Supplementary Information for

Underwater superoleophobic graphene oxide coated mesh for the separation of oil and water

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Experimental section

Materials

Graphite flake (natural, 325 mesh) was purchased from Alfa Aesar. Stainless steel meshes with different mesh sizes were commercially available. All chemicals were used as received without further purification.

Synthesis of graphene oxide (GO) using a modified Hummers method

A 500 mL reaction flask was charged with 0.5 g of natural flake graphite and a stir bar. 23 mL of concentrated sulfuric acid was added into the reaction flask. Next, 0.5 g of NaNO₃ was added under stirring and cooled in an ice-water bath. The flask was then slowly charged with 3 g of KMnO₄ in order to avoid rapid increase of solution temperature. Once mixed, the flask was transferred to a 35 °C oil bath and stirred for about 1 h. Then, 40 mL of water was slowly added dropwise, forming a thick paste and increasing temperature. Meanwhile, the temperature of oil bath was raised to 90 °C, and the solution was stirred for 30 min. Finally, 100 mL of water was added, followed by the slow addition of 5 mL of H₂O₂ (30%). The solution was centrifuged at 9000 rpm. The solid was then washed by 5% HCl and water. Next, washing was done with water, until there were some flocks above the precipitates. The upper water was carefully removed. The precipitates were then dispersed into 200 mL of water, followed by low-power sonication for 30 min (Branson 1510E-MT) to form atomically thin GO.

Preparation of underwater superoleophobic GO coated mesh

Clean stainless steel mesh was immersed into GO aqueous solution with low-power sonication for 10 min, then dried at 60 °C. The mesh was immersed into GO aqueous solution again for another 10 min, and then dried at 60 °C. GO was easily coated onto stainless steel mesh surface.

Characterization

GO aqueous solution was examined on a UV-visible spectrophotometer (Agilent Cary

60). Morphology of as-obtained GO was measured with a transmission electron microscope (TEM, FEI Tecnai G2 TF20). TEM samples were prepared by dropping the homogeneous suspensions on a copper TEM grid that was coated with a thin amorphous carbon film, and dried at room temperature. The exfoliated GO monolayer and their thickness were characterized by an atomic force microscope (AFM, AIST-NT SmartSPM). The nanosheets were coated on freshly cleaved mica surface through a drop-casting method, and vacuum dried at 60 °C. Infrared spectrum of GO was collected on a Nexus 870 Fourier transform infrared (FTIR) spectrometer (Nicolet). Morphology of uncoated and GO coated meshes was measured with a field emission scanning electron microscope (FESEM, Hitachi S4800). Water contact angles and oil contact angles were obtained on a DSA100 contact-angle system (Kruss Company, Germany) at ambient temperature by measuring at five different positions on the same sample.

Supplementary Figures



Fig. S1 TEM images of GO at different magnifications.



Fig. S2 AFM image (left) and height profiles (right) along the lines shown in AFM image of GO.



Fig. S3 Photographs of neat (left) and GO coated (right) meshes before and after immersing into water, then taking out from water.



Fig. S4 Photographs of a water droplet on neat (left) and GO coated (right) meshes in oil (hexane).



Fig. S5 Relationship between the pore diameters of neat and GO coated meshes and the dichloroethane contact angles.



Fig. S6 Photographs show that a dichloroethane droplet rolls off the sloping surface of GO coated mesh (up) but adheres to that of neat mesh (down), the pore diameter is about 425 μ m (40 mesh size).



Fig. S7 Photographs of an oil droplet on neat (left) and GO coated (right) meshes in air.



Fig. S8 Effect of coating times, GO concentration and pore diameter of neat mesh on water flux. Water fluxes were determined by calculating the volume of water permeation in unit time using the following equation: flux = V/St, where V is the volume of water permeation, S is the valid area of the films, and t is the testing time. GO concentration was controlled by dispersing GO precipitates into 200 (A) and 250 (B) mL water.



Fig. S9 The dichloroethane contact angles of GO coated meshes versus the cycles of oil-water separation. The insets are photographs of an oil droplet on GO coated meshes in aqueous solution.

Supplementary Movies

Movie S1 A dichloroethane droplet rolls off the sloping surface of GO coated mesh with an average pore diameter of about $425 \mu m$.

Movie S2 Oil-water separation by using GO coated mesh with an average pore diameter of about 53 μ m.

Movie S3 Oil-water separation by using neat mesh with an average pore diameter of about 53 μ m.

Movie S4 Oil-water separation by using GO coated mesh with an average pore diameter of about 425 μ m.