

# Closing Letter of RILEM TC 282-CCL: Calcined Clays as Supplementary Cementitious Materials

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## Abstract

With over 8% of global carbon emissions worldwide, the cement industry is challenged to lower its carbon footprint. Replacement of clinker in cementitious systems becomes crucial. Sound research proved that kaolinitic clays with as low as 40% kaolinite can have a high reactivity as SCM. Further research studies found a synergy between the aluminates in calcined clays and the carbonates in limestone that led to the proposal of a ternary binder called Limestone Calcined Clay Cement, LC3, consisting of 50% Portland Cement, 30% calcined clay and 15% limestone. This paper presents the efforts of a group of members from 41 universities and 17 industrial partners through the RILEM Technical Committee 282 – CCL: Calcined Clays as Supplementary Cementitious Materials. The work was oriented to fill existing information gaps on characteristics of clay minerals, the process of clay calcination, hydration of cementitious systems containing calcined clay and limestone, fresh and hardened properties of concrete, standardization, and durability of concrete produced with binders containing calcined clay and limestone. The TC 282-CCL has published 10 whitepapers, with a strong contribution to a better knowledge and understanding of the role of calcined clay in cement and concrete.

**Keywords:** Calcined clay; Calcination technology, Hydration, Calcined clay cement; Concrete; Durability.

## 1 Introduction

The cement industry is in the pursuit of sustainability. With over 8% of global carbon emissions worldwide, the industry is challenged to lower its carbon footprint. Among several measures, replacement of clinker - a material produced through burning of limestone and clay - in cementitious systems becomes crucial. [1,2] For decades, industrial by-products such as Pulverized Fly Ash, PFA, and Ground Granulated Blast Furnace Slag, GGBFS, have successfully been used as clinker replacement materials. However, phasing out of coal power plants has decreased the availability of PFA, while the increasing use of recycled metal in the iron and steel industry has brought a decrease of the availability of GGBS [3,4].

Since 2004, the research groups LMC EPFL and CIDEM UCLV in Switzerland and Cuba started a study to assess the viability of using low grade kaolinitic clays as Supplementary Cementitious Materials (SCM). Sound research proved that kaolinitic clays with as low as 40% kaolinite can have a high reactivity as SCM [5,6]. Further research studies detected a synergy between the aluminates in calcined clays and the carbonates in limestone to form Afm phases in the form of

carboaluminates. This breakthrough led to the proposal of a ternary binder called Limestone Calcined Clay Cement, LC3, consisting of 50 mass-% Portland Cement, 30% calcined clay and 15% limestone [7–9]. The development of such ternary systems involving limestone and a SCM led to several research studies around the world in this subject.

As SCM, (calcined) clays and limestone have been looked at mostly, as clays and limestone are widely available worldwide, with special emphasis on the tropical belt, where most of current cement production takes place. LC3 does not demand high purity materials, so a low grade kaolinitic clay and limestone would be suitable. The reserves of suitable clays are huge, often as a waste of industrial processes for extraction of high grade clay minerals such as kaolin, and also during dolomitic limestone, as a waste of clinker production.

The study of ternary combinations such as LC3 opened a new avenue for the sustainable production of cement. However, as a “new” material, there are many gaps in information of how the material can be used. They could be grouped in the following categories:

1. Origin and characteristics of clay minerals, including characterization techniques.

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2. The process of clay calcination, technologies available, calcination parameters.
3. Hydration of cementitious systems containing calcined clay and limestone.
4. The use of binders containing calcined clay and limestone in applications. Fresh and hardened properties. Standardization.
5. Durability of concrete produced with binders containing calcined clay and limestone.

These information gaps were filled through the collective effort of a group of members from 41 universities and 17 industrial partners through the Technical Committee 282 – CCL: Calcined Clays as Supplementary Cementitious Materials. Five workgroups were created to address the identified gaps. 10 white papers were published in the Materials & Structures journal between 2022 and 2024.

## 2 Outputs of the Technical Committee

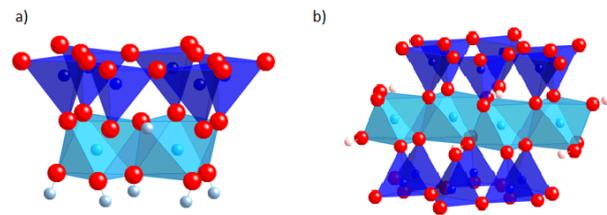
The knowledge generated by the work of the TC can be summarized as presented below:

- Information on clay structure and how it impacts the reactivity of the material, as well as other physico-chemical parameters; discussion of main techniques for clay characterization [10,11].
- Discussion of clay calcination and how it can impact the properties of the calcined product; temperature window for calcination; impact of technology for calcination and real industrial examples of clay calcination projects [12,13].
- Discussion on the main mechanisms for hydration of cementitious systems containing calcined clays and limestone and comparison with traditional cementitious systems [14].
- Collection of experience on the use of binders containing calcined clay and limestone in concrete; impact on fresh and hardened properties of concrete; current state of standardization for cements containing calcined clay and limestone [15–17].
- Discussion on the performance of concrete produced with binders containing calcined clay and limestone under various exposure conditions such as chloride and carbon dioxide exposure, chemical attack, alkali silica reactivity etc. [18,19].

Each of these outputs are discussed in more detail in the subsequent sub-sections.

### 2.1 Clay minerals

The properties and occurrence of clay resources that are potentially suitable for use as SCM were discussed. Great emphasis was placed on kaolinitic clays, known as 1:1 clays, but also the 2:1 clays, which are also abundant but less reactive. Kaolinitic clays are found across the world, but are most abundant in tropical and subtropical regions, the same regions where housing and infrastructure will likely be highest in the twenty-first century. 2:1 clays are mainly found in mild climates such as the northern part of Europe. Figure 1 presents the structural order of 1:1 clays and 2:1 clays.



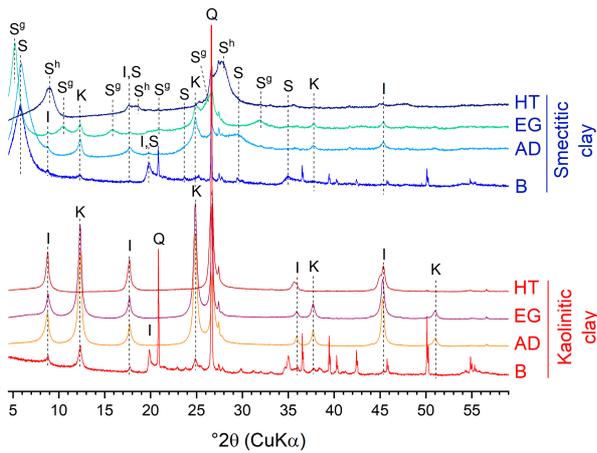
**Figure 1.** a) A 1:1 or T-O layer showing the connection between the tetrahedral and the octahedral sheets. b) A 2:1 or T-O-T layer showing an octahedral layer with a tetrahedral layer on either side. Figure made using CrystalMaker Software® [11].

The identification of kaolinitic clays was a focus of the paper, since prevalent classification schemes of kaolin deposits are focused almost exclusively on high-grade deposits. A mapping of reserves of kaolinitic clays suitable as source of SCM was presented, including mining tailings of current high-grade kaolin exploitation, which are expected to be much more abundant than currently exploited high-grade kaolin.

Reactivity of the clays was also a subject of attention. A content of 40 mass-% of kaolinite - defined by an equivalent strength performance argument for the so-called limestone-calcined clay cements of 50 mass-% clinker replacement - is proposed as minimum threshold value to qualify kaolinitic clays as suitable for use as SCM after calcination.

Relevant characterization techniques were also discussed in a second paper [10]. Most relevant techniques identified were X-Ray Diffraction (XRD), thermal analysis and Infra-Red (IR) spectroscopy. For each of the techniques, clay specific sample preparation and data collection routines were described together with guidelines to the interpretation and analysis of the collected data.

XRD (see Figure 2) can provide both highly resolved identification of clay and other minerals and accurate phase quantification. To obtain such information, specific laboratory routines and analytical software are required, and using these correctly relies on somewhat advanced understanding and analytical experience. Thermal analysis can be used more readily and easily, yet does not provide the same level of detail as XRD. Overlap between mass loss events for different clay minerals can be significant. IR spectroscopy should mainly be seen as a complementary qualitative technique that provides information on structural (dis)order or averaged octahedral layer composition that is more difficult to obtain using XRD and thermal analysis [10].

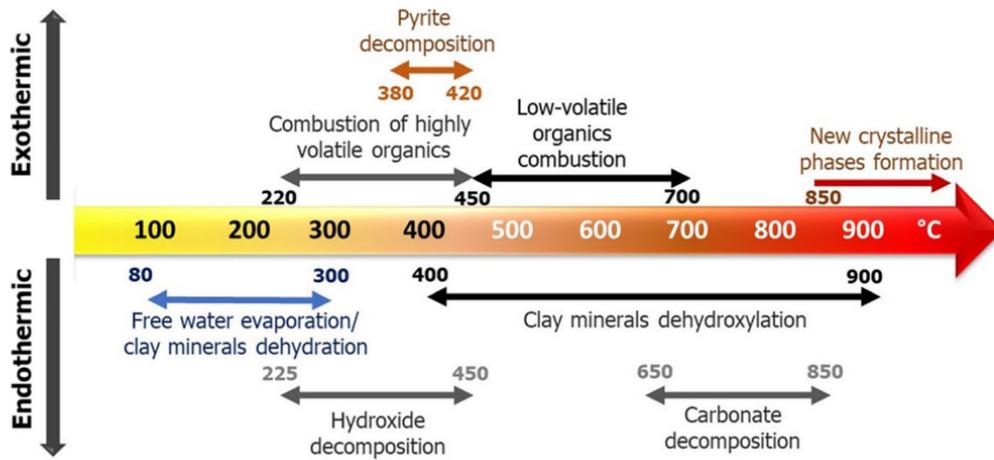


**Figure 2.** Interpreted XRD measurements of the case study impure kaolinitic and smectitic clays. XRD measurements were made on randomly oriented bulk samples (B), and on air-dried (AD), ethylene

glycol solvated (EG) and 550 °C heat treated (HT) oriented samples of the clay size fraction (< 2 μm). The main reflection peaks are labelled as follows: I stands for illite, K for kaolinite, S for smectite, S<sup>g</sup> for glycolated smectite, S<sup>h</sup> for heat treated, collapsed smectite, and Q for quartz [10].

### 2.2 Clay calcination

The entire process of clay calcination was discussed in a paper [12], from the quarry to the industry. The importance of fully understanding the clay available before designing the extraction, pre-processing, and calcination processes was discussed. The various processes were discussed, as well as their impact on CO<sub>2</sub> emissions and energy consumption. Figure 3 presents a schematic showing the thermal transformations occurring during clay calcination.



**Figure 3.** Temperature range of typical reactions taking place during the calcination of clays in conventional calcination systems. This Figure serves only as a guide; actual calcination steps need to be revealed locally based on the type of clays and processing [12].

The main technologies that could potentially be used to further improve the sustainability of clay calcination and use as cement replacement were highlighted. Flash calciners and rotary kilns (see Figure 4) were assessed viewing their impact on the reactivity of the calcined material. Modelling of clay calcination was also discussed, as well as colour control of the calcined product [12].



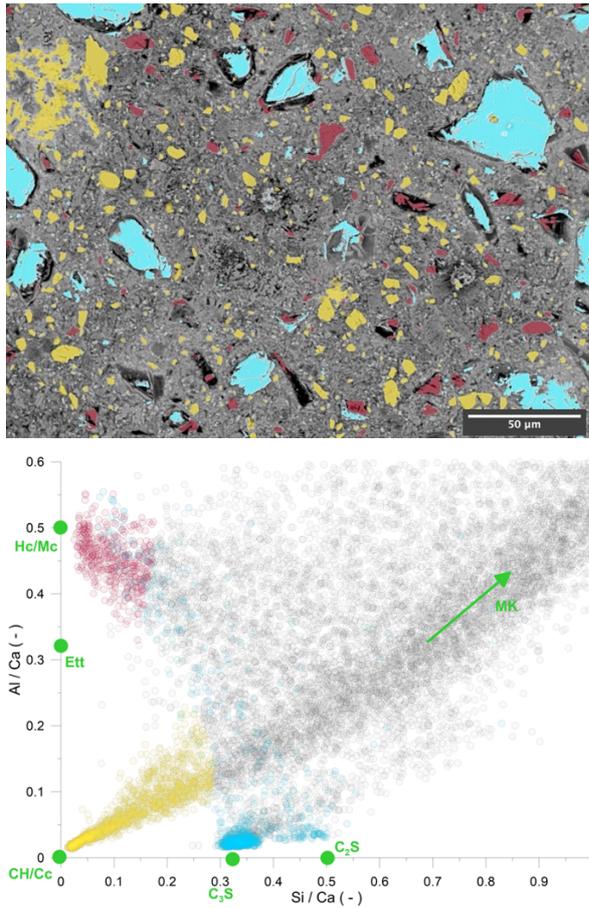
**Figure 4.** Retrofitted rotary kiln for clay calcination at Porto Velho, Brazil [13].

operation. The study highlighted the role of calcined clay, especially in a context where other SCMs are scarce or have a high price. It also stressed the need of converting old clinker kilns into clay calciners, as a low CAPEX alternative, and the need to move to alternative fuels in the new endeavours [13].

### 2.3 Hydration of systems containing calcined clay and limestone

The impact of the use of calcined clays in blended cements was discussed. Hydration follows a similar pattern to Portland cement in terms of hydration, space filling and mechanical strength development (example shown in Figure 5). Portlandite is consumed by the pozzolanic reaction of calcined clay. The composition of C-(A)-S-H changes, incorporating Al in its structure and with a lower Ca/Si compared with the C-S-H observed in plain cement. In the presence of limestone, a higher amount of carboaluminate hydrates is obtained thanks to the alumina provided by calcined clay [14].

A second paper discussed the experiences at industrial level on clay calcination based on the evaluation of existing pilot projects and some few industrial projects on commercial



**Figure 5.** Top: Pore filling due to formation of carboaluminate hydration production in limestone-calcined clay combination. Bottom: corresponding chemical composition of the microstructure, as reported in [20]. Note: Portlandite + limestone in yellow, Hc + Mc in red, anhydrous cement grains in cyan [14].

The mechanism of hydration was also discussed. The reaction of calcined clay slows down because of the lack of (large) solution-saturated pores. The precipitation of hydrates continues but much more slowly in the fine saturated pores. Higher supersaturation is required to compensate for the higher curvature of the smaller crystal. The precipitation may also keep occurring in the pore solution film lining the surface of the pores [14].

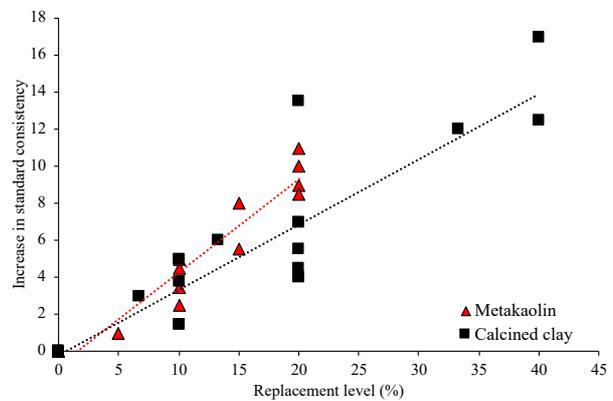
Methods to monitor the reaction were also discussed. Solid state NMR and PONKCS are the most direct methods. Mass balance and thermodynamic modelling are both suitable, but the results depend on reliable inputs from XRD-Rietveld (and SEM-EDS for C-A-S-H composition for mass balance). Other methods are not recommended due to their lack of precision [14]. Other factors, such as the type of cement used, were also discussed. Overall, it was concluded that a highly reactive clinker or cement, with  $N_{a_{eq}}$  in the range of 0.3-0.6 mass-% can provide higher early strength in such systems.

## 2.4 Applications

The impact of calcined clays with/without limestone on the fresh and hardened properties of concrete were discussed.

The fresh properties of concrete are affected by calcined clay particle size and surface area, internal porosity in sintered particles, and presence of incompletely calcined 2:1 clays or mineral impurities, with an impact on slump, and increase in the yield stress, viscosity, and thixotropy of cementitious systems. This might increase the need for admixtures.

Common PCE structures may be considered efficient enough for PC-calcined clay blends to provide control of the initial fresh state properties. However, new variants of PCE in modified forms were found to produce improved retention behaviour. The polymer structure has the same influence as on straight PC systems; hence, an adsorption-electrosteric repulsion dual mechanism is still involved. However, most studies show that the polymer consumption is higher than for PC systems, most probably as a result of the much (several times) higher specific surface area and faster reactivity of calcined clays, which is indicated by the increased standard consistency as shown in Figure 6 [16].



**Figure 6.** Increase in standard consistency of cement pastes with CC or MK. Note: CC – if  $A/S < 0.7$ ; MK if  $A/S > 0.7$  [16].

Calcined clay systems have produced conflicting changes in setting time compared to straight PC systems as measured by the Vicat needle test. However, this is primary due to higher w/b than control mixtures to achieve a standard consistency. When measured on mortar or concrete, calcined clay without any superplasticizers can reduce setting time. A slump retainer, i.e. a retarding agent could be used to control setting behaviour of calcined clay and calcined clay-limestone binders.

The presence of calcined clays modifies the hardened properties of concrete. Calcined clay strength was shown to be a function of the kaolinite content in the clay. Calcined clay with at least 40% kaolinite content can be used to produce concrete with similar 28-day strengths as control Portland cement only with up to 30 mass-% replacement of calcined clay and 15 mass-% of limestone. Early age strength, such as 1 day strength are lower than in PC concrete, but from 3 days on and up to 28 days it can meet or exceed the strength of concrete made only with Portland cement [15].

Creep and shrinkage appear to be a function of the clay kaolinite content and cement replacement level. Lower reactivity calcined clays used at high replacement percentages significantly increase the concrete creep, while low-to-moderate replacement levels of high-purity

metakaolin appear to reduce creep compliance. Calcined clay usage tends to reduce concrete drying shrinkage, partly because of the water absorption properties and dense microstructure of the material [15].

Calcined clay has been defined in many national material standards as a manufactured natural pozzolan with minimum pozzolanic activity performance standards and some compositional standards such as LOI and  $SO_3$  limits. Standards also exist in many locations to use calcined clay in a blended cement, with many also allowing it to be used in combination with limestone fines to make an LC3 material. [17].

Some standards or building codes have limits on the maximum SCM or limestone fine replacement levels that limit the amount of clinker reduction that can be obtained. Current building codes often contain limits on w/cm, strength, or minimum cement contents for durability. Because calcined clays may increase water demand but also improve the transport properties, concrete could potentially be made with a higher w/cm without compromising durability. Adoption of performance-based standards in national codes for durability in lieu of these prescriptive requirements would allow for calcined clay to be used more economically while still protecting the safety of the public [17].

## 2.5 Durability

The impact of calcined clays on the durability performance of concrete in binary and ternary blended cementitious systems was assessed. The pore structure alteration due to the replacement of cement by calcined clays was explicitly highlighted as the main contributor to the observed transport properties of such systems. Calcined clay modifies the pore structure by the refinement of sizes rather than reducing the total pore volume. Due to pore refinement, durability indices such as resistivity, chloride diffusivity (see Figure 8), and absorption rate are improved significantly in calcined clay concretes, more specifically in concretes prepared with kaolinite clay. When kaolinite content in the raw clay was above 40%, there is no significant difference in pore structure in terms of the pore size distribution [18].

The carbonation rate in calcined clay concrete is expected to increase with clay replacement level. A statistical analysis of existing data showed that the carbonation rate could be 3–4 times higher than plain cement concrete. Due to the limited portlandite reserve, decalcification of C-(A-)S-H and other  $AF_t$  and  $AF_m$  phases is the primary interaction with atmospheric  $CO_2$  in calcined clay systems. [18].

Sulphate resistance improved for calcined clay systems in sodium sulphate exposure for both kaolinite and non-kaolinite clays. The addition of limestone with calcined clay did not lead to any thaumasite attack as in PLC. Limited studies exist on seawater exposure, physical sulphate attack, magnesium sulphate attack for calcined clay systems, which could be explored in further research on leaching and decalcification of alumina rich low Ca/Si ratio C-A-S-H expected to form in calcined clay systems [18].

The tendencies for ASR were effectively reduced by calcined clay addition with metakaolin (at lower replacement level of

10–15%) and calcined clay–limestone combination. The use of non-kaolinite clay for ASR reduction is not well reported. While the refined pore structure is seen to have a significant impact on most durability properties, it is seen that certain phenomena such as carbonation and magnesium sulphate attack result in reduced performance compared with plain OPC systems, owing to the lack of the calcium hydroxide buffer that is typically present in plain cement systems [18].

In the specific case of chloride attack, the use of calcined clay has a significant impact. Calcined clays can be an ideal SCM for use in chloride environments with excellent chloride resistance. This is due to a combination of factors including pore refinement, lower pore solution conductivity and changes in chloride binding characteristics. In general, the performance of calcined kaolinite clay with about 40–50% kaolinite content is certainly improved compared to OPC, and for calcined kaolinitic clay with 50–70% kaolinite content, the performance is significantly improved [18].

Studies on cement paste, mortar or concrete all converge to show nearly an order of magnitude lower chloride diffusion coefficient for calcined clay system, highlighting the dominant role of pore refinement in the binding matrix. Concrete containing calcined clay (with 50–70% kaolinite content in clays) is typically found to be less dependent on curing than other blended cement concrete containing fly ashes or slags [18].

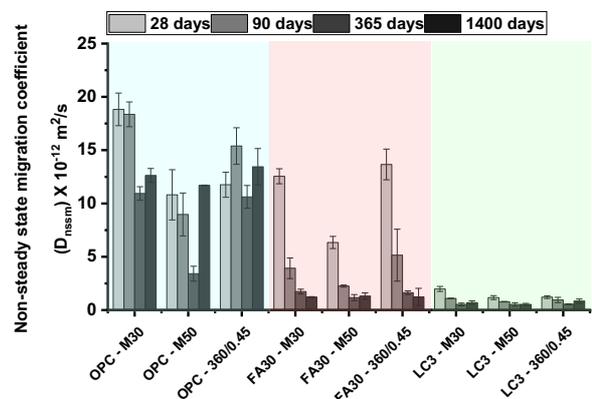


Figure 7. Chloride resistance of different strength grades of concrete with plain PC, fly ash replaced PC and LC3 [19].

Calcined clay-based concretes with binary or ternary blended cementitious systems (i.e., with and without limestone) show promising results with significantly delay in the onset of corrosion, i.e., longer corrosion initiation despite reduced chloride threshold, and also lower corrosion rate which highlight the benefits of using calcined clay concrete in marine exposure conditions using existing service life estimation tools utilized in the concrete industry [18].

## 3 Research gaps

There are still many research gaps that are clearly identified in each of the white papers published. These are:

- The requirements for kaolinitic clay resources for use as SCM have not yet been integrated in systematic geological resource surveys, not even at regional levels. As soil profiles typically cover only several meters of

- depth, it is clear that very large subsoil reserves would be ignored. The challenge to define selection criteria case for 2:1 clay mineral resources is likely to be formidable, if not unfeasible, as there is many 2:1 clay minerals and even mixed-layer clay minerals that are relatively abundant. Further research on the use of 2:1 clays is much needed since there are countries where these are the only clay resources available. [11].
- The process of clay calcination is still not completely understood. There are areas which need further investigation, such as
    - o Interaction of clays with moisture, drying and grinding process and electrical interactions during grinding clay for preparation for calcination.
    - o The influence of cooling and heating rates on clay reactivity, the influence of process atmosphere on clay calcination (other than colour control), the calcination of 2:1 clays for use as SCMs, thermodynamic and kinetic data derivation for clay calcination and hydration, novel techniques for the stabilisation of magnetite and/or the separation of iron from raw or calcined clay, large-scale application of microwave calcination, design and development of electric or solar powered furnaces for clay calcination, and other ways of activation such as mechano-chemical activation [12].
  - Understanding the hydration of cements with calcined clays still lacks information and research on areas such as the relationship between 1:1 and 2:1 clays and how it can influence the hydration, the sulphate aluminate interactions at early ages, the impact of early reactivity on hydration of multiple blends, the impact of dolomite as source of carbonates, the need to get equivalent strength level at 2 days, the control of particle size during grinding, the use of calcined clays in other types of cements, such as calcium sulfoaluminate cements (CSAs), calcium aluminate cements (CACs) and alkali-activated cements (AAC), and the properties of ternary blends containing clinker, calcined clay and slags or fly ashes or natural pozzolans [14].
  - In terms of fresh properties of concrete containing calcined clays, the areas identified for further research include a better understanding of the physical chemistry, the ionic conditions of the pore solution and the microscopic phenomena at the origins of workability loss (agglomeration, thixotropy, yield stress increase) which may lead to more efficient molecules, and thus a lower admixture cost; we also have to consider the role of ettringite vs CASH precipitation at early ages, which may be significantly impacted if we for example utilise 2:1 clays as well; research on the mechanism of slump loss in concrete is needed, including if thixotropic or yield stress growth is responsible; study on the impact of calcined clay as retarders; systematic work on the effects of calcined clay on bleeding is needed. This should include a fundamental understanding of the interaction of calcined clays and water, including agglomeration [16].

- In terms of hardened properties of concrete containing calcined clays, the areas identified for future research include the need to establish a clear impact of parameters such as kaolinite content of original clay, calcined clay–limestone ratio, gypsum content, clinker substitution rate, and w/cm ratio on strength of concrete [15].
- The barriers for the standardization of low and ultra-low clinker cements need to be addressed. Many standards have legacy requirements for durability such as maximum w/cm limits that may have worked well with Portland cement, fly ash, or slag cement, but may be hindering adoption of calcined clay; further, the role of available calcium needs to be addressed, since consumption of portlandite may reduce the performance gain and reactivity of the clays in the respective cements. There are also barriers in the application of new, lower carbon cements in concrete or mortar applications in specific exposure classes, and the approval process can take years, even if the cement composition is already covered by standards [17].
- The challenge for durability is to better understand transport properties of concrete containing calcined clay and how it can influence the performance of concrete in different environments (chloride, carbonation, sulphate, ASR), with strong emphasis on long term durability.

#### 4 Final comments

The work of the RILEM TC 282 – CCL: Calcined Clays as Supplementary Cementitious Materials has contributed to a better knowledge and understanding of the role of calcined clay in cement and concrete. The large availability of suitable clays makes them a viable alternative for SCM in a context where other SCMs (PFA, GGBS) are slowly getting depleted in several parts of the world.

The main process for clays is calcination under fixed parameters. There is a great variety of technological options in place for clay calcination that will enable a swift development of the industry in the forthcoming years. The hydration mechanisms of cements containing calcined clays are well understood, and the similarities and differences with traditional cements have been established. Cements containing calcined clays mainly behave similarly to a traditional Portland blended cement.

The roll out of industrial projects poses challenges for the use of cements containing calcined clays, such as the use of powerful admixtures to counteract increase in water demand, but very good mechanical performance. The use of calcined clays is widely accepted in the standards almost everywhere. Current clinker content allowed is in a range between 40-50%, for ternary binders containing limestone (LC3).

Durability studies present excellent performance of concrete containing calcined clay, with special emphasis on chloride aggressive environments.

#### Authorship statement (CRedit)

**Fernando Martirena:** Conceptualization, Writing – Original Draft, Writing – Review & Editing. **Manu Santhanam:**

Conceptualization, Writing – Original Draft, Writing – Review & Editing.

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