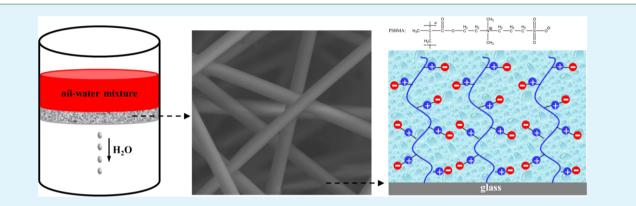
Superhydrophilic and Underwater Superoleophobic Poly(sulfobetaine methacrylate)-Grafted Glass Fiber Filters for Oil– Water Separation

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Supporting Information



ABSTRACT: Oil-water separation is a major problem in industries such as oil production and wastewater treatment, where millions of gallons of oil-contaminated water are produced. Zwitterionic poly(sulfobetaine methacrylate) (pSBMA) is a superhydrophilic polymer due to its strong interaction with water via electrostatic interactions. By coating surfaces of filter media with such a superhydrophilic polymer, it is expected that one can effectively separate oil and water. In this work, pSBMA was grafted onto glass fiber surfaces using surface-initiated atom transfer radical polymerization (SI-ATRP). The in-air water contact angle of the pSBMA-treated glass was $8-15^{\circ}$, as compared to 31° for the control untreated glass, whereas the underwater-oil contact angle of the pSBMA-grafted glass was $162-169^{\circ}$, as compared to 142° for the control pristine glass, suggesting that the pSBMA-grafted glass slides are superhydrophilic and underwater superoleophobic. Such superhydrophilicity and underwater superoleophobicity were realized by modifying surface chemistry only, with no need to create rough surfaces. The pSBMA-grafted glass fiber filters demonstrated exceptional results at separating oil from water without even allowing miniscule amounts of visible oil to permeate through.

KEYWORDS: glass fiber, oil-water separation, pSBMA, SI-ATRP, zwitterionic

1. INTRODUCTION

Oil–water separation has been a worldwide challenge for a long time, due to the increasing environmental awareness and tighter regulations, as well as increased production of industrial wastewater and accidental oil spills.¹

In recent years, for effective oil–water separation, attention has been paid to create both hydrophobic–oleophilic "oilremoving" surfaces and hydrophilic–oleophobic "water-removing" surfaces. However, due to the intrinsic oleophilic characteristic resulting from high surface energy, oil-removing surfaces are easily fouled and blocked up by oils. This creates a major disadvantage because the adhered oils greatly affect the separation efficiency and are harder to remove, therefore increasing oil and material waste.^{2–4} Therefore, it is essential to develop novel surfaces or materials for oil–water separation that are easily produced and recycled, have high resistance to organic contaminants such as oils, and have high separation efficiency and low operating costs. Various substrates have been studied for oil-water separation including filter paper,⁵ manganese oxide nanowire,⁶ metal meshes,^{1,7-11} polymer composites,¹² textiles,¹³⁻¹⁵ silicon,^{16,17} and plastics.¹⁸ Some drawbacks exist over using these substrates, such as low flexibility of metal meshes and poor mechanical stability of filter paper and polymer films. Several groups have successfully achieved hydrophilicoleophobic surfaces^{16,17,19,20} on the types of substrates mentioned above; however, these surfaces have only been used to study self-cleaning and antifouling properties but not for oil-water separation.

Wettability of surfaces can be changed via surface modification techniques to achieve antifouling surfaces, using methods such as vapor deposition,^{6,7,14,16} dip coating,^{8,13} spray

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coating,^{9,11} spin coating,^{19,20} and polymer grafting.^{15,21} Superoleophobic surfaces are generally shown to require the use of both hydrophilic material and hierarchical surface structures, a combination of surface chemistry and surface roughness.¹⁵

Zwitterionic poly(sulfobetaine methacrylate) (pSBMA) is a novel superhydrophilic polymer and has been so far widely explored as an ultralow biofouling material to resist adsorption from proteins, cells, and bacteria for biomedical applications.²² Zhang et al. also have shown that pSBMA-grafted glass slides resist fouling from marine microorganisms.²³ These characteristics are ascribed by its strong hydration ability due to the electrostatic attractions between charges on the side groups of the polymer and the water molecules. As found by the low-field nuclear magnetic resonance experiments, a single SBMA monomer tightly interacts with eight water molecules.²⁴ We hypothesize that effective oil—water separation can be achieved, if surfaces of filter media are coated with such a superhydrophilic polymer.

Herein we report a simple novel technique to fabricate, for the first time, a superhydrophilic and underwater superoleophobic pSBMA-grafted glass fiber filter for the separation of oil and water from oil-water mixtures. The grafting was realized by surface-initiated atom transfer radical polymerization (SI-ATRP). In addition to the polymer being covalently anchored to the surface, ATRP has several distinct advantages over the other mentioned techniques such as controllable uniform film thickness, narrow polymer molecular weight distribution, and high surface coverage.²⁵ Glass fiber filter medium was chosen due to its strong mechanical properties, recyclability, low cost, and easily modifiable surface. The glass fibers, augmented with electrospun polymer nanofibers, have been used before to create filter media for separation of water droplets out of oil.²⁶ To the best of our knowledge, modified hydrophilic glass fiber filters have not been utilized as oil-water separation media.

2. EXPERIMENTAL SECTION

2.1. Materials. Unwoven glass fiber mats with an average fiber diameter of 6.5 μ m and thickness of 1.4 mm were provided by Hollingsworth and Vose. The glass slides were obtained from Corning (2947–3 in. × 1 in.). The monomer *N*-(3-sulfopropyl)-*N*-(methacryloxyethyl)-*N*,*N*-dimethylammonium betaine (sulfobetaine methacrylate, SBMA, H₂C=C(CH₃)COOCH₂CH₂N-(CH₃)₂(CH₂)₃SO₃), 2,2'-bipyridyl (bpy, 99%), copper(I) bromide (99.99%), ethyl 2-bromoisobutyrate (EBIB), tetrahydrofuran (THF), methanol, and oil red-O dye were purchased from Sigma-Aldrich and used as received. Hexadecane (99%) was purchased from Alfa Aesar. Sodium hydroxide was purchased from Fisher Scientific. Ethanol (absolute 200 proof) was purchased from Pharmco-AAPER. DI water was purified to a minimum resistivity of 18.0 MΩ-cm by a Millipore filter system.

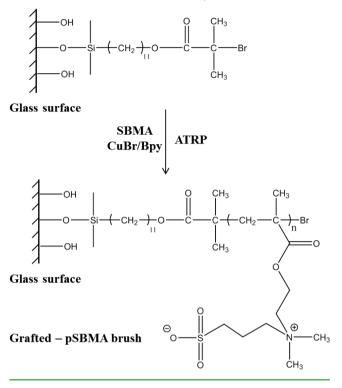
The ATRP initiator, (11-(2-bromo-2-methyl)-propionyloxyl) undecyltrichlorosilane (Br-PUCS), was synthesized through the reaction of 1,3-propane sultone and 2-(dimethylamino)ethyl methacrylate using a method published previously.²⁷ ¹H NMR (300 MHz, CDCl₃) δ : 1.23–1.45 (br m, 16H); 1.54–1.75 (m, 4H); 1.93 (s, 6H); 4.16 (t, 2H) ppm.

2.2. Immobilization of the ATRP Initiator. Glass fiber mats were cut into 1 in. (diameter) pieces from an 8.5 in. \times 11 in. sheet, while glass slides were cut into 1/2 in. \times 1 in. pieces. Prior to the ATRP initiator immobilization, the glass substrates were cleaned with water and ethanol, soaked in a 20 wt % NaOH solution for approximately 24 h, washed subsequently with DI water and ethanol multiple times, and then air-dried. For the initiator immobilization, four to six substrates were immersed in 15 mL of THF solution containing 10 μ L of BrPUCS initiator and left for approximately 24 h. The substrates were

then washed with THF twice and air-dried prior to the pSBMA grafting.

2.3. Preparation of pSBMA-Grafted Substrates. The pSBMA brushes were grafted on glass surfaces via SI-ATRP according to Scheme 1. The glass substrate modified with the Br-PUCS initiator,

Scheme 1. Grafting of pSBMA on Glass Surfaces via Surface-Initiated Atom Transfer Radical Polymerization



along with the SBMA monomer (0.296 g), bpy ligand (0.062 g), and Cu(I)Br (0.031 g), were placed into a test tube. The tube was then sealed with a natural rubber septum and quickly filled with nitrogen. A deoxygenated solution of methanol (7 mL) and DI water (7 mL) was then added into the reaction vessel using a syringe under nitrogen protection. The solvents were deoxygenated by passing a continuous stream of dry nitrogen through it for 30 min prior to the addition. After the ATRP reaction for the predetermined time, the substrates were removed and sequentially washed with DI water and ethanol twice and air-dried before use.

To estimate the molecular weight of the pSBMA brushes, EBIB was added to the initial reaction mixture as the free initiator with the SBMA/EBIB ratio of 100:1. After polymerization, the molecular weight of pSBMA in solution was determined by gel permeation chromatography (GPC) using a distilled water mixture containing 10 mM lithium bromide as eluent and poly(ethylene glycol) as the standard.

The grafting density (chains/nm²) of pSBMA brushes, σ , was estimated using the following equation: $\sigma = h\rho(N_A/M_n)$, where *h* is the dry thickness of the polymer layer determined by ellipsometry, ρ the density of the dry polymer (1.30 g/cm³ for pSBMA),²⁸ N_A Avogadro's number, and M_n the number-average molecular weight of polymer chains on the surface, estimated from molecular weight of free pSBMA in solution determined by GPC.

2.4. X-ray Photoelectron Spectroscopy (XPS). Surface elements of the initiator and pSBMA-modified (after 1 h polymerization) substrates were analyzed using XPS (VersaProbe II Scanning XPS Microprobe, Physical Electronics). Samples were completely dried in vacuo before the measurement. The binding energy (BE) scale was corrected using C_{1s} as a reference at BE of 285 eV. Three elements characteristic of the initiator or polymer were identified from

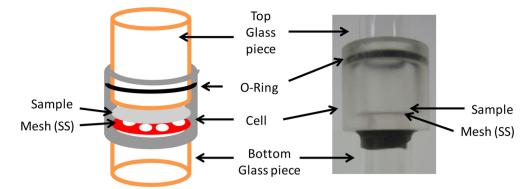


Figure 1. Schematic of the separation cell, with the filter sample and supporting stainless steel (SS) mesh held in place inside the cell between the top and bottom glass tubes.

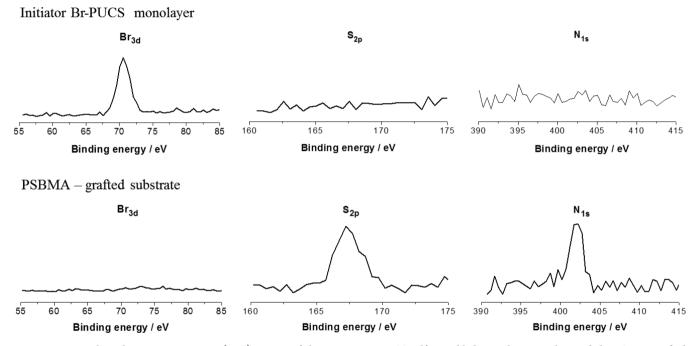


Figure 2. X-ray photoelectron spectroscopy (XPS) spectra of the initiator Br-PUCS self-assembled monolayer on glass and the pSBMA-grafted substrate.

their peak positions: bromide (Br_{3dr} BE \sim 70 eV), sulfur (S_{2pr} BE \sim 168 eV), and nitrogen (N_{1sr} BE \sim 403 eV).

2.5. Film Thickness Measurements. Film thicknesses of the grafted polymer layers were measured using a variable-angle spectroscopic ellipsometer (J. A. Woollam Co.). The phase factor (Δ) and amplitude factor (Ψ) were measured in air at room temperature in the wavelength range of 500–700 nm at a 10 nm interval. The measurement was repeated for incident angles of 65, 70, and 75° and averaged. For the ellipsometry measurement purpose, the back sides of the glass substrates were roughened using sandpaper to prevent backside reflections.²⁹ Measurements were made on the smooth sides of the samples. Fitting of the experimental data was done using WVASE32 software.

2.6. Contact Angle Measurements. Contact angle measurements of the glass slides were made using a Rame-Hart Goniometer. A cell containing a prism stand was used for the underwater measurements. The glass sample was placed on the stand with the polymer-modified side facing down. The cell was subsequently filled with DI water. The tip of a curved needle containing hexadecane was placed under the sample, and the solution was pushed out of the syringe until an oil droplet formed. In doing this, the oil droplet is "captured" underneath the sample, and the contact angle can then be measured.

The in-air contact angle measurements used an approximate water drop size of $3-5 \ \mu$ L, and the underwater contact angle measurements used an approximate oil drop size of $10-15 \ \mu$ L. At least two areas were measured on each sample. The underwater-oil contact angles were analyzed with the Drop-Image software, whereas the in-air water contact angles were analyzed using ImageJ.

2.7. Oil/Water Separation Tests. Oil/water separation tests were performed using the apparatus shown in Figure 1. A piece of glass tube (bottom piece) was embedded permanently into the bottom of a Plexiglas cell (ID = 1 in.) using epoxy glue. A 1 in. stainless steel (SS) mesh (1/8 in. pore size) was placed inside the cell for structural support. The glass fiber filter sample (1 in. diameter) was then placed on top of the stainless steel mesh followed by an additional glass tube (top piece, OD = 1 in., height = 3 in.) to securely hold the fiber sample and supporting mesh in place. An O-ring was embedded inside the cell to prevent any leakage of solution between the glass tube and the cell. The whole apparatus was held vertical using a clamp which was attached to the bottom glass tube.

For the oil/water separation, 10 mL of DI water was first poured into the tube to hydrate the filter samples. A mixture of hexadecane and DI water (10:20 mL), which was prepared by stirring the solution at 1200 rpm for 30 min followed by additional vigorous hand shaking, was poured immediately down the top glass tube. The permeate was

then collected in a graduated cylinder from which the levels were recorded at various times for the water, oil, and total permeate solution. The oil red-O dye was added to the oil for visual purposes.

3. RESULTS AND DISCUSSION

Surface-initiated ATRP is a robust and versatile method to prepare polymer brushes with controlled thickness and welldefined structure on the surfaces. In this work, a "graft-from" method via ATRP (i.e., grafting polymer brushes from the surface with immobilized initiators) was utilized to prepare pSBMA brushes, following the formation of the initiator Br-PUCS monolayer on glass (Scheme 1).

The modified surfaces were characterized by XPS (Figure 2). A peak centered at 70 eV was detected on the initiator-modified surface. The peak is attributed to bromide 3d, suggesting that the Br-PUCS initiator was anchored on the surface. After the ATRP polymerization, XPS signals appeared at 403 and 168 eV for the pSBMA-grafted substrate which were from nitrogen 1s and sulfur 2p, respectively, revealing the existence of SBMA moieties on the modified surface. On the other hand, the signal of bromide 3d was significantly reduced after the polymerization, indicating that the surface was effectively coated by polymers. XPS results suggest that pSBMA was successfully grafted on the surface.

The ellipsometry results (Figure 3) show that the pSBMA thickness increases with the ATRP time, further confirming that

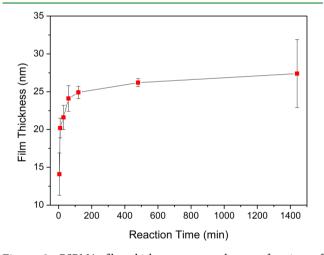


Figure 3. PSBMA film thickness measured as a function of polymerization time using ellipsometry.

the grafting of pSBMA onto the substrates is successful. The film thickness increases quickly within the first 2 h to \sim 24 nm; thereafter, a gradual increase is seen up until 24 h with a film thickness of \sim 27.4 nm. The much slower film thickness increasing rate can be due to either the deactivation of materials

and/or termination of the long polymer chains via selfassociation at later stages of the polymerization. Other groups have reported thickness of the ATRP-generated pSBMA brushes ranging from 5 to 90 nm, $^{23,30-32}$ where the differences could be due to the use of different materials (initiators, catalyst/ligand), substrates (gold, glass, or silicon), or experimental conditions (concentration, reaction time).

The molecular weight (Mn) of the pSBMA brushes, estimated by GPC, was 12.1, 15.5, and 19.8 k, respectively, after 1, 2, or 8 h polymerization. The grafted chain density was determined according to a previously reported method.²⁸ The relationship between the polymer film thickness and molecular weight was first plotted. Using the equation described in the Materials and Methods, the grafting density σ can be derived from the slope of the curve. The grafting density of the pSBMA chains in this work was estimated to be 0.35 chains/nm².

The wettability of the pSBMA grafted glass slides was characterized via contact angle measurements as shown in Figures 4 and 5. The average in-air water contact angle (Figure 5a) of the control glass slide, represented as the 0 reaction time, was 31°, while the water wettability increased toward superhydrophilicity with contact angles of $\sim 8-15^{\circ}$ for the pSBMA-grafted glass slides. Even though the pSBMA film thickness increases over polymerization time, as shown in the ellipsometry results, the in-air water contact angles do not become more hydrophilic with time.

Similarly, underwater-oil contact angles (Figure 5b) on the pSBMA-grafted glass slides resulted in an increase toward underwater superoleophobicity with contact angles of 162–169°, while the control pristine glass slide had a contact angle of 142°. This increase in superoleophobicity is acquired due to water being trapped at the solid–oil–water interface by the polymer layer, thus effectively preventing strong adhesion of oil. It is also observed that if the prism stand of the goniometer for samples was not perfectly leveled the oil droplet would start moving, thus suggesting low tilt angles for the pSBMA-grafted samples. Notice that the contact angle characterizations of the pSBMA-grafted glass fiber mats were not possible due to flexibility and intrinsic absorbing property of the substrates.

It is remarkable that we have achieved underwater superoleophobicity, with an oil contact angle of $162-169^{\circ}$, only by modifying surface chemistry. Current literature generally needs to use both surface chemistry and surface roughness to achieve similar effects. For example, a flat copper surface modified with hydrophilic OH-terminated thiol led to an underwater-oil contact angle of 129° ; only after roughening surfaces with nanostructures, the superoleophobicity (163°) was realized.³³ In our work, a nanostructured surface is not needed.

The pSBMA-grafted glass slides exhibit the underwater antifouling (oil-resistant) characteristics. When a pSBMAgrafted glass slide was placed in water after being dipped in



Figure 4. Images of the in-air water contact angles (a) and underwater hexadecane contact angles (b) at different pSBMA polymerization times.

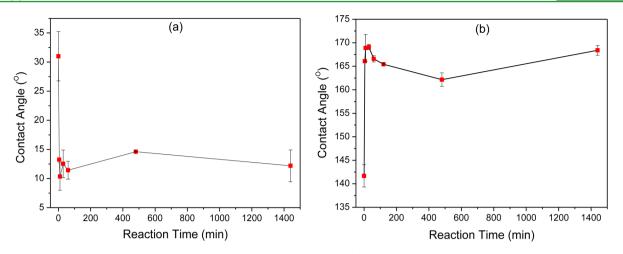


Figure 5. In-air water contact angle (a) and underwater hexadecane contact angle (b) for pSBMA-grafted glass substrates at different reaction times. Sample with 0 reaction time refers to the control glass without pSBMA modification.

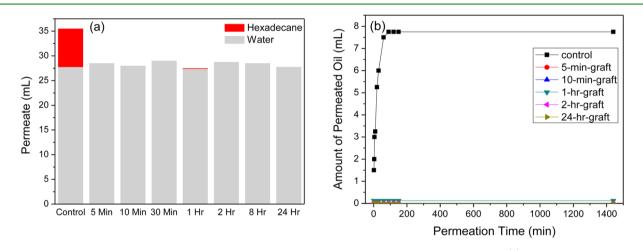


Figure 6. Oil-water separation data of the pSBMA-grafted glass fiber filters with various polymerization times. (a) Amount of permeated water, permeated oil, and total permeate after 24 h permeation. (b) Amount of permeated oil with increasing permeation time. Water (10 mL) was used to first hydrate the filters, followed by the addition of a mixed water/hexadecane solution (20 mL:10 mL).

oil, the oil droplets easily floated up (see video am502302g_si_001.avi in Supporting Information). Conversely, the oil droplets gathered together onto the surface of the control pristine glass slide (video am502302g_si_002.avi in Supporting Information). Even after mild agitation in water, the control sample still retained some amount of oil on its surface. These observations suggest that the pSBMA-grafted surfaces are self-cleaning and oil-resistant underwater. A similar observation was observed with fish scales, where fish exhibit superoleophilicity in air and superoleophobicity underwater and therefore are self-cleaning in oil-polluted water.¹⁷ Furthermore, the pSBMA-grafted glass fiber mats, after being hydrated and placed in an oil environment, were not fouled; therefore, they are also self-cleaning underwater (video am502302g_si_003.avi in Supporting Information).

As shown in the experimental setup for oil—water separation (Figure 1), the pSBMA-grafted glass fiber sample was placed inside the Plexiglas cell on a SS mesh that was used for structural support purposes. After hydrating the sample with water, the water/hexadecane mixture was poured into the top glass tube. It was found that the water selectively permeated through the pSBMA-grafted filter, while the oil was retained above the sample (Figures 6 and 7, video am502302g_si 004.avi in Supporting Information). Even samples grafted

with pSBMA for 5 min can be effectively used to completely prevent the oil from permeating through the filter while allowing the water to quickly permeate within seconds. In contrast, for the control fiber filter without pSBMA coating, most of the oil permeated through within 1 h along with water (Figure 6a and 6b), while the oil was retained on the top for the pSBMA-grafted samples for over 24 h (Figure 7d).

With the rigorous stirring and mixing, the hexadecane/water mixture used in the work can remain stable for about 2 min before spontaneous separation (no surfactant was added into the mixture). On the other hand, oil—water separation through the pSBMA-modified membranes started almost instantaneously and mostly completed within 1 min, so the separation was mostly through the membrane, not just a spontaneous process.

Not only the pSBMA-grafted filters effectively separate oil from water but also they can be easily cleaned with water and ethanol and reused, and the separation requires no additional force other than the gravitational force, suggesting that such filters are good candidates for industrial oil-water separation.

Due to the in-air oleophilic property of the pSBMA-grafted samples, the filters were first hydrated with water before the oil-water separation test. This prevents the samples from absorbing the oil on contact and further inhibits the permeation of oil through the filter by capillary effects. Hexadecane, an

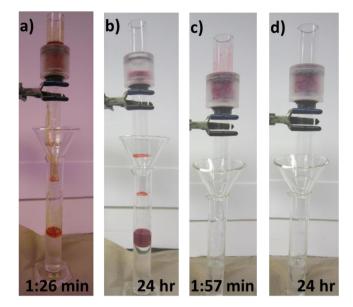


Figure 7. Images of the oil/water separation apparatus with the control filter (a, b) or the pSBMA-grafted filter (c, d). A mixture of DI water and hexadecane was poured into the top glass tube. With the pSBMA-grafted glass filter, water selectively permeated through the filter, while oil, in red, was retained on the top (c, d).

aggressive foulant with a low surface energy of 26.9 mN/m,¹⁹ was used as the model oil for our studies. During the oil–water separation, the underwater superoleophobic property of the pSBMA-grafted samples, which is due to the presence of a strong hydration layer associated with the polymer brushes between the surface and oil and the high repellency between the nonpolar (oil) and polar (water, pSBMA) molecules, prevents oil droplets from adhering, therefore making oil separation and material recycling very easy.

If not initially hydrated, the pSBMA-coated samples can still separate oil and water to some extent. That is, a small amount of oil permeated through the filters during the initial stage after pouring the oil-water mixture; the oil stopped permeating thereafter, presumably when the entire sample was quickly hydrated with water due to its superhydrophilic property. The oil did not further permeate for over 24 h. Nevertheless, to achieve the best oil/water separation, i.e., no permeation of a single drop of oil, pSBMA-coated samples need to be prehydrated. With the control glass fiber filter, if not initially hydrated, the oil rapidly permeated the filter within a few minutes.

To get a different perspective on the hydrophilicity of the pSBMA-grafted fibers, water absorption experiments were performed (Figure 8). The water absorption level was determined based on the weight difference of the hydrated and dry filters as follows: water absorption (%) = $[(W_{\rm S} - W_{\rm D})/$ $W_{\rm D}$ × 100%, where $W_{\rm S}$ represents the weight of the filter after water uptake and W_D represents the weight of the dry filter. Overall, the pSBMA-grafted fiber filters exhibited marginally more water absorption, when compared to the nongrafted control glass fiber sample that absorbed 1011% water. The 5 min, 10 min, 1 h, and 24 h pSBMA-grafted samples resulted in 22, 21, 6, and 6% more water absorption than the control sample, respectively (no statistical difference). Water absorption can be attributed to several factors: capillary effect, hydrophilicity of glass, and superhydrophilicity of pSBMA. Because of the hydrophilicity of the glass substrate and capillary

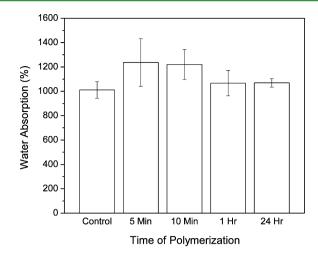


Figure 8. Water absorption of the control glass fiber filter and the pSBMA-grafted glass fiber filters with different grafting times. The percentage reported was determined from the weight difference of the hydrated and dry filters normalized to the dry filter weight.

effect, even the control glass fiber sample itself can absorb 1011% water, which made it difficult to probe the effect of pSBMA coatings on water adsorption for our case. A previous report shows that the hydrophobic ePTFE (i.e., expanded polytetrafluoroethylene) microporous membrane had zero hydration capacity, whereas the pSBMA-grafted ePTFE membrane had significantly improved hydration capacity, demonstrating the ability of pSBMA surface grafting to retain water.³⁴ In another work, it was found from NMR that a single SBMA monomer can bind eight water molecules.²⁴ The increased swelling of the hydrated pSBMA-grafted samples may have helped to achieve the oil–water separation.

Scanning electron microscopy (SEM) was used to characterize surface morphologies of the glass fibers, both before and after pSBMA grafting. Figure 9a is a typical SEM image of the control glass fiber with no pSBMA modification, showing a porous unwoven fiber structure with an average fiber diameter of 6.5 μ m and a pore range of 50–350 μ m. Figures 9b and 9c are for the 5 min and 1 h pSBMA-grafted samples, respectively. The diameters of the pSBMA-grafted glass fibers remain about 6.5 μ m, thus showing no significant change after the ATRP grafting step. There are also no materials within the pores, therefore ensuring free passage of solutions through the pores. Surfaces of the modified fibers appear smooth with no visible topographical features at the 5 μ m scale (Figure 9c, inset). Overall, the pSBMA grafting does not cause significant morphological changes of the glass fibers, implying that the superhydrophilicity and underwater superoleophobicity are mostly achieved via surface chemistry, not by surface roughness.

The fact that amphiphilic surfaces in air turn to be superhydrophilic–superoleophobic underwater indicates that oil contaminants can be easily washed away when immersed in water; therefore, such a coating is self-cleaning and can be used against marine ship fouling.³⁵ Furthermore, studies have found that upon heating or addition of salt pSBMA shows enhanced water wettability due to the self-association state being reversed.^{36,37} The novel pSBMA-grafted fiber filters are therefore expected to perform even better and can be used under harsh conditions such as high temperatures and high salt concentrations. The presented polymer coating can be used for potential applications in oil spill cleanup, wastewater treatment,

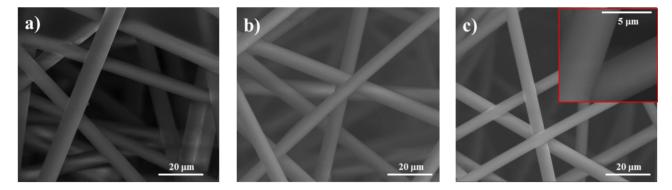


Figure 9. SEM micrographs of (a) the control pristine glass fiber sample, (b) the pSBMA-grafted sample with polymerization time of 5 min, and (c) the pSBMA-grafted sample with 1 h polymerization time.

underwater marine antifouling coating, self-cleaning coating, and antibiofouling surface.

4. CONCLUSIONS

In conclusion, novel superhydrophilic and underwater superoleophobic pSBMA-grafted glass fiber mats have successfully been engineered for the use of oil—water separation. PSBMAgrafted glass slides also exhibit underwater superoleophobicity, and therefore self-cleaning properties are ascribed, which can be used against fouling from oil as well as marine life. With the nontoxic and inexpensive materials, easy fabrication, rapid and efficient separation, easy cleaning, and low energy requirement, the technique counteracts some of the disadvantages of the current membrane filtration technologies such as membrane fouling, low efficiency, and high operation costs.

ASSOCIATED CONTENT

S Supporting Information

Four videos are provided. am502302g_si_001.avi and am502302g_si_002.avi show the underwater-oil resistance of the pSBMA-grafted glass slide and underwater-oil nonresistance of the control pristine glass slide, respectively. am502302g_si_003.avi shows the underwater-oil resistance of the pSBMA-grafted glass fiber mats. am502302g_si_004.avi demonstrates oil—water separation through the pSBMA-grafted glass fiber filter, with oil retained above and water passing. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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