

Regular Article

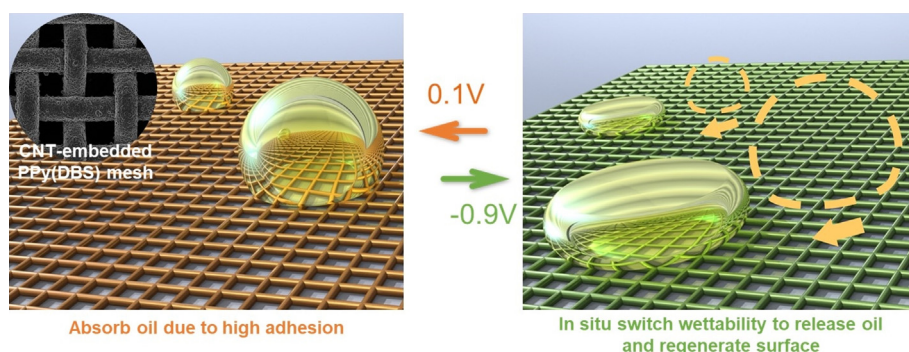
A carbon nanotube-embedded conjugated polymer mesh with controlled oil absorption and surface regeneration via *in situ* wettability switch

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GRAPHICAL ABSTRACT



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ABSTRACT

This paper presents a mesh-type absorbent made of a carbon nanotube (CNT)-embedded polypyrrole dodecylbenzenesulfonate (PPy(DBS)) surface for controlled absorption and release of oils and regeneration of polymer surfaces toward continuous oil/water separation. The mesh absorbs dichloromethane (DCM) under oxidation in aqueous environment and releases them under reduction via *in situ* switching of underwater wettability. CNTs were grown out of stainless steel surfaces and embedded into the PPy (DBS) film, which enhanced the switchable wettability as well as the surface regeneration. The CNT-embedded morphology improved oil retention by a factor of two in an oxidized state and decreased wettability switch time by 16% in a reduced state over the mesh without CNT embedding during 250 redox cyclic testing. A rolled 2 cm × 3 cm CNT-embedded PPy(DBS) mesh was furthermore used to demonstrate the continuous absorption and release of oils, during which DCM of 16 times of the absorbent weight was collected in 50 redox cycles.

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1. Introduction

There is an immense need for low-cost, efficient and reliable methods for oily water treatment and/or oil recovery. Recent decades, materials with special wetting properties and corresponding strategies have been developed for oil/water separation [1,2]. The

oleophilic/hydrophobic or underwater oleophobic properties were utilized to separate water from water/oil mixture via absorption or filtration [2–4]. Materials with special wettability, including polymers [5,6,15,7–14], metals [7,16–23], carbon [24–28], silicon [5,14,15,22,29–34], or fluorine [5,11,19,23,34,35]-based compounds were fabricated on various substrates, such as meshes [8,9,35,10,12,15,18,22,27,28,31], fabric or textiles [5,7,14,16,20,21,30,33], sponges [6,29,32], foams [24,34], membranes [13,19,23,26], and aerogels [25], demonstrating oil/water

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separation. The special wetting properties were further enhanced by incorporating macroscopic morphologies inherited from the substrates or microscopic structures via microfabrication using chemical etching [18,19,23,24,32,34], nanoparticles [7,10,14,16,17,29] or sol [20,30,31]. These structures enhanced the separation efficiency by introducing higher surface areas for oil absorption (Wenzel's model [36]) or entrapped air phase for oil rejection (Cassie–Baxter [37]).

While the synergistic effect of surface chemistry and structures enables the materials with special wetting, a strategy for the continuous operation would further enhance the efficiency of oil/water separation. For example, an uninterrupted filtration is possible using well-designed apparatus [7,11,15,17,18,31]. But, typically the oil absorbents gradually lose their wetting capability or reach the limit in absorbing capacity due to fouling or saturation. The degeneration of these materials warrants either the enhancement of their capacity via adding auxiliary receptacle such as bags [30,33] or skimmers [24], or the regeneration or cleaning of saturated materials via rinsing [6,9,30], squeezing [6,32], heating or burning [7,25], UV radiation [7,19,22], or pumping [29,30]. However, all these regeneration methods accompany the process down-time or require additional materials and labors. Various functional materials may enable continuous operation by allowing absorbed oil to be released 'on demand' using switching wettability via external stimuli such as temperature, pH, light, electrical, or magnetic field [38–44]. Materials with special wettability capable of *in situ* switching their wettability would enable controlled trap and release of oils toward continuous oil/water separation.

Here we demonstrate continuous absorption and release of dichloromethylene (DCM) via *in situ* switch of underwater wettability

(i.e., affinity for organic liquid in an aqueous environment) of a carbon nanotube (CNT)-embedded polypyrrole dodecylbenzenesulfonate (PPy(DBS)) mesh. We first grow CNTs on the stainless steel mesh surface, and subsequently electropolymerize PPy (DBS) atop CNTs. We oxidize or reduce PPy(DBS) meshes (with and without CNT embedment) in 0.1 M NaNO₃ to absorb and subsequently *in situ* release DCM, while simultaneously regenerating the polymer surface. We then prove that the CNT embedment enhances the surface wettability and longevity of the PPy(DBS) mesh. Lastly, we show a proof-of-concept oil collection and surface recovery of the polymer surface via *in situ* underwater wettability switch.

2. Working mechanism

Fig. 1 shows the mechanism of absorption of DCM and release of once-absorbed DCM using the PPy(DBS) mesh, and *in situ* regeneration of the PPy(DBS) surface (i.e., regeneration without moving the mesh out of electrolyte or using any additional material to the system). PPy(DBS) switches its underwater wettability *in situ* upon application of voltages as low as ± 1 V [45–47]. When a positive voltage (e.g., 0.1 V) is applied, PPy(DBS) surface is oxidized with a strong adhesion toward oils (therefore the PPy(DBS) mesh can 'absorb' oils by adhering the oils on the PPy(DBS) surface (Fig. 1a-i, ii). When a negative voltage (e.g., -0.9 V) is applied, the PPy(DBS) surface is reduced, where previously attached oil droplets roll off from the mesh (Fig. 1b-i) or permeate through the mesh (Fig. 1b-ii). This switching of underwater wettability, as has been previously reported [46–48], is attributed to the reorientation and desorption of DBS⁻ molecules [49,50]. Being amphiphilic,

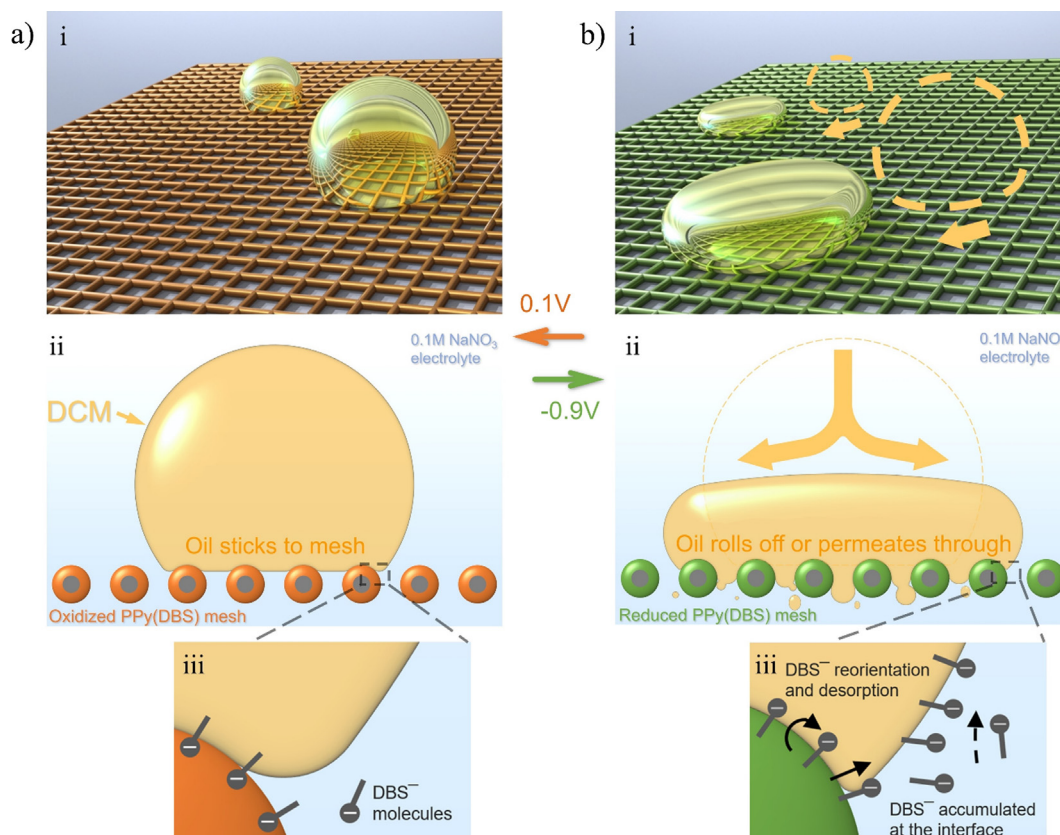


Fig. 1. Schematic illustration of the mechanism of oil absorption and release using a PPy(DBS) mesh via *in situ* wettability switch. (a) Oxidized PPy(DBS) surface shows strong affinity to oils [49], adhering the oils on the polymer surface. Therefore, the PPy(DBS) mesh can be used to 'absorb' oils. (b) Upon reduction, PPy(DBS) surface *in situ* switches underwater wettability, allowing oils to be released from the mesh via rolling off or permeating through the pores (i, ii). This wettability switch is attributed to the reorientation and desorption of DBS⁻ molecules [49,50] (iii).

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