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Biobased super-hydrophobic coating on cotton fabric fabricated by spray-coating for efficient oil/water separation

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ABSTRACT

Super-hydrophobic materials for oil/water separation are usually prepared from non-renewable feedstocks, which does not coincide with the sense of sustainable development. Herein, we report a sustainable super-hydrophobic material fabricated via a simple spray-coating method from biobased feedstocks including epoxidized soybean oil, sebacic acid, stearic acid, zinc oxide and cotton fabric. Rough surface structure was successfully constructed by spraying epoxidized soybean oil (ESO) with sebacic acid (SA) and nano-ZnO. After modification with stearic acid (STA), the surface became super-hydrophobic with a water contact angle (WCA) of 155°. The super-hydrophobic fabric shows excellent stability without losing super-hydrophobicity after immersion in water and oil for several days. The super-hydrophobic fabric can selectively separate water mixtures with various oily liquids with separation efficiency higher than 97.5%. With high separation efficiency and excellent stability, the biobased super-hydrophobic fabric can be used as a sustainable and eco-friendly oil/water separation material.

1. Introduction

The increasing emission of industrial oily wastewater and frequent oil spill accidents have caused both environmental pollution and waste of resources, which has become one of the most urgent global concerns [1–3]. Therefore, it is necessary to develop long-term durable and high efficient oil/water separation materials to directly separate oil/water mixtures or selectively absorb oil from oily wastewater [4–6]. In recent years, much attention has been paid to super-hydrophobic materials, which are able to separate oil/water mixtures or absorb oil from wastewater [7–12]. Various materials with permeability or absorbability such as textiles [13–17], metal meshes [18–20], sponges [21–25] and aerogels [26–28] can be used as possible oil/water separation materials. After super-hydrophobic modification, those materials showed excellent oil/water separation behavior due to the special water-resistance and specific oleophilicity.

Super-hydrophobicity is realizable through imitation of the surface structure of some naturally occurring super-hydrophobic living organisms, such as lotus leaves and water striders, of which the surface structures are well known for micro/nanoscale roughness with low surface energy [29–32]. Thus, the various techniques to prepare super-

hydrophobic surfaces usually consists of two steps, i.e., rough surface structure creation and subsequent low surface energy modification [33–36]. One versatile strategy involves coating nanoscale particles onto a substrate surface to create a micro/nanoscale structure, and subsequently modification of the surface with low energy substances such as long-chained hydrocarbon or fluorine-containing substances [37–39]. Dip-coating and spray-coating methods can easily construct micro/nanoscale rough structures on various substrates, thus, both coating methods are widely used to fabricate super-hydrophobic materials [40–42]. Adhesives are required to locate between the surface nano-particles and the substrate to make the particles and the substrate combine tightly to exhibit good durability. Metal meshes, sponges, aerogels and synthetic fiber fabrics [8,26,40] are widely used as the substrate, and diglycidyl ether of bisphenol-A (DGEBA) based epoxy thermosets, poly(methyl methacrylate) (PMMA), polystyrene (PS) and polydimethylsiloxane (PDMS) [38,42–45] were usually used as the adhesive. Most of those materials are derived from nonrenewable resources and are unable to biodegrade after being discarded, which not only increases precious nonrenewable resource consumption but also causes secondary environmental pollution [40,46,47].

Therefore, renewable and biodegradable materials, such as cellulose

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based materials and poly(lactic acid) based non-woven fabrics, have attracted increasingly attention in fabricating super-hydrophobic oil/water separation materials [45,48–51]. Although the substrates are renewable and biodegradable, the other components such as adhesives and surface low energy modifier used in those studies were still non-renewable and non-biodegradable [52–54]. So, it is highly desirable to develop a new generation of super-hydrophobic oil/water materials which are composed of renewable feedstocks and are able to biodegrade after their life time.

In this study, we report a super-hydrophobic oil/water separation material fabricated via a spray-coating method with cotton fabric as the substrate, sebacic acid cured epoxidized soybean oil (CESO) as the adhesive, nano-ZnO particles as the rough structure component and stearic acid as the low surface energy modifier. Dicarboxylic acid cured epoxidized soybean oil (CESO) is able to biodegrade and has excellent adhesive properties [55], thus can be used as a potential renewable and biodegradable adhesive to replace nonrenewable and non-degradable adhesives. Both cotton fabric and stearic acid are renewable and biodegradable, and nano-ZnO is also a biodegradable substance with low cost [56–58]. Therefore, the super-hydrophobic material with all raw materials biodegradable and renewable can be used as an eco-friendly alternative to traditional oil/water separation materials.

2. Experimental section

2.1. Materials

Epoxidized soybean oil (ESO) with epoxy value of $\geq 6.0\%$ was purchased from Micxy Chemical Co., Ltd (Chengdu, China). The average number of epoxy group per an ESO molecule is 4.1 as calculated by NMR analysis. Sebacic acid (SA, 98.5%), stearic acid (STA, $\geq 99\%$) and the catalyst 1, 8-Diazabicyclo [5.4.0] undec-7-ene (DBU, $\geq 98\%$) were obtained from the Micxy Chemical Co., Ltd. Nano-ZnO (99.7%, 50 ± 10 nm) and silicon oil were received from Aladdin Industrial Corporation. Nano-ZnO was dried in an 80°C oven for 1 h prior to use. Decane was purchased from Shanghai Titanchem Co., Ltd. Chloroform, toluene, petroleum ether and absolute ethanol were obtained from Chuandong chemical plant (Chongqing, China). Cotton fabric was bought from a local store.

2.2. Preparation of the super-hydrophobic cellulosic fabric

The super-hydrophobic cotton fabric was fabricated by a spray-coating method. The substrates in absolute ethanol were treated with ultrasound for 10 min to remove possible impurities and then dried at 80°C for 1 h prior to use. The adhesive solution was prepared by dissolving 20 wt% ESO plus SA and 0.2 wt% DBU into absolute ethanol. The weight ratio of SA to ESO was 0.31:1 to get -COOH/epoxy molar ratio of 0.7:1 [59,60]. ZnO nanoparticles (1.0 g) were dispersed in 25 ml of absolute ethanol under ultrasound for 30 min to obtain a homogeneous suspension. Firstly, the substrates were spray-coated with the prepared adhesive solution and then the well spray-coated substrates were moved into a fan-assisted oven for 10 min to remove the ethanol. The nano-ZnO suspension was sprayed onto the desired substrate. Then, the coated substrates were dried and cured at 150°C for 3 h. The super-hydrophobic cotton fabric was obtained after immersing in 0.2 mol/L STA ethanol solution for 6 h followed by drying at 120°C for an hour. Spray gun (with a 0.5 mm inside diameter spray nozzle) was used to spray the prepared solution and nano-ZnO/ethanol dispersion onto cotton fabric substrate ($15\text{ mm} \times 15\text{ mm}$) for 5 s with 0.2 MPa pressure. The spray gun was cleaned with absolute ethanol and dried after each spraying process. The thickness of the pristine and coated cotton fabric was measured with a thickness gauge (CH-1-ST, Liuling instruments, Shanghai, China).

2.3. Oil/water separation

Hexane, decane, petroleum ether, toluene, chloroform and silicone oil were selected as the oil phases and all were colored red. All oil/water mixtures were composed of 20 ml dyed oil and 20 ml water. The oil/water separation capability was evaluated by using a filtering process with the as-prepared super-hydrophobic cotton fabric as the filter membrane. Before the separation process, a small amount of oil was poured onto the separation material to pre-wet the super-hydrophobic cotton fabric. Then, mixtures were slowly poured into the separation system, oil could easily penetrate through the fabric only driven by gravity, while water was repelled at the upper surfaces. After the separation, the weight of collected oil was measured to evaluate the separation efficiency. The separation efficiency (W) was calculated according to the equation:

$$W(\%) = \left(\frac{M_1}{M_2} \right) \times 100 \quad (1)$$

where M_1 and M_2 are the weight of the collected oil and the initial weight of oil, respectively. The super-hydrophobic cotton fabric was used to separate the same oil/water mixture several times to determine the reusing efficiency.

The flux of different oils penetrating the super-hydrophobic cotton fabric was calculated by:

$$F = \frac{V}{St} \quad (2)$$

where V is the volume of the collected oil, S is the surface area of separation material, and t is the time.

2.4. Durability measurement

The variation of WCA with time was measured after immersion in water and oil for several days to study the durability of the super-hydrophobic cotton fabric. Seven solutions with pH of 1, 3, 5, 7, 9, 11 and 13 were prepared to study the acidic or alkaline resistance of the super-hydrophobic cotton fabric.

2.5. Characterization

The water contact angle was measured by a contact angle meter (JC2000C1, China) at room temperature employing $5\ \mu\text{L}$ distilled water droplet. For each sample, five measurements were performed and the mean value was used as the final result. Scanning electron microscopy (SEM, S-4800 Hitachi, Japan) was used to investigate the surfaces morphology of the obtained super-hydrophobic cellulosic fabrics. The samples were sprayed with a layer of platinum prior to observation. The chemical composition of the as-prepared surfaces were analyzed by X-ray photoelectron spectroscopy (XPS, Escalab 250Xi). All samples were totally dried before the test.

3. Results and discussion

3.1. Surface morphology, wettability and coating thickness

Fig. 1 shows the SEM and WCA images of pristine and spray-coated cotton fabrics. For simplicity, CF, CESO/CF, ZnO/CESO/CF and STA/ZnO/CESO/CF were used to designate the original cotton fabric, the CESO coated fabric, the nano-ZnO and CESO coated fabric and the final super-hydrophobic cotton fabric, respectively. The CF shows a porous network weave structure (Fig. 1a₁) with smooth cellulosic fibers (Fig. 1a₂). The water droplet infiltrates (Fig. 1a₃) the pristine CF immediately during WCA measurement due to the hydrophilicity of the CF. After coated with ESO, the weave structure of CF remained almost unchanged compared to pristine CF (Fig. 1b₁). However, although the microfibrils are still smooth, the individual microfibrils were difficult to

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