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Mask-assisted electrospray for superoleophobic surfaces: An experimental and numerical study

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ABSTRACT

This paper presents both experimental work and numerical simulations of formation of superoleophobic surfaces created by mask-assisted electrospraying, followed by a second layer overlay and fluoropolymer treatment. The primary electric field focusing in the mask-assisted electrospray effectively guides the electrosprayed particulates into the mesh openings, forming characteristic pyramid-shaped pillars. The secondary focusing occurs during the overlay deposition when the electrosprayed particulates favorably deposit onto the pre-patterned pillars. Systematic studies were conducted on the effects of mask-substrate-gap and duration of the overlay deposition on the pattern morphology and wetting performance. A shorter mask-substrate-gap results in a stronger focusing effect and pillars with a larger aspect ratio. The overlay deposition firstly increases the pillar height and then changes the pillar shape from pyramids to domes with overhangs due to electrostatic interactions. All the surfaces are superhydrophobic, however, superoleophobicity varies. Surfaces that have tall pillars and overhang structures demonstrate robust superoleophobicity when compared to their counterparts with shorter pillars and absence of overhang structures. The primary and secondary electric field focusing effects exerted by the mask and the pre-patterned pillars, and their roles in pattern formation have been numerically investigated by COMSOL Multiphysics simulation. A reasonable agreement has been obtained between the numerical predictions and experimental results.

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1. Introduction

Electrospray is a low-cost, facile, and effective coating process by which liquids are sprayed onto a substrate through electrostatic interactions to form functional micro/nano structured coatings [1,2]. A liquid meniscus forms at the nozzle exit, the final shape of which is determined by the combinational forces of surface tension, hydrostatic pressure, and coulomb repulsion due to charge accumulation at the meniscus surface. A very thin jet develops at the apex of the meniscus when coulomb repulsive force overcomes surface tension and further breaks up into fine droplets when Rayleigh limit is reached [3]. These droplets carry the same polarity charges as the electric potential applied to the nozzle. When the charged droplets move away from the nozzle tip, they expand into an electrospray plume [4]. As the droplets deposit onto the substrate and the solvent evaporates, micro/nano structured coatings can be obtained. Usually the substrate needs to be conductive, so that the deposited charges will be dissipated quickly without affecting the subsequent deposition.

As a potentially scalable coating technique, electrospraying has been utilized in fabricating superhydrophobic surfaces. Simsek et al. [5] created superhydrophobic surfaces with dual scale roughness by electrospraying a copolymer of styrene and a perfluoroacrylate monomer in good/poor solvent systems. Hu et al. [6] co-electrosprayed poly(L-lactide) and modified silica nanoparticles to form superhydrophobic surfaces with convex and concave microstructures and investigated its anti-bioadhesion properties. Superhydrophobic surfaces with hierarchical roughness have also been fabricated with organosilane-coated alumina particles [7] and silica micro particles decorated with gold nanoparticles via electrospray deposition [8]. Similar to other coating techniques, e.g. air brush spray [9–12], plasma spray [13,14], chemical vapor deposition [15], and electrochemical deposition [16], these research works have only demonstrated superhydrophobic surfaces with micro/nanoscale random roughness. However, superoleophobicity is more challenging since low surface tension liquids tend to wet textured surfaces unless a re-entrant and/or overhang structure is provided [17–20]. In addition, patterned surfaces with larger spacing, which are particularly beneficial in providing larger slip length and drag reduction [21,22], have not yet been fabricated through these large area coating techniques.

Our previous investigation on the surfaces created by mask-assisted electrospray [23] demonstrated both superhydrophobicity and superoleophobicity with water and hexadecane; direct evidence was provided that both water and hexadecane droplets exist in the Cassie-Baxter state, "sitting" on the apex of the pillars. In this study, we

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experimentally and numerically investigated the processing-structure-performance relationships through mask-assisted electrospraying and subsequent overlaying process, pillar morphology, and surface wetting property characterizations. Specifically, the effects of mask-substrate-gap, and duration of overlay on the pillar morphology and wetting performance were systematically studied. The electric field and pattern formation process were simulated by COMSOL Multiphysics. The wetting robustness of the superhydrophobic and superoleophobic surfaces were also discussed.

2. Material and methods

2.1. Materials

A SiO_2 suspension AERODISP W7512S was obtained from Evonik containing 11–13% solid concentration and with a primary particle size of ~13 nm. Poly(diallyldimethylammonium chloride) (PDADMAC), 20 wt.% in water was purchased from Sigma Aldrich. A fluoropolymer, 1H, 1H, 2H, 2H-Perfluorodecyltrichlorosilane 96% purity (FDTS) was obtained from Alfa Aesar. Deionized (DI) water with resistivity of 18.2 MΩ·cm was produced by Barnstead Smart2Pure water purification system (Thermo Scientific). Hexadecane (99% pure, ACROS) was purchased from Fisher Scientific. All materials were used as received without further purification.

2.2. Methods of fabricating superoleophobic surfaces via mask-assisted electrospray

Fig. 1a shows a schematic of the electrospray experiment setup. A 250 μL syringe from Hamilton was connected to a stainless steel nozzle of 0.5 mm inner diameter by a PTFE tubing. A syringe pump (NE-4000) from New Era Pump System was utilized to control the electrospraying flow rate. A vacuum sample holder was attached to a fine-adjust cross-slide table. Both the table and the attached sample holder were grounded. A positive voltage was applied to the nozzle by a high voltage power supply (Bertan Series 230) from Spellman. A light source (Fiber-Lite MI-152) from Dolan-Jenner and a CCD camera from Edmond (EO-2013) were used to monitor the meniscus and to ensure a stable electrospray. A PTFE coated stainless steel wire mesh, having a ~36 μm wire diameter and a ~79 μm center-to-center distance, was purchased from TWP Inc. and used as mask to assist the fabrication of patterned surfaces.

In this study, a Fisher Scientific microscope glass slide with a 1 mm thickness was used as a substrate to fabricate superhydrophobic and superoleophobic surfaces. The substrate was first cleaned by following the order of hot soapy water, Acetone, and Isopropanol then rinsed with DI water and dried by clean compressed air. The glass slide was further treated by a plasma cleaner (PDC-001-HP-115V from HARRIC PLASMA) for 5 min to assure that the substrate was thoroughly cleaned. A thin layer of PDADMAC (~52 mg/mL in DI water) was spin coated onto the previously cleaned glass slide by a spin coater (SCS 6800 Specialty Coating Systems) at 3000 rpm. The as-received SiO_2 dispersion was diluted with Isopropanol to a concentration of 15 mg/mL and used for the electrospray process. The mesh was attached to the glass slide at various gaps using an insulating spacer. A bias electric potential was applied to the mesh mask. In the present study, the working distance, nozzle voltage, bias voltage, and suspension flow rate are 50 mm, 10 kV DC, 2 kV DC, and 3 μL/min, respectively.

The procedure for generating patterned microscale roughness of SiO_2 nanoparticles on the substrate is illustrated in Fig. 1b. A 3 μL/min flow rate of SiO_2 dispersion was electrosprayed through the mask for 90 min. Then the mesh was removed, followed by overlaying a layer of electrosprayed SiO_2 without the mask for various durations of time. The sprayed sample was baked for 1 h at 140 °C in a convection oven (Isotemp 700 from Fisher Scientific) to dry off any solvent in the coating. Finally the samples were plasma-cleaned for 5 min before FDTS treatment through a vapor deposition process at 90 °C for 15 min. The detailed procedures have been given in our previous report [23].

2.3. Surface characterization

An ultra-high resolution scanning electron microscope (HITACHI SU-70 FE-SEM) with 5 kV and 15 mm scanning distance was used to characterize the morphology of fabricated textured surfaces. To minimize charging effect of silica particles, the samples were coated with platinum using a platinum sputter (Denton Vacuum Desk V) for 60 s. The pillar height was characterized by a laser scanning microscope (Zeiss, LSM 710). Five groups of 10 pillars were measured and the average pillar height was obtained.

Contact angle and sliding angle measurements were conducted on a goniometer (OCA 15) from Dataphysics. For apparent contact angle measurements, 3 μL droplets of DI water and hexadecane were gently dispensed on the testing surface. The Young-Laplace fitting was used

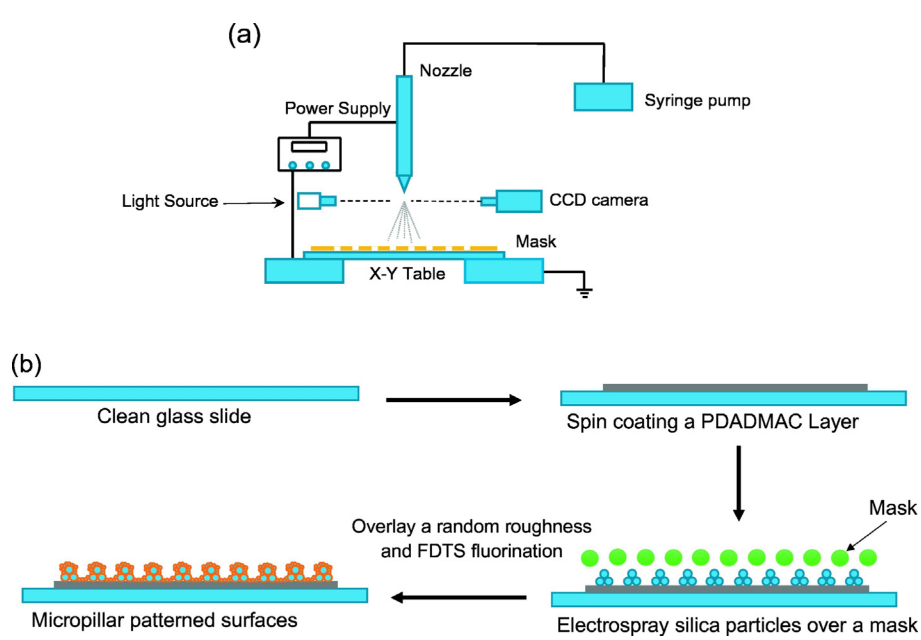

Fig. 1. Schematics of (a) the mask-assisted electrospray experimental setup; (b) fabrication procedure of superoleophobic surfaces with patterned multiscale roughness.

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