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One-step candle soot-PDMS dip-coated superhydrophobic stainless steel mesh for oil-water separation

Rajaram S. Sutar [a], Sanjay S. Latthe [a,b,*], Xinna Wu [a], Bairu Shi [a,c], Nikhil N. Pargaonkar [d], Sagar S. Ingole [d], Anand N. Biradar [b], Saravanan Nagappan [e], Yong Hyun Kim [f,g], Appasaheb K. Bhosale [d], Viswanathan S. Saji [h], Shanhu Liu [a,*]

[a] College of Chemistry and Molecular Science, Henan University, Kaifeng 475004, China
[b] Department of Physics, Vivekanand College (Autonomous), 416003 Maharashtra, India
[c] PetroChina Research Institute of Petroleum Exploration & Development, Beijing 100083, China
[d] Department of Physics, Raje Ramrao College, Jath, Sangli 416404, Maharashtra, India
[e] Industry-University Cooperation Foundation, Pukyong National University, Busan 48513, Republic of Korea
[f] Department of Smart Green Technology Engineering, Pukyong National University, Busan 48513, Republic of Korea
[g] School of Electrical Engineering, Pukyong National University, Busan 48513, Republic of Korea
[h] Interdisciplinary Research Center for Advanced Materials, King Fahd University of Petroleum & Minerals, Dhahran 31261, Saudi Arabia

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ABSTRACT

We presented a superhydrophobic stainless steel mesh (SSM) developed by facile one-step dip coating of candle soot (CS)-polydimethylsiloxane (PDMS) using chloroform as solvent for effective and durable oil-water separation. SSM dip coated with chloroform solution of 200 mg CS and 0.5 mL PDMS yielded water contact angle (WCA), oil contact angle (OCA) and sliding angle (SA) of $162 \pm 2°$, $0°$ and $4 \pm 1°$, respectively. In series of oil-water separation studies, superhydrophobic mesh revealed >98 % separation efficiency with high separation flux of 7810 $L \cdot m^{-2} \cdot h^{-1}$. The superhydrophobic mesh revealed excellent mechanical, chemical and thermal durability.

1. Introduction

Oily effluent from chemical factories and oil spills threatens marine life and environment [1]. Practical and affordable technologies for treating oily wastewater are urgently needed [2]. Superhydrophobic materials separate oil and water from oily wastewater by repelling water and allowing oil to pass through them. Superhydrophobic surfaces for oil–water separation have been created using porous substrates like metal meshes, sponges, and textiles [3–5]. Stainless steel mesh (SSM) is a better choice due to its durability, affordability, and versatility [6,7].

Several recent reports utilized CS to develop superhydrophobic surfaces [6–11]. For example, Zhang et al. used two-step approach where CS layer was deposited on SSM using candle flame. They have used xylene solvent for subsequent PDMS deposition. PDMS acted as binder and low surface energy modifier. It could separate heavy oil (dichloromethane) from water with 94 % efficiency [7]. However, authors have not evaluated durability of the developed mesh for reusability or their permeation flux with different viscosity oils. Liu et al. used multi-step approach where candle flame deposited CS/SSM was

treated with SiO_2 and subsequently with low surface energy silane [10]. Khosravi et al. deposited CS on SSM via candle flame, vapor-deposited polypyrrole, and then modified it with stearic acid [6]. Song et al. deposited CS layer on glue-coated SSM by two-step [11]. Developing superhydrophobic/superoleophilic SSM with facile approach, one-step, durable, and high permeation flux with viscous oils is essential for practical applications.

In this context, we developed CS-PDMS coated SSM via a simple one-step dip coating in CS-PDMS using chloroform solvent. Chloroform is better than other organic solvents such as xylene (used in Ref. 7) for uniformly dispersing CS particles and dissolving PDMS. Chloroform has higher solubility constant and lower evaporation temperature [12]. The one-step coated SSM was evaluated for its wetting ability, surface and chemical structure, durability and oil–water separation using different viscous oils.

2. Materials and methods

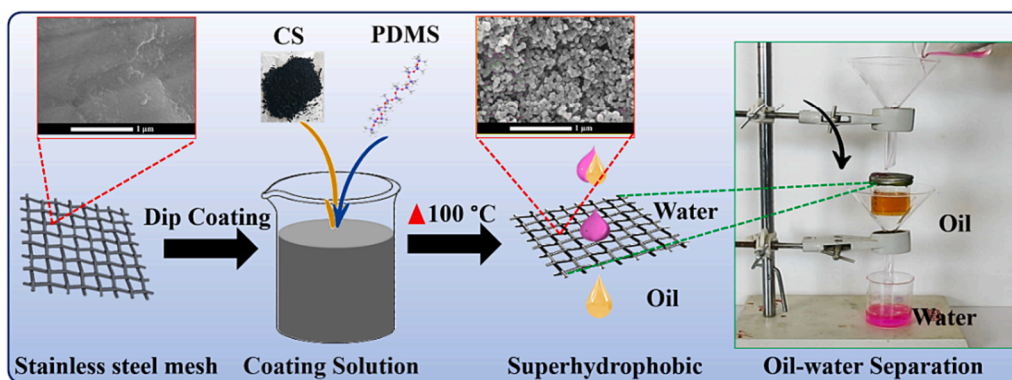Before use, the 4×4 cm^2 SSM pieces were ultrasonically cleaned

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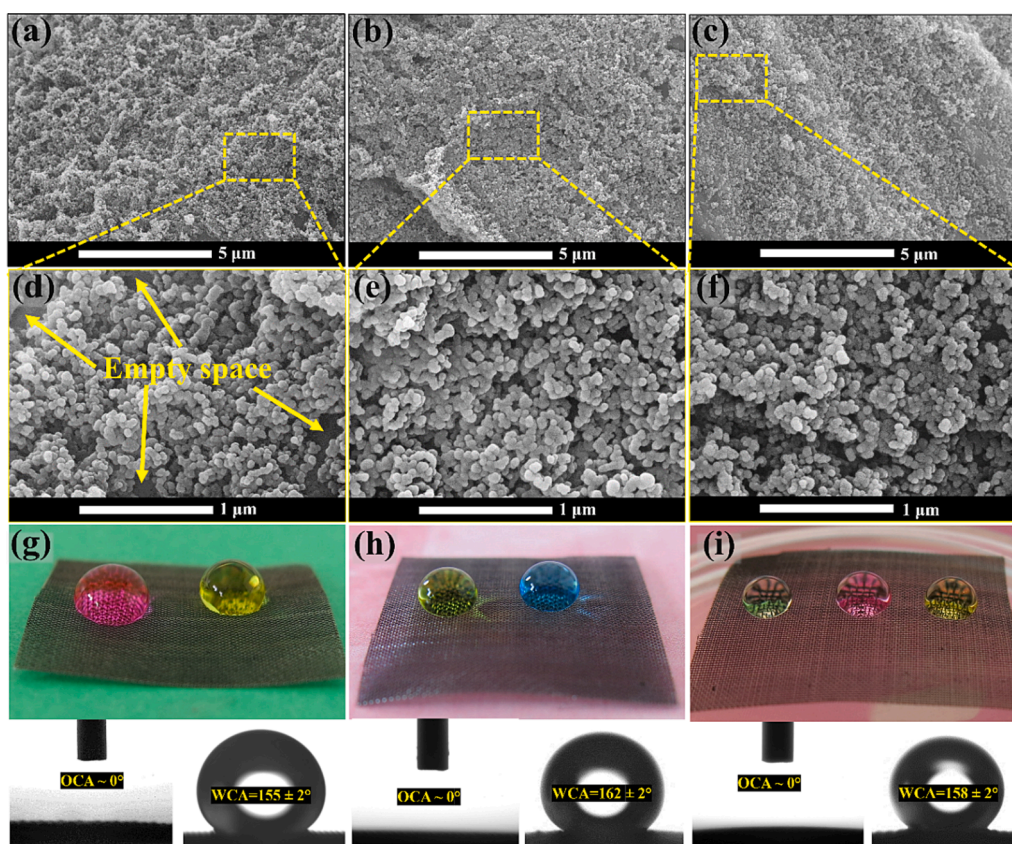**Fig. 1.** Fabrication of superhydrophobic SSM.

Fig. 2. Low and high magnification SEM images of (a&d) CPM-1, (b&e) CPM-2 and (c&f) CPM-3. WCA, OCA, and coloured water droplets on (g) CPM-1, (h) CPM-2, and (i) CPM-3.

with ethanol and distilled water for 5 min each and dried at 80 °C for 10 min. 0.5 mL PDMS was stirred in 20 mL CHCl$_3$ at 100 rpm. After 30 min of stirring, 100–300 mg CS particles were added and stirred for an additional 30 min. SSM was submerged for 20 min in the prepared solution and drawn out slowly at 50 mm/s, dried at room temperature for 10 min and heated at 100 °C for 1 h (Fig. 1). The coated meshes were named CPM-1, CPM-2, and CPM-3, corresponding to 100, 200, and 300 mg of CS used. More experimental details are provided in Supporting Information (SI).

3. Results and discussion

The CS nanoparticles formed network-like structure on coated SSM, as seen in SEM micrographs. Fig. 2a&d illustrates the composite is not evenly distributed throughout mesh due to low CS concentration; empty spaces were evident. At 200 mg of CS, coating spread evenly on the mesh (Fig. 2b). The linked CS particles in PDMS network formed micro/nano-scale surface roughness (Fig. 2e). The nanopores could trap air, which is essential for superhydrophobicity [13]. The gaps in the CS particle aggregates may be due to the rapid solvent evaporation during heat treatment [14]. A higher CS concentration led to more aggregation and intermittent pores in the composite coating (Fig. 2c&f). Table S1 and Fig. S1 provide elemental percentage and EDS spectra. The high C content is associated with CS, and the Si content is with PDMS incorporation.

CPM-1 sample showed 155 ± 2° of WCA due to lesser extent of CS-PDMS deposition on SSM (Fig. 2g). The droplet had trouble rolling freely on this surface. CPM-2 sample showed better WCA of 162 ± 2° and SA of 4 ± 1°, which can be attributed to more uniform surface coverage with desirable hierarchical nano/micro surface roughness
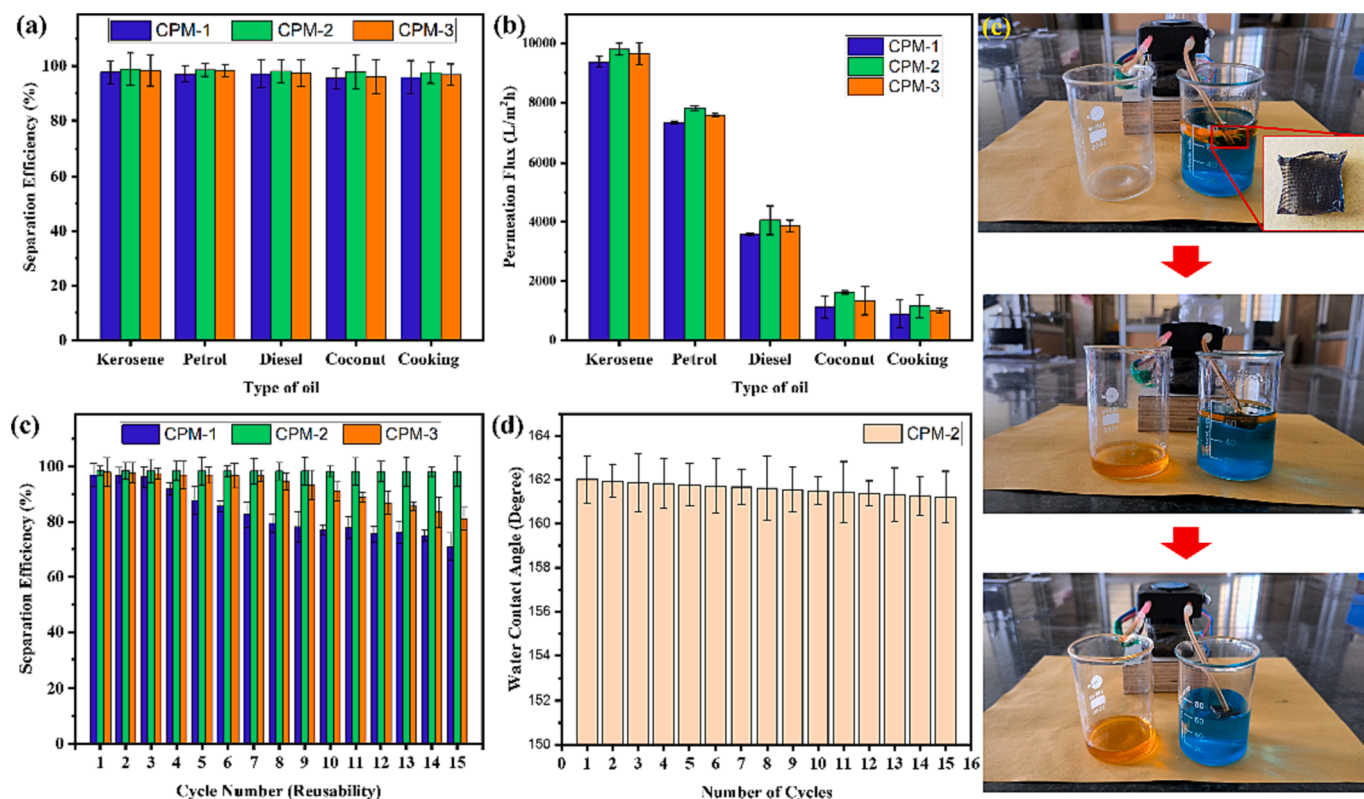
Fig. 3. (a) Separation efficiency and (b) permeation flux for various oil–water mixtures. (c&d) Variation of separation efficiency and WCA against number of kerosene-water separation cycles. (e) Continuous petrol-water separation.
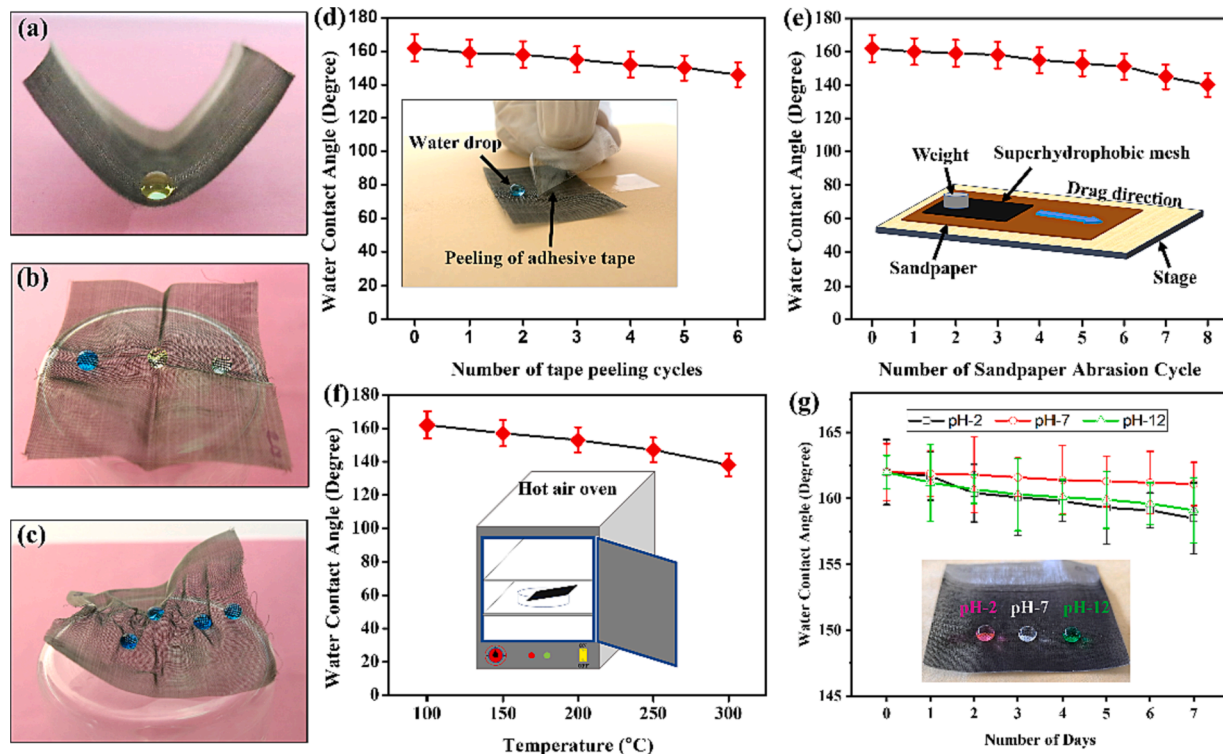

Fig. 4. Photographs of (a) bending, (b) folding, and (c) twisting of CPM-2. (d–f) WCA variation during, (d) tape peeling, (e) sandpaper abrasion and (f) temperature and (g) immersion in different pH solutions.

(Fig. 2h). Further increment of CS particles in coating reduced WCA to $158 \pm 2°$ (Fig. 2i). All the samples exhibited an OCA of nearly $0°$ (Fig. 2g-i).

In the gravity-based oil–water separation, mesh filtered oil into enclosed beaker, and water was collected in separate beaker under the funnel (Fig. S2). As-coated meshes had separation efficiency exceeding 98 % (Fig. 3a). All meshes showed separation flux of 7810 ± 780 $L \cdot m^{-2} \cdot h^{-1}$ for kerosene and 1160 ± 120 $L \cdot m^{-2} \cdot h^{-1}$ for cooking oil, the disparity associated with viscosity (molecular density) variance (Fig. 3b). Compared to CPM-1 and CPM-3, the CPM-2 sample maintained separation efficiency >98 % even after 15 kerosene-water separation (Fig. 3c). CPM-2 sample demonstrated superior separation compared to CPM-1 and CPM-3. Hence, CPM-2 was selected for further studies. Additionally, we examined wetting stability during reusability (Fig. 3d). Beyond the filtration mode, CPM-2 sample demonstrated superior oil–water separation in continuous mode (20 mL petrol collected within 38 s) (Fig. 3e). The oil–water separation mechanism is provided in SI file.

Mechanical bending, folding and twisting, tape peeling, sandpaper abrasion, and chemical and thermal tests are used to determine durability [15,16]. Superhydrophobic mesh stayed intact even after bending at 90-180° (Fig. 4a). WCA and SA remained at $160 \pm 2°$ and $5 \pm 1°$, after being folded and twisted (Fig. 4b-c). The superhydrophobicity retained with WCA of $151 \pm 2°$ and SA of $7 \pm 1°$ after 5 tape peeling cycles and the water drops maintained spherical shape (Fig. 4d). With further increase of peeling cycles, WCA reduced and the water drops get stuck on surface. The superhydrophobicity was retained for 6 abrasion cycles with WCA of $152 \pm 2°$ and SA of $6 \pm 1°$ (Fig. 4e); however, WCA reduced to $\sim 140°$ after 8 cycles, attributed to significant loss of coated composite. The coated mesh sustained superhydrophobicity up to $200 °C$, with WCA of $153 \pm 2°$ and SA of $6 \pm 1°$ (Fig. 4f). CPM-2 showed minimal WCA variation even after immersion in pH 2, 7, and 12 solutions, indicating high chemical stability (Fig. 4g). The durability studies support strong amalgamation of one-step dip-coated CS and PDMS using chloroform solvent, contributing to enhanced oil–water separation efficiency and durability to re-use. Table S2 compares results with reported similar works.

4. Conclusions

The one-step candle soot – PDMS dip-coated SSM developed in this work using chloroform solvent presented excellent oil–water separation capability. The mesh showed separation efficiency of 98 % and separation flux of 7810 $L \cdot m^{-2} \cdot h^{-1}$. The superhydrophobicity was retained even after multiple bending, folding, twisting, sandpaper abrasion, tape peeling, and temperatures up to $200 °C$. The simple and low-cost approach is helpful for industrial oil–water separation.

CRediT authorship contribution statement

Rajaram S. Sutar: Conceptualization, Methodology, Writing – original draft. **Sanjay S. Latthe:** Supervision, Writing – review & editing. **Xinna Wu:** Methodology. **Bairu Shi:** Methodology. **Nikhil N. Pargaonkar:** Methodology. **Sagar S. Ingole:** Methodology. **Anand N. Biradar:** Methodology. **Saravanan Nagappan:** Writing – review & editing. **Yong Hyun Kim:** Writing – review & editing. **Appasaheb K. Bhosale:** Writing – review & editing. **Viswanathan S. Saji:** Writing – review & editing. **Shanhu Liu:** Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matlet.2023.135791.

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