Supporting Information

Poly(ε-caprolactone) Microfiber Meshes for Repeated Oil Retrieval

*Joseph S. Hersey, Stefan T. Yohe, Mark W. Grinstaff**

Additional Methods

Contact angle analysis. A Kruss DSA100 contact angle goniometer was used to quantify the contact angles of water and oil (4 μl) over time on the surface of the electrospun meshes. Each droplet was recorded at 5 frames per second for 1 minute and the contact angle was analyzed frame-by-frame.

Mechanical testing. A Instron 5848 Micro-tester tensile testing apparatus was used to generate a σ vs. ε curve for 20% electrospun PCL meshes either before or after 10 cycles of oil exposure and retrieval under vacuum (700 mbar). The meshes were cut into 1.5 cm by 6 cm rectangles and the thicknesses for each mesh were measured using a calibrated microscope (Olympus IX70) using a 10x magnification. The sample was fixed to the apparatus using sandwiched pressure tape reinforced with super glue. The sample was elongated at 1 mm per second corresponding to a ~2.5% strain rate to ~120% strain while recording the load across the sample at 10 Hz using a 100 N load cell. The elastic region of the mesh was defined as $\varepsilon < 0.1$ and the elastic modulus was calculated as the slope of the linear regression of this elastic region.

Scanning electron microscopy. A Zeiss SUPRA 55VP field emission SEM was used to image the surfaces of the meshes. The samples were affixed to an aluminum sample stub using copper tape and were coated with 5 nm of Au/Pd prior to imaging and imaged at an accelerating voltage of 2 kV.

Rheometry. An Advanced Rheometer (AR1000) was used to determine the viscosity of the pump oil and crude oil at 25 °C using a 40 mm aluminum cone geometry by first performing a

stress sweep and then a frequency sweep to determine the appropriate shear stress (1 Pa) and frequency (1 Hz) to determine the viscosity of the fluids.

Supporting Figures

Table S1. Electrospinning parameters.

PCL Concentration (wt. %)	Applied Voltage (kV)	Distance to Collector (cm)	Flow rate (mL/hr)	Needle Gauge	Solvent ratio (Chloroform:Methanol)
10	12	8	10	18	5:1
15	14	16	10	18	5:1
20	17.5	24	10	18	5:1

Fig. S1 Representative SEM image illustrating the nanometer scale (< 1000 nm) roughness on each electrospun fiber in a 20% PCL mesh (Scale bar: 10 μm).

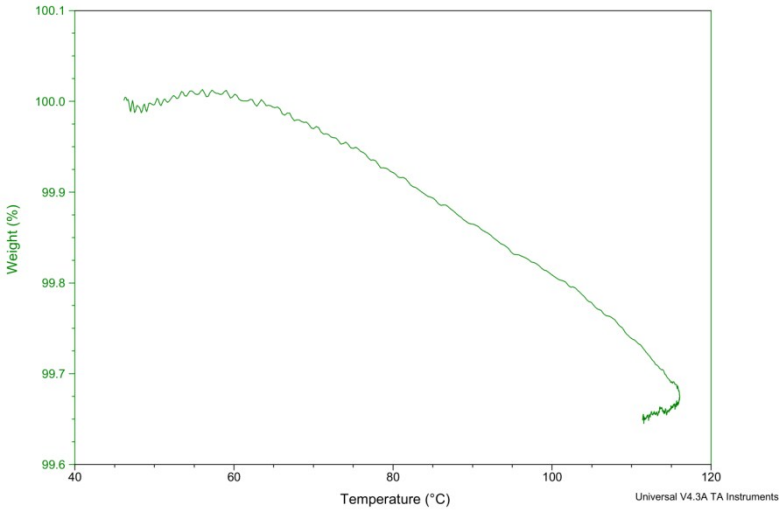


Fig. S2 Sample TGA plot of a 20% by wt. PCL mesh after being exposed to a 1:1 oil:water mixture. After being heated to >110 °C for 5 minutes, only ~0.35% of the sample's mass was found to be water.

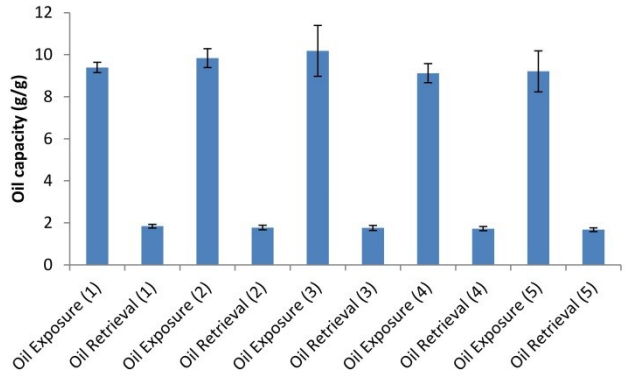
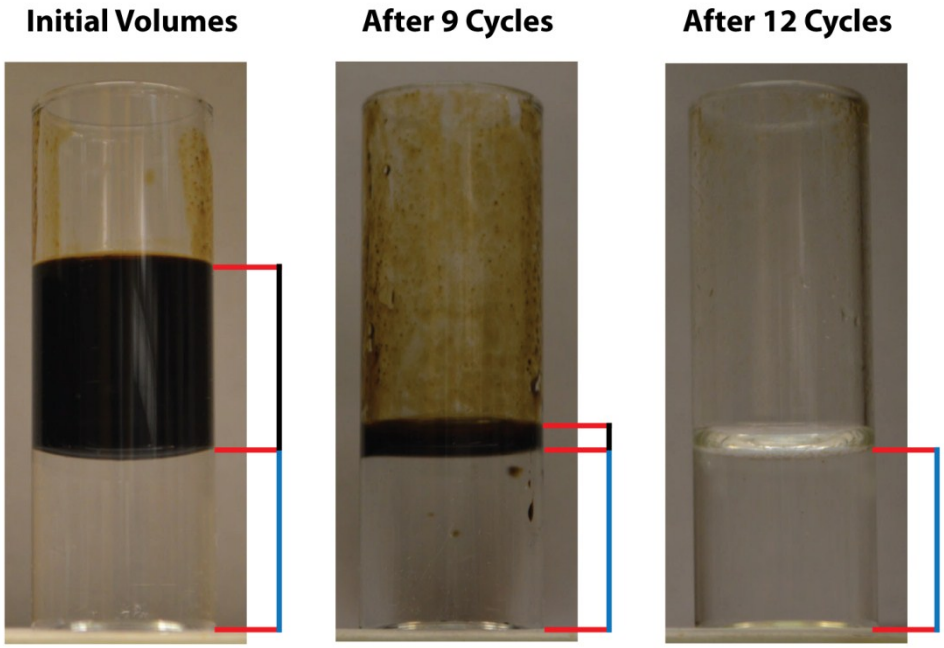


Fig. S3 20% by wt. PCL electrospun meshes maintain their oil removal capacity (g/g) over five oil exposure / vacuum retrieval cycles. The meshes were exposed to 1:1 oil and water mixtures during each oil exposure and the oil was retrieved by applying a vacuum to the mesh to retrieve the oil from within the mesh. (Avg±SD, n=3) ($p>0.05$ for cycle 1 vs. cycle 5 using a two-tailed T-test)



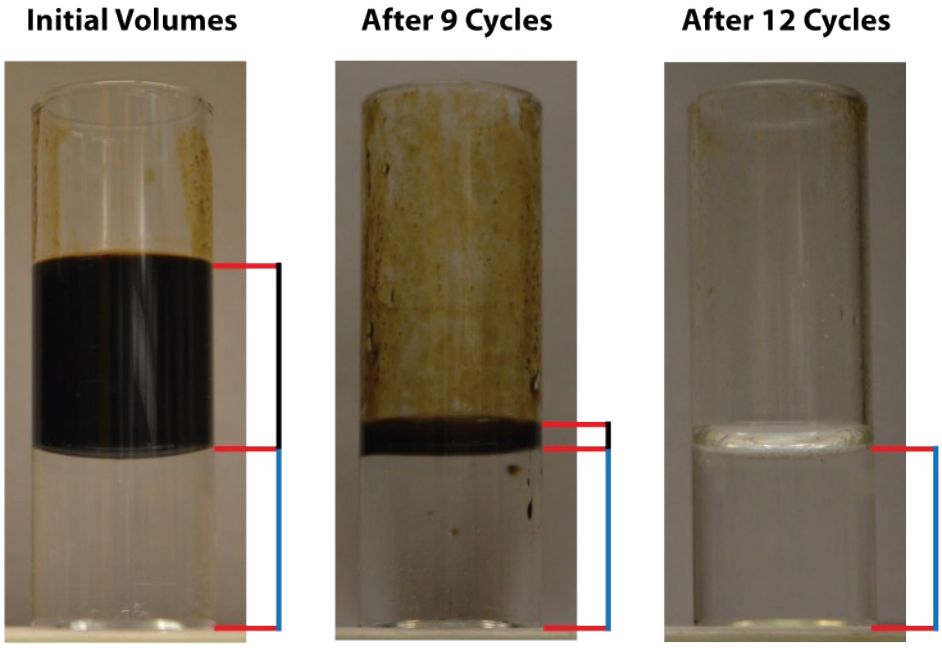


Fig. S4

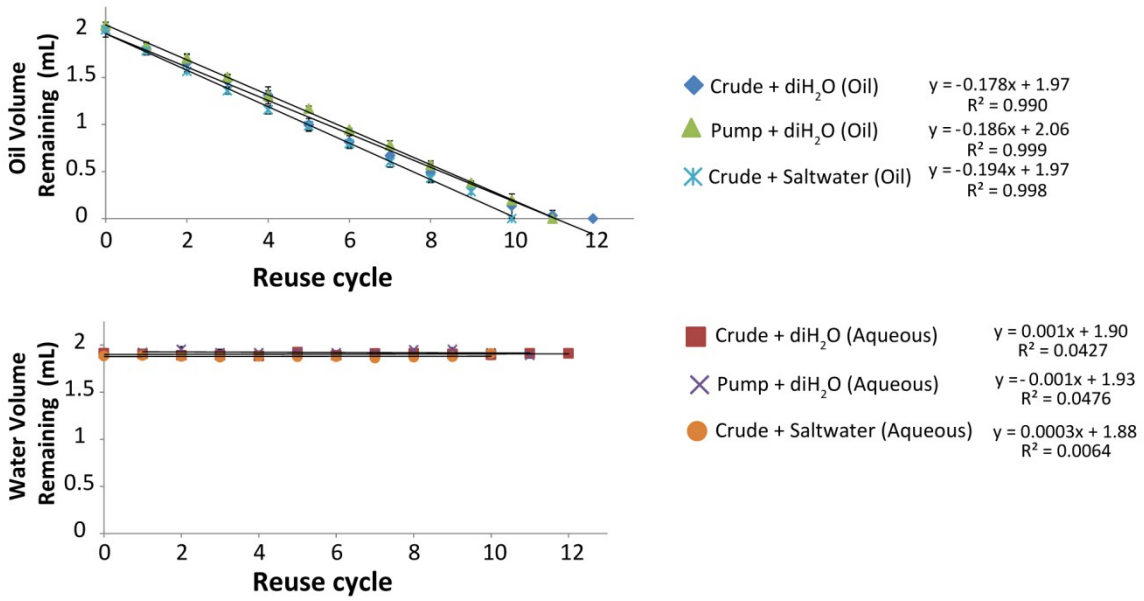


Fig. S5 Oil (top) and water (bottom) volumes after each oil retrieval cycle. Linear fits were applied and the slopes correspond to the average volume removed per reuse cycle. The oil retrieval fits have statistically significantly different slopes ($p=0.002$) and the water volume slopes are not statistically different from zero ($p>0.6$). (Avg±SD, n=3)

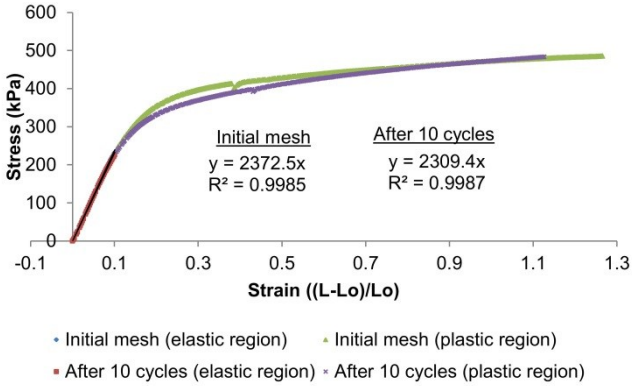


Fig. S6 20% by wt. PCL electrospun meshes maintain their mechanical properties over ten oil exposure / vacuum retrieval cycles. The meshes were exposed to 1:1 oil and water mixtures during each exposure and cleaned by applying a vacuum to the mesh to retrieve the oil from within the mesh. The elastic modulus of the mesh remains constant at approximately 2.3 MPa after repeated use.

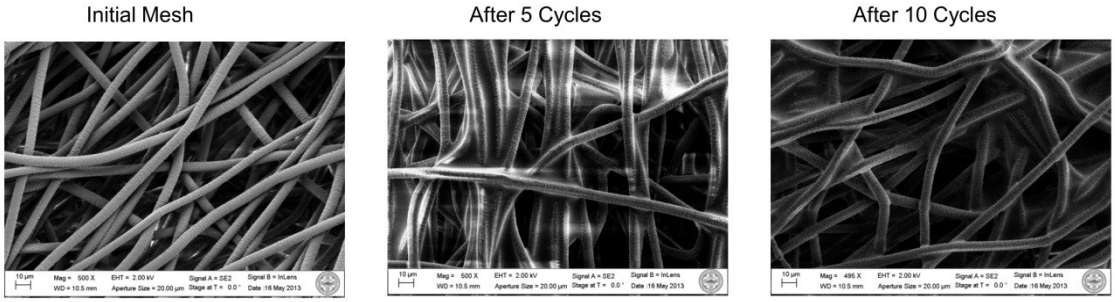


Fig. S7 SEM images of the initial 20% PCL mesh and the mesh after 5 and 10 cycles of oil exposure and oil retrieval under vacuum. Some oil remains adsorbed onto the fibers of the mesh causing sub-optimal imaging conditions. The meshes maintain their morphology and porosity after several cycles. (Scale bar: 10 μm)

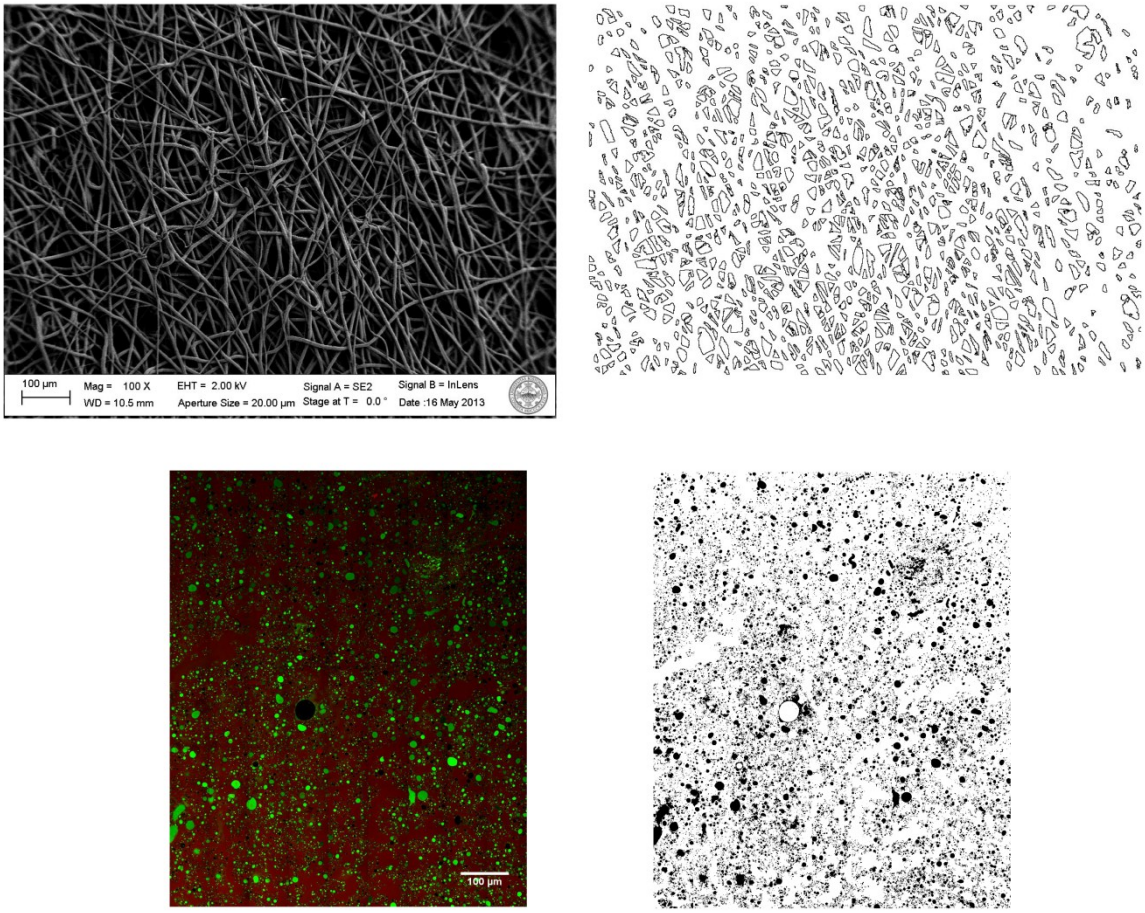


Fig. S8 (Top) SEM image of 20% PCL mesh (left) and segmented image with outlined pore geometry (right). (Bottom) Confocal microscopy image of 0.5% span 80 stabilized water-in-oil emulsion highlighting oil in red and water in green (left) and specifically the water emulsion in black (right). (Scale bar: 100 µm)

Table S2. Water-in-oil emulsions before mesh exposure.

Span80 Concentration (wt.%)	Average Diameter (µm)	Standard Deviation (µm)
0.5	10	11.3
1.0	11.3	15.5
5.0	11.9	15.3