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# Opening Letter of RILEM TC-MCA: Mechano-chemical activation of cementitious constituents

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This article presents an opening scientific statement of RILEM TC MCA.

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## Abstract

Mechano-chemical activation has gained attention in recent years for its capacity to increase the reactivity of cementitious constituents and valorise waste streams. We define mechano-chemical activation as a change in the structure and/or chemistry of a material which accelerates its dissolution and participation in subsequent formation of reaction products, that is caused via mechanical energy supplied through a mechanical treatment. The potential advantages of mechano-chemical activation have driven growing numbers of promising laboratory-scale studies on numerous materials. Yet several knowledge gaps still need to be resolved. We highlight five key knowledge gaps in this topic: the a priori susceptibility of different material categories to mechano-chemical activation; influence of processing parameters across mill types; structural/property indicators; performance indicators; and, processing-property-performance relationships. The approach of TC-MCA is to carry out interlaboratory testing experiments to resolve specific outstanding questions within these knowledge gaps, that are challenging to address by a single laboratory. Improved understanding of mechano-chemical activation at the laboratory scale is a necessary preliminary step to assess its likely value at industrial scale.

**Keywords:** Mechano-chemical activation; Reactivity; Supplementary cementitious materials; Waste streams; Byproducts.

## 1 Aim and scope

This article presents an opening scientific statement of RILEM TC-MCA. The aim is to introduce the motivation, conceptual framing, and intended direction for RILEM TC-MCA, rather than to summarise existing knowledge. In the following sections we outline: definitions and terminology around mechano-chemical activation; the distinction between mechanical treatment and mechano-chemical activation; the rationale and potential benefits for using mechano-chemically activated materials in cementitious systems; the current status and outlook for mechanically-treated cementitious constituents; a description of outstanding research gaps; and, the intended aims and scope of TC-MCA. This opening letter is not conceived as a review. Where detailed reviews on a topic already exist, we refer to these; where reviews on a topic do not already exist, we identify these areas as future opportunities for the work of the TC, rather than addressing them here. The perspectives presented in this letter reflect the views of the authors and have been reviewed and endorsed through the approval process of the members of RILEM TC-MCA.

## 2 Definitions of key terms

### 2.1 Mechano-chemical activation

Numerous definitions of "mechanochemistry" and "mechano-chemical reactions" are found within sub-disciplines of Chemistry. Unsurprisingly, there is no unanimously agreed definition. Proposed definitions are typically quite specific in terms of mechanistic description, e.g. "reaction that is induced by the direct absorption of mechanical energy" [1]. Yet in practice, use of the term mechanochemistry in research "has grown to include any transformation that is observed during or after any type of mechanical treatment, regardless of the exact role of the mechanical action." [2].

The versatility of mechanochemistry has led to a surge of interest in recent years across numerous fields, including pharmaceuticals [3], photovoltaics [4] and polymers [5].

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Increasing interest in mechanochemistry is partly driven by the potential to improve upon conventional methods, such as reducing waste generation, improving energy efficiency and avoiding solvent use for syntheses [6]. Yet mechanochemistry itself is not new [7]. The first known mechano-chemical reaction is proposed to be the reduction of mercury sulfide to mercury via grinding, recorded in 315 BCE [8].

In the context of cementitious materials, activation refers to a process which increases the reactivity of a constituent in a cementitious system. In turn, reactivity is the extent of dissolution and participation in reaction product formation.

We define mechano-chemical activation in the context of cementitious materials, to be: A change in the structure and/or chemistry of a material which accelerates its dissolution and participation in subsequent formation of reaction products, that is caused via mechanical energy supplied through a mechanical treatment. The first part of this definition describes the mechanism (i.e. a change in the structure and/or chemistry of a material) which hence results in activation (i.e. the acceleration of its dissolution and participation in subsequent formation of reaction products). The second part of this definition refers to the nature of the energy supplied and the type of treatment (i.e. mechanical), distinct from other activation treatments (e.g. thermal, chemical). The terms 'structure' and 'chemistry' are used here sensu lato, given that the precise determinants of reactivity are not yet completely understood. However, from current knowledge, relevant changes regarding structure are expected to include: distribution of coordination states; concentrations of defects; and, types of defects. Relevant changes regarding chemistry are expected to include: chemical composition; character of bonding; and, surface charge.

Using this two-part definition, we can 1) describe an experimental procedure that involves a mechanical treatment (intended to achieve activation), and 2) describe whether the mechanism of activation is demonstrably mechano-chemical in nature, through the characterisation of the processed material and its performance in a cementitious system (Figure 1). This conscious distinction between treatment and mechanism enables us to avoid using mechano-chemical activation as a catch-all term to 'include any performance improvement that is observed after any type of mechanical treatment, regardless of the exact role of the mechanical action' (purposefully amending the original statement of [2]). Whilst some degree of comminution usually occurs in parallel, this is not the defining feature of mechano-chemical activation. When performance improvements are attributed to size and surface area effects alone, we recommend to use the term 'mechanical activation' (see details in Section 2.2).

Our aim here is to offer a framing that helps authors and readers separate the description of a treatment (what was done) from the description of a mechanism (what changed and how it affected reactivity). We suggest here our guidelines for terminology that can help with the conscious choice of terminology around mechano-chemical activation, within the field of cementitious materials. The long history and multi-disciplinary interest in mechanochemistry shows the ongoing debates around terminology [2]. Given the existing range of terminology and definitions in the field, there is a risk that more nomenclature simply leads to more noise. So, why do we think there is value in proposing a definition that is specific to the field of cementitious materials?

First, much of the mechanochemistry literature has been developed for synthesis reactions (e.g. A + B → C), whereas many of the relevant mechano-chemical reactions for cementitious materials are decomposition or amorphisation reactions which do not involve syntheses . Second, the term "activation" in general chemistry has a specific, yet application-agnostic meaning: "Input of external energy into a chemical system is said to bring about activation of the system" [9]. Whereas for cementitious materials, activation is used in a more application-specific sense, as described above. Third, the term "activation" also has ambiguity within the field of cementitious materials. For example, in alkali-activated materials the term activation refers to the processes of dissolution and polycondensation, rather than a treatment to increase reactivity of a constituent. Fourth, the majority of activation research for cementitious materials follows an experimental structure of 'Investigate effect of treatment X on characteristics of material Y, and then test its performance in cementitious system Z'. In this type of exploratory research, the activation treatment is known a priori (via choice of process equipment); whereas often it is not possible to state with confidence whether the activation mechanism is mechano-chemical sensu stricto. This is particularly the case for investigations of complex mineral resources, in which accurately describing the change in mineral characteristics is challenging; and, in studies which infer activation solely from mortar strength results. Hence, for the cementitious materials community, we believe there is value in distinguishing between descriptions of activation mechanism and activation treatment.

The use of a hyphen (i.e. mechano-chemical, instead of mechanochemical") is a matter of personal taste. We prefer the use of a hyphen, as 1) it aligns with existing International Union of Pure and Applied Chemistry (IUPAC) terminology for "mechano-chemical reaction", and 2) it helps to 'future-proof' terminology for emerging developments in sequential activation treatments. For example, a 'thermo-mechano-chemical treatment' is more legible than a 'thermomechanochemical treatment'.

## 2.2 Mechanical activation

The previous section established why reactivity must frame any discussion of activation; now we return to the practical question of how mechanical treatments differ from mechano-chemical activation.

A common question is 'how does mechano-chemical activation differ from 'just grinding' ?'. The aim, and mechanism, of conventional grinding is comminution, i.e. reducing particle size to increase surface area (Figure 1C). In many cases, comminution alone does not meaningfully alter

structure or chemistry. Nonetheless, an increase in specific surface area typically increases reactivity. For the context of cementitious materials, we define mechanical activation as "a mechanical treatment that increases the reactivity of a cementitious constituent via reduction in particle size, but does not cause any meaningful chemical or structural changes beyond surface defects" [1].

In other cases, a mechanical treatment can also introduce detectable and non-negligible changes in material chemistry and/or structure (inc. defects, strain, partial amorphisation, or surface chemistry changes) which increase reactivity. This type of treatment would be classified as mechano-chemical activation (Figure 1B). We therefore view mechanical treatments as a continuum: at one end, outcomes are dominated by comminution (surface-dominated); at the other, by structural or chemical modification (bulk-dominated) that changes how the material dissolves and participates in product formation.

We provide these definitions to clarify how we conceptualise mechano-chemical activation within the scope of this TC. In practice, defining the outcomes of a given mechanical treatment as mechano-chemical activation treatment sensu stricto may require minimum thresholds for the extent of measured changes in structural aspects and reactivity. However, in this opening letter we use these definitions sensu lato, mainly to set the boundaries of investigation for this TC. In some studies, the term 'mechanical activation' is used to refer to mechano-chemical activation (as we define here) [10, 11]. Conversely, other prior studies use the term 'mechano-chemical activation' or 'mechanochemical effects' to describe mechanical treatments which do not fulfill our definition here. For example, using grinding to accelerate the ambient carbonation reactions of slags [12, 13]. From the evidence presented in these studies, the carbonation reaction here is not caused by the input of mechanical energy; it is just accelerated, as the grinding process creates new surface area which can be carbonated. We would therefore describe this mechanical treatment as 'grinding-accelerated carbonation' – mechanical activation, that increases the rate of an ambient (not mechanically-induced) carbonation reaction. In contrast, some criteria for mechano-chemical activation.

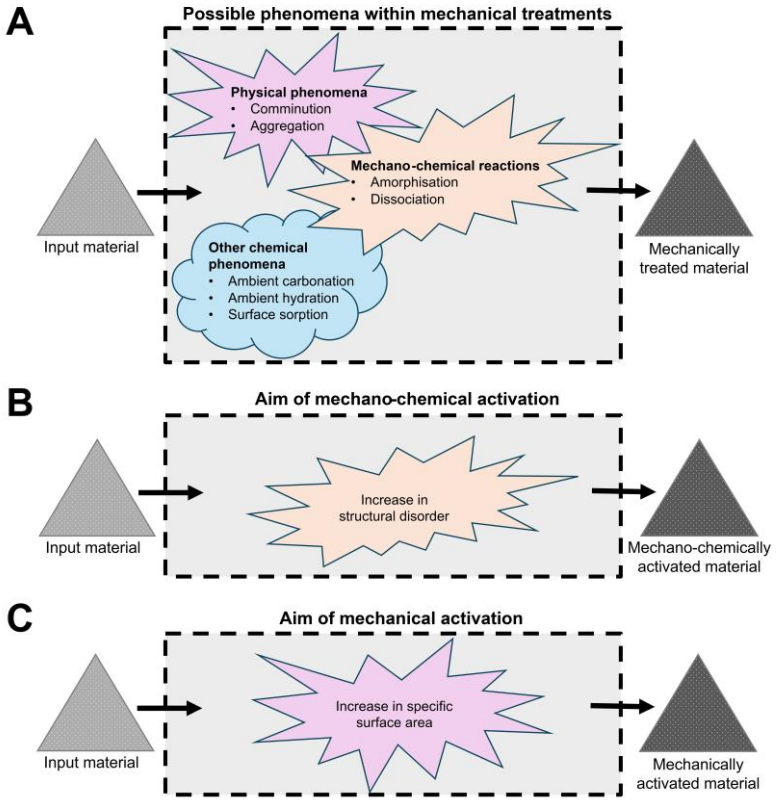



**Figure 1.** A) During a mechanical treatment of a material, several phenomena can take place – these can include mechanical-chemical reactions, but are not limited to these. The aims of B) mechano-chemical activation and C) mechanical activation are primarily limited to specific phenomena.

[1] In the existing mechanochemistry literature, the term mechanical activation "*is widely used in relation to thermal reactions that are facilitated by mechanical pre-treatment*" (Michalchuk et al., 2021): this existing use is distinct to how the term is used here in the context of cementitious materials.

These studies on recycled concrete fines show evidence for how calcium carbonate polymorphs are structurally disordered during the grinding process [15] (fulfilling the 'mechano-chemical' part of our definition), and how these disordered calcium carbonate phases then react more quickly with available Al in a blended cement [14] (fulfilling the 'activation' aspect of our definition).

We do not suggest to revisit every prior study which has used the term mechano-chemical activation, and test whether it meets the definition proposed here. Research studies are useful for the value of their findings, regardless of the terminology used. The precision and practical use of terminology across applications naturally evolves over time. However, the examples above demonstrate the ambiguities in the use of the term 'mechano-chemical' which have proliferated in the absence of a definition tailored to the field of cementitious materials. As interest in this topic continues to increase, we believe that the definitions proposed above can help bring more clarity within the community for communicating research aims and findings.

## 3 Applications of mechano-chemical activation for cementitious materials

There are two main potential routes to impact from using mechanical treatments for activation of cementitious constituents (Figure 2). Here, we define 'cementitious constituents' as solid materials used to make anhydrous cements: this term is inclusive of clinker, SCMs, fillers, and other solids which do not fall into the previous categories.

For conventional cementitious constituents, mechanical treatments have the potential to accelerate their reactions in hydrating cementitious systems. This has been widely demonstrated for mechano-chemically activated clays (in comparison to thermally activated clays) [17, 18], and for mechano-chemically activated clinker phases (in comparison to conventionally ground clinker phases) [19-21]. Whilst not yet widely validated in terms of mechanical properties, this route has the potential to lower the minimum viable clinker factor by achieving higher strength development at early ages (i.e. ≤2 d). In turn, lower clinker factor in blended cements (for equivalent strength development) can lead to lower embodied carbon per unit volume of concrete.

For inert or low-reactivity cementitious constituents, mechanical treatments have the potential to substantially increase their reactivity. This has been demonstrated for micaceous minerals [11, 22], natural pozzolans [23, 24] and slags [25]. The primary route to impact of this approach is to valorise by-products in higher value products (e.g. SCMs) than they currently are used for (e.g. backfill materials, aggregates); or, to valorise materials as products which would otherwise be treated as wastes (e.g. excavation waste, mining waste). Thus, this approach can help resolve two industrial challenges simultaneously: 1) reducing the volume of mineral wastes going to landfill, and 2) increasing the global supply of SCMs.

A commonly encountered question is 'why do we need mechano-chemical activation when we already have thermal activation (i.e. calcination) ?'. This question is most commonly encountered within the field of calcined clays: since clay calcination is already used at scale, is mechano-chemical activation not simply a solution in search of a problem? The question can be answered in two parts – one specific to activated clays, and the second applicable to cementitious constituents in general.

For activated clays, calcination is a widely used and scalable process that is highly effective for kaolinitic clays. It is also well-known that clays with a non-kaolinitic mineralogy (e.g. smectitic, illitic) typically have substantially lower pozzolanic reactivity than kaolinitic clays once calcined [26-29]. For regions in which non-kaolinitic clays are widespread, mechano-chemical activation could give better reactivity performance for the clays available. Furthermore, mechano-chemical activation offers potential advantages over calcination in specific aspects, e.g. accelerated early age dissolution of the activated clay, avoiding process emissions from the decomposition of carbonate associated minerals, the use of an electrified process, and avoiding the need for a subsequent grinding step.

For cementitious constituents more broadly, thermal activation is well-established for clays and has also been used for waste concrete fines (i.e. hydrated cement paste) [30]. However, these two mineral resources represent special cases in which a thermal treatment induces a decomposition reaction which leads to higher reactivity; for the majority of other cementitious constituents (and inert or low-reactivity waste streams), thermal treatments are not effective at increasing reactivity. In contrast, the amorphisation of most minerals via a mechanical treatment increases their reactivity to some degree.

Whilst thermal activation is highly effective and scalable for kaolinitic clays, and will undoubtedly make up the market majority of activation treatments in the future, mechano-chemical activation is in principle more widely applicable to a diverse range of mineral streams.

## 4 Current knowledge

The effects of prolonged and/or extended mechanical treatments on the structure and properties of minerals have been studied for many years, for a variety of applications. Several pioneering mechano-chemical activation studies relevant to construction materials were carried out in the second half of the 20th century; unfortunately, due to a lack of English translations and online accessibility, many of these studies are not widely known (See [31] and references within). Nonetheless, whilst numerous existing studies show proof-of-concept or provide characterisation data, knowledge is still relatively limited regarding the applications of mechano-chemically activated materials in cementitious systems.

In the following two sub-sections, we give a brief description of the mechano-chemical activation mechanisms most relevant to cementitious constituents, and then provide a brief overview of what is already known for different categories of cementitious constituents.

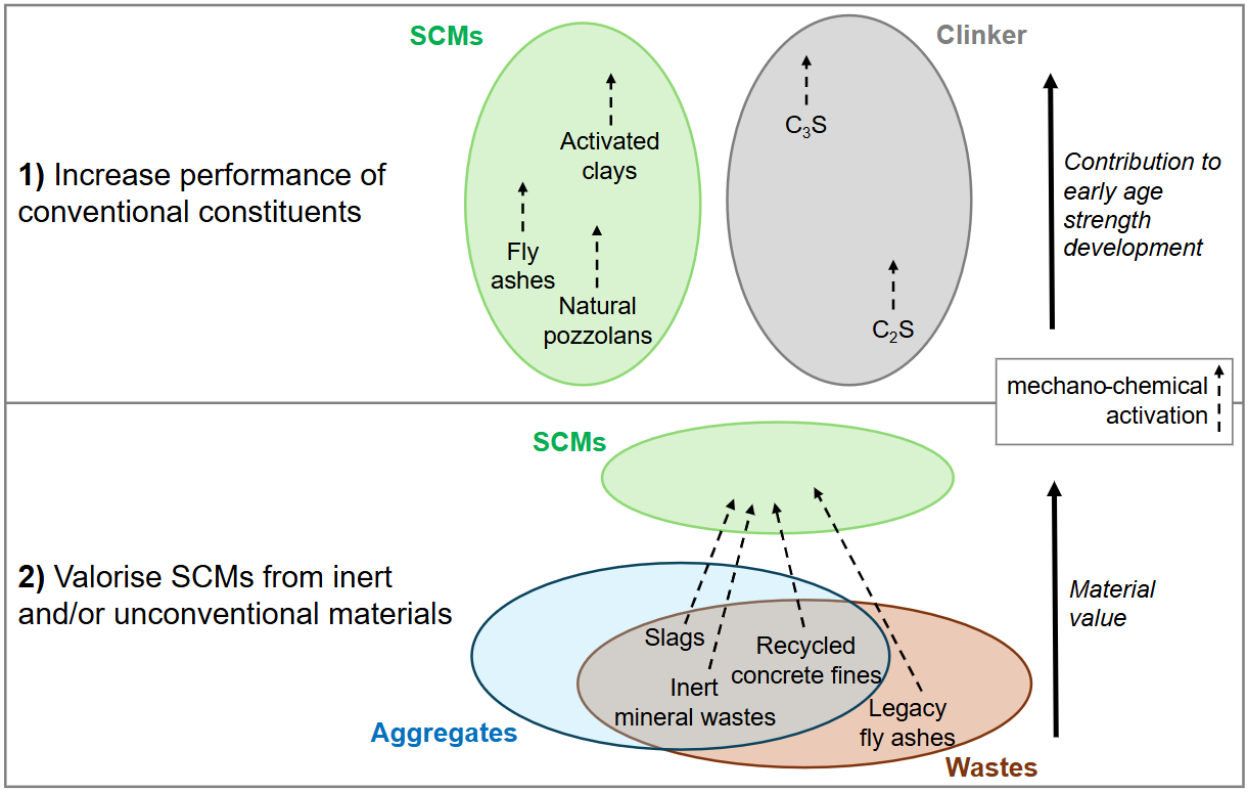



**Figure 2**. Mechano-chemical activation through mechanical treatments can enhance the value of cementitious constituents in two main ways: increasing the early age strength development of conventional constituents, and valorising SCMs from materials typically used as aggregates or left as wastes.

## 4.1 Mechanisms of mechano-chemical activation

### 4.1.1 Mechanically-induced amorphisation

Over prolonged and/or intensive mechanical treatments, minerals accumulate structural defects and amorphous regions develop. Quartz is a simple mineral whose mechanically-induced amorphisation has been well-studied (early work is reviewed in detail in Chapter 5 of [31], and [32]), and is also highly relevant for cementitious constituents. Here, using the example of quartz, we explain the basic thermodynamic principles that determine how mechanical treatments lead to amorphisation, and how amorphisation leads to faster dissolution kinetics.

The basic principle of mechanically-induced amorphisation has been known for >100 years: in their experiments on quartz, Ray described as early as 1921 how "the action of grinding modifies the crystalline material, and partially converts it into the vitreous form" [33]. This phenomenon is not limited to a specific set of loading conditions. At one extreme, complete amorphisation of quartz has been achieved at pressures >30 GPa under quasihydrostatic loading conditions in a diamond anvil cell [34]; at the other extreme, partial amorphisation of quartz is achievable over prolonged grinding periods using a simple automated agate mortar grinder [35]. However, the mode of mechanical treatment does affect the type and distribution of defects generated. For grinding treatments (distinct from shock loading and diamond anvil cell experiments), Si-O covalent bonds are broken; this destroys long range order in the surface layer of quartz particles [36]. The radicals (i.e. unpaired valence electrons) generated by Si-O bond rupture can then react with $O_2$ and/or $H_2O$ depending on the grinding atmosphere [37]. The end result of a prolonged mechanical treatment is a mechanically-amorphised surface layer in the quartz particles.

Amorphous silica has a less negative enthalpy of formation, and a higher entropy of formation, compared to quartz [38]. The enthalpy change leads to a less negative free energy, whereas the entropy change leads to a more negative free energy; in this case, the effect of the enthalpy change outweighs the effect of the entropy change, leading to a smaller free energy of formation of amorphous silica compared to quartz [38]. Consequently amorphous silica has a higher free energy of solution compared to quartz (by 9.4 kJ/mol) [38]. As a result, the dissolution rate of amorphous silica is substantially higher than quartz in alkaline solutions [39]; hence, quartz can have a substantially higher pozzolanic reactivity after an intensive mechanical treatment [40]. This general effect is widely known in the field of cementitious materials. Quartz is generally considered to be inert; whereas silica fume - also $SiO_2$ but in amorphous form and with an extremely fine particle size - is known to be pozzolanically reactive.

Individual minerals have nuances around their dissolution kinetics; however, the general principles of how mechanically-induced amorphisation ultimately leads to faster dissolution rates in quartz is broadly applicable to other minerals too (Figure 3).

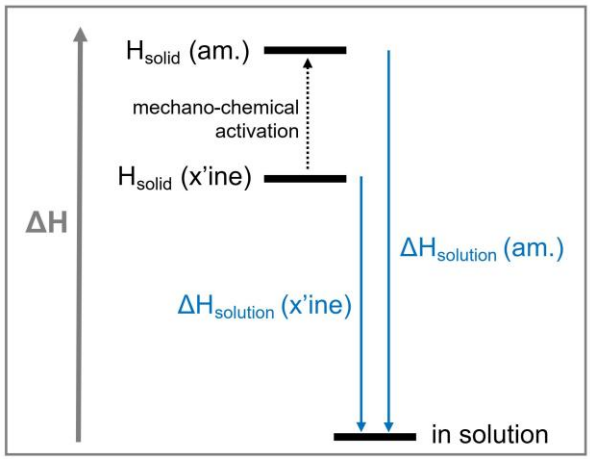



**Figure 3**. An increase in formation enthalpy (Hsolid) after mechanically-induced amorphisation leads to an increase in the enthalpy of solution (ΔHsolution). x'ine = crystalline; am. = amorphous. symbol indicates a mechano-chemical reaction, as developed by [45].

### 4.1.2 Mechanically-induced dissociation reactions

Several dissociation reactions which are achievable by heating can also be achieved via a mechano-chemical route. Mechano-chemical reactions relevant to cementitious constituents include: dehydroxylation of phyllosilicates to form meta phyllosilicates [41]; dehydroxylation of goethite to form hematite [42]; and, decarbonation of calcite to form free lime [43]. The extent and rate of these mechano-chemical reactions are dependent on the milling conditions used, including the milling atmosphere.

The mechano-chemical reaction of highest current interest for cementitious materials is the dehydroxylation of phyllosilicates, as this reaction has the potential to greatly increase pozzolanic reactivity (see sub-section below for more details on this). The currently accepted theory for the mechanism of mechanically-induced dehydroxylation is 'prototropy'. An O–H bond present in an Al–O–H octahedral site is ruptured under a mechanical force and leaves an Al–O site [44]; the radical then reacts with a neighbouring Al–O–H site to form $H_2O$. The change in coordination of Al sites then increases the driving force for dissolution. The mechanisms of other dissociation reactions are less well-investigated; but at a basic level, the activation energy required can be supplied by either thermal or mechanical means. Whilst the effect of these mechanically-induced dissociation reactions on the performance of constituents in cementitious systems is not completely understood, their existence is known.

## 4.2 Mechanically treated materials used in cementitious systems

Many published studies have investigated the application of mechanical treatments to cementitious constituents. Several reviews have been published (cited below), which typically focus on a single type of constituent. The majority of studies have investigated the use of mechanically treated materials in clinker-based cements, but many have used alkali-activated cements. We do not distinguish between the different cementitious systems in terms of assessing the findings of studies; however, we focus our outlook on blended cements due to their much larger current and projected market share. In this sub-section, we give a very brief overview of mechanically treated cementitious constituents which have been previously studied, with an emphasis on commercialisation attempts where known. And, based on current knowledge, we give our outlook on how mechanical treatments are most relevant for each constituent

### 4.2.1 Clays and phyllosilicate minerals

Clays are one of the most widely investigated materials for mechano-chemical activation, and the performance of many mechano-chemically activated clays has been validated at the cement paste and mortar scale. The dehydroxylation mechanism is described above, and reviews describing other aspects of material characteristics and performance can be found in [46, 47]. As of early 2026, thyssenkrupp-Polysius/SCHWENK have already built a pilot plant equipped with a 'booster mill' to produce Meca-clay [48], a mechano-chemically activated clay SCM product.

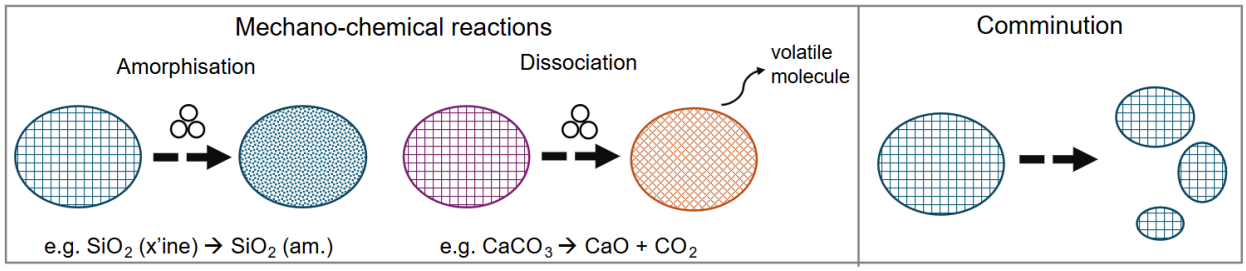



**Figure 4.** Mechano-chemical reactions are a change in the structure and/or chemistry of a material, and are caused by the input of mechanical energy. Comminution involves the creation of fresh surface area, and is not considered to be a mechano-chemical reaction. X'ine = crystalline; am. = amorphous. The 'three ball' symbol indicates a mechano-chemical reaction, as developed by [45].

As described above, thermal activation of kaolinitic clays is highly effective and industrially widespread. From the published evidence currently available, it seems unlikely that mechanical treatments could provide benefits in performance and/or embodied energy which would give a clear advantage over existing calcination routes for kaolinitic clays. However, some circumstances may offer competitive advantage for mechanical treatments. For example, in regions where there is a domestic shortage of SCMs, but there are abundant stocks or flows of mineral wastes rich in non-kaolinitic clays (e.g. excavation waste, dredging waste, mining waste). The effectiveness of mechano-chemical activation for some 2:1 phyllosilicates (particularly muscovite [11, 22, 49]) may give a performance advantage over calcination for valorising such wastes. Such waste-derived mechano-chemically activated clays could also act as a 'stopgap' solution to fulfil increasing demand for activated clay, whilst dedicated quarries for suitable kaolinitic calcined clays are being established (a process that can take up to 20 years).

### 4.2.2 Slags

Whilst mechanical treatment of GGBS has been investigated [50], the majority of research is on mechanical treatment of less reactive slags. These include: steel ladle furnace slag [25] and Cu-Ni slags [51]. Production of any slag can be a substantial volume at the local level; but at the global scale, steel slags are the most significant. Given projected declines in global GGBS availability due to increased use of recycled steel in EAFs, increasing attention is paid to valorising steel slags in higher value applications (i.e. as SCMs rather than aggregates) [52]. The changes in structure and chemistry of slags during mechanical treatment vary widely based on slag chemistry and milling conditions. Carbonation of Ca-bearing phases increases the grindability of slags. There is hence growing interest in mechanical treatments in a $CO_2$-rich environment which can increase reactivity and also result in $CO_2$ uptake by the ground slag [53, 54].

Because slags are typically formed in coarse nodules, there is a greater opportunity for comminution to increase reactivity compared to other constituents which originate as powders (e.g. clays). This can lead to ambiguity around the actual mechanism through which reactivity is increased: for example, referring to enhanced comminution of steel slags using grinding aids, and the subsequent increase in reactivity, as a "mechanochemical effect" [12]. Slags are hence a material category where vigilance is needed around terminology. A reaction that is not caused by the input of mechanical energy, but simply accelerated via use of a mechanical treatment through the ongoing generation of new surface area (e.g. carbonation) is not a mechano-chemical reaction sensu stricto. Nonetheless, for the combined goals of increasing SCM supplies, reducing waste streams and maximising the value of valorisation applications, mechanical treatments to activate slags are a promising area of research.

### 4.2.3 Fly ash and other ashes

The mechanical treatment of fly ash has been investigated in numerous studies, as reviewed by [55-57]. Amorphous phases typically make up a large proportion of fly ashes; mechanical treatments can increase the total amorphous content, via mechanically induced amorphisation of crystalline phases (e.g. quartz, mullite) [56]. However, in some cases the reactivity gains from a mechanical treatment are simply due to increased specific surface area from comminution, and not from any mechano-chemical reactions [58].

Global production of fly ash is projected to decrease as less coal is used as fuel in industrial combustion processes. Yet there remain many large stockpiles of 'legacy fly ash' which have been disposed underground or in open piles. In contrast, production of ashes from other combustion processes (e.g. municipal solid waste incineration) is increasing. In this context, mechanical treatments have two potential benefits.

First, to improve the reactivity of legacy fly ashes and facilitate the valorisation of these waste stockpiles. The weathering process of fly ashes over time leads to formation of portlandite and carbonates [59], which increases their Loss on Ignition (LOI) and is also expected to reduce their reactivity. Nonetheless, legacy fly ashes can still meet standards requirements for fly ashes [60], and so mechanical treatments which improve their reactivity could help lead to market acceptance.

Second, to help immobilisation of heavy metals in combustion ashes. Mechanical treatments have been widely investigated as an immobilisation route for soils contaminated with heavy metals, with mechanisms for heavy metal immobilisation including chemisorption on freshly exposed surface sites, entrapment between aggregated particles, and chelation with organic matter [61]. Mechanical treatments also seem effective for heavy metal immobilisation in both fly ash [62] and municipal solid waste incineration ash [63]. Whilst fly ashes and combustion ashes may not be a major global source of SCMs by volume in the coming years, mechanical treatments offer value from a perspective of circular economy and environmental remediation.

### 4.2.4 Natural 'raw' pozzolans

Natural 'raw' pozzolans are a diverse array of mineral resources, in which the reactive component is usually a silicate or aluminosilicate amorphous phase [64]. Natural pozzolans investigated under mechanical treatments so far include pumice [23, 65] and basaltic fines [66]; [67] has provided a review of majority of reported literature in this topic. Mechanical treatment has the potential to cause mechanically induced amorphisation of crystalline phases (e.g. feldspars [68], zeolites [69]); this can lead to increased reactivity [65, 67, 70] and higher compressive strength [23].

Natural pozzolans are abundant in some world regions, but their use requires the primary extraction of mineral resources. Mechanical treatments could be highly relevant for regions where the use of natural pozzolans is well-established and supplies are abundant (e.g. regions of North America and East Africa); as improving their pozzolanic reactivity could help to reduce minimum viable clinker contents beyond what is currently feasible.

### 4.2.5 Inert minerals and phases

Much general research has been done on the mechanically-induced amorphisation of minerals which would generally be considered inert in a cementitious system (inc. quartz, feldspars, iron oxides); however, relatively little of this existing research is directly relevant to cementitious materials. Beyond a few studies on the mechanical treatment of individual inert minerals (e.g. quartz [40]), the majority of sector-specific studies have been carried out on industrial wastes [71, 72]. A major non waste-derived mineral resource investigated is igneous rocks [23, 66, 73].

The main value of studying individual inert minerals is to gain a better understanding around the potential of mechanical treatments on assemblages of inert minerals (e.g. mining wastes, natural rocks), in order to improve predictive power and avoid repetitive trial and error studies. Reactivity gains can be meaningful in relative terms (i.e. compared to the as-received mineral), but are still modest in absolute terms (i.e. compared to conventional SCMs). In addition, there are outstanding questions about whether it makes sense to expend energy to manufacture a low reactivity SCM from an inert material. These concerns notwithstanding, investigation of the mechanically-induced amorphisation and reactivity of 'inert' minerals is valuable from the perspective of valorising large volumes of industrial mineral wastes.

### 4.2.6 Recycled concrete fines

Similar to low-reactivity slags, recycled concrete fines have been seen as potential replacements for fine aggregates for many years, but there is now increasing focus on valorising them as SCMs. Mineral phases arising from fine aggregates (e.g. quartz, feldspars, phyllosilicates) can be partly amorphised during an intensive mechanical treatment [74-76]. Intensive mechanical treatment of C-S-H in isolation is known to increase its reactivity [77]; however, the effect of mechanical treatments on C-S-H within recycled concrete fines is so far largely unknown. Nonetheless, mechanical treatments do have a beneficial effect on reactivity [78] and compressive strength [74-76].

Also similar to slags, the reactive Ca content of recycled concrete fines (i.e. the calcium silicate hydrate (C-S-H) gel in hydrated cement paste) offers an opportunity for carbonation. Hence, mechanical treatments in a $CO_2$-rich environment are a growing focus for recycled concrete fines [15]. Since grinding is already used to process concrete demolition waste, investigation of mechanical treatments to enhance reactivity and/or accelerate carbon uptake is worthwhile from a waste valorisation and circular economy perspective.

### 4.2.7 Clinker phases

The partial amorphisation of clinker phases during intensive mechanical treatments has been known for many years both for clinker itself [31] and for individual clinker phases [19]. In the previous section, the explanation of mechanically-induced amorphisation used the example of quartz: a thermodynamically stable mineral at ambient conditions, that is transformed to an amorphised state with a higher enthalpy of formation. However, the metastable nature of clinker phases leads to behaviours which deviate from this simple example. The transition of β-$C_2S$ to γ-$C_2S$ can be achieved by mechanical treatment ([79] cited in [31]); here, the application of mechanical energy leads to formation of a phase that is more thermodynamically stable, not less stable. The hydration reaction is accelerated in amorphised $C_3S$, yet the mechanically-induced amorphisation actually makes $C_3S$ more thermodynamically stable compared to its crystalline form [21]; this leads to a lower heat of hydration in amorphised $C_3S$ [20]. Knowledge from mechanical treatment of other minerals is therefore not completely transferrable to clinker.

There have been several innovative mechanical treatments for clinker, such as 'Energetically modified cements' [80] and similar approaches involving co-grinding clinker with other constituents [81], which improve early age strength development [82]. Again, although the term "mechanochemical" is used to describe the changes which improve performance [81, 83], the mechanisms described are a combination of enhanced comminution and enhanced dispersion. These phenomena are enhanced and facilitated by the mechanical treatment, but as described they are not considered as mechano-chemical reactions sensu stricto.

The benefit of intensive mechanical treatments is clear in terms of hydration kinetics from calorimetry curves. However, it is not yet clear how beneficial the mechanically-induced amorphisation of clinker phases may be for early age strength development, above and beyond the gains achieved simply by finer comminution. Industrial clinker grinding

processes have low energy efficiency, and cement companies are under pressure to reduce the embodied carbon of their clinker. With this in mind, mechano-chemical activation of clinker is unlikely to be one of the 'low-hanging fruit' for cement producers. Yet, for specific products or applications which require a blended cement with extremely low clinker contents, mechano-chemical activation to boost the early age hydration of cement may be a useful approach. Scientifically, exploring the potential of mechanical treatments to engineer clinker reactivity is very interesting, but is unlikely to have as much impact as mechanical treatments for SCMs; as far as the production of clinker needs to be carried out by thermal treatment and cannot be simply replaced by a mechanical treatment.

## 5 Knowledge gaps

From the potential benefits and outlooks for different constituents given above, we next describe five key knowledge gaps that are important for meeting the potential of mechano-chemical activation as a strategy to help facilitate decarbonisation and waste reduction in the cementitious materials sector. These are primarily based on our own personal expertise and informed opinions. Each of the following sub-sections describes a knowledge gap shown in Figure 5.

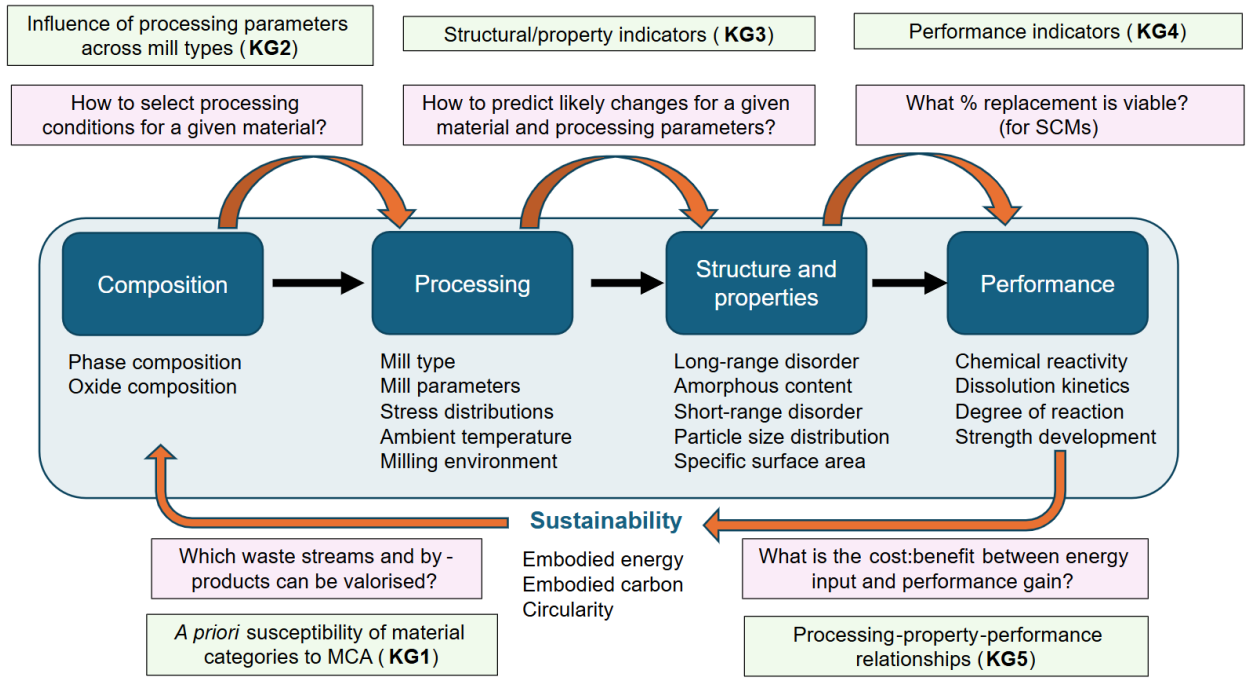



**Figure 5.** Schematic diagram showing the key measurable characteristics regarding mechano-chemical activation of a cementitious constituent divided into topics of: composition, processing, properties, performance and sustainability. Knowledge gaps (KG#) are stated in green boxes and practical questions are stated in pink boxes. These characteristics focus within the boundary of a given cementitious constituent; at the boundary of a cementitious system, other characteristics will also be relevant e.g. rheology of fresh mortars, durability of cured systems. The schematic diagram is based on the 'materials square pyramid' concept developed by Donahue [84].

### 5.1 A priori susceptibility of material categories to mechano-chemical activation

One of the most basic uncertainties is how the inherent characteristics of the material influence whether mechanical treatment leads mainly to comminution or whether it can cause deeper changes in structure or chemistry. Within the laboratory devices and treatment windows considered in the focus area of cementitious materials, there is no widely accepted, predictive pre-screening criterion to judge, before any processing, whether a material is even capable of being pushed into a mechano-chemical regime. This remains a fundamental limitation for interpreting past studies and for planning meaningful experiments.

A first gap concerns mineralogy, because the way different minerals respond to mechanical energy can vary greatly. For layered phyllosilicates, low-severity milling may yield either physical effects or, in some cases, alter hydroxyl groups or interlayer interactions, whereas framework silicates usually maintain their long-range structure unless the treatment becomes more severe. Although these tendencies are generally known, they remain mostly qualitative or semi-quantitative impressions. For example, there is some evidence that susceptibility to amorphization is inversely correlated to minerals' Mohs hardness [85]. We do not yet have well-established, simple criteria that tell us which minerals are likely to undergo structural change under realistic treatment windows, or which minerals will mostly undergo size reduction with minimal modifications. This makes it difficult to judge when mechanical treatment is expected to cross into mechano-chemical activation.

A related gap concerns the prior history of a material. Minerals rarely come into a milling device in a pristine state. They may contain defects created by earlier thermal treatments, weathering, partial hydration during storage, or even by routine handling and transport. These inherited features may make a mineral easier or harder to disorder, but at present we have no systematic way to recognise or quantify this. Two powders that look identical in composition may behave differently, whether this reflects differences in inherited defects remains uncertain here. This can shift the outcome of processing in ways we cannot currently predict, yet it is rarely documented.

A third issue is the behaviour of mixed or complex mineral assemblages, such as those present in clays, excavation waste, industrial byproducts, recycled concrete fines or natural pozzolans. These materials contain several minerals with different responses to mechanical energy, and the interactions between them during treatment are still poorly understood. A component that is "inert" by itself may exhibit different reactivity in a mixture, but whether such changes arise from interactions with more disorder-prone phases is uncertain in our context. Without better understanding of how these mixtures behave, we cannot reliably anticipate which fraction controls the overall response or whether the material as a whole will benefit from mechano-chemical treatment.

Altogether these gaps show that we lack a clear way of assessing the susceptibility of a material to mechano-chemical activation. In practice, it is not established that materials respond similarly to mechanical treatment: their behaviour may depend on mineralogy, inherited structure and the presence of other phases. This limits our ability to design experiments, compare studies, or decide whether a material is a good candidate for mechano-chemical activation.

## 5.2 Influence of processing parameters across mill types

A major gap lies in understanding how the mechanical treatment itself influences the outcome. We know that different milling devices impose different balances of impact, shear and attrition, but we still cannot describe these differences in a way that makes results easy to compare. Simply listing rpm, milling time or a device name does not tell us what kind of mechanical environment the powder actually experienced. As a result, nominally similar settings may not guarantee comparable mechanical environments or outcomes across studies.

One difficulty is that we do not yet know whether treatments can be compared through a single measure of "severity". Work on planetary mills has shown that energy transfer depends strongly on geometry, filling ratio, ball size and motion. Thus, the same amount of time at the same speed may correspond to different stresses on the powder. We do not yet know which operating parameters truly matter for predicting structural change, and which are only incidental.

Another source of uncertainty is the contact history and the micro environment inside the mill. We cannot tell in advance whether a given high-energy treatment can yield only physical (surface-area) effects or will also introduce structural disorder that alters dissolution per unit area, and we do not yet have a simple way to describe these differences for comparison across devices and experiments.

Temperature and atmosphere during milling introduce further complexity across all mill types. Each collision produces some local heating; unless a mill is equipped with an active cooling system the bulk temperature of the powder rises during long treatments. Higher temperature can promote the formation of defects, but it could also accelerate relaxation processes that partially heal them. As a result, two treatments that appear similar may have different effects on the free energy of the processed material. Atmosphere interacts directly with freshly created surface sites. Oxygen can react with radicals formed by bond breakage, and water vapour or humidity (present either in the starting environment within the vessel, or released from the material during milling) can influence reconstruction of the surface. For calcium and/or magnesium-bearing materials, carbon dioxide within the vessel environment (either atmospheric or enriched concentrations) may carbonate the surface during milling. These changes may not be visible in long-range structural measurements, when they are confined to the surface, but they can still influence how the material later dissolves. At present we do not have clear guidance on when the atmosphere or temperature during milling meaningfully affect the outcome, and when they simply create temporary states that disappear once the material enters a cementitious environment. Nonetheless, deliberately engineering the grinding environment (e.g., controlled gases, humidity, or solvent traces) may offer a route to improve effectiveness and energy efficiency (analogous to the role of grinding aids in clinker grinding), but its benefits and limits for MCA remain insufficiently explored and will require systematic, comparable studies.

## 5.3 Structural/property indicators

A central difficulty in understanding mechano-chemical activation is that mechanical treatment causes both physical and structural changes, usually at the same time. Particles become smaller and new surfaces form, but bonds may also break or distort, local structures can rearrange, and in some cases the first signs of amorphisation may develop. When a material shows higher reactivity after milling, we currently cannot separate how much of that change comes from increased surface area and how much comes from genuine structural modification inside the solid.

It is often helpful to distinguish between effects that increase access, such as comminution, and effects that increase stored energy, such as the creation of defects or local disorder. Comminution mainly exposes new surface and shortens diffusion paths, while structural modifications change the internal energy landscape of the material itself. The challenge is that the tools commonly used to analyse processed powders do not allow us to quantify these contributions in a straightforward way.

A further uncertainty comes from how we use the term "amorphous." Loss of long-range order and changes in short-range structure are not the same. A material may lose its long-range periodicity yet still keep most of its local bonding environment, or it may preserve its diffraction pattern while the short range structure becomes distorted. These differences matter because dissolution and early hydrate formation appear to be governed primarily by the local atomic environment. It is also not yet known whether the structural disorder and defects generated during intensive grinding undergo any degree of annealing under the storage conditions and timescales relevant to cementitious constituents.

Another uncertainty is how these structural changes influence behaviour in a real hydration environment. It is not established whether, small variations in defect density or local bonding can alter the early heat release or the identity of the first hydration products, and substantial changes in long-range order may not reliably correspond to differences in performance. Currently, there is no shared understanding of which types of structural changes are most important for the reactions that occur in cementitious systems.

## 5.4 Performance indicators

A major uncertainty is how to interpret changes in the behaviour of mechanically treated materials once they enter a real cementitious environment. At present it is very difficult to tell whether an observed improvement in hydration or early reactivity is driven mainly by an increase in surface area or whether it reflects a genuine structural modification inside the solid. Because mechanical treatments nearly always change both, most measurements blend these effects together.

One of the clearest examples is the use of solution-based reactivity tests such as the $R^3$ method. These tests are widely used, but they do not necessarily reflect whether an observed gain in reactivity reflects increased surface area or genuine structural modification. This makes it difficult to use $R^3$ type results as evidence that a mechano-chemical change has taken place. Other test methods also face distinct challenges to isolate changes in chemical reactivity due to structural modifications. For example, in the strength activity index (SAI) test, contributions to mortar strength development can also arise from enhanced filler effects and particle packing in finely ground materials.

Another uncertainty is that we do not yet know how mechanically-induced disorder influences early hydration in different binder chemistries. This means it is not established whether a structural change that appears to speed dissolution in a Portland system would behave similarly in an alkali activated system. Likewise, a treatment that could seem ineffective in Portland cement systems may still influence gel formation in an alkali-activated binder. At the moment we cannot predict these differences. Regardless of binder chemistry, the effect of using mechano-chemically activated constituents on longer-term performance aspects (e.g. durability) of cementitious systems is still largely unknown.

Lastly, there is relatively little published strength data for cementitious systems incorporating mechano-chemically activated constituents. This is likely due to the limit on how much material can be ground in a planetary ball mill, making it time-consuming to generate sufficient material for mortar production. The relationship between strength and chemical reactivity deserves investigating, as physical contributions to strength development (i.e. filler effect, particle packing effects) may become more significant for intensively ground materials.

## 5.5 Processing-property-performance relationships

Questions around processing-property-performance relationships are inseparable from questions around industrial scalability. To assess the ratio of costs (i.e. embodied energy and carbon) vs. benefits (i.e. potential higher reactivity) for mechano-chemical activation, numerical values are required. Yet, the numerical values obtained from laboratory studies, using laboratory-scale milling devices, are not representative of industrial-scale milling equipment or operational practices. This situation has the potential to lead to stasis – we need representative values for embodied energy to progress to larger scales, but we cannot progress to larger scales until we have representative values for embodied energy.

A useful workaround is to focus on relative trends in processing-property-performance relationships, rather than absolute values. For example: using a given mill type and fixed set of milling conditions, to identify the trends in $R^3$ reactivity vs. embodied energy of milling for a range of cementitious materials. Whilst the absolute values of embodied energy are not useful, the trends can indicate the level of energy input at which a reactivity asymptote is reached [86]. Comparing trends across different material categories can then indicate where mechano-chemical activation gives greatest 'bang for its buck' (i.e. gives the greatest increase in reactivity for the mechanical input energy).

The scalability of mechano-chemical activation is a challenge to be resolved for all relevant manufacturing sectors [87, 88]. The scale of production required in the cementitious materials sectors is orders of magnitude larger than some other sectors in which mechano-chemical activation is being adopted e.g. pharmaceuticals. Scaled-up production of mechano-chemical activation processes by several companies in the cement sector (e.g. thyssenkrupp-Polysius/SCHWENK [48], Celitement[2], Matterup[3]) shows that this topic is not confined to the laboratory. High-energy mills have been upscaled to some extent, e.g. the booster mill, a variety of stirred media mill. It is so far unknown whether existing grinding equipment in the cement sector (e.g. horizontal axis ball mills, vertical roller mills) could be repurposed to achieve similar outcomes as laboratory-scale high-energy milling devices (e.g. planetary ball mills, disk mills).

The sheer scale and cost of industrial-scale milling devices are obstacles to their use in research studies. However, modelling approaches offer a useful route to obtain indicative data. Numerical modelling approaches have been developed for numerous mill types, including planetary ball mills [89] and vibratory disk mills [43]. Adopting these existing models can indicate the conditions under which different mill types would need to operate in order to achieve the energetic regime conducive to mechano-chemical activation.

# 6 Approach of TC MCA

Many specific questions within the knowledge gaps described above can be adequately addressed in conventional studies undertaken in a single laboratory. However, a clear limitation of the conventional 'single laboratory' approach is the lack of comparability between studies, i.e. different materials, different milling equipment, different characterization, different performance measurements. Inter-Laboratory Tests

[2] https://celitement.de/en/

[3] https://www.materrup.com/

are best placed to give comparable datasets to answer some key outstanding questions. The planned approach of TC MCA follows five threads of action that correspond to the five knowledge gaps described above[4].

1. Material susceptibility: to clarify how different materials respond under shared, controlled treatments.
2. Structural indicators: to establish which physical and structural changes can be measured reproducibly across laboratories.
3. Processing environment (mechanical and hydration context): to stabilise and characterise the mechanical and chemical treatment environment across devices and laboratories, enabling meaningful comparison of mechano-chemical responses.
4. Early reactivity and hydration response: to identify early-stage reactivity measures that reliably reflect mechano-chemical modification across representative binder environments.
5. Energy input and activation efficiency: to relate mechanical energy input to the degree of structural change and early reactivity, providing a basis for assessing the efficiency of laboratory-scale activation.

Together, these threads provide a coherent framework that connects material susceptibility, processing conditions, structural change and early performance. The TC therefore concentrates on questions where coordinated testing can reduce uncertainty and support shared understanding. Within this framework, the TC will determine reproducible trends in material response, develop a compact set of processing descriptors that improve comparability, identify early reactivity measures sensitive to mechano-chemical modification, and relate energy input to observed activation. It is anticipated that the knowledge generated through this TC will ultimately support the testing and development of scaled-up mechano-chemical activation processes, and also inform assessments of embodied energy and carbon.

The TC will not attempt comprehensive screening of mineralogical diversity, detailed mechanistic studies at atomic or structural scale, extended hydration or strength testing in mortars and concretes, industrial milling assessments, or optimisation of activated materials for specific binder systems. Hydration measurements are included only insofar as they clarify early chemical/mechano-chemical effects.

## 7 Conclusions and outlook

Mechano-chemical activation is not a new concept, but has drawn increasing attention in recent years for its capacity to increase the reactivity of cementitious constituents and valorise waste streams. These advantages have driven growing numbers of promising laboratory-scale studies on numerous materials. Mechano-chemical activation will not be the most effective, efficient or affordable option for all material streams, in all locations, for all applications. Yet, mechano-chemical activation does not need to be a 'one size fits all' solution in order to be valuable: it is one approach that – used wisely - can help the cementitious materials sectors meet their decarbonisation and circular economy goals.

The majority of research studies within this topic follow a typical approach: grind a new material, characterise its structural and physical changes, measure its reactivity, and test its performance in a cementitious system. This empirical approach is well-suited to exploratory research, in which the potential for mechano-chemical activation of a new material is not yet established. However, the research community soon reaches an asymptote of knowledge. We know what is possible for a specific material and set of processing conditions, but we do not have greater predictive power for what is repeatable beyond this.

Inter-laboratory studies are hence an essential part of the research community's efforts to identify repeatable findings at the laboratory scale. We therefore believe that TC-MCA will make a unique and necessary contribution to the cementitious materials research community, that is necessary to accelerate the progress of mechano-chemical activation in Technology Readiness Level (TRL). Only when pilot industrial scale production is reached (TRL 7) will we find out the true industrial value of mechano-chemical activation. Amongst cementitious constituents, mechano-chemically activated clays have the most extensive track record in research; commercial scale-up of mechano-chemically activated clays is now underway. Other cementitious constituents have been less extensively researched. For these, a better understanding of mechano-chemical activation potential at the laboratory scale is necessary, in order to generate the preliminary evidence required to assess its likely value at industrial scale.

## Authorship statement (CRediT)

**Alastair T.M. Marsh:** Conceptualization; Writing – Original Draft; Writing – Review & Editing; Visualization. **Arezou Babaahmadi:** Conceptualization; Writing – Original Draft; Writing – Review & Editing; Visualization.

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[4] These are planned to be addressed through the four planned working groups (WGs) within the TC: WG1: repeatability and characterisation; WG2: universality of mechano-chemical activation processes; WG3: effects of milling environment; WG4: modelled and measured energy inputs.

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