

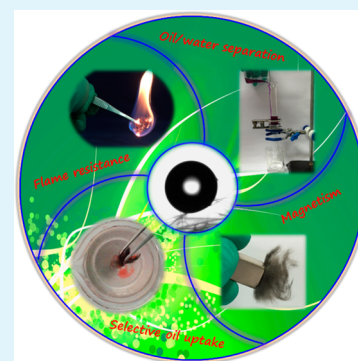
Ultrathermostable, Magnetic-Driven, and Superhydrophobic Quartz Fibers for Water Remediation

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Supporting Information

ABSTRACT: A quartz fiber based 3D monolithic materials was fabricated, which combines ultrahigh thermostability, remote controllability, mechanical flexibility, high water/oil selectivity, high processing capacity, and regeneration ability. This material exhibited great potential in water remediation, such as large absorption capacity (50- to 172-fold weight gain) toward oil standing in front of all magnetic sorbents and remarkable oil/water separation performance.



KEYWORDS: quartz fiber, thermostable, magnetic, superhydrophobic, water remediation

1. INTRODUCTION

With increasingly aggravated environmental problems, especially water pollution throughout the world because of oil spills and discharge of industrial effluents, the subject of water remediation such as oil sorption and oil/water separation has received growing attentions in past few years.^{1–4} From the view of practical applications, some key attributes should be considered for materials design in this field. First, high oil/water selectivity is required for achieving high efficiency. Second, a large processing capacity is preferred for large-scale applications. Third, remote controllability is essential to facilitate materials handling. Fourth, good mechanical properties are important for handling and reuse of materials. Last, materials with excellent tolerance under harsh conditions (e.g., a wide working temperature range) is extremely desirable for adapting to diverse work conditions, achieving long lifespan, and avoiding secondary pollution.

In recent years, 3D hydrophobic materials (3D-HM) have emerged as rising stars to address both oil sorption and oil/water separation, for their large processing capacity.^{5,6} 3D-HM can be generally divided into two classes according to the synthetic routes. In the first class, 3D-HM are fabricated via bottom-up assembly, such as the preparation of carbon nanotubes (CNT) sponges,⁷ carbon-based aerogels,^{8–12} and microporous polymer aerogels.^{13,14} In this class, certain carbon-based materials can acquire high absorption capacity up to 1000-fold weight gain. However, only a few of them possess sufficient mechanical strength and flexibility for easy handling.⁷ In the second class, 3D-HM are derived from the modification of commercial foams^{2,5,6,15–20} or fibers.^{21–23} Taking advantage

of as-existed supports, the materials often enjoy excellent mechanical properties that facilitate handling and regeneration process. Additionally, this architecture also allows the better control of microscale structures for improved water/oil selectivity. For instance, we have elaborately devised a series of well-defined microstructures with diverse dimensions on melamine foam, from which the wettability of materials could be controllably modulated and systematically improved.⁶ To realize further the remote controllability, magnetism has also been introduced in 3D-HM by both bottom-up assembly of magnetic building blocks (e.g., magnetic CNTs) or post-modification of magnetic components (e.g., Fe₃O₄) onto as-obtained 3D networks.^{24–33} For instance, Gui et al.²⁶ reported magnetic carbon nanotubes (CNTs) sponges with high recyclability by in situ filling of iron nanowires in tubes during chemical vapor deposition (CVD) growth, which showed an oil absorption capacity of 49- to 56-fold weight gain. In another work, polyurethane/Fe₂O₃ nanoparticles/submicrometer polytetrafluoroethylene particles was fabricated by Calcagnile et al.,²⁴ which exhibited high oil/water selectivity and an oil absorption capacity of ~13.25-fold weight gain. Actually, because of the additional introduction of magnetic components, the as-obtained 3D-HM always possess relatively low absorption capacity (typically less than 30-fold weight gain) as a result of increased density, which is one of the great challenges that retards the practical use of those magnetic

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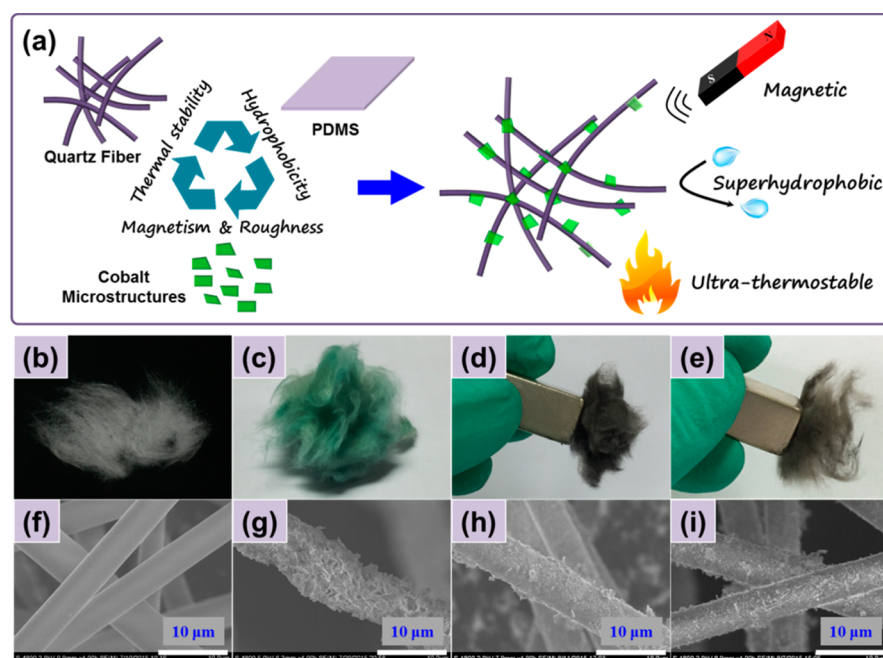


Figure 1. (a) Schematic illustration of the synthetic route toward TMS-QF. (b–e) Digital photos of (b) pristine QF, (c) cobalt(II) hydroxide loaded QF, (d) cobalt-loaded QF, and (e) TMS-QF. (f–i) Corresponding SEM images of materials in b–e, respectively.

materials. To address this issue, we recently fabricated superhydrophobic melamine foams by engineering magnetic components into hybrid dimensional microstructures, which simultaneously realizes the magnetism integration and high oil/water selectivity, thus realizing high absorption capacity (60- to 160-fold weight gain) and remote controllability at the same time.³³

Another consideration for 3D-HM is their thermostability. Until now, few works have achieved 3D-HM with sufficiently high thermostability (e.g., 900 °C in air). In this context, the thermostability is referred to the stability of 3D networks. We note that although hydrophobic layer would be decomposed upon heating even in an inert environment the hydrophobicity could be recovered by a second-round treatment if the 3D networks could survive high-temperature treatment. Several carbon-based 3D-HM are reported to exhibit high thermostability of up to 1000 °C in inert environment.^{9,10,17} However, they are unable to withstand high temperature (e.g., > 600 °C) in air even after annealing, which affects their use in real condition. Some metal foam based materials, such as nickel foams and copper foams, can retain the 3D frameworks against high-temperature treatment although suffering from oxidation. However, their huge densities (typically >300 mg cm⁻³) would greatly retard their specific processing capacity in water remediation, making them impossible for industrial applications. Hence, it is still a big challenge to create 3D-HM with highly thermostable networks. Thus, the fabrication of 3D-HM with combined high water/oil selectivity, remote controllability, superior mechanical properties, good regeneration ability, and high thermostability is still quite a challenge. To address the above issues, new materials need to be created.

It is known that quartz fiber (QF), a commonly used material for gas filter and thermal insulation,^{34,35} possesses excellent flexibility and ultrahigh thermal stability over 1000 °C in air, which overwhelms nearly all reported materials such as polymer sponges (Figure S1), CNT aerogels, and graphene frameworks.^{9,10,12} Therefore, we hypothesize that the QF can be

utilized as a superior 3D thermostable and mechanically flexible substrate to sequentially load magnetic microstructures and hydrophobic coatings, thus producing a 3D-HM satisfying the requirements mentioned above. In this way, except for providing magnetism for remote control, the microstructured magnetic components can also account for the significantly enhanced water/oil selectivity in conjunction with hydrophobic coatings by boosting the surface roughness. Additionally, the increased surface roughness can also accelerate the oil uptake process and enable oils to easily penetrate into inside space of fibers, thus greatly promoting the oil absorption capacity. More importantly, the QF provides a mechanically flexible and highly thermostable substrate, which not only facilitates easy handling and diverse deformation during practical use but also enables the reservation of 3D networks of materials after experiencing extreme temperature and allows the regeneration afterward. Therefore, by this method, the resultant 3D materials could simultaneously meet nearly all requirements for water remediation as mentioned above. Herein, we introduced commercial QF as a substrate for water remediation for the first time. To integrate magnetism and amplify the intrinsic wettability of materials, magnetic cobalt microstructures were introduced on to the QF. After coating of a hydrophobic layer, an ultrathermostable, magnetic-driven, superhydrophobic, and flexible QF (denoted as TMS-QF) was obtained. The resultant materials not only showed great potential in magnetic-driven selective oil uptake (absorption capacity of 50- to 172-fold weight gain, which stands in front of all that of magnetic sorbents) and high-efficiency oil/water separation performance (separation ratio >99%) but also ultrahigh stability in wide temperature range from -196 to ~1150 °C in air. Moreover, this material exhibited an interesting self-healing-like behavior even in a dry state, facilitating its facile handling and recycling in practical applications.

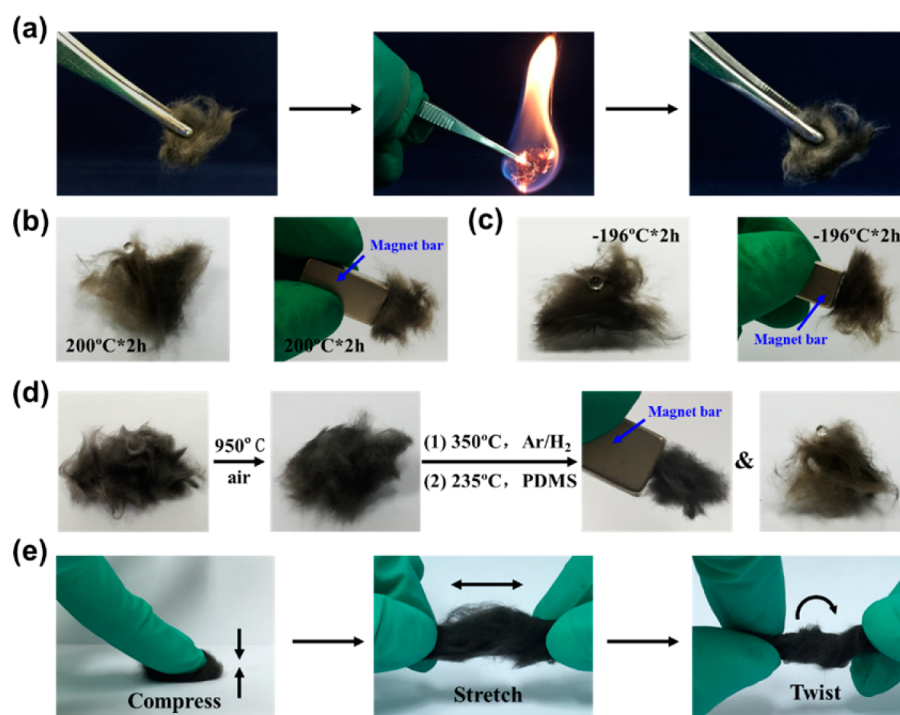


Figure 2. (a) Illustration of the flame resistance of TMS-QF. (b and c) Representation of the hydrophobic and magnetic properties of TMS-QF after experiencing normal high-temperature (200 °C in air for 2 h) and low-temperature (−196 °C in liquid nitrogen for 2 h) environments, respectively. (d) Recovery of hydrophobicity and magnetism of TMS-QF after treatment at ultrahigh temperature. (e) Tolerance of TMS-QF toward different deformation.

2. RESULTS AND DISCUSSION

2.1. Synthesis and Basic Characterizations. The synthesis route is shown in Figure 1a. Briefly, the QF was first loaded with cobalt(II) hydroxide by controlled precipitation using epichlorohydrin as the inert proton scavenger (Supporting Information).^{6,36,37} Then, the $\text{Co}(\text{OH})_2$ -loaded QF was transformed to cobalt-loaded QF by reductive annealing in hydrogen/argon atmosphere. Via the above two steps, the cobalt microstructures were introduced to provide both magnetism for remote control and microstructures for enhanced surface roughness. Then, a low-energy polydimethylsiloxane (PDMS)-based conformal layer was deposited on the cobalt-loaded QF by a simple coheating method,^{6,8,20,33,38} thus creating the final product. It should be noted that during this step, the PDMS deposition can prevent the as-existing cobalt from the oxidation in air,³³ thus retaining the magnetism of the cobalt in cobalt-loaded QF. The chemical composition of loaded matters was determined by X-ray powder diffraction (XRD) measurement (Figure S2), which verified above analyses.

The macroscopic appearance and microscopic morphology of the material in each step are shown in Figures 1b–i and S3. As shown Figure 1f, 3D macroporous networks of QF were assembled from randomly aligned individual fibers via van der Waals (VDW) interaction and interfiber entanglement, which was similar to that of CNT sponges.²⁶ Because of the low bonding strength of interaction, the pore cannot maintain a regular shape. After the loading of $\text{Co}(\text{OH})_2$, the fiber was partially covered with micrometer-size flat sheets. Following reductive annealing, most sheets were converted to smaller-size irregular microstructures, thus producing highly rough surfaces. Notably, the final PDMS coating process did not affect the morphology of cobalt-loaded QF, giving rise to the hydro-

phobic surface retaining original microstructures. Because fibers are covered by PDMS layer, interactions between fibers can only be attributed to the interactions between PDMS. From the chemical structure, no covalent bonds or hydrogen bonds could be formed between PDMS molecules during the self-healing-like process. Hence, only VDW interactions could account for the attraction between fibers in final materials.

On the basis of the combination of the low-energy surface endowed by PDMS coatings and the high roughness provided by microstructures, the resultant TMS-QF exhibited remarkable superhydrophobicity. As shown in Figure S4a,b, TMS-QF possessed a high static water contact angle of $\sim 165.4^\circ$, which is about 20° higher than that of pristine QF after direct PDMS coating (146.8°). The value of the contact angle is also higher than that of a series of 3D-HM such as carbon-based aerogels ($140\text{--}156^\circ$),^{7,8,26} microporous polymer aerogels ($126\text{--}135^\circ$),^{13,14} and some polymer foam based materials ($140\text{--}149^\circ$).^{2,17,18} Additionally, TMS-QF also possesses superoleophilicity, which can be evidenced from a zero contact angle of toluene (Figure S4c). This high water/oil selectivity is essential for its applications in water remediation.

For practical use, materials with high stability in a wide temperature range in ambient atmosphere is essential. The temperature tolerance of TMS-QF is carefully characterized step by step (Figure 2a–d). First, the TMS-QF can maintain both the superhydrophobicity and magnetism after treatment at normal high temperature (200 °C) and low temperature (−196 °C) environments (Figure 2b,c), which facilitates its direct use in most conditions. At higher temperature, the hydrophobicity and the magnetism would be lost in a stepwise manner because of the decomposition of PDMS layer and the oxidation of cobalt microstructures (220–300 °C). This is also common for other 3D hydrophobic materials. Fortunately, thanks to the

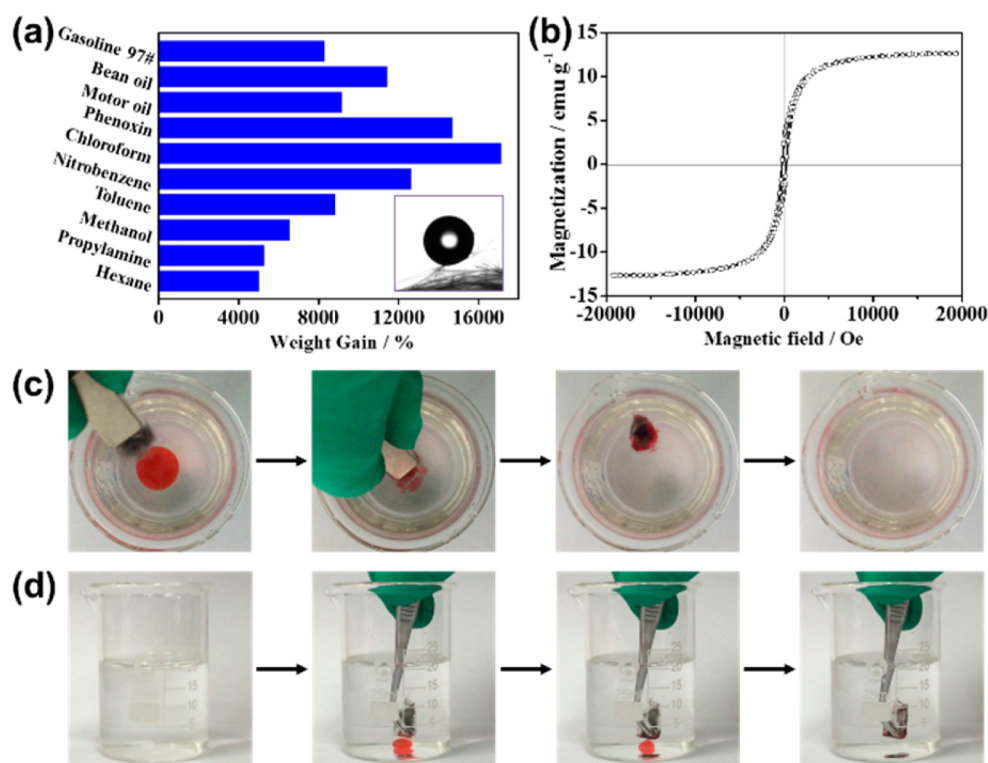


Figure 3. (a) Oil uptake capacity of TMS-QF toward a series of organic solvents and oils. Inset shows that a water droplet can maintain a perfect sphere shape on the TMS-QF. (b) Magnetization curves of TMS-QF. (c and d) On-water and underwater absorption of organic solvents by using TMS-QF, respectively. For on-water absorption, a magnet was used to drive the motion of TMS-QF for remote control sorption. The organic solvents (dyed by Sudan III) used in c and d were toluene and chloroform, respectively.

ultrahigh thermostability and flame resistance inherited from the original QF, the 3D networks of TMS-QF can survive in air at extremely high temperatures. As shown in Figure 2a, anhydrous ethanol saturated pristine QF and TMS-QF were lighted in air, neither of which showed visible change before and after burning. This means that the sorbent can withstand a temperature at least several hundred degrees Celsius. To test further the limiting service temperature, the TMS-QF was placed into the muffle furnace and heated at 950 °C for 30 min in air. After ultrahigh temperature treatment, the material still remained its marvelous mechanical flexibility. In this case, the magnetism and hydrophobicity of resultant material can be facily recovered by a second round of reductive annealing and PDMS coating (Figure 2d). At a further increased temperature of 1150 °C, the QF can still retain its mechanical properties, although the fibers turned purple because of the reaction of cobalt oxides and QF. Despite this, the TMS-QF can still be regenerated by reloading of fresh cobalt microstructures and PDMS layers, indicating its pronounced regeneration ability. However, further increased temperature (e.g., above 1200 °C) would soften the QFs and permanently degrade their mechanical properties. In summary, TMS-QF can maintain its 3D networks and regenerate even after treatment up to 1150 °C in air, suggesting its extraordinary thermostability. If the normal regeneration process is involved, then the as-prepared TMS-QF can exhibit an ultrawide working temperature range from -196 to 1150 °C, overwhelming all existing materials in the field of water remediation.

Apart from superhydrophobicity/superoleophilicity and ultrahigh stability in a wide temperature range, TMS-QF also displayed remarkable mechanical properties because of non-covalent-bonded 3D networks constructed by randomly aligned

fibers, which is similar to that of CNT sponges.²⁶ As mentioned above, the 3D network of TMS-QF is held together by both VDW interactions and interfiber entanglement. As shown in Figure 2e, TMS-QF could withstand a wide range of mechanical deformations such as compressing, stretching, and twisting, which facilitates their handling under real conditions. It should be noted that the materials' properties and performance will not be obviously affected after immersing in various solution (Figure S5). For example, the thermostability, magnetism, hydrophobicity, and oil absorption performance were well-maintained after treatment. However, it should be noted that the elongated time could cause the dissolution of cobalt in acid environment.

2.2. Selective Oil-Uptake Performance of TMS-QF.

Combining all the above excellent properties, TMS-QF could find great potential in selective oil sorption. As depicted in Figure 3a, TMS-QF showed a high sorption capacity toward a wide range of organic solvents and oils (50- to 172-fold weight gain), comparable to or higher than that of other type nonmagnetic fibers such as carbon fiber aerogel, electrospun fibers, and superhydrophobic kapok fibers (7- to 190-fold).^{21–23} Although the value is still lower than some nonmagnetic carbon nanotube or graphene-based aerogels (from 10- to 1000-fold weight gain),^{7,8,10,11} the unique superior mechanical flexibility and magnetic-driven ability of TMS-QF can more or less compensate for this weakness. Actually, except for very few examples,^{25,29} the absorption capacity of TMS-QF was much higher than that of most reported magnetic 3D hydrophobic materials (typically less than 30-fold).^{24,26,28,30–32} Apart from the prominent absorption capacity, the TMS-QF could be directly used in treatment of oil/water mixture such as that in a real system. As shown in Figure 3c,d, the fibers can

easily remove the organic solvents either on water or underwater. Because of the adequate magnetism (saturation magnetization $\sim 12.7 \text{ emu g}^{-1}$, Figure 3b), the on-water oil removal process can be readily driven by a magnet, which facilitates remote control and easy collection after sorption.

The recyclability is another important aspect by which to evaluate the sorbent. As shown in Figures S6 and S7, thanks to the high thermostability and remarkable mechanical flexibility, the organic solvent saturated TMS-QF can be regenerated by either heating, combustion, or squeezing, depending on the properties of adsorbates. For regeneration by heating and combustion, over 70% absorption capacity can be preserved. Notably, the mass of original TMS-QF remained nearly unchanged after 10 combustion cycles, much more stable than carbon-based sorbents such as nitrogen-doped CNT aerogels ($\sim 40\%$ loss after the first combustion cycle).⁹ The regeneration ability toward viscous oils absorption has also been tested, with viscous motor oil (15W-40, with the viscosity over two magnitudes higher than that of common organic solvent) was used as the model adsorbate. As shown in Figure S8, TMS-QF showed the capacity retention of $\sim 45\%$ and $\sim 73\%$ for recycling by combustion and squeezing, respectively.

The capacity loss during the regeneration process can be explained by the interactions between QFs (Figures 4a and

because of the increased density.⁸ For motor oil, the more obvious capacity loss might be attributed to the incompletely combustion due to its larger number of carbon atoms per molecule compared with that of acetone, which could leave carbon species in the network after burning and thus reduce the accommodation room for adsorbates in the next round of absorption. The regeneration process by squeezing leads to lower capacity retention for chloroform absorption, which can be attributed to the increased packing density induced by the squeezing force (Figures 4b,c and S6c,d). By contrast, the relative higher capacity retention toward motor oil absorption could be attributed to its high viscosity, which makes it difficult to be removed from TMS-QF by squeezing thus more or less suppressing the aggregation of fibers. Actually, over 30 wt % absorbed oil remained in the fibers' networks by simply squeezing. Although TMS-QF showed certain capacity fading after cycling, it still possesses relatively high sorption capacity (~ 70 -fold weight gain for chloroform) even after several regeneration rounds.

The weak VDW interaction in TMS-QF can also bring some unexpected bonuses. For example, because the VDW interaction is so weak that it allows easy and reversible breaking and recombination, the QF-based materials can behave like self-healing materials such as certain hydrogels.³⁹ To distinguish this VDW interaction induced behavior from traditional self-healing behavior, we called it "self-healing-like behavior". As shown in Figure S9, after breaking up, the two resultant pieces of the fibers can be easily adhered together by simple pressing, which is attributed to the formation of new cross-linking junctions between fibers through VDW interactions. Interestingly, the fibers can readily "heal" in a dry state, which is impossible for conventional self-healing wet gels. It should be noted herein that the interfiber entanglement might be less effective than VDW interaction because it is hard to form complex mechanical entanglement during a simple self-healing-like process in air. The mechanical strength at the healed junction is always weaker than that of original state, which might also indicate the reduced interfiber entanglement after adhering. Nevertheless, the mechanical strength could be further improved by dropping some liquid (e.g., ethanol) on the fracture region, which can be attributed to the promotion of the fibers' mobility thus forming more cross-linking junctions in fractured region. This intriguing self-healing-like behavior is useful to facilitate the handling and recycling process of materials.

2.3. Oil/Water Separation Performance of TMS-QF.

Apart from the selective oil sorption, TMS-QF can also be used as an efficient filter for oil/water separation because of its ability to block water penetration selectively (Figure 4d,e). During the separation process, the droplet bears the gravity (G), static pressure (F_p) applied by solution above it, and the Laplace pressure (F_L) applied by the filter. Given the area of the filter, the F_p is dependent on the height of liquid column, whereas F_L relies on the surface wettability of the filter. F_L is in the opposite direction to the G and F_p for the water droplet but in the same direction for the oil droplet. Hence, for the oil droplet, it could easily penetrate the filter without resistance. For water, it would be blocked at the filter by F_L , where we have

$$F_L = F_p + G \quad (1)$$

With the accumulation of water during separation process, F_p will improve in proportion to the height of liquid column, causing the gradual wetting of the substrate. With the increased

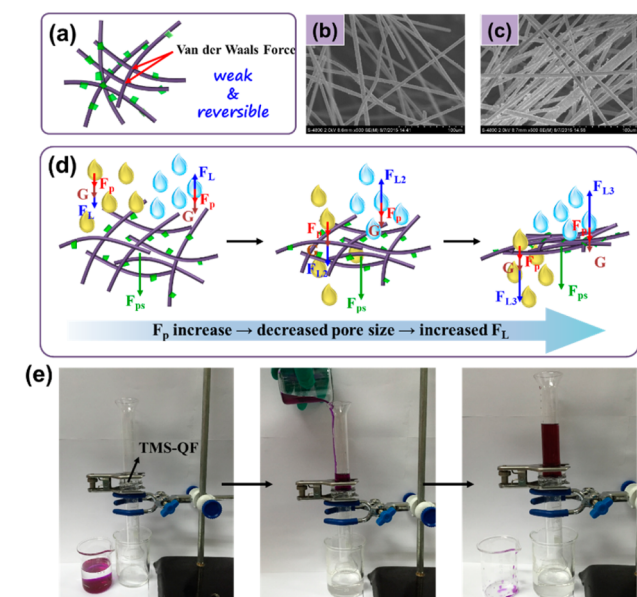


Figure 4. (a) Illustration of possible interactions in TMS-QF. (b and c) SEM images of TMS-QF before and after absorption (followed by drying via squeezing) of chloroform. (d) Proposed mechanism and analysis of oil/water separation by using TMS-QF. (e) Digital photos of oil/water separation process using TMS-QF as the filter.

S6e). Unlike commercial polymer foams (e.g., polyurethane foams and melamine foams) that are constructed by 3D covalent-bonded networks, the network of QFs is held together mainly through VDW interactions. Similar to that of hydrogen bonds,³⁹ the VDW interaction is much weaker than that of covalent bonding, leading to reversible breaking and recombination behavior. During the regeneration process by heating or combustion, the fibers will undergo great surface tension change before and after drying,¹³ easily causing the aggregation of fibers and leading to the increase of material's density. As a result, the sorption capacity decreased with repeating cycles

solid–liquid contact area, F_L will also be enhanced correspondingly. In this way, below a certain liquid column height (corresponding to the intrusion pressure F_i), the oil/water mixture can be easily separated. Above F_i , the filter cannot provide sufficient high F_L to balance $F_p + G$, thus being completely wetted and losing the separation ability.⁶ In this case, we have

$$F_L = F_i < F_p + G \quad (2)$$

Herein, the VDW interaction between fibers showed its power again. For covalent cross-linked polymer foams, the pore structures cannot deform significantly because of the stiff structure fixed by the strong covalent bonds. Hence, during the separation process, the Laplace pressure will not undergo a large change. In contrast, for the VDW force cross-linked QFs, it can easily deform and aggregate together under the static pressure generated by the liquid column. As schemed in Figure 4d, with enhanced aggregation, an increasingly dense filter will result in smaller pore size in the material, thus generating higher F_L . In this way, the water repellence can be automatically enhanced with increasing liquid column height by this “self-densifying” behavior, thus exhibiting high intrusion pressure. In our case, a separation ratio above 99% was calculated from filtering either hexane/water mixture (v/v: 40/10), chloroform/water mixture, or motor oil/water mixture by using ~20 mg QFT-MS according to a previously reported method.^{6,14} The separation process is conducted at ambient pressure. The water flux was calculated to be about 36 000 L m⁻² h⁻¹ for the former two systems, which is much higher than that of third system (~1500 L m⁻² h⁻¹). This is attributed to the significant viscosity difference between common organic solvents and viscous motor oils. The intrusion pressure of ~1 kPa was obtained for the filter, comparable to that of hydrophobic copper foams, melamine foams, and porous nitrocellulose membranes.^{5,6,40} Theoretically, the easy deformability also allowed the insertion of more fibers as the filter, which can further improve F_i and thus enlarge processing capacity. More importantly, even if the filter is penetrated by the water due to the high static pressure, it could be easily regenerated by ethanol washing and drying, which is promising for repeated use.

3. CONCLUSIONS

By utilizing QF as the substrate to support magnetic cobalt microstructures and a hydrophobic PDMS layer, an ultra thermostable, magnetic-driven, superhydrophobic, flexible, and regenerable 3D monolithic material was successfully fabricated. Herein, the QF provided high thermostability and mechanical flexibility, and cobalt microstructures contributed to both magnetism and high surface roughness. In this way, the resultant TMS-QF combined nearly all important elements required for water remediation in practice. The 3D networks of TMS-QF could tolerate an ultrawide temperature range from -196 to ~1150 °C in air, covering most practical conditions. Additionally, TMS-QF not only showed great potential in selective, remote control, and recyclable oil uptake behavior with stunning sorption capacity (50- to 172-fold weight gain) but also can serve as a high-efficiency filter in oil/water separation with a unique self-densifying behavior. More interestingly, thanks to the reversible interactions between QFs, the resultant material also exhibited self-healing-like behavior even in a dry state, considerably facilitating handling and recycling process. The presented materials may open a new

avenue in fabricating high-performance, highly stable, and remote controllable materials for water remediation.

4. EXPERIMENTAL SECTION

4.1. General. All reagents, such as cobalt acetate tetrahydrate, epichlorohydrin, and QF (diameter: 1–3 μm) were purchased from commercial suppliers (Alfa-Aesar, TCI or Aladdin Chemistry Co., Ltd.) and used without further purification. Thermogravimetric analysis (TGA) was conducted via Q600 STD with heating rate of 10 K min⁻¹ in air. Scanning electron microscopy (SEM) was carried out on a Hitachi S-4800 field-emission-gun scanning electron microscope. Contact angle measurement was directly conducted on the aerogel at room temperature. The data was collected by OCA20 (Dataphysics). More than five positions were measured per sample to receive a mean contact angle.

4.2. Preparation of Magnetic-Driven, Superhydrophobic, and Highly Thermal Stable Quartz Fiber (TMS-QF). The QF was first dipped into the cobalt acetate tetrahydrate aqueous (0.1 M) containing epichlorohydrin (10 vol %) and heated at 50 °C for ~24 h. Then, the resultant QF was washed with water and frozen at -18 °C for ~12 h before freeze-drying for 24 h, thus obtaining cobalt(II) hydroxide loaded QF (C-QF).

The C-QF was transferred into a quartz tube and annealed at 350 °C for 1 h under a flow of Ar/H₂ of 300/50 (sccm), thereby acquiring cobalt-loaded magnetic QF (M-QF).

The resultant M-QF was coated with polydimethylsiloxane (PDMS) by a simple vapor deposition process. In short, M-QF was coheated with PDMS at 235 °C for 15 min in a sealed container, thus producing superhydrophobic and magnetic QF (TMS-QF).

4.3. Oil Uptake Measurement. Typically, a piece of TMS-QF (~2 mg) was dropped into organic solvent or oil for several hours to achieve complete absorption equilibrium. Then, the weight gain (%) was calculated by

$$q_m = 100 \times (m - m_0) / m_0$$

where m_0 and m represent the mass of TMS-QF before and after absorption, respectively. The regeneration of TMS-QF was achieved by either heating at ~100 °C for several minutes or squeezing using tweezers.

4.4. Oil/Water Separation. A piece of TMS-QF was used as filter and assembled into a separation device as in our previous report. In brief, TMS-QF was sandwiched between two hollow glass cylinders with a clamp. The oil/water mixture was prepared by mixing hexane and deionized water (dyed by xylenol orange) together. For measurement, the oil/water mixture (50 mL, o/w: 4/1) was directly dropped on the device.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsami.5b11341.

Additional results (SEM, contact angle, and XRD). (PDF)

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Notes

The authors declare no competing financial interest.

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