

# Barrier performance of silane–clay nanocomposite coatings on concrete structure

Ricky S.C. Woo<sup>a</sup>, Honggang Zhu<sup>b</sup>, Michael M.K. Chow<sup>a</sup>,  
Christopher K.Y. Leung<sup>b</sup>, Jang-Kyo Kim<sup>a,\*</sup>

<sup>a</sup> *Department of Mechanical Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Hong Kong, PR China*

<sup>b</sup> *Department of Civil Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Hong Kong, PR China*

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## Abstract

The barrier performance of silane/clay nanocomposites as a coating material on concrete structure has been evaluated under different accelerated weathering tests, including moisture permeability and salty water spray. The silane/clay nanocomposite was fabricated by curing the silane–organoclay mixture through hydrogen bonding with concrete. XRD analysis indicated an improved intercalation of clay after adding into the silane solution. SEM examination of the coated concrete surface confirmed that the nanocomposite can effectively cover the pores and voids present on the concrete surface. The rheological study revealed a linear increase in viscosity with the addition of clay. Wetting properties were evaluated via contact angle measurements. The moisture permeability test showed that the permeability was substantially reduced due to the presence of clay of high aspect ratio. The salty water spray tests indicated the distinct barrier characteristics of silane/clay nanocomposite coating on concrete structure.

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## 1. Introduction

The deterioration of concrete due to exposure to environment and mechanical loads requires continuous repair and rehabilitation. Among many degradation mechanisms, corrosion of steel rebar inside concrete is one of the most significant and detrimental. The chemical reaction in concrete produces calcium hydroxide, which provides an alkaline environment. Thus, a stable oxide film is formed on the steel surface, protecting the rebar from corrosion. However, the penetration of chloride ions, from sea water or de-icing salts, can break down the protective film and expose the steel bar to corrosion [1]. To delay the chloride penetration and hence to prolong the service life of concrete structures, surface treatment such as coating is commonly employed.

Silane has widely been applied to glass fiber surface as the coupling agent with polymer resins [2]. Silane, once applied as coating on concrete, penetrates into the concrete pores and thus forms an impervious layer on the surface. This protective action greatly improves the resistance to environmental attacks, such as chloride ions diffusion, water permeation [3,4]. Polymer–nanoclay composites have emerged as a new class of advanced organic–inorganic materials with excellent mechanical properties and barrier characteristics with only a few percent of well-dispersed clay reinforcements [5,6]. Clay nanocomposites find many potential applications in civil engineering [7–9]. Apart from enhanced modulus, strength and fracture toughness, the nanoclay can offer an excellent barrier capability with significantly reduced permeability of chemicals, moisture and gases [10–12]. The reduction of chemical and moisture absorption can suppress the corrosion of reinforcing materials in concrete, giving rise to better long-term durability.

\* Corresponding author. Tel.: +852 2358 7207; fax: +852 2358 1543.  
E-mail address: [mejkkim@ust.hk](mailto:mejkkim@ust.hk) (J.-K. Kim).

This paper is part of a larger project on clay nanocomposites with excellent moisture barrier properties for applications as adhesive, coating and matrix for fiber reinforced composites in construction. As a continuation of previous work [8–13], this paper studies the barrier characteristics of silane–clay nanocomposites that are applied onto concrete as the protective coating. Several accelerated weathering tests, including moisture permeability test and salty water resistance test, were employed to assess the performance of silane coating and organoclay reinforced silane nanocomposite coating, relative to that of plain concrete without a protective coating.

## 2. Experiments

### 2.1. Materials and sample preparation

The silane used in this work is SILRES BS 1701 (supplied by Wacker Silicones Inc.), a mixture of isomeric octyltriethoxysilane and isooctyltriethoxysilane as main components. The chemical structure of octyltriethoxysilane shown in Fig. 1 indicates that the silicon atom is attached with three hydrolyzable alkoxy groups and an organofunctional group. Silane is normally applied as a water-repellent primer and hydrophobic impregnating agent for concrete. The silane reacts with atmospheric moisture or pore water in the concrete, generating active ingredient to lower the water absorbency of the treated concrete.

Two types of montmorillonite-based clay with different organic modifiers (Fig. 2), namely Cloisite 20A (dimethyl dehydrogenated tallow quaternary ammonium modified, supplied by Rockwood Specialties) and I.30P (primary octadecylamine modified, supplied by Nanocor Inc.), were added into silane to prepare nanocomposites. The clay–silane mixture was ultrasonicated (Ultrasonic Processor XL2020) at 70 W and 42 kHz for 3 h, and was applied as coating onto concrete surface, which was cured at room

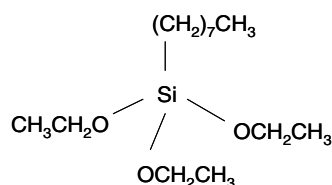


Fig. 1. Chemical structure of octyltriethoxysilane.

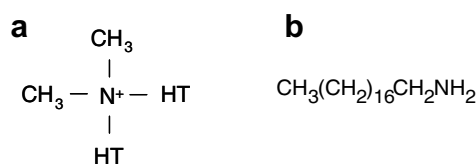


Fig. 2. Chemical structures of organic modifiers in (a) Cloisite 20A (HT is hydrogenated tallow having around 65% C18, 30% C16 and 5% C14) and (b) I.30P.

temperature for at least seven days before test. The surface morphologies of concrete surfaces with and without coating were examined using a scanning electron microscope (SEM, JEOL 6300).

### 2.2. Characterization of nanocomposites

The silane/clay mixtures after ultrasonication were analyzed on an X-ray diffraction analyzer (XRD, Philips PW1830) that consisted of a Cu anode and a graphite monochromator to measure the intergallery distance of organoclay before and after the incorporation into silane. XRD spectra were obtained at room temperature on a  $\theta$ – $\theta$  diffractometer equipped with an intrinsic germanium detector system using CuK $\alpha$  radiation (1.540562 Å) at a scanning rate of 0.01°/min from 2° to 6°. Differential scanning calorimetry (DSC, DSC 92 Setaram 90/39324) was used to study the curing behavior of silane with the application of heat from around room temperature to 240 °C at a heating rate of 10 °C/min in a nitrogen environment. Any sudden change in heat flow would indicate the physical transformation of the material. The viscosities of the silane–clay mixtures with different clay contents were measured on a rotational rheometer (Paar Physica US 200) with controlled shear rates at room temperature. The torque applied to rotate a spindle at a constant speed of 200 rpm was measured while the spindle was immersed into the sample fluid of about 6 g.

Wetting properties of silane containing different types and contents of organoclay were determined by static contact angle measurement at room temperature using a goniometer, with an accuracy of  $\pm 0.2^\circ$ . A single droplet of 2–4  $\mu$ l was dispensed at a time on the surface of concrete substrate using a motorized syringe. The concrete sample was prepared from cement mortar consisting of water:cement:sand = 0.6:1:0.6 by weight ratio and PVA fiber:cement mortar = 0.02:1 by volume ratio, which was moist-cured at 25 °C and 98% RH for 14 days. The average contact angles were recorded immediately after the droplets touched the substrate and at least five measurements were conducted for each test. The theoretical considerations of wetting are based on the Young–Dupre equation [14] that defines the work of adhesion,  $W_a$ , between the liquid and solid as a function of contact angle  $\theta$

$$W_a = \gamma_S + \gamma_L - \gamma_{SL} = (1 + \cos \theta)\gamma_{SL} \quad (1)$$

where  $\gamma_S$  and  $\gamma_L$  are the surface free energies of the solid and the liquid, respectively; and  $\gamma_{SL}$  is the interfacial free energy. The thermodynamic work of adhesion is defined as the work required to separate the interface between two phases from their equilibrium states.

### 2.3. Accelerated weathering tests

The salty water spray resistance test was performed according to Model Specification for Protective Coatings for Concrete, Appendix 4 from Civil Engineering Depart-

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