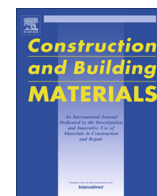




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Studies on early stage hydration of tricalcium silicate incorporating silica nanoparticles: Part II

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HIGHLIGHTS

- SNPs shows its maximum nucleation effect at 8 h when the rate of product formation is high than the control.
- Maximum pozzolanic effect of SNPs observed at 24 h when the quantity of CH was lower than the control.
- ~25% addition C-S-H formed in the presence of SNPs at 24 h.
- More polymerized and well ordered crystalline C-S-H is formed in presence of SNPs.
- SNPs formed more compact and dense microstructure at 24 h which is responsible for the higher early age mechanical strength.

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ABSTRACT

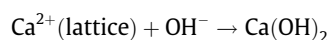
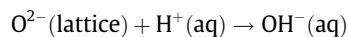
The beneficial role of silica nano-particles (SNPs) during the early stage hydration of tricalcium silicate (C₃S) has been investigated. ICP results show that SNPs significantly accelerate the hydration rate as well as the growth of hydrated products, by providing additional nucleating sites. XRD results also show a dominant nucleation effect of SNPs up to 10 h. While, at later stage i.e. from 15 to 24 h, the pozzolanic effect of SNPs was dominant. Further, TGA results reveal that, in SNPs incorporated samples, quantity of CH is higher (~61% at 8 h) up to 10 h, and lower (~32% at 24 h) from 15 to 24 h than the control, showing the nucleation and pozzolanic effect of SNPs respectively. FTIR and XRD results further reveal an accelerated formation of crystalline C-S-H (jennite and tobermorite) with SNPs. All these results i.e. accelerated hydration, crystalline C-S-H formation and lower C/S (~1.9) lead to enhanced durability of cementitious materials.

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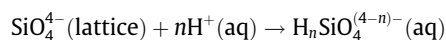
1. Introduction

Recently, nanotechnology based applications have gained wide-spread interest and concrete technologists are exploring different nano-materials such as SiO₂, TiO₂, Fe₂O₃, Al₂O₃, CNT and etc., to improve the performance of cementitious materials [1–7]. Precise understanding of hydration products formed as result of the addition of these nano-materials is essential for the strategic modification of conventional cementitious materials. These nano-materials accelerate the hydration process by providing additional nucleating sites and showing their maximum effects during induction & acceleration period of hydration [8–10]. The chemistry of nano-particles incorporated in cementitious materials at this stage

is still not well established, in fact, the hydration chemistry in pure system (Portland cement) is itself a complex process. There are four main stages that occur during the hydration i.e. pre-induction, induction, acceleration and deceleration period [11–13]. On contact with water, C₃S undergoes an intense and short-lived reaction, known as the pre-induction period. Oxygen ions from the surface of the C₃S lattice react with protons in the water and form hydroxide ions, which in turn combine with Ca²⁺ leading to form calcium hydroxide (CH) [14]:



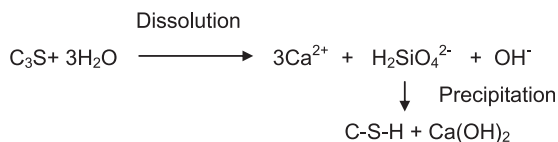
At the same time, silicate material from the 'C₃S' lattice surface enters into the liquid phase:



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A complete reaction may be represented as:



The coupled dissolution and precipitation reactions that occur when C_3S grains come in contact with water, determine a number of practically important properties, such as setting time, rate of heat release and etc. Thermodynamic studies consider that a metastable C-S-H is formed during the initial stage of hydration exhibiting a greater solubility than that of final stable C-S-H and lesser solubility than C_3S . At the end of induction period, this metastable C-S-H is converted to a more stable C-S-H [15–18]. In contrast of this, Scrivener et al., 2008–2012 [19–23] have reported that the slowdown in the initial reaction of C_3S is caused by the changes in ionic concentration in the solution and not by the presence of inhibiting layers formed on the surface of C_3S grains. Further, Scrivener et al., 2012 [24] have reported that the hydration of alite is well simulated by two mechanisms, namely: Solution Controlled Dissolution (SCD) up to the end of the induction period and Nucleation with Densifying Growth (NDG) to capture the main heat evolution peak. They also proposed a scheme for an early stage hydration and according to that, as C_3S grains come in contact of water, some primary hydrates are precipitated on the surface of C_3S grains, but they do not form a continuous layer [25].

Onset of acceleration is still a point of discussion because at this time two main reactions occur simultaneously: precipitation of CH and rapid growth of C-S-H. The role of precipitation of CH as a key mechanism was dismissed, because CH cannot act as a seed, since it quickly dissolve as soon as it comes in contact with water. However, the CH precipitation decreases the calcium hydroxide concentration contributing a rise in the rate of reaction, Fig. 1 [26,27].

According to C-S-H nucleation theory, the growth of C-S-H, plays an important role during the acceleration period, which is more gradual and prolonged than any other cementitious system i.e. calcium aluminate hydrate, ettringite and calcium sulpho aluminate etc. Earlier reviews on cement hydration have concluded that after the formation of a metastable C-S-H layer on C_3S surfaces, a stable form of C-S-H is formed which nucleate the hydration process [28–32]. Garrault and Nonat [33] use the slow dissolution hypothesis and interpreted a decrease in silicate concentrations in the beginning of hydration, as a result of C-S-H

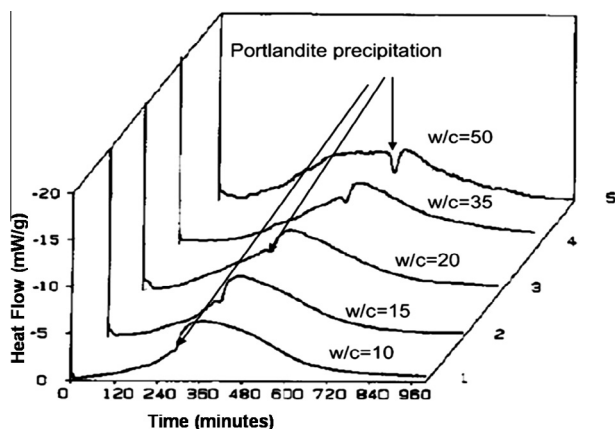


Fig. 1. Heat evolution rate curves for the C_3S hydration in lime saturated solutions with different solution to C_3S ratios. The portlandite precipitation revealed by an endothermic peak in the curve, occurs later and later when the w/c increased [26,27].

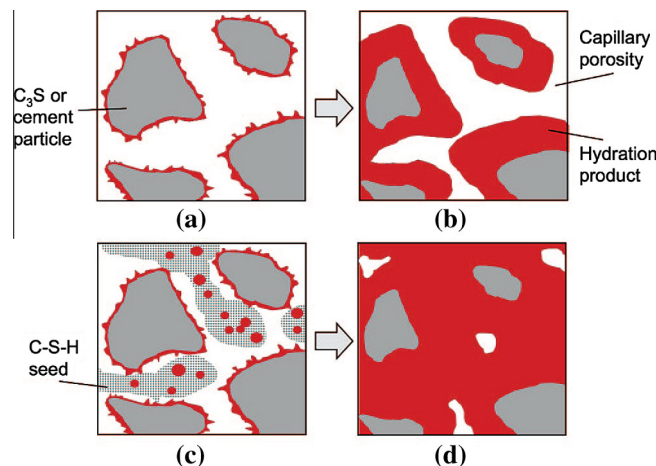


Fig. 2. Schematic of the hydration process. (a) Normal unseeded paste after a few minutes of mixing. (b) Nucleation and growth. (c) Paste with C-S-H seed, a few minutes after mixing. (d) After several hours the overall extent of early hydration is greater, and there is much less capillary porosity [39].

nucleation. Applications of the Avrami model to isothermal calorimetry or QENS data require assuming an induction period of at least 1 h prior to any C-S-H nucleation to obtain good fits [34,35]. But Thomas's, 2007 [36] application of a boundary Nucleation and Growth (BNG) model to fit isothermal calorimetry data and its application in by Scherer et al. [37,38], to fit chemical shrinkage data indicate that initial C-S-H nucleation occurs close to the time of mixing.

Thomas et al. reported that a stable C-S-H nucleation as a controlling factor in the early-age hydration of C_3S , wherein C_3S was seeded by adding a reactive form of C-S-H. In these experiments, the induction period was essentially eliminated and hydration progressed to nucleation and Growth (N + G) kinetics immediately and at a higher rate than in an unseeded paste, Fig. 2 [39]. Without seeding, more time was required for the natural nucleation and growth process to provide enough C-S-H surface area for appreciable growth rates to be observed.

In previous study [40] we focused the preinduction and induction period of hydration, and observed that in the presence of SNPs, additional C-S-H is formed and the supersaturation stage of Ca^{2+} was found to be delayed by 30 min. In the present study, influence of SNPs on hydration during acceleration and deceleration period of C_3S was examined using ICP, XRD, FTIR and SEM/EDX techniques.

2. Materials and methods

2.1. Sample preparation of pure and SNPs incorporated C_3S for hydration studies

To study the effect of SNPs during hydration, 10% (w/w) of SNPs (30–70 nm) was added to the laboratory prepared powder C_3S [40]. For the aqueous phase study the mixture was hydrated with a w/b 5.0. The hydrated mixtures were stirred continuously in a magnetic stirrer. These hydrated mixtures were filtrated at different time intervals starting from 1 to 24 h. For the paste study the mixture was hydrated with a w/b ratio 0.4. Hydration was stopped at different time intervals starting from 4 to 24 h covering the acceleration and deceleration period of hydration.

2.2. Characteristics techniques

In the present study, influence of SNPs on hydration during the acceleration and deceleration period have been studied. The techniques, Inductive Couple Plasma (ICP) (model Prodigy XP, Teledyne Leeman lab, USA) for the determination of ionic concentration, Powder X-ray diffraction (model DMax-2200, Rigaku, Japan) was used for the study of different phases formed during the hydration at early stage. All the experiments were carried out at room temperature using copper radiation ($\text{Cu-K}\alpha$) at 40 kV/40 mA. Scanning was performed with a step width of $0.02^\circ 2\theta$ over an angular range from 5° to 80° with a scanning rate of $0.5^\circ/\text{min}$ throughout the

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