



Superhydrophobic coatings prepared from methyl-modified silica particles using simple dip-coating method

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Abstract

A simple and time-saving approach for preparing self-cleaning superhydrophobic silica coatings using a dip-coating technique is reported in this study. Commercially available silica particles were modified with methyl groups using methyltrichlorosilane as a modifying agent. By adopting a multi-layer deposition process, a superhydrophobic silica coating with a water contact angle of $153^\circ \pm 2^\circ$ and roll-off angle of $8^\circ \pm 1^\circ$ was obtained. The prepared silica coating exhibited excellent self-cleaning performance; moreover, it was able to maintain superhydrophobicity under the impact of a water jet. This method could be an effective strategy for fabricating self-cleaning superhydrophobic surfaces for promising industrial applications.

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1. Introduction

Many biological surfaces, particularly some plant leaves [1,2], exhibits remarkable water repellence (superhydrophobicity). As a result, they are highly self-cleaning. The well-known high water repellency and self-cleaning properties of lotus leaves [3] have attracted a lot of attention and have generated much interest in fundamental research, as well as in industrial applications. In the practical design of multifunctional surfaces, artificially mimicking the functionalities of the lotus leaf surface for solid surfaces could help meet the increasing need for self-cleaning materials in daily real-world and industrial applications. It is well known that the wettability of a solid surface can be controlled by its chemical

composition and morphological structure (i.e., surface roughness) [4]. An appropriate combination of the surface roughness and low surface energy is one of the key criteria for preparing artificial superhydrophobic surfaces. Numerous methods for developing artificial superhydrophobic surfaces and their possible applications have been summarized in recent review articles [5–10]. Many studies have attempted to synthesize rough or patterned solid surfaces to obtain superhydrophobic properties using expensive equipment and tedious procedures. Moreover, some methods for creating superhydrophobic surfaces require the repetition of the entire experimental process, which can be time-consuming and laborious reducing their applicability [11]. Therefore, the development of a simplified synthetic method for preparing superhydrophobic surfaces has remained a challenge. Therefore, in this article, we discuss a simple, economical, and time-saving method for preparing superhydrophobic silica coatings on glass substrates using dip coating. Silica was chosen as the coating material because it exhibits excellent intrinsic characteristics such as non-toxicity, high

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thermal and mechanical stability, and easy structural regulation [12]. Hence, it becomes very popular and is widely used in the field of coating technology, especially for preparing superhydrophobic silica coatings. Different hydrophobic organosilane compounds, in combination with silica precursors, have been applied to various substrates for preparing superhydrophobic silica coatings using different methods [13–18]. Gao and McCarthy [19] prepared a perfectly hydrophobic surface with a water contact angle of $> 170^\circ$ on a silicon wafer by submerging it in a toluene solution of methyltrichlorosilane (MTCS) at room temperature. Khoo and Tseng [20] described in detail the hydrophobic engineering of three-dimensional nano-architectures with various shapes, morphologies, and sizes using the phase separation of MTCS on glass and silica substrates. Khalilabad and Yazdanasheenas [21] demonstrated a dip-pad-dry coating method for depositing graphene oxide on cotton fibers. When combined with a subsequent reaction with MTCS, the method yielded polymethylsiloxane (PMS) nanofilaments on the fiber surface, which converted highly water-absorbing insulator cotton fabrics into textile-based substrate conductors with superhydrophobic properties. Shirgholami et al. [22] recently reported the deposition of polymethylsilsesquioxane (PMSQ) nanostructures onto the surface of cotton fabric and adopted the subsequent modification in a toluene solution of MTCS at room temperature with different duration times to achieve superhydrophobic cotton fabrics. The present work offers an effective and simple method for preparing self-cleaning superhydrophobic coatings on glass substrates. In our experiment, silica particles were easily modified by methyl groups using MTCS as a modifying agent; they were then applied to a glass substrate using a simple dip-coating technique.

2. Experimental

2.1. Materials

Silicon dioxide (SiO_2) particles ($\sim 99\%$) with diameters in the range $0.5\text{--}10\text{ }\mu\text{m}$ ($\sim 80\%$ in the range $1\text{--}5\text{ }\mu\text{m}$), MTCS ($\geq 97\%$), and toluene (anhydrous, 98.8%) were purchased from Sigma Aldrich, U.S.A., and all the chemicals were used as received without any further purification. Glass micro-slides (Blue Stars[®]) were purchased from Polar Industrial Corporation (India).

2.2. Preparation of superhydrophobic silica coating

Prior to coating, the glass micro-slide substrates were cleaned thoroughly using the procedure described in our earlier report [23]. The method employed for preparing the superhydrophobic silica coating was as follows: initially, 1 g of pristine SiO_2 particles was dispersed in 20 ml of toluene and ultrasonicated for 15 min. In the next step, 0.6 ml of MTCS was added slowly to this mixture, which was again ultrasonicated for another 2 h to obtain homogeneous solution. Finally, the coatings were prepared using a simple dip-coating method at room temperature ($\sim 27^\circ\text{C}$). A schematic of the dip-coating process is shown in Fig. 1. A well-cleaned glass substrate was dipped into the coating solution for 5 s and then

withdrawn at a constant speed of $\sim 5\text{ mm/s}$ after which was kept in air at room temperature for 5 min. The same coating procedure was repeated two more times under a fixed condition with the same sample to obtain a uniform and rough-textured silica coating. All the prepared coatings were first kept in room temperature for an hour to enhance the adhesion of the coating material to the substrate, and then annealed in air at 150°C for 5 h with a ramping rate of 2°C/min to eliminate the residual solvent. Finally, silica coatings prepared after one, two, and three dips were characterized in detail by suitable techniques.

2.3. Characterizations

The surface morphologies of the coatings were analyzed using a field emission scanning electron microscope (FE-SEM) (Hitachi, S-4800), and surface chemical analyses of the coatings were performed using the energy dispersive X-ray (EDX) method for a sample area of $100\text{ }\mu\text{m}^2$. The surface topographies and roughness values of the prepared coatings were analyzed using laser microscopy (KEYENCE, VK-X200 series). Here, the surface roughness of each coating was measured for a $50 \times 50\text{ }\mu\text{m}^2$ planar area in the non-contact mode. The surface roughness of each coating was measured in at least ten different areas, and the average value of these measurements was taken as the final value. Optical photographs were obtained using a Canon digital camera (G 15 series). The static water contact angles and sliding angles were measured over five different areas on a sample using a contact angle meter (Ramehart Instrument Co., USA), and the average value of these measurements was selected as the final value. The angle at which a placed water droplet started to move from the coated surface was recorded as the sliding angle value.

3. Results and discussion

3.1. Surface morphology and chemical composition

In general, rough-textured solid structures with a low surface energy demonstrate strong non-wetting characteristics. Fig. 2(a–c)

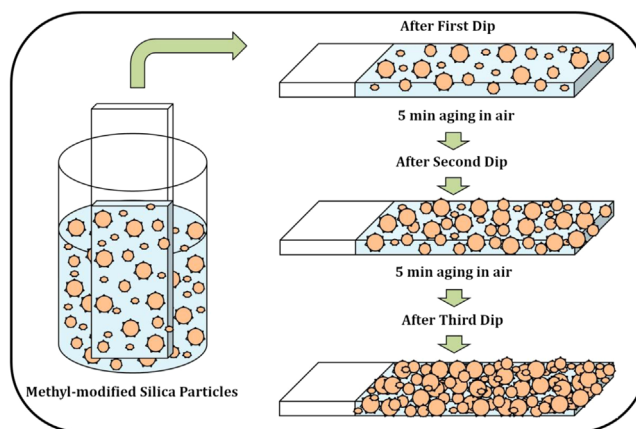


Fig. 1. Schematic showing simple dip-coating process for deposition of methyl-modified silica particles on glass substrate.

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