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Current measurement methods and limitations**

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Carbonation depth of lime-based binders: Current measurement methods and limitations

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Abstract. Given the crucial role of carbonation in the hardening of lime-based binders, accurate measurements of carbonation depths are essential for analysing both carbonation kinetics and carbon sequestration capabilities. This study employed both the conventional phenolphthalein spray method and the profile-based method to determine carbonation depths in four types of binders. Unlike the distinct carbonation front observed in cementitious materials, lime-based binders displayed a transition zone between fully carbonated and uncarbonated areas. Meanwhile, the remaining portlandite content in some specimens did not necessarily increase with depth, and typical Liesegang patterns were observed. Compared to phenolphthalein spray method, profile-based methods provide more quantitative evidence for further analyses, but inevitable slice interval can also lead to errors in carbonation depth estimation. Therefore, developing a more precise and convenient method remains essential for a deeper understanding of the carbonation behaviour in lime-based binders.

Introduction and Experiment setup

Currently, phenolphthalein spray method is widely used to measure carbonation depths of cementitious materials. However, when applied to lime-based materials, this method can result in misleading patterns, such as Liesegang rings [1] and large partially carbonated zone [2]. Furthermore, it is impractical to do any quantitative analysis with these patterns, as the relationship between the colour and the carbonation degree is rather unclear.

In order to gain more insights into the carbonation process, the profile-based method was proposed for cementitious materials [3]. By characterizing powder samples collected at different depths, the spatial distribution of carbonation products could be roughly determined. This study employs both the phenolphthalein spray and profile-based methods to explore the carbonation behaviour of lime-based materials. Four kinds of paste, C100L0 (pure cement paste), C67L33 (67 wt.% cement and 33 wt.% lime), C33L67 (33 wt.% cement and 67% wt.% lime) and COL100 (pure lime paste), were prepared following the curing scheme in EN 1015-1. After 28 days' curing, the other five sides of prism specimens were sealed with the aluminium tape, leaving only one side exposed to the accelerated carbonation environment (1.0% CO₂, 65% RH). Following 28 days of exposure, prism specimens were cut apart and powder samples were collected at different depths for thermogravimetric analyses (TGA) and X-ray diffraction (XRD).

Results and discussions

As is shown in Fig. 1, some highly-carbonated zones could be found in lime-cement binders, but they are not necessarily close to the exposure surface (top side). Similar phenomena were also reported in some previous investigations [1], and they might be attributed to the Ostwald ripening and recrystallization of carbonation products.

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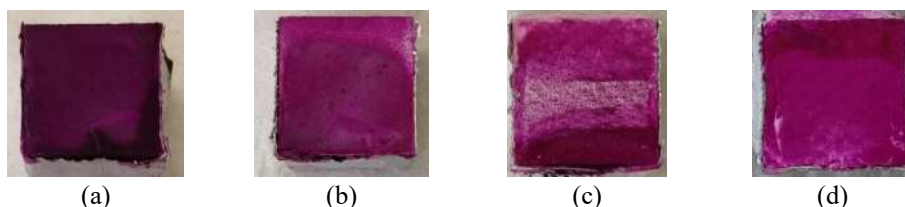


Fig. 1. Phenolphthalein spray test results: (a) C100L0; (b) C67L33; (c) C33L67; (d) C0L100

These Liesegang patterns could be further quantified with TGA. As shown in Fig. 2, the CaCO_3 content in C33L67 firstly increases with depths and then decreases, and the remained $\text{Ca}(\text{OH})_2$ content also fluctuates. However, in mixtures with less lime, both contents varied monotonically with the depth, and sharp carbonation fronts could be clearly defined.

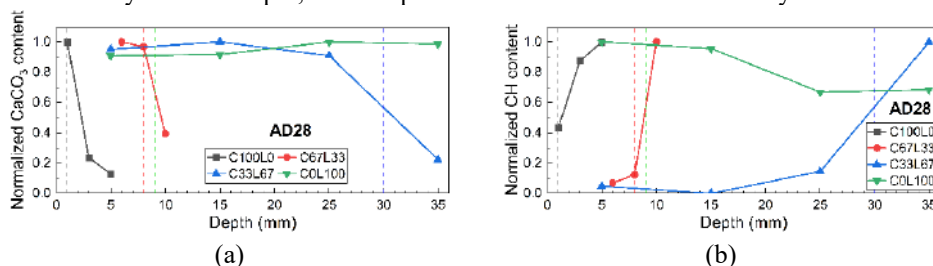


Fig. 2. Normalized TGA results: (a) normalized CaCO_3 contents; (b) normalized $\text{Ca}(\text{OH})_2$ contents

Phase assemblages of carbonation products could be obtained with XRD (shown in Fig. 3). In lime-cement binders, AFm phases also got carbonated, leading to the formation of calcium carboaluminate hydrates. However, due to the inevitable slice interval, it is hard to determine exact spatial distributions of carbonation products. More novel testing methods, such as confocal Raman microscopy tests [3], could be considered in future studies.

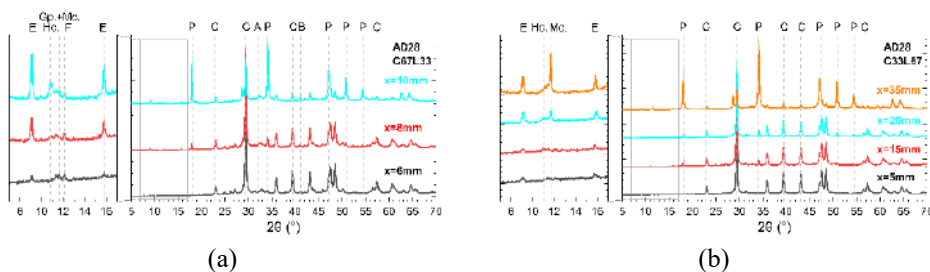


Fig. 3. XRD patterns of representative powder samples: (a) C67L33; (b) C33L67. E=Etringite, Gp.=Gypsum, Hc./Mc.=Hemi/Mono-carboaluminate hydrates, P=Portlandite, C=Calcite

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