Investigation on Superhydrophobicity and Piezoresistivity of Self-sensing Cement-Based Sensors Using Silane Surface Treatment

W. K. Dong, W. G. Li, X. Q. Lin, and S. P. Shah

Abstract Cement-based sensors are highly susceptible to the effects of watery environments due to the hydrophilic properties of the cement matrix. In this paper, we applied a surface treatment using a silane/isopropanol solution to graphene/cement-based sensors to achieve superhydrophobicity and mitigate piezoresistive instability in watery environments. After treatment, impressive water contact angles of 163.4° and 142.0° were achieved for the surface and inner cement-based sensors, respectively. Moreover, the piezoresistivity of the coated cement-based sensors exhibited greater stability compared to their untreated counterparts. These results provide valuable insights into the piezoresistivity of hydrophobic cement-based sensors in moist environments, offering promising prospects for future structural health monitoring applications.

Keywords Cement-based sensors · Graphene nanoplates · Piezoresistivity · Silicone hydrophobic powder

1 Introduction

Piezoresistivity-based self-sensing cementitious composites have attracted increasing attention recently, for their ability to achieve structural and pavement health monitoring automatically. Cement-based sensors have been widely

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investigated, ranging from their types and content of conductive filler, matrix, additives, curing, and drying methods to field application conditions [1, 2]. However, due to the hydrophilic and porous structure of the cement matrix, the electrical conductivity and piezoresistivity of cement-based sensors can be easily affected by the working environment, especially watery, and humid conditions [3, 4]. Previous studies have attempted to remove the influence of penetrated water on the piezoresistivity of cement-based sensors. The water absorption of cement-based sensors was significantly reduced in the early age, but the efficiency was relatively low, with unstable piezoresistivity in the long term [5].

Basically, using waterproofing materials to treat the surface of cement-based sensors can prevent water penetrating the cement matrix [6, 7], which can reduce the interference of water molecules on electrical resistivity and piezoresistivity. In this study, we propose a special surface treatment of graphene/cement-based sensors by immersing the sensors in a silane/isopropanol solution. The hydrophobic silane is expected to penetrate the cement-based sensors and improve the waterproofing properties.

2 Methods

In this paper, graphene nanoplate (GNP) is used to achieve intrinsic self-sensing ability and piezoresistivity of cement-based sensor. The GNP is commercially available, and its specific properties are documented [8]. The raw materials consisted of general-purpose cement, silica fume, superplasticizer, GNP, silane hydrophobic powder (SHP), and tap water. The addition of SHP can enhance the waterproofing and hydrophobic behaviors of cement-based sensors. The silane used was aqueous trichlorosilane for surface modification, and the solvent isopropanol was chosen to disperse the silane and control the concentration. The physical and chemical properties of silane and isopropanol are listed in Table 1.

To remove surface impurities and smooth the surface, the cement-based sensors were firstly polished and cleaned using sandpaper before coating. The surface modification followed the steps shown in Fig. 1. Isopropanol was prepared in a measuring beaker, and 4% silane by volume of isopropanol was added, followed by 5 min of

Table 1 Physical and chemical properties of silane and isopropanol

Product	Appearance	Color	Relative density	Formula	Molecular weight
Trichloro (1H, 1H, 2H, 2H-perfluorooctyl) silane	Clear liquid	Colorless	1.3 g/cm^3	$C_8H_4Cl_3F_{13}Si$	481.54 g/mol
2-Propanol	Clear liquid	Colorless	0.785 g/mL	C_3H_8O	60.10 g/mol

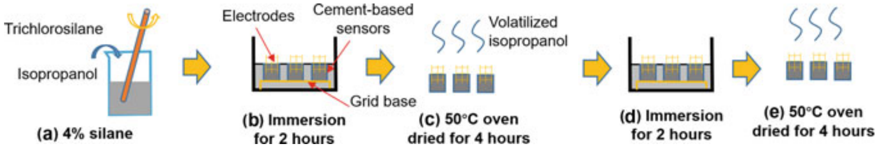


Fig. 1 **a**–**e** Treatment procedure of cement-based sensors

mechanical mixing to dissolve and disperse the silane. The cement-based sensors were placed above a copper mesh in a plastic container at a distance of 5.0 mm, so all surfaces had continual contact with the silane/isopropanol or silane solution. The mixed solution was gently poured into the container until the top surfaces of cement-based sensors were just covered. It should be noted that the electrodes of the cement-based sensors were not immersed in the silane/isopropanol solution, to ensure excellent conductivity of the electrodes. The container was sealed with plastic film to avoid volatilization of isopropanol. The cement-based sensors were immersed for 2 h to ensure the thorough entrance of the solution into the cracks and pores of the cementitious material. Finally, the cement-based sensors were dried in an oven at 50 °C for 4 h to volatilize the isopropanol.

The water contact angle (CA) measurements of the cement-based sensors before and after surface modification were performed with an optical tensiometer (Attension Theta). The test liquid was deionized water with a volume of 0.2 μL for each water drop. The water CA of the intact surface of the cement-based sensors represents the hydrophobic coating efficiency. To obtain the hydrophobic behavior of the inner sensor, the CA tests were also performed on cross-sections of the cement-based sensors.

3 Results and Discussion

3.1 Hydrophobic Behavior

Figure 2 shows the surface water CA of the cement-based sensors before and after surface modification at the time of 0, 1, 5, and 9 s from water dropping to stabilization. The cement-based sensors without a coating shown in Fig. 3a exhibited hydrophilic behavior with an initial CA of 79.2°. Subsequently, the water CA gradually decreased over time until the smallest value of 70.1°, which implied that the water molecules penetrated the cement-based sensors due to the hydrophilic behavior and porous structure of the cement matrix. Consequently, the altered water content would be able to permanently affect the electrical and piezoresistive properties of the cement-based sensors, which indicates the necessity to coat them. For the coated cement-based sensors, it was observed that they exhibited hydrophobic behavior, with a final CA of 163.4°. In addition, the cement-based sensors without a coating showed

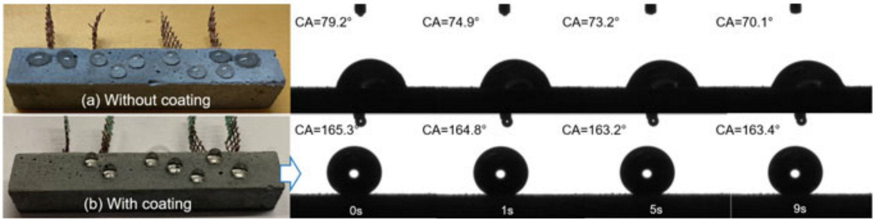
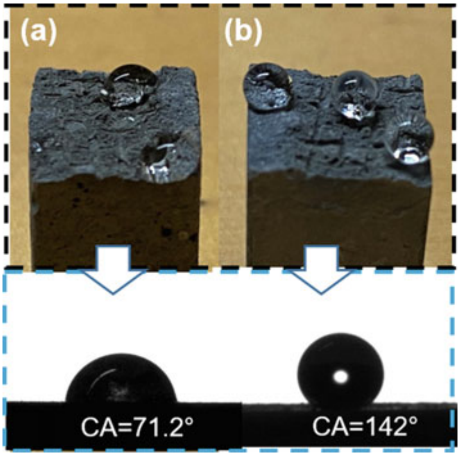


Fig. 2 Water contact angles (CAs) of cement-based sensors **a** without and **b** with surface treatment

Fig. 3 Water contact angles (CAs) of the interior of cement-based sensors: **a** without coating and **b** with coating

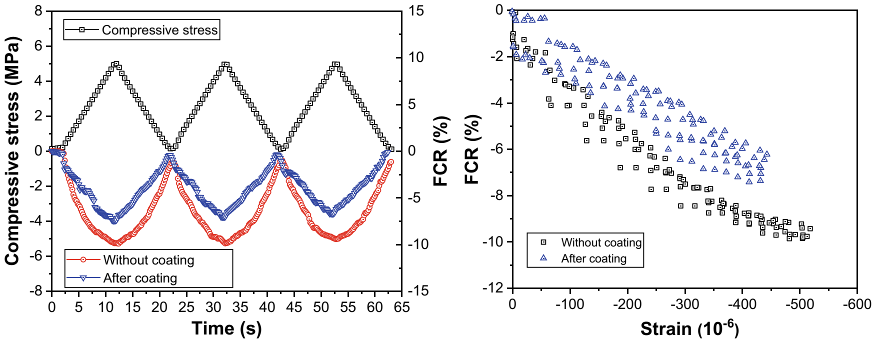


decreasing CA, whereas their coated counterpart only showed a slight fluctuation rather than continual decline.

Figure 3a, b shows the water CAs the cross-sectional surface of the cement-based sensors before and after treatment, to display the hydrophobic or hydrophilic behavior of the interior of the sensors. The cement-based sensor without a coating displayed hydrophilic behavior and a similar CA of 71.2° to that of surface. In contrast, the interior of the sensor became hydrophobic with a CA value of 142.0°, which demonstrated that the silane/isopropanol solution could penetrate into the core of cement-based sensors through micropores and cracks, resulting in hydrophobicity of the cut cross-section.

3.2 *Piezoresistivity*

The stress-sensing performance of the cement-based sensors before and after surface modification is shown in Fig. 4. Fractional changes of resistivity (FCR) of the cement-based sensors exhibited an excellent relationship to compressive stress, with first a



(a) Stress self-sensing

(b) Strain self-sensing

Fig. 4 **a** Stress- and **b** strain-sensing capacities of cement-based sensors before and after surface modification. FCR, fractional change of resistivity

decrease and then returned resistivity in the loading and unloading processes. This finding demonstrated that the silane-based surface modification did not eliminate the piezoresistivity of the cement-based sensors. The graphene-filled cement-based sensor without a coating showed the highest FCR value of 12.6%, followed by an average FCR of 7.2% for the cement-based sensors after surface treatment. These data implied that the stress-sensing efficiency might be weakened by the silane modification. In addition, small fluctuations can be seen for the silane-coated cement-based sensors, mainly due to the brittleness and heterogeneity of the cementitious materials, which led to sudden changes of electrical resistivity. Secondly, the intruded silane aggravated the fluctuation because of its poor electrical conductivity. For the strain-sensing performance, the FCR showed an excellent relationship with compressive strain and showed a similar changing mode to compressive stress.

4 Conclusions

Cement-based sensors can easily absorb water molecules because of their porous structure and hydrophilic behavior. In this study, a silane-based surface modification was applied to improve the waterproofing and superhydrophobic behavior, while maintaining excellent piezoresistivity of the cement-based sensors. The final water CA significantly increased to 163.4°, and the piezoresistivity was relatively well maintained. The piezoresistivity of the cement-based sensors seemed to decrease after the surface modification, with slightly poorer linearity and repeatability, lower gauge factor, and higher hysteresis. Despite this, the cement-based sensors exhibited acceptable linearity and repeatability.

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