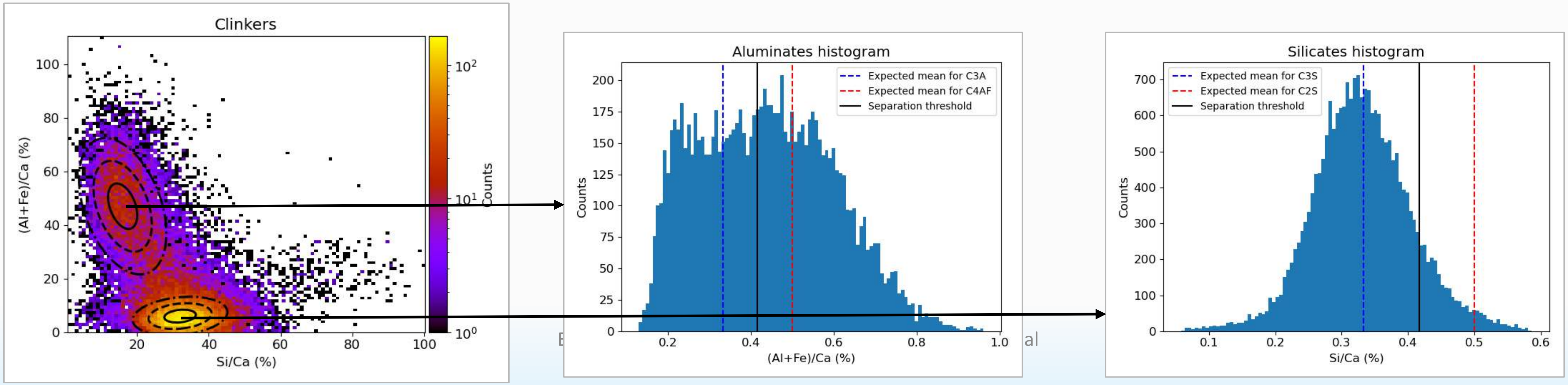
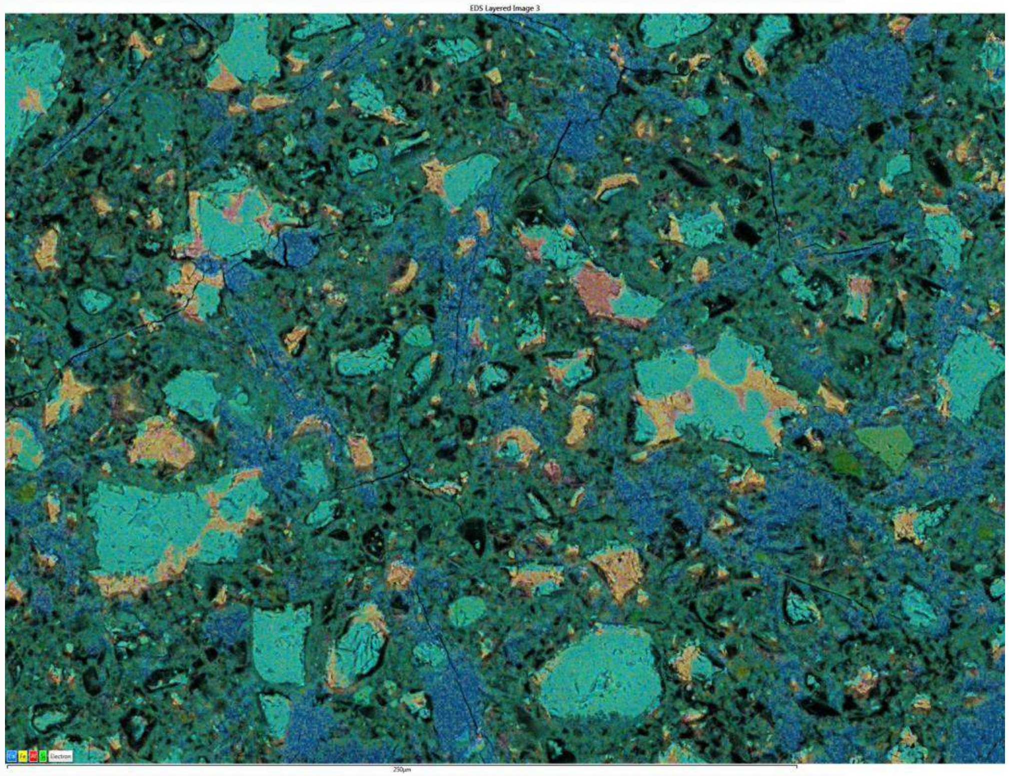


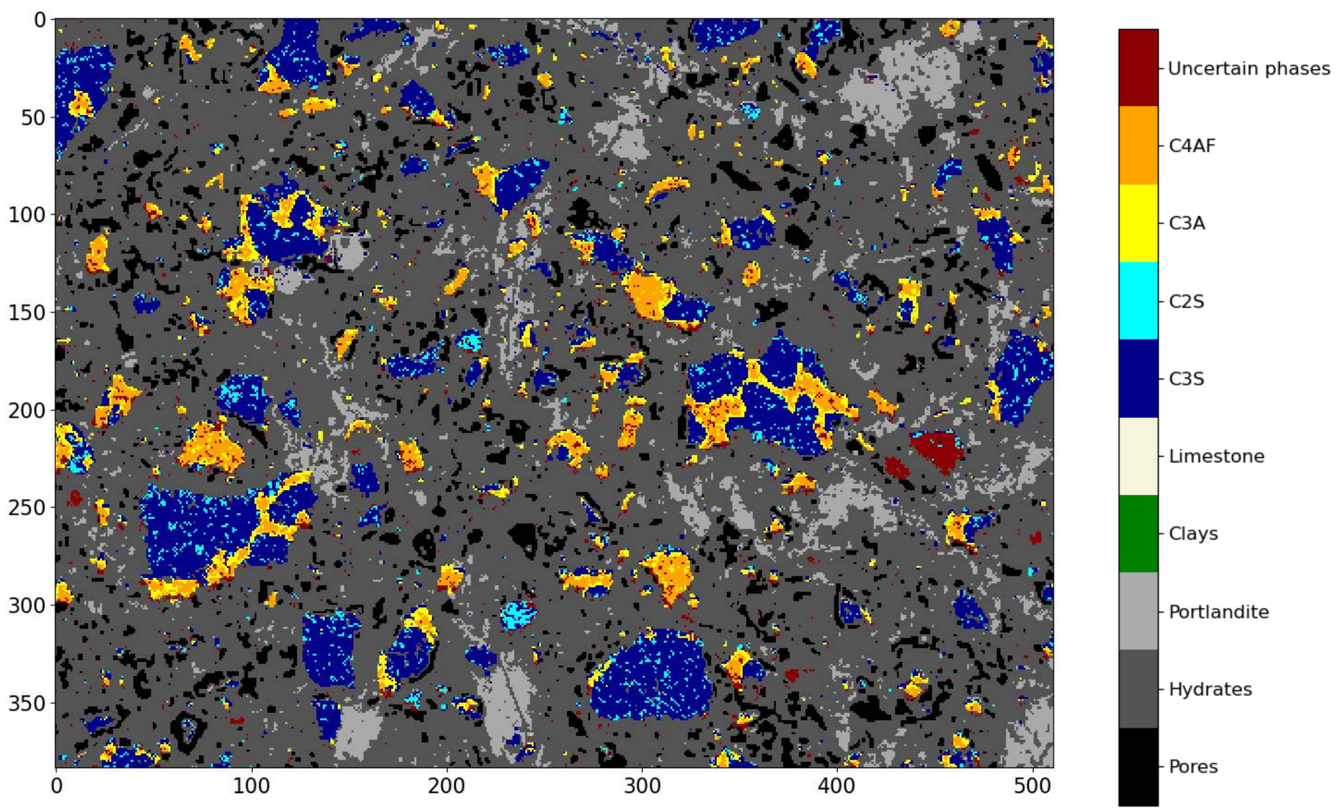


Novelty of the method

Final result: Portland cement (OPC), 2 days

Phase	Pores	Hyd phase	Portland.	Uncert. phases	C3S	C2S	C3A	C4AF
Quantity (%)	8,18	67,58	6,23	1,70	9,89	1,29	2,38	2,75





Novelty of the method

Analysis of the results

Comparison XRD and SEM

Phase	SEM	XRD
C3S	7,1	7,2
C2S	1,3	3,2
C3A	1,3	0,8
C4AF	2,6	5,1
CH	10,1	13,0

Possible sources of errors:

- The SEM measurement considers porosity, not the XRD
- XRD relative error is high in some region of the spectrum (C2S, and C3A are not problematic, but C3S and C4AF might be)

Next steps:

- We will have more in-depth characterization in the next batch of samples by December
    - We will be able to correct for porosity (MIP and DVS measurements)
    - We will make better image acquisitions

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Conclusions

Unsupervised segmentation

- Difficulty for verification: ground truth not known
- Rely on expert's knowledge for interpretability
  - Thermodynamics, limitation of each characterization method
- Data-driven method:
  - Necessitates to consider data management and availability
  - Sensitivity of the data

Novelty of the method

Next steps

- Understanding the XRD / SEM difference
  - Improving the SEM acquisition (wider EDX + BSE maps)
  - Adding some other characterization technique (MIP and DVS for porosity, possibly calorimetry?)
- Uncertainty quantifications
  - From acquisitions:
    - images to images sampling variability
    - 5kV vs 15 kV
  - From modelling methods:
    - Current method to expand to hydrates
    - Brutal clustering on the RGB image
    - "Bicoloured balls in jar" method
    - CNN
- Improving the workflow to make it as seamless as possible
  - Seamless vs flexibility challenge

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Collaborations potentials

- Sharing needs
  - What are your academic & industrial needs / what is the problem you are trying to solve
- Sharing data
  - What is the data you are generating?
    - Electron microscopy images? How many do you have? Would it be possible to share them?
    - What types of ML algorithms do you use? Would it be possible to share them?
- Beyond this meeting
  - Interviewing the R&D departments of big EU companies about their materials characterization and methods needs, challenges, bottleneck.
    - Current needs and challenges
    - Foreseen one on the short / medium / long term