Supporting information

A Cellulose Based Hydrophilic, Oleophobic Hydrated Filter for

Water/Oil Separation

Kathleen Rohrbach,^{a[+]} Yuanyuan Li,^{a[+]} Hongli Zhu,^a Zhen Liu,^a Jiaqi Dai,^a Julia

Andreasen,^a and Liangbing Hu^{a*}

^aDepartment of Materials Science and Engineering, University of Maryland College Park,

MD 20742 USA

[+]These authors contributed equally to this work

E-mail: binghu@umd.edu

Contents

- S1. Nanofibrillated cellulose (NFC) preparation
- S2. Filter Fabrication
- S3. Emulsion Preparation
- S4. Oleophobic Behavior
- S5. Characterization

S6. Determination of the Filtration Rate and Resulting Oil Concentration in Separated Water

S7. Digital images of filter paper fabricated before and after trials

Experimental

S1 Nanofibrillated cellulose (NFC) preparation

The experimental conditions are similar to the process used in Y. Li as stated before.¹⁷ In this study, 78 mg TEMPO, 514 mg sodium bromide (NaBr) and 5 g Kraft bleached softwood pulp was added together in a carbonate buffer solution (pH=10.5) and then mixed. During mixing, 30 mL of 12% NaClO was added to the mixture drop wise to initiate TEMPO oxidation of the cellulose under gentle agitation, while maintaining a pH of 10.5. NaClO being fully consumed signified the end of the reaction. After the TEMPO treatment, the fibers were thoroughly rinsed with distilled water to remove any chemicals and then disintegrated by one pass through a Microfluidizer M-110EH (Microfluidizes Ind., USA) to obtain a NFC suspension. The suspension was retained for filter modification.

S2 Filter Fabrication

As reported in the paper, unbleached wood pulp filters were cut into 4 cm by 4 cm square samples. The samples were dipped into the NFC suspension retained in S1 to form a hydrogel coating of NFC around the filter's fibers. This dipping process created an average loading of 6.4 mg of NFC on the filter. After dipping, the filters were left to dry at 25 °C for 24 hours and then placed in an oven at 80°C to initiate a crosslinking reaction between the NFC hydrogel coating. The final filter had an average pore size of 23 μ m when hydrated.

S3 Emulsion Preparation

For testing the separation abilities of the paper an emulsion was prepared using 5 mL of water and 5 mL of hexane. To create a visual contrast between the water and hexane, the hexane used in this experiment was dyed red by adding 5mg oil red-o in per mL hexane. A ratio of 2.5 mg of sodium dodecyl sulfate (SDS) to 1 mL of water was used as an emulsifier.⁴ The hexane, water, and SDS were added to a glass vial mixed for 2 hours to create an even emulsion, determined by the consistent pink color of the mixture.

S4 Oleophobic Behavior

To compare the repulsion and contact angle of the oil on a modified and unmodified filter, 5 μ L drops of hexane and 5 μ L of water where place on a hydrated modified filter, and a dry unmodified filter. The test is shown in Figure S1.

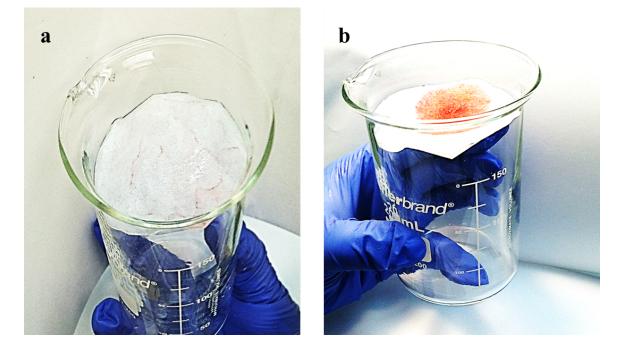


Figure S1: Image of a) the hydrated NFC modified filter with hexane droplet (red) on top and b) the unmodified filter, showing absorbance of oil (red).

S5 Characterization

The NFC used to modify the filter was characterized using atomic force microscopy (AFM) in tapping mode. The surface morphology, adhesion, resulting pore size and surface roughness of the filter were determined using scanning electron microscopy (SEM) and optical microscopy imaging. A Hitachi SU-70 Analytical UHR FEG-SEM was used to take the SEM images. The SEM samples were prepared with a layer of Ag using a vacuum coater. Filter paper average pore size was calculated according to Hagne-Poisueille Law.

$$\Delta P = \frac{8\eta l V}{\pi r^4}$$

Where: ΔP = Pressure difference between the ends of the paper, l = paper thickness, η = the dynamic viscosity of liquid, V = the volumetric flow rate, r = the radius of the pore. Microscope was used to obtain the optical image. Figure S2 is the typical microscope image of the hexane-water emulsion.

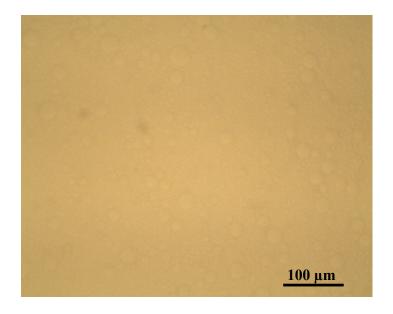


Figure S2 Typical microscope image of the hexane-water emulsion

S6 Determination of the Filtration Rate and Resulting Oil Concentration in Separated Water

The filter created in S2 was saturated with water and attached to the bottom of a glass graduated cylinder funnel, which was suspended over a beaker placed on a scale, as seen Figure S3. An effective seal was created between the filter and funnel, through the use of plastic attachment fitted to the bottom of the funnel with a 3.5 cm hole punched into the bottom. The emulsion was poured into the glass graduated cylinder filtration funnel and then a covering was placed over the filtration funnel to prevent evaporation. The flow rate was determined by timing the water passing through the filter. The filtered amount was determined by the weight displayed on the scale and the known density of the water (1 g /mL). The data of weight percent filtered water vs time was plotted using origin and the rate was determined by taking the slope of the linear fit for the plot.



Figure S3: Experimental setup of water/oil emulsion separation.

The water filtered was collected and the absorption analyzed using an UV-vis spectrometer. The filtered water sample was compared to the absorption of a control sample of pure water, hexane, and o-red hexane to determine the concentration of oil in the filtered water.

S7 Inspection of the filter before and after filtration

The filter was visually inspected in the dry and hydrated state both before and after filtration. The filters were rinsed before final inspection. As seen in Figure S4, there was no observed change in the filter's color after the trials were performed, indicating no oil clogging or fouling occurred.

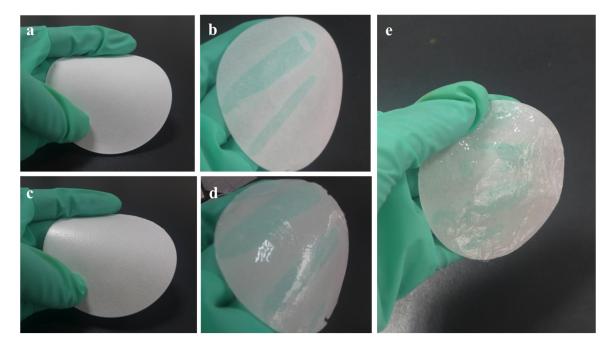


Figure S4: a-b) Digital images of a regular filter in dry and wet state, c-b) the modified filter in dry and hydrated state, and e) the rinsed filter after trials shows that there's no oil fouling on the filter.