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Materials Characterisation & Modelling

ML for Microstructural Image Analysis and Materials Characterization of Cement

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Questions & Answers

1. WHY DO WE CARE?

Microstructure is the cornerstone of our understanding of materials properties and strategy for performance optimisation. To characterise quantitatively microstructures is therefore of critical importance for materials scientists.

2. WHAT IS NEW?

The development of machine learning (ML) algorithms for image analysis has not yet been imported in our field. The literature dedicated to electron microscopy and image analysis for materials still relies mostly on lengthy histogram-based, morphological and spectral operations.

3. WILL IT WORK?

Our preliminary work on cement pastes is promising. Clustering algorithms allowed us to better understand the kinetics of low carbon cements. Still, slight differences with XRD raise questions.

4. WHAT IS NEXT?

a) The slight difference with XRD need to be explained: is it simply do to porosity or does either method has some intrinsic limitations?
b) From the segments, the porosity and PSSD of each mineral phase can be recovered and plugged into micro-mechanical models to predict material strength.
c) Ensemble and uncertainty algorithms will be implemented.

Motivations & challenge:

Materials science aims at bridging the gap between materials elaboration processes and their macroscopic properties through an understanding of the key microstructural features. To understand the process – microstructure – properties relationships then enable to predict and optimise materials performance. The microstructural origin of compressive strength of cement pastes is for example dictated by the porosity and mechanical properties of each constituent phase [1]; in metals, the mechanical properties depend on the grain sizes [2, Chapter 2]. To characterise microstructures precisely is therefore a critical task. Scanning electron microscopy (SEM) is one of the workhorses of materials scientists. SEM generates a variety of images that need to be analysed to yield relevant information. **→ Answer 1.**

Despite the importance of SEM image analysis, there does not yet exist a related textbook that takes advantage of machine learning (ML) techniques. The three references closest to SEM image analysis may be [3], [4] and [5]. These textbooks focus on traditional image analysis techniques that rely on geometrical, morphological and spectral operations; only very recent ones ([5] published in 2023) include a chapter on deep learning. According to Merchant [5], **the main bottleneck is the absence of an available wide dataset of labeled SEM images** [5, p449]. **→ Answer 2**

1 Pichler, B., Hellmich, C., & Eberhardsteiner, J. (2009). Spherical and acicular representation of hydrates in a micromechanical model for cement paste: prediction of early-age elasticity and strength. *Acta Mechanica*, *203*(3-4), 137.
2 Ashby, M. F., Shercliff, H., & Cebon, D. (2018). *Materials: engineering, science, processing and design*. Butterworth-Heinemann.
3 Wojnar, L. (2019). *Image analysis: applications in materials engineering*. Crc Press.
4 Heilbronner, R., & Barrett, S. (2013). *Image analysis in earth sciences: microstructures and textures of earth materials* (Vol. 129). Springer Science & Business Media.
5 Merchant, F., & Castleman, K. (Eds.). Second edition (2023). *Microscope image processing*. Academic press.

Materials & Methods:

Three types of cement were analysed: Portland Cement (PC), PC blended with Limestone (PLS) and Limestone Calcined Clay Cements (LC3). The cements were cast and cured for either 2 days or 28 days, after which they were stopped by solvent exchange and characterized by XRD and SEM analysis.

The workflow for image analysis is illustrated below. The input data is the stack composed of the BSE (black and white image) and EDX (atomic concentration of each relevant element). After a preprocessing step, it was found that Gaussian Mixture Models (GMM) could not directly yield the phase assemblage, the workflow had to be broken in two steps. In the first one, the porosity (black segment in figure 2), the hydrates (intermiate greys) and the clinker anhydrous phases (whiter phases) had to be separated. In the second one, the focus was put on the clinker phase, since the phase assemblage can be computed afterward.

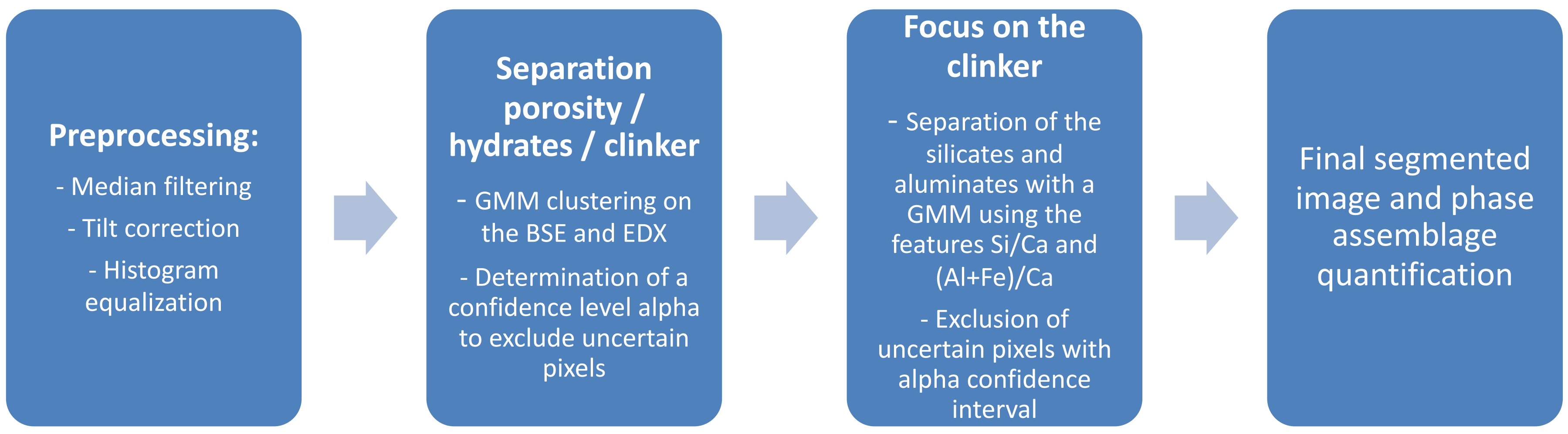

Figure 1: Workflow of the image analysis scheme.

Preliminary results:

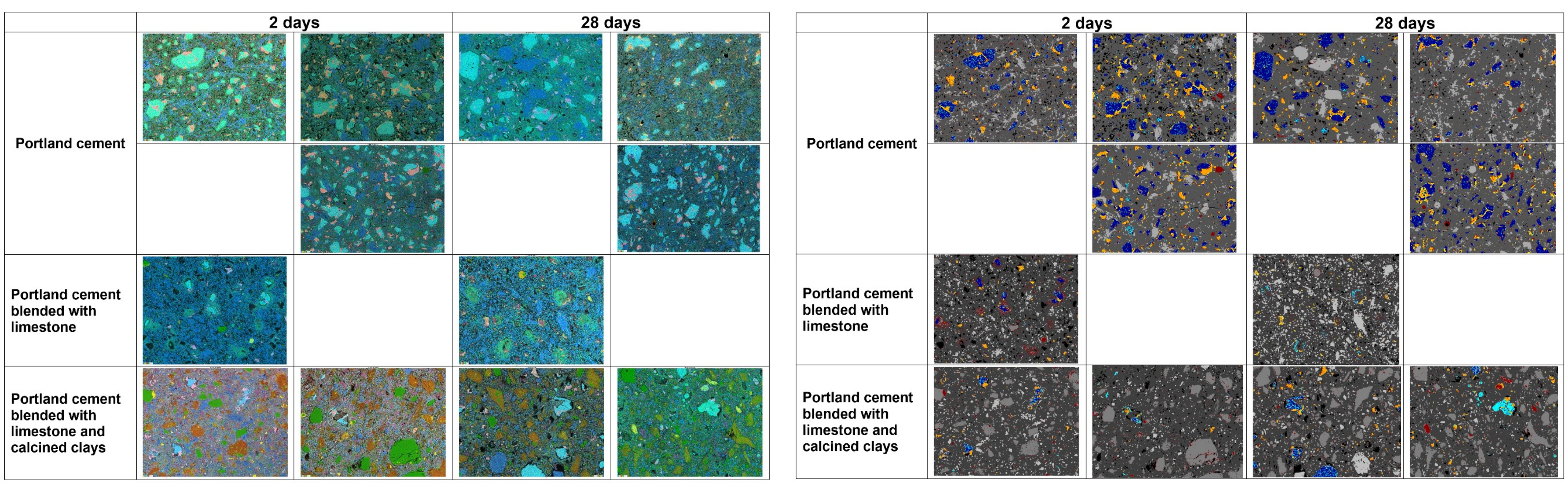

Figure 2: Comparison of the acquired images (left) and segmented ones (right). They visually seem to be well captured but some details raise questions. Belite (light blue segment) almost always appears as noise (« salt and pepper ») within the alite particles (dark blue particles); the same comment applies to C3A embedded within C4AF.

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

Table 1: Comparison of weight fraction measured by SEM and XRD (average over 12 samples). Because XRD does not measure porosity, the expected difference is about 30% (more for XRD). This could explain the jump from 10,1% to 13% for CH, but the equality of C3S raise concern. Also, the C4AF value measured by XRD is larger than by SEM. **→ Answer 3**

Conclusion and next steps:

SEM BSE-EDX images were segmented by means of the GMM algorithms with promising results. Yet our dataset needs to be extended to gain more robust conclusions and hopefully explain the slight discrepancy observed when comparing with XRD. Next steps also include the calculation of the particle size and shape distribution of each phase and uncertainty estimations. **→ Answer 4**

__We look for partnerships and synergies__ with other European projects to build case studies and transversal algorithms for all classes of materials: metals, polymers, ceramics, geomaterials, etc.
In a first step, we simply need to build a database of images: come discuss with us over a coffee ☺.

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