



## Superhydrophobic rice husk ash coating on concrete



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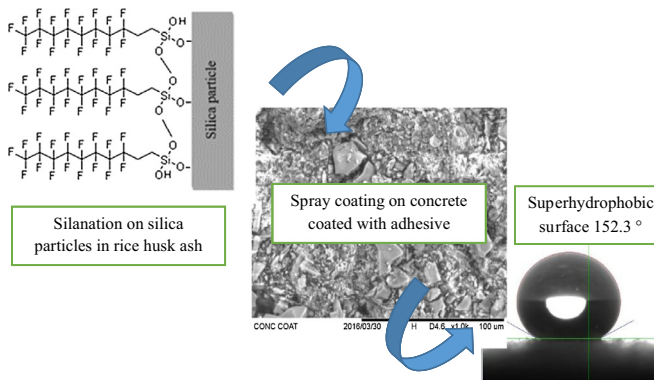
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### HIGHLIGHTS

- Silica particles in rice husk ash was modified using fluoroalkyl silane.
- The hydrophobic silica particles were spray coated on concrete coated with adhesive.
- Water contact angle of  $152.3 \pm 0.5^\circ$  on the coated concrete was observed.
- The cumulative water uptake was reduced as much as 40.38%.
- The water sorptivity was reduced up to 44.44%.

### GRAPHICAL ABSTRACT



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### ABSTRACT

The silica particles in rice husk ash are useful to create the roughness required by the superhydrophobic coating on concrete. The superhydrophobic coating was prepared using rice husk ash dispersed in the ethanolic solution containing fluoroalkyl silane, 1H,1H,2H,2H-perfluorodecyl triethoxy silane (2 vol.%). The ash solution was sprayed on a layer of commercial adhesive coated on the concrete. The water contact angle of  $152.3 \pm 0.5^\circ$  on the coated concrete was observed. The cumulative water uptake was reduced as much as 40.38% while the water sorptivity was reduced up to 44.44%. The penetration of water into the coated concrete under the water pressure of 500 kPa was successfully reduced after 72 h, but not fully prevented. The ash coating did not affect the surface hardness or the compressive strength significantly, but the ash coating resulted in a higher ultrasonic velocity due to the reduction of surface porosity.

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### 1. Introduction

Concrete is a very strong and durable material in construction industry, but it can suffer from the loss of strength due to water

penetration. Water can penetrate easily into the porous and hydrophilic concrete. The water penetration not only generates the distress in concrete, the water also carries other aggressive substances that can cause the corrosion of reinforcement steels. Furthermore, the rust occupies a great volume in concrete, resulting expansion and tensile stress. The cracking, spalling and delamination of concrete are the subsequent effects of expansion and tensile stress formed in the concrete.

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The silicon-based compounds such as silanes, siloxanes and silicones are frequently used as the water-repellent agents in the construction industry [1]. The alkyl groups of these silicon-based compounds reduce the interaction between concrete and water greatly. Silanes are the monomeric silicon compounds which hydrolyse and condense into siloxanes with the stable Si-O-Si bonds. The siloxanes can be further crosslinked into polysiloxanes or silicones. Tittarelli and Moriconi [2] reported that blending 1 wt % of butyl-ethoxy silane solution into cement enhanced the polarization resistance of the galvanized steel plates embedded in the resulted concrete as much as three times higher than the polarization resistance of the galvanized steel plates in the unmodified concrete. The concrete specimens could be also submerged in the alkyltrialkoxysilane solution for 24 h to ensure the formation of complex siloxane species, namely silsesquioxanes [3]. Sobolev and Batrakov [4] used polyethyl hydrosiloxane and polymethyl hydrosiloxane based admixtures which release hydrogen to form the hydrophobic voids in concrete. In the construction industry, the silanes and siloxanes are more commonly used to form the hydrophobic coatings on concrete in order to achieve water proofing. They are supplied in the organic solvents or dispersed in the water. Since the silanes are highly volatile and low in viscosity, the silanes are usually applied at a minimum concentration of 92% to prevent aqueous chloride ingress into highway structure [1]. Meanwhile, siloxanes are less volatile and they react faster than silane. The siloxanes concentration is usually adjusted based of the substrate permeability and pH [1]. The mixture of silanes and siloxanes is more commonly used instead of using the silanes or the siloxanes alone. On the other hand, the silicone resins with high molecular weight are more frequently used as the hydrophobic sealant.

In the recent years, the superhydrophobic coatings and admixtures for construction materials received a lot of attention since they offer anti-fouling [5], anti-corrosion [6] and anti-icing properties [7]. Besides introducing the hydrophobic agents as mentioned earlier, the nanoparticles are commonly used to create the required roughness for superhydrophobic surface [8]. The air captured on the rough surface can reduce the contact between solid-water, which is classified as the Cassie-Baxter state. For instance, the commercial metakaolin and silica nanoparticles [9] were further blended into the polymethyl hydrogen siloxane solution to form the cementitious composites with superhydrophobicity. The superhydrophobic composite not only showed a reduced absorption of water, but also exhibited the insignificant changes of compressive strength. A recent study [10] described hybrid sol of TiO<sub>2</sub> and SiO<sub>2</sub> nanoparticles, functionalized with a fluoroalkyl-functional water-borne oligosiloxane in aqueous solution and deposited over the bricks. Although the surface hydrophobicity of this TiO<sub>2</sub>-SiO<sub>2</sub> coating reduced under UV irradiation, the surface hydrophobicity was restored in the darkness and the photocatalytic properties were maintained. Wong et al. [11] used the superhydrophobic paper sludge ash to form water-resisting coating or admixture for concrete. The water absorption and sorptivity of concrete reduced more than 84% when the superhydrophobic ash was applied on the concrete surface or blended into the concrete before curing. The waste ash has been long used in concrete to replace cement, fine aggregate, coarse aggregate and reinforcing materials partially in order to reduce the building cost and environmental impacts [12]. The alkaline ash can be even used for carbon capture and the carbonated ash enhances the mechanical properties of concrete including tensile and compressive strength [13].

In Malaysia, about two million tons of rice husk are produced annually and the combustion of rice husk are widely practiced for energy generation [14]. The major by-product, rice husk ash contains more than 90% of amorphous silica which serves as the

ideal substituent of commercial nanoparticles in the superhydrophobic coating. In our previous work, the superhydrophobic rice husk ash was sprayed on the adhesive layer which applied on the glass [5]. The ash coating repelled foulant satisfactory when it was immersed into the slurry solution containing kaolin and methylene blue dye. The major aim of this work was to evaluate the feasibility of superhydrophobic ash coating on concrete to reduce the water absorption of concrete. In addition, the concrete properties such as water contact angle, compressive strength and wall strength were measured.

## 2. Materials and methods

### 2.1. Materials

Ordinary Portland cement (Tasek Corporation Berhad, Malaysia) complying with BS 12:1996 or ASTM-C150-Type 1 was used as the main binder in all the concrete samples. The fine aggregates was procured from river sand (LH Lean Hup Sdn. Bhd., Malaysia) and sieved using 0.6 mm sieve, while the 20 mm granite (Kwad Sdn. Bhd., Malaysia) was used as the coarse aggregate. Tap water was used in the preparation of concrete samples. The raw rice husk from a rice mill located in Perak, Malaysia was used in the preparation of superhydrophobic rice husk ash coating. The anhydrous citric acid, absolute ethanol and kaolin were acquired from Merck, Malaysia. The fluoroalkyl silane, 1H,1H,2H,2H-perfluorodecyltriethoxysilane (HFDS) was purchased from Gelest Inc. while the analytical grade of stearic acid, C<sub>18</sub>H<sub>36</sub>O<sub>2</sub> (SA), with 95% purity was purchased from Sigma Aldrich. The spray adhesive, 3 M Spray Mount™ Artist's adhesive was used to attach the ash particles on concrete firmly.

### 2.2. Concrete preparation procedure

The concrete samples used in this study was prepared from the mix containing 1264.8 kg/m<sup>3</sup> of coarse aggregate, 595.2 kg/m<sup>3</sup> of fine aggregate, 380 kg/m<sup>3</sup> of cement and 190 kg/m<sup>3</sup> of free water. The free water/cement (w/c) ratio was fixed at 0.5. The homogenized fresh concrete was cast and vibrated in the cube mould (100 × 100 × 100 mm cube mould). During the preparation of concrete mix, the slump test was carried out consistently with BS 1881: Part 102: 1983 to determine the workability of fresh concrete. The concrete in a cube was cured for 28 days according to BS 1881: Part 111: 1983.

### 2.3. Preparation and characterization procedure of superhydrophobic coating

The raw rice husk was washed with tap water, followed by another 3 times of washing using distilled water. Then, the rice husk was dried at 110 °C for 24 h. The dried rice husk was further treated with citric acid to initiate the hydrolysis of celluloses and hemicelluloses based on literature [15]. About 20 g of dried rice husk was immersed in 500 ml of citric acid solution (5 wt.%) and stirred for 2 h at 50 °C. The treated rice husk was then filtered, rinsed and dried. In order to obtain rice husk ash which is rich of silica, the rice husk was calcined in a muffle furnace (Carbolite) at 800 °C for 30 min under atmospheric condition. The superhydrophobic coating solution was obtained by dispersing about 3 g of rice husk ash into 50 ml of HFDS/ethanol mixture (volume ratio 1:50). The solution was stirred for 1 h at room temperature before spray coating. The homogeneous ash solution was manually sprayed on to a layer of adhesive on the concrete cube at a constant distance of 10 cm until the surface was wetted, using at least 278 ml/m<sup>2</sup> of solution. The coating morphology was studied using the scanning electron microscope (SEM, Quanta FEG 450).

### 2.4. Concrete test procedure

#### 2.4.1. Hydrophobicity measurement and water repellence test

The concrete hydrophobicity was quantified in term of water contact angle. The water contact angle of the coatings was measured using a goniometer (Ramé-Hart Instruments Co., 250-F1). The water droplets were placed on the coated concrete or the uncoated concrete and the water contact angle was measured after 30 s. The average value of water contact angle was determined using three concrete samples with three replications of measurement. The water droplet containing methylene orange dye was also placed on the concrete surface to conduct the visual inspection on the surface hydrophobicity. Furthermore, the concrete was placed under a tap with running water to demonstrate the water repellence. The samples were tilted to repel the water.

#### 2.4.2. Capillary water absorption test

The capillary water absorption test was used to quantify the ability of the concrete cubes to absorb water by capillary suction in accordance with ASTM C1585. The capillary test was conducted by placing three concrete samples on two Perspex rods in a tray of water of 2–5 mm depth. All the square surface of concrete samples

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