Selective oil/water filter paper via a scalable one-pot hydrothermal growth of ZnO nanowires

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Experimental Detail

A commercial filter paper (Whatman, Grade 4 Qualitative filtration paper with pore size of 20-25 μm) was cleaned by ultrasonication in ethanol for 30 mins. A ZnO precursor solution was prepared by dissolving zinc nitrate hexahydrate (Sigma-Aldrich, 80 mM), hexamethylenetetramine (HMTA, Sigma-Aldrich, 25mM), polyethylenimine (PEI, Sigma-Aldrich, 5 mM) and ammonia solution (Sigma-Aldrich, ACS reagent, 28.0-30.0 %, 5 mL) in 100 mL DI water. The solution was magnetically stirred in beaker at room temperature. The obtained clear solution was heated up to 90 °C and covered by watch glass. Then, the cleaned filter paper was placed at the interface between air and the solution. After keeping heating for 10 min, the cover glass was removed. Sudden drop in vapor pressure causes the formation of ZnO nuclei particle layer on the fiber surface. Once the high-density nuclei layer was formed, the cover was closed again and kept for further ZnO nanowire growth. The as-grown filter paper was thoroughly rinsed by DI water and ethanol, dried for further use.
Low magnification and high magnification scanning electron microscopy (SEM) images were recorded by Vega-3 (Tescan, Czech) and field emission SEM (FESEM, Zeiss SIGMA, German), respectively. Thin film X-ray diffraction (XRD) pattern were performed by X-ray diffractometer (Rigaku ultima IV, Japan) with Cu-Kα radiation. The XRD data was collected over a 2θ of 20° to 80°. Contact angle was measured by VCA Optima (AST Products Inc., US) at room temperature. For underwater oil contact angle measurement, the as-grown filter paper was firstly placed into a transparent chamber with water. A 5 μL 1,2-dichloroethane (DCE) was then dropped onto the paper substrate and the contact angle was measured. UV-vis absorption spectra were recorded on a UV-vis spectrophotometer (Perkin-Elmer NIR-UV, USA).

The as-prepared filter paper was pre-wetted with water and attached to a glass graduated funnel by effective seal. The filter paper was also fixed on bottom of the funnel by a plastic sheet with proper size of hole. The surfactant-free oil/water mixture (50 v/v%) were poured onto the as-prepared filter. The filtration was achieved by weight the collection in beaker.3,4

Surfactant-stabilized oil/water emulsion was prepared by adding sodium dodecyl sulfate (SDS, electrophoresis grade, 98%, Sigma-Aldrich, 2 mg/mL) to a kerosene/water (50 v/v%) mixture solution, following by 30 mins sonication. Finally, a white and milky solution was obtained. The prepared emulsion solution was poured onto the as-modified filter paper and the water permeated through the filter paper which was driven by gravity.

**Supporting Figures**

To determine the absence of oil after filtration, oil (kerosene) was dyed with Oil Red O (1 wt%, Sigma-Aldrich) with typical absorption peak at around 518 nm.7,8 The filter and reference water were also measured by UV-visible absorption spectra (Fig. S3), and no obvious dye peak
was observed of the filtered solution. The UV-visible absorption results prove that the filtered sample contains no oil.

![Image](image1.png)

**Fig. S1** SEM images of filter paper after ZnO nucleation.

![Image](image2.png)

**Fig. S2** Optical images of dynamic contacting process of water droplet onto the as-coated filter paper surface.
Fig. S3 UV-visible spectroscopy for reference water, filtered water and dyed oil.

Fig. S4 A plot of the permeated water weight (%) over the separation time.

The water separation flux $J$ is calculated by the following equation:\textsuperscript{3,7,9}

$$J = \frac{V}{A\Delta t}$$

Where, $V$ is the volume of the permeated water, $A$ is the separation area and $\Delta t$ is the permeated time.
Fig. S5 Optical micrographs of the oil/water emulsion feed (a) and filtrate (b).

Fig. S6 Change in the flux with increasing cycle number using as-modified filter paper.

Reference


