



Effects of silanes and silane derivatives on cement hydration and mechanical properties of mortars



Huajun Feng^{a,b,*}, Hoang Thanh Nam Le^a, Shasha Wang^a, Min-Hong Zhang^a

^aDepartment of Civil and Environmental Engineering, Faculty of Engineering, National University of Singapore, 1 Engineering Drive 2, Singapore 117576

^bSchool of Engineering, Republic Polytechnic, 9 Woodlands Avenue 9, Singapore 738964

HIGHLIGHTS

- Silane derivatives used as bridging agent to improve cement and mortar structure.
- Silanes retard cement hydration whereas silane derivatives mitigate the retardation.
- Strengths and E-modulus of mortars are increased by silanes and silane derivatives.
- Silane derivatives show different effects on cement hydration compared to silanes.

ARTICLE INFO

Article history:

Received 31 March 2016

Received in revised form 8 September 2016

Accepted 1 November 2016

Available online 9 November 2016

Keywords:

Cement
Elastic modulus
Hydration
Mortar
Silane
Silane derivative
Strength

ABSTRACT

An experimental research was conducted with the objectives to study the effects silanes (amino, vinyl, and epoxy-based) and their derivatives (silane oligomers and silane nanoparticles) on cement hydration and mechanical properties of mortars. Silanes and silane derivatives are used as coupling agents to combine different compositions in mortars and to chemically react with calcium silicate hydrates to introduce organic component into C–S–H structure. The use of silanes retarded the cement hydration. However, silane derivatives were found to effectively mitigate the retardation. Liquid-state ²⁹Si nuclear magnetic resonance (NMR) spectroscopy indicated that silanes and silane derivatives have different hydrolysis and condensation products in the solutions of cement pastes. Further analysis of silicon concentration in the solution indicated that the hydrolysis and condensation products of silane oligomer is opt to be in the solution, instead of adsorption onto the surface of cement hydration products, to retard the cement hydration.

Ordinary mortars with the silanes and silane derivatives showed general enhancement in flexural strength and some improvement in compressive strength after curing for 7 days and 28 days. Due to good strength properties, negligible hydration retardation, and simple preparation method, epoxy-based silane oligomer was selected for further study in high-strength mortar. Results indicated that the compressive strength, splitting tensile strength, and elastic modulus of the high-strength mortar were increased by 20%, 38%, and 13%, respectively, compared with the control mortar.

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1. Introduction

Improving the mechanical properties of hardened cements, mortars, and concretes by adding small amount of organic molecules or polymers in an inorganic matrix has drawn considerable attentions in recent years [1–4]. This method is initially inspired by natural bio-composites, such as bones and shells. For instance,

nacre, a natural composite, is composed of 99 vol% hard aragonite (CaCO₃) layers separated by 1 vol% soft polymer layers [5]. The inorganic/organic framework of nacre can simultaneously provide strength, hardness, and toughness: with about 1 vol% polymer, nacre is twice as hard and 1000 times as tough as its constituent CaCO₃ phase [6].

For cement-based materials, the addition of organic molecules or polymers has been proven efficient to improve their mechanical performance. Calcium aluminate cement-polyvinyl alcohol polymer (CAC-PVAP) system is one of the examples characterized by very high tensile strength (up to 70 MPa) [7–9]. Micro-structural analysis shows that CAC-PVAP system is composed of inorganic

* Corresponding author at: Department of Civil and Environmental Engineering, Faculty of Engineering, National University of Singapore, 1 Engineering Drive 2, Singapore 117576.

E-mail address: fhjtiger@gmail.com (H. Feng).

calcium aluminate cement grains embedded in polymer matrix. The polymer is believed to serve as pore filler and act as a rheological aid to optimize particle packing and to reduce the porosity. However, the increase of CAC-PVAP system in fracture resistance is limited [10]. This is due to the poor bonding of polymer with cement hydration products [10], which is believed to be the key for the fracture toughness of nacre [11].

Calcium silicate hydrate (C–S–H) is the major phase in ordinary Portland cement paste responsible for cohesion and mechanical properties [12,13], and occupies the largest space among cement hydration products [14]. The classical C–S–H has a randomly layered structure (Fig. 1a) consisting of different minerals, such as imperfect Jennite and tobermorite [15]. About 1/4 of the C–S–H structure is occupied by gel pores of various sizes formed during cement hydration [14], and about 1/3 of the bond in the C–S–H is weak van de Waal's bond [16]. Recent studies showed several successful examples to bind organic components with C–S–H layer through strong covalent bonds [10,13,17]. A schematic model on the organically-modified C–S–H layer structure is shown in Fig. 1b. Some silicate tetrahedral was replaced by organic molecules from silane. X-ray diffraction and ^{29}Si NMR results proved the existence of organic compositions at the inter-layer of C–S–H without disrupting the integrity of C–S–H framework [10].

Silane is the key chemical to bridge between organic phase and inorganic C–S–H matrix. Typical structure of silane molecules is shown in Fig. 2. The structure contains two types of groups: “X” group and “RO” groups. The “X” group may provide various organic groups, e.g. alkyl, amino, mercapto, epoxy, and vinyl among others. The three “RO” groups are used to form covalent bonds with inorganic materials through chemical reactions. Fig. 2 also presents the schematic reaction mechanism of silane with C–S–H based on the studies of previous publications [10,13,17]. Silane is an active chemical under alkaline aqueous condition (which is typical for the environment of cement paste, mortar, and concrete). As shown in Fig. 2, three “RO” groups of silane may react with water by a hydrolysis reaction under alkaline condition to form intermediate with three “HO” hydroxyl groups. The “HO” hydroxyl groups may couple with the same “HO” groups on the surface of the calcium silicate sheets of C–S–H to establish covalent bonds. As a result, the organic phase is introduced into the C–S–H structure through the “X” group of silane. If the three “HO” groups of silane react with different C–S–H sheets, these sheets will be coupled together by the bridging effect of silanes.

In addition to the C–S–H, the “HO” hydroxyl groups exist on the surface of other components in cement pastes and mortars. These components can be coupled together by the use of silanes as well based on similar reaction mechanism. Therefore, the coupling effects of silanes are showed in three ways: (1) bridging effects between organic and inorganic components; (2) linking between different C–S–H layers; and (3) coupling between the

C–S–H and other component in mortars. As a comprehensive result, the use of silanes is promising to improve the inner cohesion and toughness of cement paste and mortars.

In past years, silane has been mixed in cement pastes as admixture [18,19], or coated on solid aggregates as modification agent [20,21]. In most published research, silanes were directly used in cement pastes or mortars. Positive effects of silanes include better workability [20], better mechanical properties [20], and decreased drying shrinkage [22] of cement composites. However, the use of silanes was reportedly reduce the hydration rate of cement [23], which will affect strength development and construction progress. Therefore, it will be intriguing to find other silane-like chemicals with positive effects on mechanical properties without compromising the rate of cement hydration.

From the chemistry viewpoint, silane may go through a series of self-condensation reactions to form various silane derivatives, including both silane oligomers and silane nanoparticles (Fig. 3). These silane derivatives are different to silane although they are derived from the same silane. Silane has one silane unit (monomer) with low molecular weight. However, silane oligomers consist of a few silane units with limited molecular weight. For instance, silane dimer and trimer are silane oligomers composed of two and three silane units, respectively. Silane nanoparticle is another kind of silane derivative with thousands of silane units and high molecular weight. The physical sizes of silane oligomers and nanoparticles may be in the range of nano or sub-nano meters, which are much larger than that of silane monomer. The physical state of silane oligomers is viscous liquid or soft polymer material depending on the number of silane units; however, the state of silane nanoparticles is solid. In addition, silane derivatives have much more “RO” hydroxyl groups in their molecular structures than silane monomers to couple with C–S–H and other compositions in cement paste and mortar. These characteristics of silane derivatives may potentially influence cement hydration rate and mechanical properties of mortars in different ways compared with corresponding silane monomers.

The objectives of this study are to study the effects of different types of silanes and silane derivatives on hydration process in cement paste and mechanical properties of mortars. Three types of silanes and their corresponding silane oligomers and nanoparticles were used. Silane oligomers and silane nanoparticles were synthesized by chemical methods in the laboratory. Effects of silanes and silane derivatives on physicochemical properties of cement pastes were investigated, including rate of heat development of cement pastes, elemental concentrations and pH values of cement paste solutions at early age. Liquid-state ^{29}Si NMR spectra and inductively coupled plasma optical emission spectrometry (ICP-OES) methods were used to investigate chemical status evolution of silanes and silane derivatives in fresh cement paste solutions. Effects of silanes and silane derivatives on compressive and flexural strengths of ordinary mortars with a water-to-cement

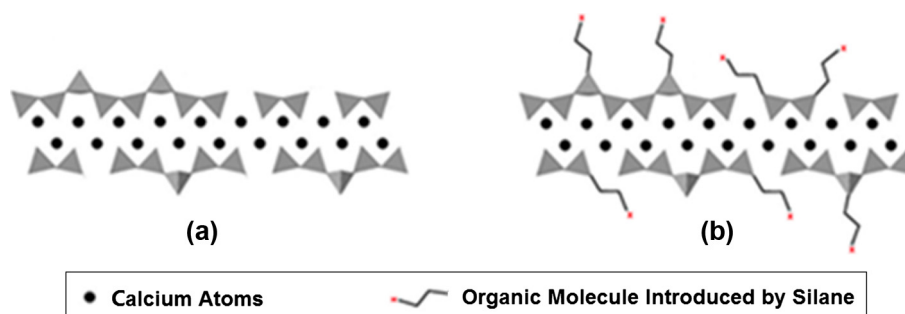


Fig. 1. Schematic layered models of (a) classical C–S–H structure and (b) C–S–H structure with the incorporation of organic compositions from silane.

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