

The impact of accelerating admixtures on blended cement hydration for early age strength enhancement

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Abstract: With an ever-increasing focus on sustainable concrete, the use of supplementary cementitious materials (SCMs) has grown and, although SCMs provide improved later age properties, their use can result in reduced early age strength. Chemical admixtures, particularly those that accelerate hydration processes, have the potential to address these deficiencies. Examples of these opportunities for greater early age strength systems have been identified in the literature, whether through the development of new research and commercial products, or through the creative application of existing admixtures. Conventional inorganic and organic accelerators can be applied in novel high order combinations that exhibit a synergistic effect on early strength. A major focus in the literature at present is the use of C-S-H seeds in superplasticiser suspensions or composites, which can elicit very early strength improvement. Such nucleating agents provide sites for hydrate product growth, potentially leading to denser microstructures and improved early age mechanical properties. In this paper, key studies from the past decade are reviewed, covering the application of a range of accelerating admixtures to the enhancement of early age strength in blended cements.

Keywords: supplementary cementitious materials, accelerating admixtures, early strength, seeding

Introduction

The use of supplementary cementitious materials (SCMs) has increased in recent years to meet the growing worldwide demand for low-carbon concrete. Currently, cement is the second most used material in the world after water, and its production is estimated to contribute up to 8% of anthropogenic CO₂ emissions (1). A recent study initiated by the United Nations Environment Program identified two key material-based strategies to reduce emissions, those being (2):

“1. Increased use of low-CO₂ supplements (SCMs) as partial replacements for Portland cement clinker.

2. More efficient use of Portland cement clinker in mortars and concretes.”

A host of long-term mitigation strategies were identified, including carbon capture and storage, improved energy efficiency, use of alternate clinkers, and recycling streams. While these alternatives do highlight a promising future for the cement life cycle, none offer the affordability, viability, and applicability of SCM use at present. SCM substitution targets a very key point in the cement production chain - up to 70% of cement-related CO₂ emissions can be directly attributed to the combustion and decomposition of calcium carbonate in Portland cement clinker during calcination. Reducing cement use through substitution is thus a conceptually simple strategy for reducing CO₂ production.

SCMs commonly include industrial by-products like fly ash (FA; a by-product of coal combustion), ground granulated blast furnace slag (GGBFS; a by-product of the production of pig iron), rice husk ash (RHA; a by-product of rice milling), or fumed silica (SF; a by-product of silicon metal production). FA and GGBFS are the primary SCMs used on the East coast of Australia, at common substitution levels of 25% and 50% by binder mass, respectively. Other potential substitutes are often limited in application due to their cost. Unlike Portland cement (PC), these materials do not require clinkering and so produce notably less CO₂ emissions per ton of cementitious product. These materials also provide secondary benefits in that they can often improve durability, mitigate the alkali-silica reaction, limit chloride ingress, and increase sulfate resistance (3). While the environmental impact of SCM-containing binder systems

is lower than that of conventional concrete, so too is the early age strength. Elevated temperature curing is one method to increase the early age strength of precast SCM blends, particularly those containing slag. This is effective with even a moderate temperature increase, but also produces a coarse, less homogenous microstructure that inhibits later age strength development (4). Alkali activation with sodium silicate and sodium hydroxide is common and effective, but these activators can be costly and are generally unsafe to handle; current research is focused on less hazardous alternatives like sodium sulfate (5).

One approach to mitigating strength loss is the addition of chemical admixtures, which are functionally diverse and can impact cementitious systems in an assortment of ways, such as altering hydration processes, improving workability, and enhancing mechanical properties. It is difficult to clearly differentiate chemical admixtures into distinct categories, though the common functions are outlined below in Table 1. Those admixtures that influence hydration processes can generally be divided into accelerators and retarders. Other categories are also frequently used, such as water reducers (WRs; plasticisers) and high-range water reducers (HRWRs; superplasticisers); these additives are included in nearly all contemporary mix designs, either as grinding aids or during mixing, and can have a noticeable effect on hydration processes. Polycarboxylate ether superplasticisers especially are a major area of current research (6).

Table 1: Common Admixture Categories and Functions

Category	Function	Common Composition
Accelerating admixtures	Decrease setting time or heat evolution during early hydration	Soluble inorganic salts (chlorides, nitrates, thiocyanates, etc.), organic compounds, seeding agents
Retarding admixtures	Delay setting time by lengthening the induction period; may also impact heat evolution	Lignosulfonic acid salts, carboxylic acid salts, carbohydrates (monosaccharides, polysaccharides, gluconates)
Water reducing admixtures	Reduce water content by 5-10%; lower water-cement ratio can lead to higher strength concrete; often have a secondary retarding effect	Lignosulfonates, hydrocarboxylic acids
Superplasticising admixtures	Synthetic polymers that reduce water content by upwards of 10% but can suffer from slump loss after ~1 hour	Polynaphthalene sulfonates, polymelamine sulfonates, polycarboxylate ether comb polymers
Speciality admixtures	Diverse range of niche applications including air entraining, corrosion inhibition, shrinkage reduction, viscosity modification, etc.	A variety of chemical classes have unique effects on concrete properties. Common examples include starch, lignin-based compounds, surfactants, etc.

While chemical admixtures have been a mainstay in the concrete industry for decades, research is always progressing and improvements to their use are still an important topic in contemporary literature. The longevity of these additives also presents a benefit in that they are very easily implemented with minimal disruption to existing industrial processes. Admixture addition does not require specialised equipment or process modifications, is relatively cost efficient, and fits into pre-existing regulatory approval and guidelines. The focus here is on improving the early strength (primarily 1- and 3-day strengths) of blended cements containing SCMs, without degrading long term strength or altering the workability of the material too significantly. At this early stage of the hydration process, the most effective admixtures for strength enhancement are accelerators, which differ from the activators mentioned above in that they do not rely on the addition of alkalis to promote SCM dissolution and hydrate formation; they present a promising, less hazardous approach to overcoming strength adversity in blended cements and warrant deeper investigation. This review aims to consider recent advances in the application and understanding of accelerating admixtures for early age strength enhancement in blended cementitious systems and identify promising avenues for future development.

Accelerating Admixtures for Early Age Strength Enhancement

Accelerating admixtures can be broadly categorised as soluble inorganic salts, soluble organic salts, or insoluble seeding material. These additives are often employed in binary, ternary, or even higher-order combinations, creating complex systems that can have significant impacts on the hydration kinetics, microstructure, and physical properties of concrete products. Two key functions can be defined; accelerators both shorten the induction period and increase the subsequent rate of hydration. The former is sometimes referred to as “set acceleration,” and the latter as “hardening acceleration” – terms that are used often in the marketing of commercial admixtures. Setting accelerators can act on either C₃A or C₃S phases during early hydration. Those affecting the former are only used in quick setting, non-structural applications due to negative impacts on mechanical properties and durability. C₃S accelerators improve early age strength to a lesser degree, but do not compromise long term properties.

Inorganic Accelerators

The most common and most effective accelerator for both setting and hardening is CaCl₂. The addition of chloride ions may, however, increase susceptibility of steel reinforcements to corrosion. Given the importance of reinforcement in structural concrete, the use of chloride accelerators is understandably limited or avoided. While the mechanism of CaCl₂ action is not yet fully understood, certain key points have been elucidated over time. It is known to lead to the precipitation of C-S-H with a more open microstructure, lower bulk density, and increased surface area, which can improve both ion permeability during hydration and space-filling. Two main qualitative mechanisms have been proposed (7). Thermodynamically, the presence of CaCl₂ promotes the supersaturation of C-S-H in solution, activating sites that would otherwise have been unable to facilitate nucleation and lowering the free energy barriers to precipitation. Kinetically, C-S-H supersaturation from CaCl₂ addition increases the rate of precipitation, both in terms of nucleation and growth. While it is not desirable to rely on chloride-based accelerators, it is possible to emulate the mechanisms through which they function, or to use them as a point of comparison for newly developed accelerator systems. Other chemically simple inorganic accelerators are generally not as effective as CaCl₂, reflected in the often-reported cation and anion series (8):

Anions: Br⁻ ≈ Cl⁻ > SCN⁻ > I⁻ > NO₃⁻ > ClO₄⁻

Cations: Ca²⁺ > Sr²⁺ > Ba²⁺ > Li⁺ > K⁺ > Na⁺ ≈ Cs⁺ > Rb⁺

Oey et al. (9) used isothermal calorimetry to compare the ability of CaCl₂ and Ca(NO₃)₂ to accelerate hydration of OPCs with varying chemistries, as well as blended binders containing FA, slag, and limestone. The results showed that Ca(NO₃)₂ was effective in reducing the duration of the induction period, i.e. accelerating the setting time, but showed no clear impact on the rate of early hydration. It is worth noting that set acceleration of a 15% slag blend was particularly susceptible to an increase in Ca(NO₃)₂ addition. The 1-day compressive strengths of mortars made from blends including Ca(NO₃)₂ were lower than the reference mixes, though by 28 days the strengths were comparable. Interestingly, the dosages that gave the greatest set acceleration resulted in the greatest decrease in strength. It is worth mentioning here that Ca(NO₃)₂ performance is highly dependent on cement chemistry and thus the accelerating effect is highly variable (10). On the other hand, CaCl₂ proved to be both an effective set and hardening accelerator at 1-4% addition and gave higher 1-day mortar strengths than the control mix. As above, the authors attributed the superior performance of CaCl₂ to a greater ability to induce nucleation of hydration products, a claim that was supported by phase boundary nucleation and growth modelling. Two of the other most common anions for accelerators are nitrites and thiocyanates; these have been prevalent in the admixture industry over the past decades and are still in regular use today (11).

Organic Accelerators

The most common organic accelerators are triisopropanolamine (TIPA) and triethanolamine (TEA). These alkanolamines can adsorb to the surface of cement particles and thus improve rheological properties. For this reason, organic accelerators have traditionally been added as grinding aids during cement production, with their accelerating capacity being a secondary benefit. The strengthening effect of these admixtures is generally less than that of the common inorganic accelerators, but they can have favourable interactions when used in combination, as discussed in the following section. Their use has changed little in recent years, and their actions have been well categorised. TEA is known to be an efficient set accelerator and early strength enhancer, while TIPA is reported to mostly increase later age

strength (12). The effect of both is highly dependent on dose rate. For example, TEA behaves as an accelerator at 0.02% addition by weight of cement and a retarder at 0.25% and 0.5%, and can initiate flash-setting at 1.0% (13).

Li et al. (14) compared the mechanochemical properties of six alkanolamines that have been of interest in the literature recently, including diethanol-isopropanolamine (DEIPA), N,N,N',N'-Tetrakis-(2-hydroxyethyl)ethylenediamine (THEED), and ethanol-diisopropanolamine (EDIPA) to determine their suitability as grinding aids as well as accelerators. DEIPA and EDIPA were identified as particularly effective grinding aids, increasing the cement powder flow and preventing agglomeration. Both alkanolamines resulted in increased 3- and 28-day mortar strengths, though DEIPA was identified as the most efficient set accelerator. An interesting comment from the authors highlighted that the alkanolamine-containing mixes required more water to achieve the target consistency. DEIPA may prove to be an ideal additive in combination with common water reducers, which are known to have a slight retarding effect. While DEIPA could counteract this retardation, the WR could mitigate the excess water demand – a synergistic relationship that warrants further investigation.

Riding et al. (15) considered the effect of CaCl_2 and DEIPA on the 1-day strength of SCM-containing binder systems. The combination was found to enhance early strength by increasing the degree of cement hydration, with DEIPA accelerating the cement aluminate phase reactions. Additionally, portlandite content was found to decrease and C-S-H was found to form with a higher calcium-to-silica ratio. The presence of CaCl_2 increased slag hydration slightly, but the authors suggest this may simply be due to the accelerated cement reaction releasing additional alkalis. DEIPA leads to an initial decrease in the heat of hydration of slag-cement systems, followed by a marked increase at later ages. This later-age enhancement occurred significantly earlier in blends with a low-alkali cement. When using high-alkali cements, the presence of alkalis already has a significant activating effect on the slag that may overwhelm any potential DEIPA effects. Further investigation may elucidate the correlation between DEIPA addition and early age strength, making it a potential alternative to alkali activators.

Accelerator Combinations

Binary, ternary, and even higher-level accelerator combinations are common, particularly in commercial blended products. Huang and Shen (16) undertook a comparison of 15 different admixtures and their effect on early strength in an experimental and statistical program based on factorial design. A key element was the use of low doses; as explained by the authors, most literature describes addition levels that, while effective, are too high to be adopted in industry due to cost or other limitations. Most common inorganic salts had little effect at these levels, especially when compared to calcium chloride. The exception to this was sodium thiocyanate (NaSCN), which the authors found to be more effective than CaCl_2 when both were added at 0.05% by mass of cement. An interesting finding was the highly favourable interaction between NaSCN and triethanolamine (TEA). The addition of 0.05% CaCl_2 falls in line with industry standards for reinforced concrete structures. It is, however, debatable that a 0.05% addition rate represents a threshold maximum for other admixtures in a contemporary concrete industry. Given the push for increased performance and decreased cement composition, manufacturers may be more willing to consider increased additions of performance-enhancing admixtures to attain their targets.

Liu et al. (17) considered a ternary accelerating mixture comprising calcium fluoroaluminate (4% by mass of binder), calcium thiocyanate (0.1%), triethanolamine (0.02%), and a polycarboxylate WR (0.1%). Cement pastes were made with the ternary blend, a cement-only reference, and a NaCl-TEA mixture for comparison. At 1-day, the compressive strength was nearly double that of the cement reference paste. Pastes containing the ternary blend remained stronger at all ages up to 28 days, though the increase was most noticeable at 1-day, as reflected in Figure 1.

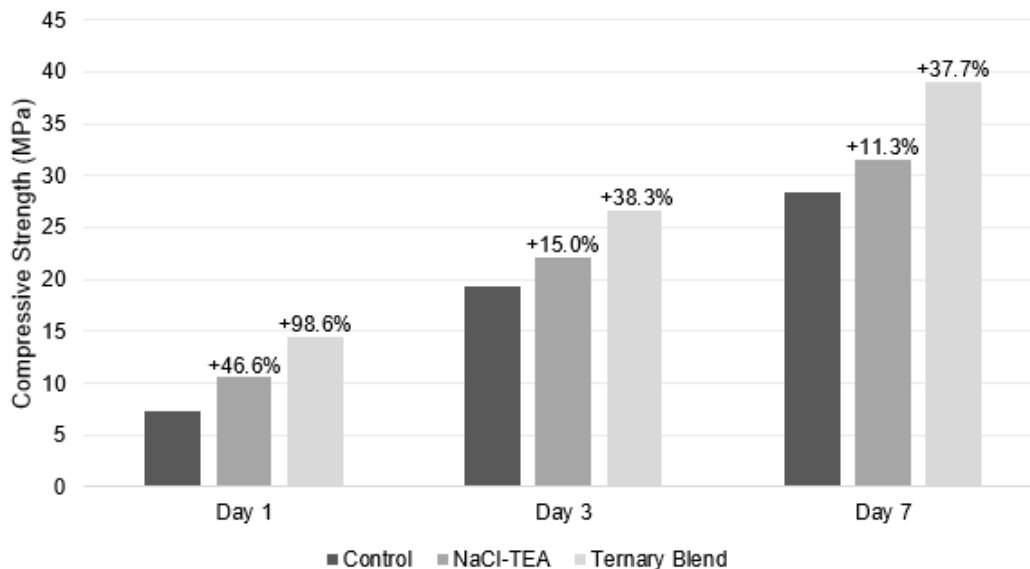


Figure 1: Compressive strength of mortar samples with binary or ternary admixture systems at 1, 3, and 7 days, including percentage strength increase relative to an OPC control (17)

X-ray diffraction analysis showed that the ternary admixture was most effective at accelerating C_3S hydration during the induction period, leading to increased quantities of hydration products like CH, C-S-H, and ettringite. This may be attributed to calcium supersaturation in the pore solution during early hydration, leading to an earlier precipitation of CH crystals and accelerating the cessation of the induction period. As the authors note, multi-component hardening blends such as this are under-represented in published literature, given that they are primarily the domain of commercial admixture producers.

Seeding Accelerators

Seeding accelerators are insoluble materials added to cementitious systems to provide nucleation sites for growing hydrate phases. In principle, the presence of these seeds lowers the activation energy for nucleation; the seeds act as heterogenous nuclei upon which crystal growth can occur, essentially diminishing the key barrier to hydrate precipitation. When added, ions from the pore solution adsorb to these nucleation sites, causing concentration gradients that subsequently affect hydration behaviours. Some seeding materials have a purely physical mechanism of acceleration, by providing nucleation sites. TiO_2 , ZrO_2 , and carbon nanotubes are common examples of these (18). Other additives can also undergo or initiate chemical reactions with ions in the pore solution. For example, nano-silica can form hydration products, $Ca(OH)_2$ alters the calcium saturation of the pore solution, which is then consumed to form C-S-H, and $CaCO_3$ interacts with alumina phases to form carboaluminates, resulting in the stabilisation of ettringite.

One particular seed material that has been of growing interest in recent years is C-S-H, which is often used in combination with polycarboxylate ethers (PCEs) either as a composite or in some cases as a suspension of C-S-H in PCE solution. John et al. (19) published a thorough review on this topic in 2018, covering synthesis methods, proposed mechanisms, and impact on mechanical properties. One key point to mention from this work is the consensus that C-S-H additives accelerate hydration setting and hardening, expediting hydrate formation, and thus greatly increasing early age strength.

Indeed, a commercial C-S-H admixture is already available for purchase as a nanoparticulate suspension in a PCE solution. Several isothermal calorimetry studies have been reported, with particular interest in the impact on early hydration in cement paste. In a comparison of 12 different accelerators (including $CaCl_2$) or nucleating agents, this product was shown to be the most effective set and hardening accelerator, capable of accelerating at addition rates between 0.1% and 3% (by dry weight of cement) (20). A separate study extended this to 5% and found it resulted in further set acceleration but a reduction in the degree of hydration at both 60 hours and 28 days post-mixing (21). This was reflected in the apparent activation energies, as shown in Figure 2, which are higher at 5% than at 0.5% or 2.0%, though still lower than the reference. An interesting finding was that, given the increased

number of nucleation sites, C-S-H initially precipitated in a loose-packed configuration and densified later according to curing temperature, seed concentration, etc. C-S-H was also shown to be a less effective nucleator at higher temperatures.

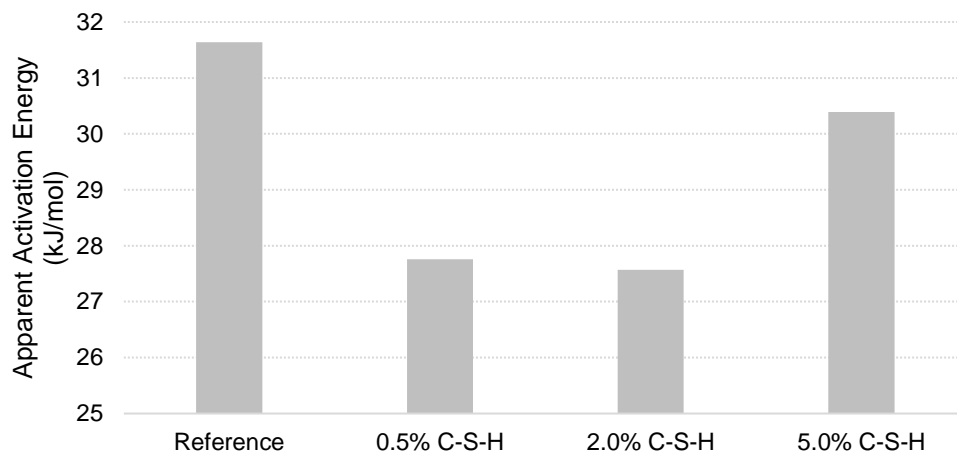


Figure 2: Apparent activation energy of cement pastes blended with C-S-H seeds at various dose rates (21)

In addition to the above studies on early hydration processes, Kanchanason and Plank (22) recently examined the effect of a 2.0% C-S-H-PCE nanocomposite (synthesised in-house) addition on the early age strength of blended cement mortars containing slag and calcined clay SCMs. For the 35% slag-cement blends containing 2.0% C-S-H additive (by weight of cement), a significant increase in strength was noticed at all ages from 6 hours to 28 days when compared to a slag-cement reference with no additive. At just 16h, the compressive strength was approximately 240% higher than that of the reference; at later times, the increase ranged from 25% to 95%. Interestingly, the blended cement mortars with C-S-H additive were also higher than a neat OPC control at all ages, which presents a promising avenue for future low carbon concrete development. The strengthening effect was also noticed in calcined clay blends, but the increase was less pronounced after 7 days. From a mechanistic study based on calorimetry and XRD, the authors suggested a three-step process for this strength enhancement:

1. Clinker silicate phase hydration is accelerated
2. Pozzolanic reactions of the SCMs are accelerated; they start earlier and proceed at a faster rate
3. Hemihydrate is formed, reducing porosity, and thus increasing compressive strength

Being a relatively new material in the concrete industry, the sustainability of C-S-H seeding has not been well investigated. Recently a life-cycle assessment was conducted to determine the impact on sustainability while also demonstrating the low-cost production and characterisation of C-S-H seeds from dilute alite hydration processes (23). It was shown that the overall energy demand and greenhouse gas emissions of producing 1 m³ of seeded cement paste (with C-S-H produced from the alite/C₃S method described) would be comparable to producing 1 m³ of unseeded paste. This does not, however, take into account the potential for SCM use; the increase in strength that can be obtained through C-S-H seeding allows for a greater degree of substitution, which can then result in a significant decrease in CO₂ emissions.

Conclusions & Future Directions

Various admixtures are available or are being developed with the potential to enhance early age strength development in SCM-blended cements. Inorganic accelerators such as nitrate and thiocyanate salts are already in common use commercially and their effects are well characterised. The greatest potential for these admixtures moving forward is in combination with organic accelerators like DEIPA. Secondary and higher order interactions are known to be essential in commercial admixture development but there is limited literature relating to their impact on low carbon binders containing slag or fly ash; these

combinations may one day present viable alternatives to the use of alkali activators. The most promising new development for early age strength improvement is the adoption of nucleating materials such as C-S-H seeds. Recent studies have shown the potential for C-S-H seeded slag-cement binders to match or even out-perform OPC control samples, which presents a promising possibility for the future of low carbon concrete. A summary of some key accelerators is presented in Table 2, alongside properties, benefits, and potential drawbacks. Further research into these new accelerators combinations and nucleating agents should include a thorough investigation into the effects on long-term properties and durability, as well as rheological performance.

Table 2: Selected inorganic, organic, and seeding accelerators and their common dose rates

Admixture	Typical Dose Rates (by weight of cement)	Benefits	Drawbacks
Chloride salts	0-4%	Efficient set and hardening accelerator	Promotes corrosion of steel reinforcements
Nitrate salts	1-4%	Effective set accelerator	Decreases 1-day compressive strengths (9)
Thiocyanate salts	1.5-3%	Effective hardening accelerator	Greater cost for reduced performance relative to chloride salts (11)
TEA	<0.025% (13)	Increases early age compressive strength; synergistic function with inorganic accelerators	Decreases late age compressive strength; can reduce fluidity when used with chloride salts (17)
TIPA	0.01-0.02%	Moderate strength enhancement after 1 day	Limited effect on strength prior to 1 day
DEIPA	0.02%	Increases early strength of slag-cement blends, synergistic effect with low chloride doses	Increases water demand
C-S-H seeds	1-3%	Significantly increases early age strength	Limited commercial availability, more research needed on durability, cost effectiveness, etc.

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