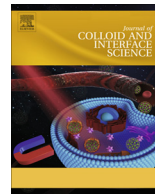




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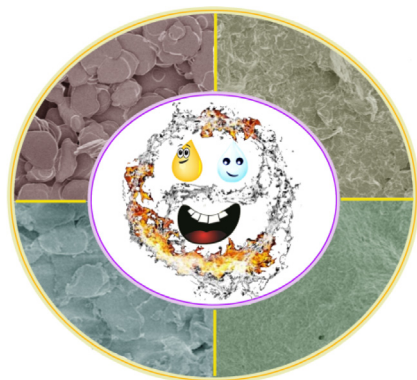
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## Regular Article

## Superhydrophobic coatings with high repellency to daily consumed liquid foods based on food grade waxes

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## GRAPHICAL ABSTRACT



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## ABSTRACT

The applications of superhydrophobic coatings in daily life are receiving increasing attention. Here, we report a general approach for preparing superhydrophobic coatings with high repellency to daily consumed liquid foods based on food grade waxes. The coatings are prepared by spray-coating the homogeneous wax suspensions in ethanol followed by annealing at 40 °C. The wax suspensions are formed by the heating dissolution-cooling precipitation-ultrasonication process thanks to the unique solubility of the waxes in ethanol. Ultrasonication of the wax suspension is helpful to improve superhydrophobicity by reducing the size of the wax microplatelets. Annealing at 40 °C could enhance mechanical stability of the coatings. The coatings are superhydrophobic with a water contact angle of 158.2° and a sliding angle of 7.3°. The coatings are resistant to intense water jetting and immersion in corrosive aqueous solutions. In addition, the coatings show excellent anti-adhesive properties for various liquid foods including cola, honey, milk and yoghurt. Moreover, the coatings are applicable onto different substrates (e.g., glass slide, PET plate and polyethylene plate) and could be prepared using different waxes (e.g., paraffin wax, beeswax and microcrystalline wax). We believe that the wax superhydrophobic coatings could find applications in various fields such as anti-adhesion of liquid foods, fruit preservation and anti-bioadhesion, etc.

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## 1. Introduction

Superhydrophobic coatings, characterized by high water contact angles (CAs > 150°) and low sliding angles (SAs < 10°), have attracted great attention. The articles about superhydrophobic coatings increase at an annual rate of ~15% according to the records of Web of Science. This is owing to the unique properties of superhydrophobic coatings, such as self-cleaning [1–3], anti-icing [4,5] and anti-corrosion [6–8]. The applications of superhydrophobic coatings cover a wide spectrum including anti-wetting fabrics [9], drag reduction of ships [10] and oil/water separation [11–13]. So, various methods have been developed to prepare superhydrophobic coatings including chemical etching [14,15], electrodeposition [16–18], nanocasting [19,20], sol-gel [21] and anodic oxidation [22]. Most of superhydrophobic coatings are prepared by construction of micro-/nanostructures on the surface of substrates followed by decreasing the surface energy of the micro-/nanostructures [23,24]. Such two-step methods are effective in preparing superhydrophobic coatings, but are inconvenient for the real-world applications in most cases. On the other hand, for preparation of superhydrophobic coatings, fluorinated compounds including perfluoroalkylsilanes and fluoroacrylic copolymers are frequently used because of their very low surface energy [25–28]. However, fluorinated compounds are causing increasing concerns because these compounds and their byproducts of degradation have potential adverse effects on human offspring and environment [29,30]. Thus, all-in-one methods for constructing surface micro-/nanostructures and decreasing surface energy using fluoro-free and water-borne compounds are of great interests [31].

In recent years, the applications of superhydrophobic coatings in daily life are receiving increasing attention. Zhu et al. prepared polypropylene fabrics super-repelling various liquids including blood, urine, milk and coffee by solvent swelling [32]. Zhang et al. prepared superhydrophobic coatings on clothes and shoes by spraying a fluorinated acrylic random copolymer micelle solution [33]. We paid our attention to reduce the waste of daily consumed liquid foods (cola, orange juice, milk, porridge, honey, jam and yoghurt) using superhydrophobic coatings, as the residual liquid foods adhered to containers are as high as 15% [34]. As mentioned above, superhydrophobic coatings have the unique self-cleaning property. If a superhydrophobic coating is applied in a facile approach to the inside wall of containers, the residual liquid foods in the containers can be greatly reduced or even eliminated. However, superhydrophobic coatings that can be brought into direct contact with food are very rare. Most of the materials for preparing superhydrophobic coatings are toxic or potentially toxic to the human body. Among all the available compounds, food grade waxes are ideal materials for preparing superhydrophobic coatings inside containers of the daily consumed liquid foods. Bayer et al. prepared a solvent-resistant superhydrophobic film using carnauba wax-alcohol emulsion [35]. Kota et al. prepared superhydrophobic coatings using suspensions of beeswax and carnauba wax in acetone [36]. However, this method is not suitable for preparing superhydrophobic coatings using the most frequently used food grade paraffin wax. Also, acetone was used as the medium, which may cause side-effects such as nausea, headache and vomit.

Here, we report a general all-in-one method for preparing superhydrophobic coatings with high repellency to daily consumed liquid foods based on food grade waxes like paraffin wax and beeswax. The coatings feature high CAs and low SAs for various liquid foods. Moreover, the superhydrophobic coatings could withstand intense water jetting and are applicable onto various substrates, which pave the way for their real-world applications in anti-adhesion of liquid foods.

## 2. Experimental section

### 2.1. Materials

Paraffin wax ( $\geq 99\%$ ), beeswax ( $\geq 99\%$ ), microcrystalline wax ( $\geq 99\%$ ) and carnauba wax ( $\geq 99\%$ ) of food grade were supplied by Yousuo Chemical Technology Co. Ltd. Shandong, China. Food grade ethanol (95%) was purchased from Lizhi Chemical Technology, Co. Ltd. Shandong, China. Glass slides (24 mm  $\times$  50 mm) were purchased from Menzel, Braunschweig, Germany.

### 2.2. Preparation of paraffin wax suspensions

Typically, 0.3 g of granular paraffin wax was dropped in 100 mL of ethanol (95%) in a conical flask in an 80 °C water bath. Paraffin wax gradually dissolved in ethanol in ~5 min under continuous stirring. Then, the flask was taken out of the water bath, and left to stand at room conditions until complete precipitation of paraffin wax in ~20 min. Finally, the suspension was homogenized using an ultrasonic cell crusher (600 W) for 2 h. After 2 s of ultrasonication, there was an interval of 1.5 s. The suspensions of the other waxes were prepared according to the same procedure. The dispersibility of the suspension is not high, and precipitates were formed in about 0.5 h. This is because the wax microplatelets are big. The suspension can be redispersed very well by magnetic stirring.

### 2.3. Preparation of paraffin wax coatings

Glass slides as substrates were washed with ethanol and distilled water, and then dried under N<sub>2</sub> flow. Then, the paraffin wax suspension (4 mL) was spray-coated onto a piece of the vertically placed glass slide using an airbrush with 0.2 MPa N<sub>2</sub>. Finally, the coating was annealed at 40 °C in an oven for 5 min to enhance the mechanical stability. The coatings based on different waxes and on different substrates were prepared according to the same procedure.

### 2.4. Characterization

The CAs and SAs of various liquids on the coatings were measured using a Contact Angle System OCA20 (Dataphysics, Germany) at room temperature. The reported data were the averages of six readings at different places on the samples. The micrographs of the samples were taken using a field emission scanning electron microscope (SEM, JSM-6701F, JEOL). Before SEM observation, the samples were fixed on the copper stubs and covered with gold (~7 nm). The surface chemical composition was analyzed by X-ray photoelectron spectroscopy (XPS) using a VG ESCALAB 250 Xi spectrometer. The spectra were recorded in the constant pass energy mode with a value of 100 eV, and all binding energies were calibrated using the C 1s peak at 284.6 eV as the reference. The Fourier Transform Infrared (FTIR) spectra of samples were collected on a Thermo Nicolet NEXUS TM spectrophotometer (Thermo, Madison, USA) in the range of 4000–400 cm<sup>-1</sup> using KBr pellets. The surface tension of various liquids was measured using an optical tensiometer (PDT60, NB Scientific Instruments, China).

## 3. Results and discussion

### 3.1. Preparation of paraffin wax suspensions and superhydrophobic coatings

The schematic illustrations for preparing the paraffin wax suspensions and superhydrophobic coatings are shown in Fig. 1.

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