



Full Length Article

Fabrication of recyclable superhydrophobic cotton fabrics



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ABSTRACT

Commercial cotton fabric was coated with SiO₂ nanoparticles wrapped with a polydimethylsiloxane (PDMS) layer, and the resulting material surface showed a water contact angle greater than 160°. The superhydrophobic fabric showed resistance to water-soluble contaminants and maintained its original superhydrophobic properties with almost no alteration even after many times of absorption-washing cycles of oil. Moreover, superhydrophobic fabric can be used as a filter to separate oil from water. We demonstrated a simple method of fabrication of superhydrophobic fabric with potential interest for use in a variety of applications.

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1. Introduction

Superhydrophobicity refers to the phenomenon that the static water contact angle of a surface is greater than 150°, and the contact angle hysteresis is smaller than 10° [1–8]. When a surface with microscale roughness superposes nanoscale roughness, water droplets can be repelled by air pockets that exist at the interface between the water and rough solid surfaces, resulting in extremely high water-repellent properties [3,9–11]. Various strategies can be used to produce superhydrophobic surfaces [2,12–16]. Among these strategies, distribution of hydrophobic nanoparticles on surfaces of various substrates can be particularly considered as the most effective way of preparing superhydrophobic surface due to its simplicity of processing. Note that nanoparticles can spontaneously agglomerate into larger sub-micrometre-scale clusters, resulting in dual surface roughness [7,17–24]. Superhydrophobic materials have a wide range of potential applications; in the present work, we aimed in the efficient treatment of wastewater contaminated by oil [25–31]. On account of cost-effectiveness and efficiency for the removal and collection of oil, using absorbent materials is considered as the most desirable way for the wastewater treatment. Some of the absorbent materials with poor hydrophobicity absorb both water and oil, and due to the absorbed water, they cannot absorb enough oil up to their maximum capacity and this makes

them non-effective for the removal or separation of oil contaminants from water [25–29,32,33]. Therefore, the superhydrophobic materials which could selectively absorb oil and oil-based organic contaminants but repel water draw much attention for their high efficiency and selectivity for removal and separation of oil from water.

The oil-saturated superhydrophobic filter should be replaced with a new filter or regenerated one. For the recycling of the oil-saturated filter, the absorbed oil in the superhydrophobic filter should be washed by easy and simple methods and superhydrophobicity of the filter should be maintained even after many times of repeated absorption-washing process [3,5,7,34,35]. In the present work, we used polydimethylsiloxane (PDMS)-coated SiO₂ nanoparticles to manufacture superhydrophobic cotton fabric. Particularly, our fabrication process for the superhydrophobic cotton fabric is simpler and less time-consuming [7,16,19,20,36–39]. Also, it is worth to emphasize that our superhydrophobic cotton fabric shows higher durability upon exposure to the water and oil environments for a long time comparing to the hitherto reported methods, i.e., the methods using only PDMS without SiO₂ nanoparticles [40,41]. In addition, the possibility for using superhydrophobic fabric as a filter for isolating oil from water was explored. The reusability of the contaminated and oil-covered superhydrophobic fabric was also demonstrated.

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2. Experiments

2.1. Materials

SiO₂ nanoparticles (Degussa, Aerosil 200, mean particle size: 24 nm), fluidic PDMS (Dow Corning, Sylgard 184) and hexane (DAE-JUNG, purity: 95%) were used as received. The polydispersity index (PDI), average molecular weight (M_n), and weight average molecular (M_w) of the PDMS used in the present work were ~ 3.3 , $\sim 4200 \text{ g mol}^{-1}$, and $\sim 14000 \text{ g mol}^{-1}$, respectively, according to gel permeation chromatography (GPC, Agilent 1100s). Cotton fabric (100-count) was purchased at a local market.

2.2. Preparation of PDMS-coated SiO₂ nanoparticles

PDMS-coated SiO₂ for the fabrication of a dipping solution was prepared using a thermal vapour deposition method [21,23,24]. SiO₂ nanoparticles and PDMS were placed in a stainless steel reactor at a weight ratio of 1:1, and they were separated using a metal mesh (30 mesh) partition (with PDMS on the bottom of the stainless steel chamber and SiO₂ on top of the mesh). During the PDMS deposition process, the reactor was sealed with polyimide (PI) tape, and the temperature of the reactor was maintained at 300 °C for 15 h. Following the aforementioned process, thin PDMS layers were deposited on the surfaces of the SiO₂ nanoparticles.

2.3. Fabrication of superhydrophobic fabric

A dipping solution was prepared by mixing solution A with solution B at a volume ratio of 29:1 (We used 29 ml of solution A and 1 ml of solution B). Solution A was obtained by dispersing 0.2 g of PDMS-SiO₂ nanoparticles in 29 ml of hexane; solution B was prepared by mixing PDMS, a curing agent, and hexane at a volume ratio of 1:0.1:8 (we used 1 ml of PDMS, 0.1 ml of curing agent, and 8 ml of hexane) [24].

Before the dip-coating process, the cotton fabric (100 counts, cut to a size of 4 cm × 4 cm, or 10 cm × 10 cm) was cleaned with sonication in acetone for 10 min and then dried at room temperature. The cleaned fabric was dipped into the prepared dipping solution for 10 s and dried for 3 min under atmospheric conditions while hanging vertically. This dip-coating process was repeated three times for each fabric sample.

Some of the dip-coated samples were annealed at 150 °C for 20 min and simultaneously pressed by a Steel-Use-Stainless-plate at a pressure of 15.2 g cm⁻². Hereafter, samples dip-coated without any additional treatment and dip-coated samples with post-annealing are referred to as “dip-fabric” and “dip-annealed-fabric,” respectively.

For the comparison of the hydrophobicity with our dip-annealed-fabric, PDMS/hexane-dip-annealed-fabric and PDMS-vapour-annealed-fabric were also fabricated. Dipping solution for the fabrication of PDMS/hexane-dip-annealed-fabric was prepared by mixing PDMS, a curing agent, and hexane at a volume ratio of 1:0.1:50 (we used 1 ml of PDMS, 0.1 ml of curing agent, and 50 ml of hexane) [40]. The dipping solution was stirred for 30 min, and the washed cotton fabric was immersed in the solution for 30 s and subsequently dried for 30 min under atmospheric conditions while hanging vertically. In the case of the fabrication of PDMS-vapour-annealed-fabric, the washed cotton fabric and PDMS (300 g) were placed in a stainless steel reactor (internal diameter: 640 mm, height: 800 mm) with PDMS on the bottom of the stainless steel chamber and the washed cotton fabric hanging vertically at the centre of the chamber [41]. During the PDMS deposition process, the reactor was sealed with aluminium foil, and the temperature of the reactor was maintained at 220 °C for 2 h. After dip-coating and

vapour-deposition, all samples were post-annealed with the same method as that for our dip-annealed-fabric.

2.4. Characterization of the dip-coated fabric

The surface structure of the PDMS-SiO₂-coated fabric prepared through dip-coating was analyzed using scanning electron microscopy (SEM, JEOL, JSM-7100F). In order to characterize the chemical functionality of the surface of the dip-coated sample, Fourier transform-infrared (FT-IR, BRUKER, Optics/vertex 70) spectroscopy was used. The baseline of the FT-IR measurement was set using bare fabric.

The resistance of various fabric samples toward water was verified by measuring the water contact angle of the sample using a Theta Optical Tensiometer model (KSV instruments, Ltd.) with a digital camera connected to a computer. The static contact angle was obtained by dropping $\sim 4 \mu\text{l}$ of distilled water onto the sample, while the dynamic contact angle was measured by gradually adding and removing water. Water contact angle hysteresis was calculated as the difference between the advancing and receding contact angles. The advancing contact angle is the maximum water contact angle value measured by adding water to a water droplet, while the receding contact angle is the minimum water contact angle value obtained during the removal of water from a water droplet. The water contact angle values denoted in the present work were obtained by calculating the average of three contact angles from different spots on each sample. In addition, in order to compare the mechanical stability of the superhydrophobic coating on the dip-fabric and dip-annealed-fabric, the contact angle of each sample was measured after ultrasonically treating each sample in soapy water for 1 h. The sonicated samples were subsequently washed with distilled water and acetone to remove any soap residues; after that, they were dried under atmospheric conditions before measurement of the water contact angle.

2.5. Washing experiment

In order to verify the resistance of our samples to water-soluble contaminants, bare and dip-annealed-fabric were floated on an aqueous methylene blue (MB, 3 ppm) solution for 6 h. After the exposure process, the dip-annealed-fabric was washed in soapy water with magnetic stirring (with a rotation speed of 800 rpm for 30 min) in order to remove the small stain of MB from the surfaces of the fabrics. The sample was rinsed with pure water and dried under atmospheric conditions, after which the water contact angles were measured.

To demonstrate that oil on the surface of dip-annealed-fabric could be removed, the dip-annealed-fabric was immersed in dyed rotary pump oil (Pochem Korea, PV-200) for 1 min and washed in soapy water under the same conditions as discussed above. Alternatively, the samples wetted with dyed oil were washed in acetone with sonication for 3 min and this process was repeated 20 times in order to verify the stability of the superhydrophobic coating on the sample. Between each absorption-washing experiment, the water contact angle of the dip-annealed sample was measured.

2.6. Measurement of weight gain

The oil and water weight gains (the maximum mass of absorbed oil or water divided by the mass of the sample) of various fabric samples (4 cm × 4 cm) were measured to verify the oil absorption capacity and resistance toward water of each sample. To measure the oil weight gain, samples were immersed in one of the four different types of oil (rotary pump oil (Pochem Korea, PV-200), hydraulic oil (Pochem Korea, POWER-32), sesame oil (Ottogi) and silicone oil (Xiameter, PMX-200)) and pressed with mesh for 1 min

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