Crucial Clay Activation Conditions

Mylène Krebs, Jean-Michel Charmet, and Damien Chudeau, Fives FCB, discuss the key factors influencing the calcination process and present insights from a calcination test centre in Northern France.

The calcination of clay is a transformative process, unlocking its potential as a reactive supplementary cementitious material (SCM). At Fives FCB, the Cement and Minerals division of Fives Group, a dedicated calcination pilot has been operational since 2021. Equipped with advanced technologies, the facility enables precise parameter optimisation for a wide range of clays. This article examines the major calcination parameters, the performance of two distinct calcination technologies, and considerations for assessing clay suitability.

Calcination equipment and methods

The Fives FCB test centre houses two calcination technologies:

- Flash calciner: Featuring the four independent process steps (i.e. heat generation, calcination, colour control, and final cooling) as in Fives' industrial plants. This technology ensures the best overall

performance and versatility across a wide range of clay qualities. It can be operated up to 1300˚C, extending the use of flash calcination to other types of materials.
- Rotary kiln: A 4 m long rotary kiln providing residence time of 30 min., suitable for producing both calcined clay and clinker.

Figure 1. Fives FCB's calcination pilot plant.

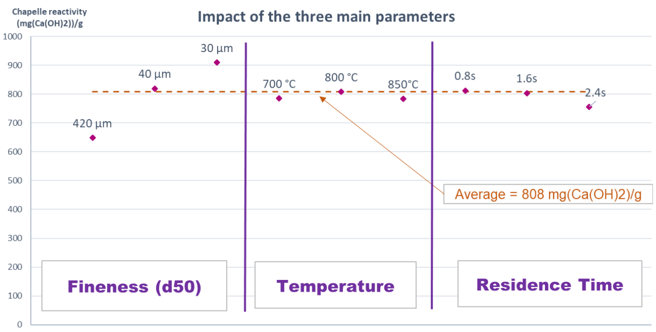

Figure 2. Calcined clay reactivity.

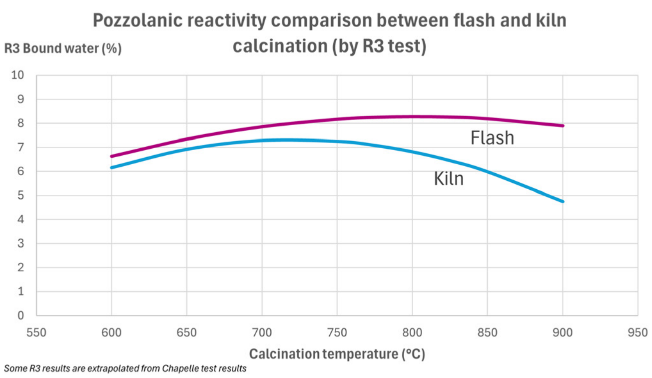

Figure 3. Comparative analysis on kiln and flash calcination.

To date, dozens of materials have been tested, resulting in a robust database of 150 samples.

Key parameters influencing calcination

Optimising the calcination process requires attention to three principal factors:

- Temperature:
  - Insufficient heating results in incomplete dehydroxylation, reducing reactivity.
  - Excessive heating induces sintering and crystallisation reactions, creating unreactive phases.
- Particle size distribution:
  - The proper grinding sequence has a strong influence on process efficiency.
  - In flash process, preliminary grinding to a d50 in a range of 30 µm to 40 µm often strikes a balance, minimising operational issues like clogging and reducing the need for subsequent grinding.
- Residence time:
  - Residence time varies significantly between technologies. Flash calciners enable short residence times (sec.), while rotary kilns require prolonged durations (30 min.). Optimal residence time correlates with particle size and ensures uniform calcination.

Experimental design for flash calcination parameter optimisation

A systematic survey of flash calcination parameters was conducted using the Taguchi method (Figure 2), a robust statistical approach to experimental design. The study evaluated the influence of the three key factors – temperature, residence time, and particle fineness – each tested at three distinct levels. This factorial design resulted in a total of 18 tests, carefully structured to assess the interactions and individual effects of these parameters. The reactivity of the calcined clay was measured using the modified Chapelle Test, following the NF P18-513 standard. This methodology ensures reliable and reproducible insights into the optimisation of calcination conditions for maximising clay reactivity.

The role of particle size distribution in calcination

The primary determinant of the calcination process efficiency and outcome is the particle size distribution of the clay. Finer particles ensure higher reactivity and consistency in the final product, as they promote uniform heat transfer and complete dehydroxylation during calcination. Conversely, coarse particles often result in insufficient calcination, leading to a lower reactivity of the material.

It is important to note that increasing the temperature or prolonging the residence time cannot fully compensate for the challenges posed by coarse particles. Such adjustments may lead to energy inefficiencies and undesirable transformations, such as the formation of unreactive phases – by sintering or crystallisation reactions. Therefore, achieving an optimal particle size distribution before calcination is critical for maximising reactivity and process efficiency.

Comparative analysis of flash calciner and rotary kiln

The Fives FCB test centre has performed comparative studies on identical clays using both technologies. Key observations include:

- Rotary kilns:
  - Advantages: adaptable for retrofitting existing kilns.
  - Limitations: difficulties to achieve uniform particle calcination.
- Flash calciners:
  - Advantages: precise control over temperature and residence time; suitable for fine clays.
  - Performance: tests confirm superior reactivity of clays calcined in flash calciners compared to rotary kilns.

As seen in Figure 3, the optimal reactivity is obtained with flash calcination at a temperature between 750˚C and 800˚C. The x-axis (calcination temperature) has been correctly set between the flash and the kiln tests by comparing with the LOI of the final product as an image of the calcination rate. The reactivity of calcined product (y-axis) has been measured performing R3 tests based on bound water. On average, the reactivity is 16% higher with flash technology compared to kiln technology. It should be noted that this conclusion on reactivity results from the comparison between the rotary kiln and the flash calciners is quite important. Indeed, final product reactivity is a key criteria for a new clay calcination plant, other important features being the energy consumption or the design efficiency related to colour control or emissions. Those tests confirm the higher efficiency of the flash calciner compared to the rotary kiln. As in every modern cement plant, calcination is performed by a flash calciner, and the kiln being used for final clinkerisation. Indeed, clay activation by dehydroxylation – consisting in breaking OH bound in the mineral structure – is a very fast kinetic process that fits perfectly with a flash calciner. It does not require the combination of several chemical components by solid-solid or solid-liquid reactions like in the clinkerisation.

Thus, a long residence time at the activation temperature, as in a rotary kiln, is not necessary

Figure 4. Erection works in the Rohrdorfer plant, Germany.

Table 1. Colour measurements.

Sample	L*	a*	b*
Raw clay 15% Fe_2O_3	63	17	32
CC without colour control	46.9	23.5	29.1
CC with colour control	43.4	1.1	4.3

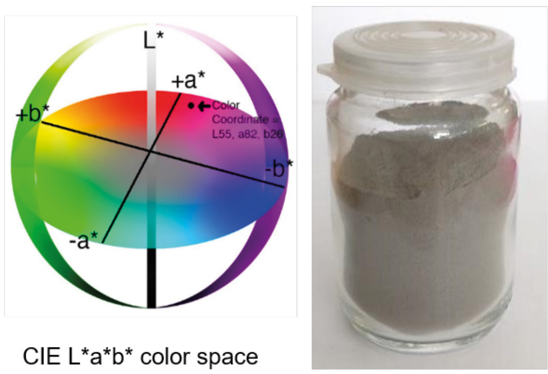

Figure 5. Fives FCB' colour control efficiency.

and can even be harmful for reactivity as sintering reactions may occur.

Clay suitability assessment

In the context of clay calcination, certain aspects can be controlled – such as the calcination parameters outlined earlier – while others, like the characteristics of the clay quarry, are more challenging to alter. A detailed evaluation of the clay is essential to determine its suitability as a candidate for calcination. Reactivity is primarily derived from kaolin, though pure kaolin is rare.

Most clays are complex mixtures of minerals, including quartz, kaolinite, illite, smectite, which can be identified through X-ray diffraction (XRD) analysis. Additionally, the chemical composition, determined via X-ray fluorescence (XRF), must be analysed to identify elements such as iron or carbonates that may impact reactivity. Thermogravimetric and differential calorimetry analysis (TGA/DSC) is also crucial for assessing the clay's thermal behaviour and suitability.

Based on extensive database insights, the suitability of a clay has been preliminarily evaluated at Fives FCB by examining the total clay content, ensuring that the loss on ignition (LOI) from dehydroxylation is sufficient, and analysing the ratios of Al_2O_3 to SiO_2.

If some parameters are suboptimal, processes such as clay beneficiation or quartz removal may be employed to enhance its quality. However, any conclusions regarding the clay's potential must ultimately be validated through pilot-scale calcination testing to ensure optimal performance.

Implementation of new calcination unit at Rohrdorfer

Fives FCB is implementing a new clay calcination unit in Germany. Rohrdorfer Zement, operating cement plants both in Germany and Austria, is actively implementing its road map to carbon neutrality by 2038. Clay calcination shall play an important role in this road map. First step will consist of testing different clays and different cement recipes.

To do so, Rohrdorfer Zement entrusted Fives FCB for the engineering, supply, and erection of a 50 tpd reliable flash calcination unit equipped with high efficiency colour control (Figure 5). Erection is under progress and commissioning scheduled for mid-2025.

Conclusion

Optimising clay calcination demands a systematic approach, balancing temperature, particle size, and residence time. Flash calcination emerges as a preferred technology for most applications, offering superior control and reactivity. Nonetheless, detailed assessments of clay mineralogy and chemistry are indispensable for choosing a suitable material and tailoring calcination strategies. ■