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Research Article

Fabricated of Superhydrophobic Silanized Melamine Sponge with Photochromic Properties for Efficiency Oil/Water Separation

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Superhydrophobic sponge as potential absorbing material for oil/water separation is attracting great attention recently. However, there are still some challenges to feasibly fabricate superhydrophobic sponge with large scale and low cost. Herein, a novel photochromic superhydrophobic melamine sponge (PDMS-SP sponge) is fabricated by facilely dip-coating and thermocuring of hydroxyl-terminated polydimethylsiloxanes mixed with photochromic spiropyran. FT-IR, EDS, and XPS results confirm the successful coating of PDMS-SP upon melamine sponge. The resultant sponge not only possesses excellent water repellency with a contact angle of 154.5° and oil-water separation efficiency with an oil absorption capacity of 48–116 folds of itself weight, but also shows photochromic phenomenon between colorless and purple when it is successively exposed to UV irradiation and visible light.

1. Introduction

Along with the rapid development of urbanization and industrialization, water pollution from oil pollutants and oil leakage is increasingly severe that has caused serious damages to human health and ecological balance. For example, about 4.9 million barrels of oil spill in the Gulf of Mexico in 2010 and cover thousands of square kilometers of sea, which cause a great harm to local marine and aquatic ecosystems [1–5]. Therefore, the effective separation of oil from water is a major concern. Traditional oil separation techniques can be summarized as physical, chemical, and biological methods, such as *in-situ* burning, dispersants, activated carbon [6], cotton fiber [7] exploited for spilled oil. Nevertheless, traditional separation materials have high cost, poor selectivity, and nonrecyclability. Fortunately, the rapid development of interface science provides a more efficient solution to separate oil from mixtures [8].

Superhydrophobic interfaces, including two-dimensional materials [9–15] and three-dimensional porous materials [16–23], can realize effective oil and water separation. The two-dimensional meshes or membranes need to be treated before oil water separation, but the three-dimensional superhydrophobic porous materials are promising solution with low energy. Various advanced 3D porous materials, such as foam, sponge, aerogel, and xerogel materials have been developed and show high selectivity and outstanding absorption capacity toward various oils and organic solvents [24–27]. Pham and Dickerson [28] explore a robust superhydrophobic 3D porous materials by the silanization of commercial melamine (MA) sponge, which reveals a water contact angle of 151.0°, good adsorption ability to a variety of organic solvents and grease, and excellent recyclability. Especially, the polysiloxane backbone exhibits a low surface tension value of around 21 mN/m that is suitable for water repellency [29]. Peng et al. [30] introduce a facile dip-coating/UV-curing method to prepare superhydrophobic and oleophilic melamine sponge, coated polydimethylsiloxane (PDMS) film onto the sponge skeleton through UV-assisted thio–ene click reactions. Dip-coating offers a robust and effective approach in large-scale preparation of a superhydrophobic sponge, which exhibits an absorption capacity of 103–179 folds its own weight. Therefore, silanized sponge is an efficient and economical method to explore three-dimensional oil-water separation materials.

In recent years, some advanced materials can respond to external stimuli (e.g., light, magnetic, thermal, and pH) and are employed in oil/water separation [31–36]. Light energy,

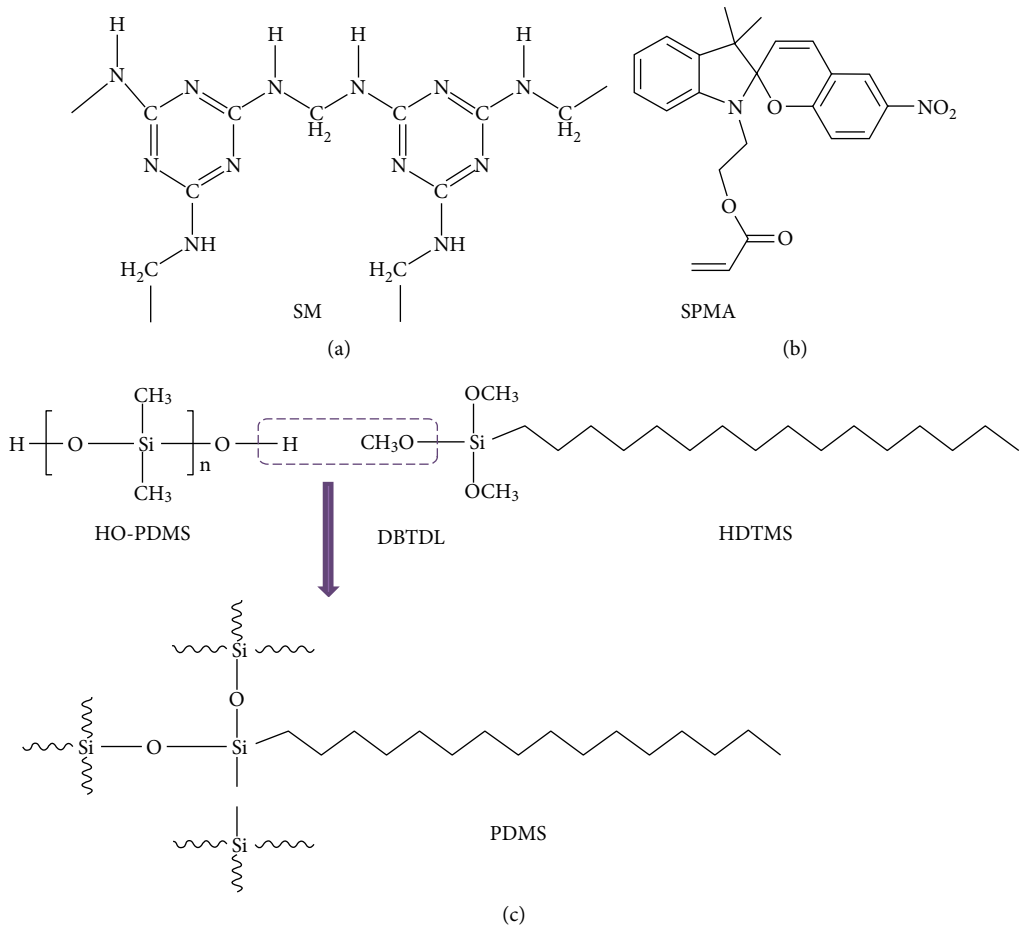

Figure 1: (a) Chemical structure of pristine melamine sponge, (b) chemical structure of SPMA, (c) schematic rout of PDMS thermocuring.

due to clean and convenient, has drawn more and more attention. Spiropyran (SP) is a photosensitive molecule featuring reversible optical-switching between two forms, the colorless ring-closed SP form and the colored ring-opened merocyanine (MC) form [37]. In our group, epoxy resin is thoroughly mixed with an SP derivative to formulate anti-counterfeiting coatings that could be applied to flexible substrates, such as food and medicine packaging [38]. Spiropyran (SP)-containing fluorinated polyacrylate (F-PA-SP) latex is prepared by emulsion polymerization, which is potential to cellulosic paper with outstanding reversible color changes and hydrophobicity [39].

Herein, a novel photochromic superhydrophobic sponge is developed by dip-coating and thermocuring of commercial melamine-formaldehyde (SM) sponge with polydimethylsiloxane (PDMS) mixtures containing spiropyran methacrylate (SPMA) as photochromic motif, and hexadecyltrimethoxysilane as multi cross-linker. After thermocuring, PDMS is covalently covered on the surface of the sponge, and SPMA is restricted in the cured rubber. Compared to other chemical methods of grafting SP to SM, this is a new simple physical way to cover SPMA on the surface of a sponge. By simple UV irradiation, PDMS-SP-coated melamine sponge transforms from colorless to purple. Next, the superhydrophobicity for oil/water separation and photochromic behavior of (PDMS)-coated melamine sponge are studied. In addition, the internal porosity, thermal, and chemical stability of SM endows PDMS-SP-coated melamine sponge with high absorption capacity (116 times) for oil absorption. These findings also exhibit potential applications to indicate the depth of UV curing three-dimensional porous materials.

2. Materials and Methods

2.1. Materials. Melamine sponge was purchased from a commercial store (7.90×10^{-3} g/cm^3). The hydroxyl-terminated polydimethylsiloxane (HO-PDMS) with a viscosity of 5000 MPa·s was provided by Gangzhou Juchen Zhaoye Organic Silicone Co., Ltd. Hexadecyltrimethoxysilane (HDTMS), dibutyltin dilaurate (DBTDL) and cetane were purchased from Aladdin Reagent Co., Ltd. (Shanghai, China). All chemicals used as received without further purification. 1′-(2-Methacryloxyethyl)-3′,3′-dimethyl-6-nitro-spiro(2H-1-benzopyran-2′,2′-indoline) (SPMA) was synthesized according to previous work [39], showed in Figure 1(b).

2.2. Preparation of Superhydrophobic PDMS-SP Melamine Sponge. The dip-coating and thermocuring process were implemented according to the reported method [30] with modification, showed in Figure 1(c). The HO-PDMS 2.5 g,

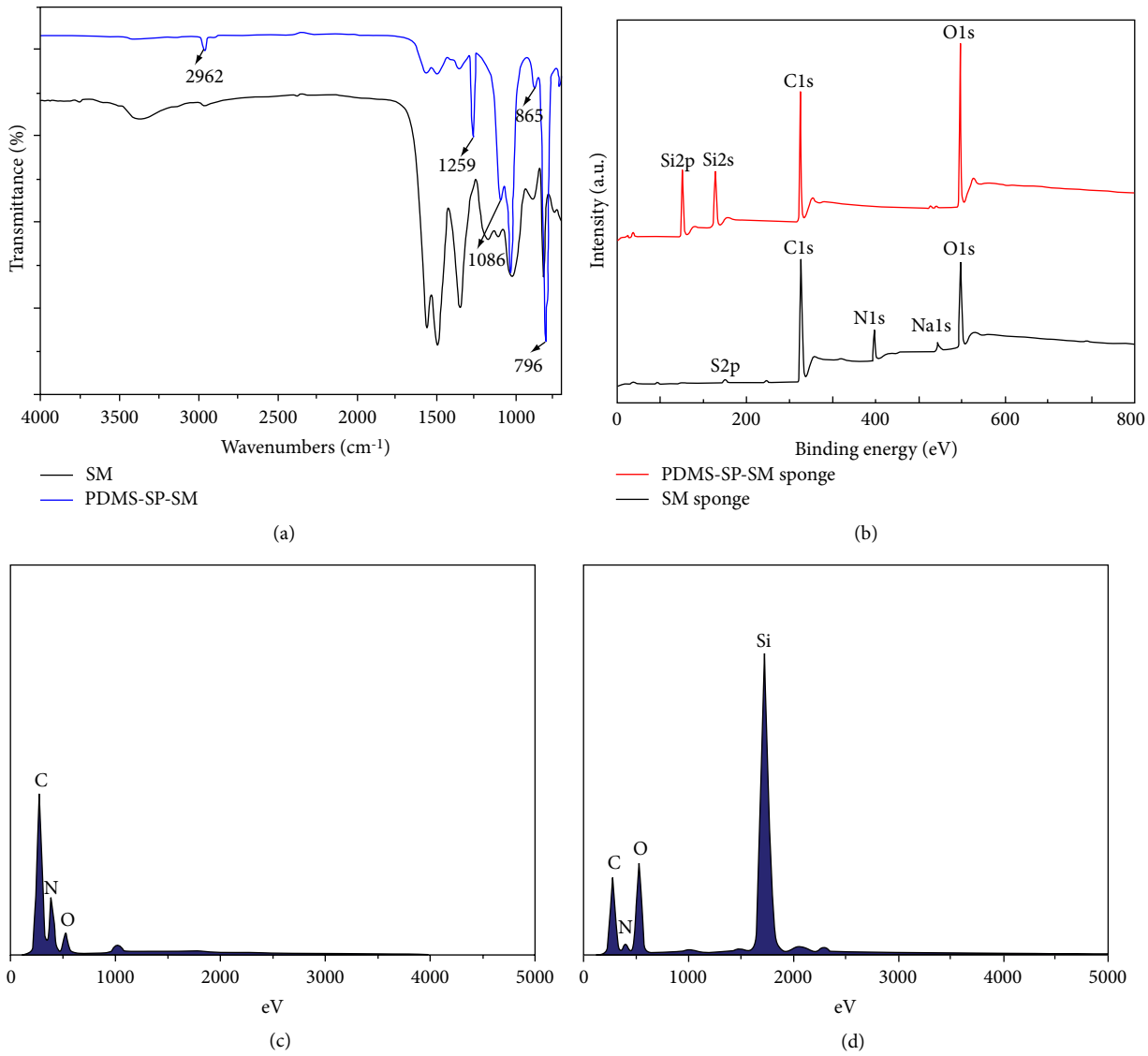

Figure 2: (a) ATR-FTIR spectra of pristine melamine sponge and PDMS-SP modified melamine sponge, (b) XPS spectra of pristine melamine sponge and PDMS-SP modified melamine sponge, (c) EDS spectra of pristine melamine sponge on the sponge framework surface, (d) EDS spectra of PDMS-SP modified melamine sponge.

HDTMS 0.25 g, SPMA 0.008 g, and DBTDL 0.05 g were dissolved in 25 mL CH_2Cl_2. The pristine melamine sponge was cut into $1 \times 1 \times 1\,cm^3$ pieces and were immersed in the above mixture for 5 min. Then removed from the solution and squeezed to extract the absorbed solution, cured at 50°C for 10 min. It was then repeatedly washed with CH_2Cl_2 using the sorption/squeezing process for the removal of any untreated starting materials. Finally, it was dried in air for 6 h, The PDMS-SP-coated melamine sponge obtained.

2.3. Oil Absorption Experiments. The absorption capacities of PDMS-SP-coated sponge for various oils and organic solvents were determined by dipping a piece of PDMS-SP-coated sponge into the liquid (oil or organic solvent) until the sponge was saturated with the liquid and left to drip for 30 s for weighting. Repeated sorption/squeezing processes were used to evaluate recyclability of PDMS-SP-coated sponge.

2.4. Characterization. Attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectra were collected 4000 and 500 cm^{-1} on a Nicolet iS50 spectrometer with a 4 cm^{-1} resolution over 32 scans. The micromorphology was observed by SEM on an SU-8010 (Hitachi Ltd., Japan) field emission electron microscope at an accelerating voltage of 5 kV. Samples were coated with a thin gold layer before SEM analysis. An energy-dispersive X-ray spectrometer fitted to the scanning electron microscope was used for chemical elemental identification. The wetting properties of PDMS-coated sponge were investigated by static contact angle measurements at room temperature using an OCA 15Pro contact angle goniometer with a droplet volume of 5 μL and each contact angle was average of 5 different positions for each sample. X-ray photoelectron spectroscopy (XPS) measurement was performed on a Kratos ESCA spectrometer (Axis Ultra DLD) with an Al Kα X source (150 W, 15 kV) at a take-off angle of 45° from the normal surface.

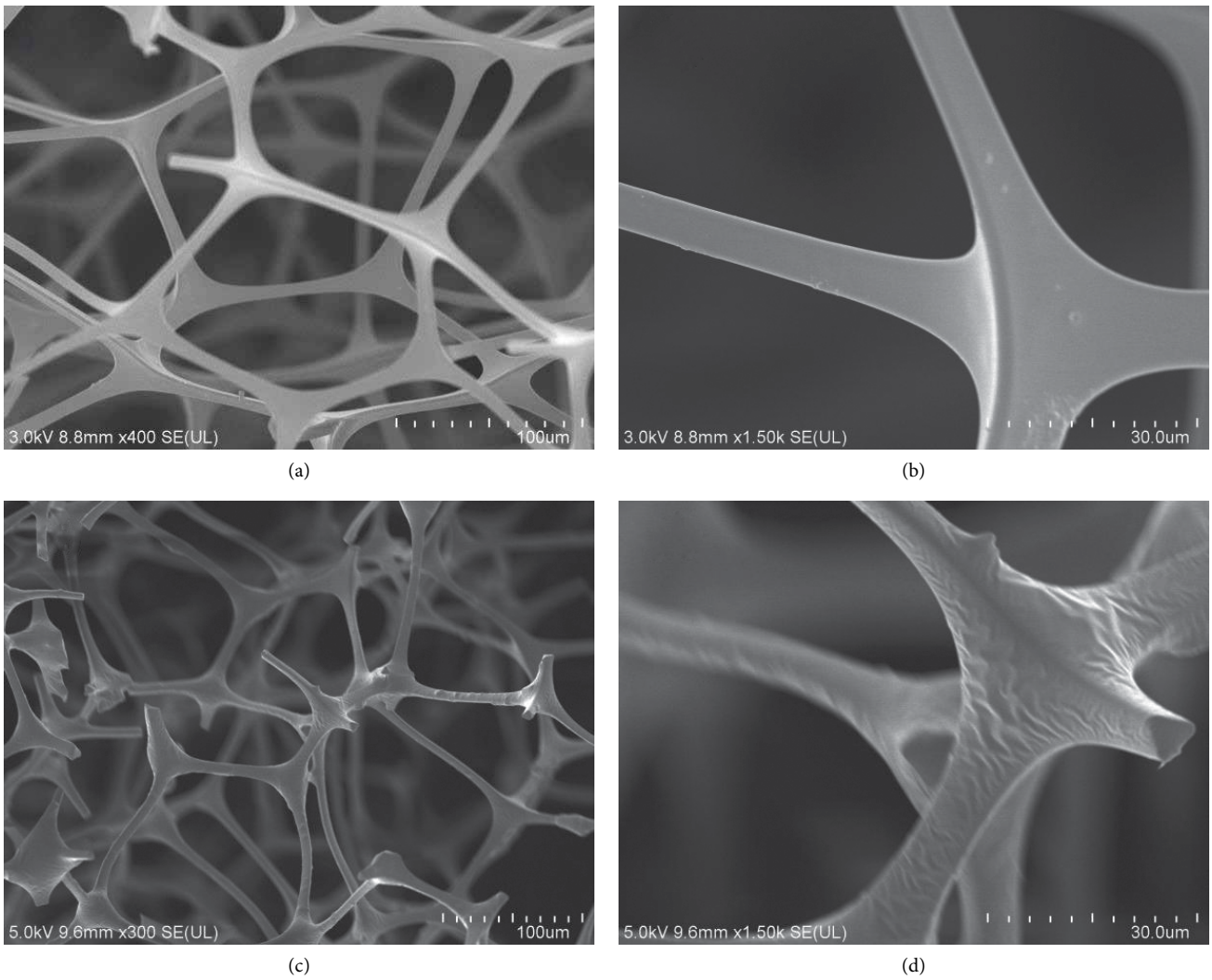

Figure 3: (a, b) SEM images of a pristine melamine sponge and (c, d) PDMS-SP modified melamine sponge.

3. Results and Discussion

3.1. Characterizations of PDMS-SP Melamine Sponge. Figure 2(a) shows the ATR-FTIR spectra of untreated SM melamine sponge and PDMS-SP-coated melamine sponge. The spectrum of SM displayed prominent peaks at 811, 1163, 1546, and 3383 cm^{-1} that are assigned to triazine ring bending, C–O stretching, C=N stretching, and N–H (of the secondary amine) stretching, respectively. Peaks centered at 1343 cm^{-1} and 1483 cm^{-1} are indicative of C–H bending. After dip-coating with PDMS, the spectrum of PDMS-SP-coated melamine sponge show obviously absorption band near 1259 cm^{-1} is attributed to symmetric deformation of the $-CH_3$ group in $-Si(CH_3)_2$ of HO-PDMS, and the bands located at 865 $cm^{-1}$and 796 $cm^{-1}$are assigned to Si–C and Si–O vibration, 2962 cm^{-1} and 1086 cm^{-1} are ascribed to stretching vibrations of $-CH_3$, and Si–O–Si groups in the HO-PDMS [40] respectively.

Figure 2(b) shows the XPS curves of untreated melamine sponge and PDMS-SP modified melamine sponge. The spectrum in Figure 1(b) SM sponge indicates five elements, including C, N, O, S, and Na which is consistent with the composition of commercial sponge [28]. Both samples exhibit a C1s peak at 284.8 eV and an O1s peak at 532.3 eV. Moreover, Si2s and Si2p peaks were found at 165.6 eV and 102.4 eV, respectively. This indicates that PDMS-SP was successfully coated to sponge frame by dipping-coating. The N1s peak located at 398.3 eV was detected on pristine melamine surface and disappeared in PDMS-SP-modified sponge spectrum. This is because that the small amount of nitrogen species is completely covered by PDMS-SP layer. As shown in Figure 2(c), three elements C, N, and O are detected by EDS in pristine melamine sponge. As expected, silicon in PDMS-SP modified melamine sponge is shown in EDS spectrum of Figure 2(d). Togethering with XPS results, it demonstrates the coating of silicone chains on melamine sponge surface.

The morphologies of melamine sponge before and after PDMS-SP-coating are examined by SEM and shown in Figure 3. The melamine sponge comprises a three-dimensional, elasticity, porous structure with pore sizes in the range of 100–150 μm (Figure 3(a)). The skeletons of the melamine sponge are smooth with an average diameter of ~10 μm (Figure 3(b)). Compared to the pristine melamine sponge, PDMS-SP-coated melamine sponge have significant changed morphologies. The porous structure of sponge is not destroyed

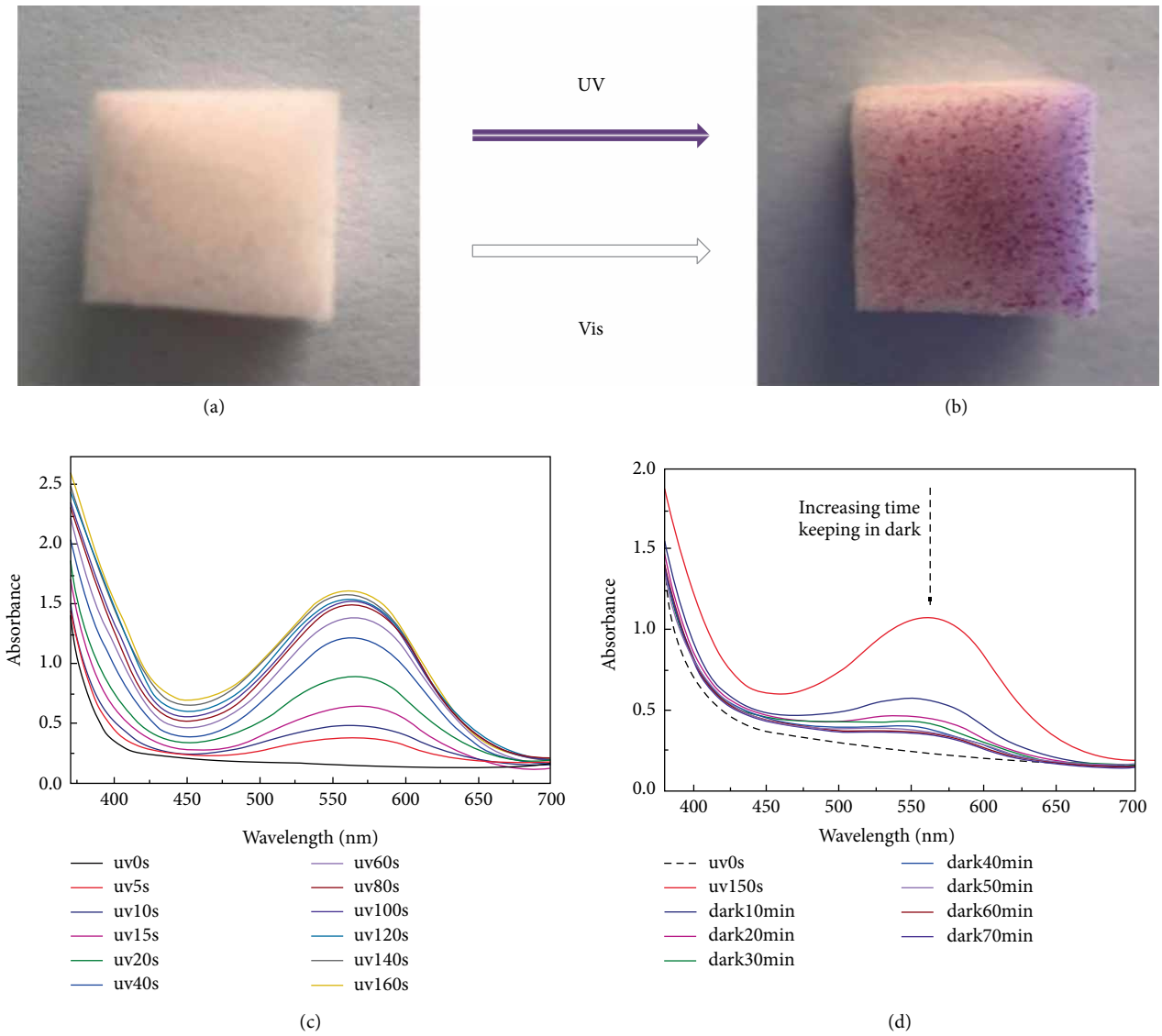

Figure 4: (a, b) Photochromic coloration of PDMS-SP modified melamine sponge under UV irradiation, (c) absorbance curves of cross-linked PDMS/SPMA after UV irradiation, (d) absorbance changes with exposure time under UV/vis irradiation.

during the dipping-coating process. However, it is obviously observed that the 3D skeleton of sponge became rough after dipping-coating and regularly arranged resembling strips are observed throughout the whole sponge skeleton (Figures 3(c) and 3(d)). Curing-induced shrinkage and migration of silicone result in undulating wrinkles with an average distance between these strips less than 3 μm, and the width of these strips is about 1 μm. These results indicate that PDMS-SP solution can be adhered to the surface of sponge skeleton fibers uniformly by thermocuring.

3.2. PDMS-SP Melamine Sponge with Photochromic Properties. As reported, spiropyran is a photosensitive molecule featuring reversible optical-switching between two forms [30]. As shown in Figures 4(a) and 4(b), PDMS-SP-coated melamine sponge transforms from colorless to purple upon exposure to UV irradiation ($\lambda = 365$ nm), while it can reversibly change from purple to colorless as irradiated with visible light. Before UV irradiation, no obvious absorbance of PDMS-SP sample in the range of 450–700 nm. Figure 4(c) illustrates the absorbance curves of the samples upon exposure to UV irradiation for 0 and 160 s. A distinct absorption peak appears at 555 nm due to the transformation of SP into MC form, the observed absorption intensity increases markedly with exposure time. When the purple sample is immediately irradiated with a fluorescent lamp, the absorption peak at 555 nm clearly decreases with the irradiation time and the latex color fades (Figure 4(d)).

3.3. Superwetting and Oil/water Separation of Sponge. The pristine melamine sponge exhibits amphiphilic properties with water contact angles (WCA) and an oil contact angles (OCA) of hexadecane closed to 0°, while PDMS-SP coated sponge was hydrophobic and oleophilic (Figure 5). The difference in PDMS-SP coated sponge is reflected in its wetting property as

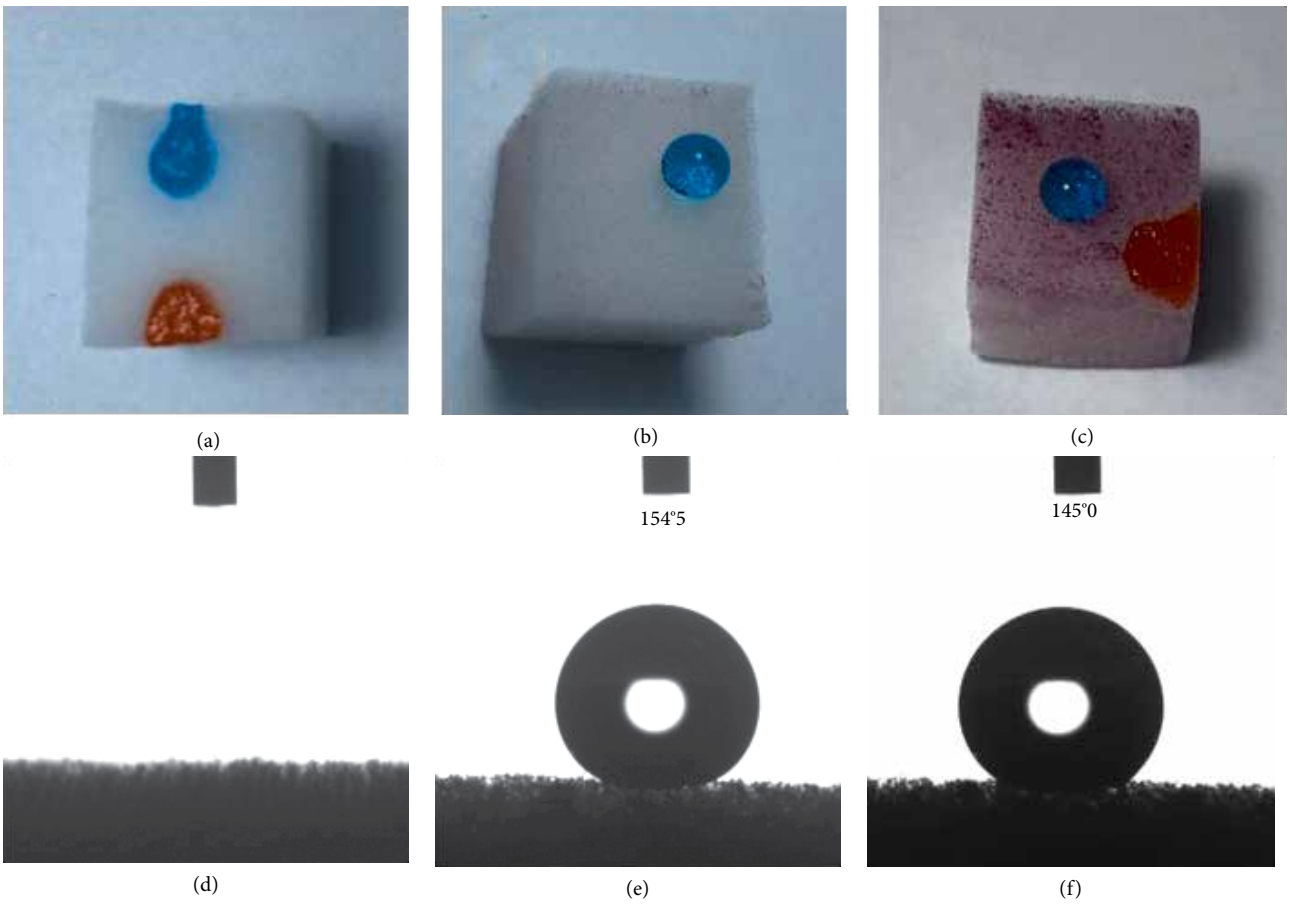

Figure 5: Superwetting of PDMS-coated sponge. (a) Amphiphilic property of pristine melamine sponge. (b) Superhydrophobic of PDMS-SP coated melamine sponge. (c) Hydrophobic and oleophilic property of PDMS-SP coated melamine sponge after UV irradiation. Contact angles of (d) pristine melamine sponge, (e) PDMS-SP coated melamine sponge, and (f) PDMS-SP coated melamine sponge after UV irradiation.

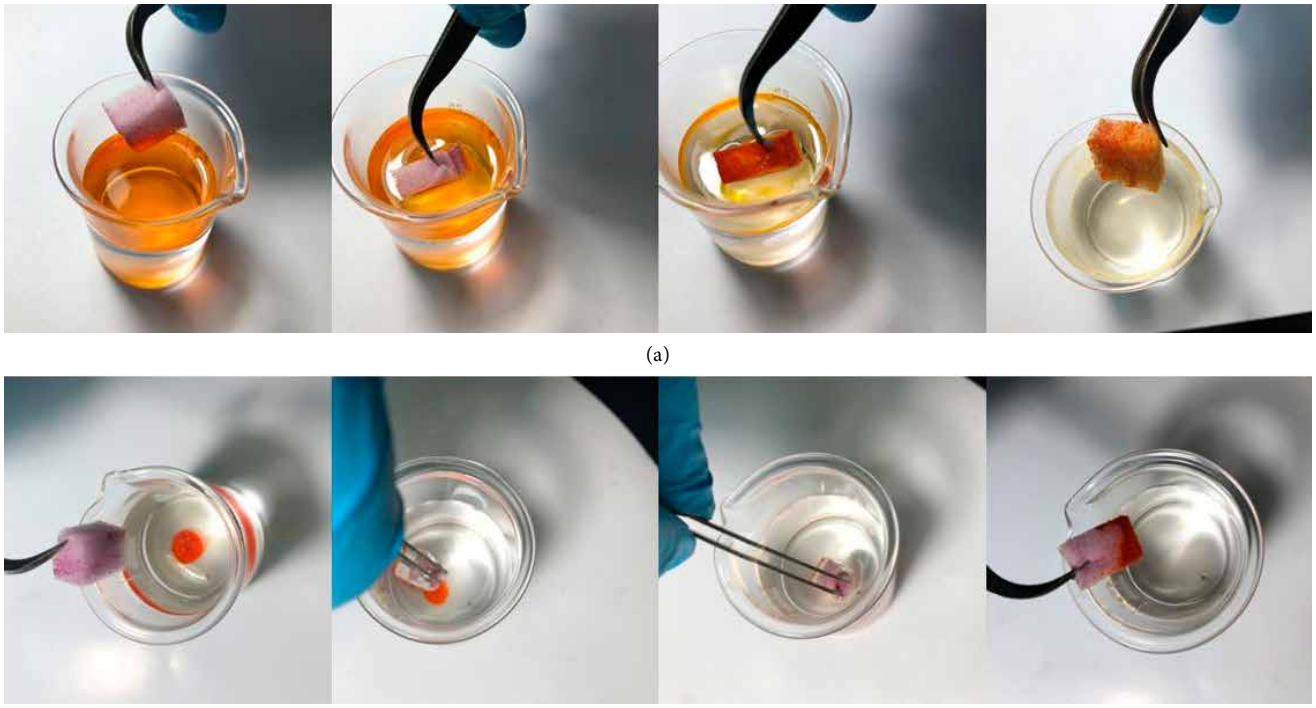

Figure 6: Photographs of the selective sorption of oil with the PDMS-SP coated melamine sponge. The oil was dyed with Sudan I. (a) Hexadecane as oil. (b) Chloroform as oil.

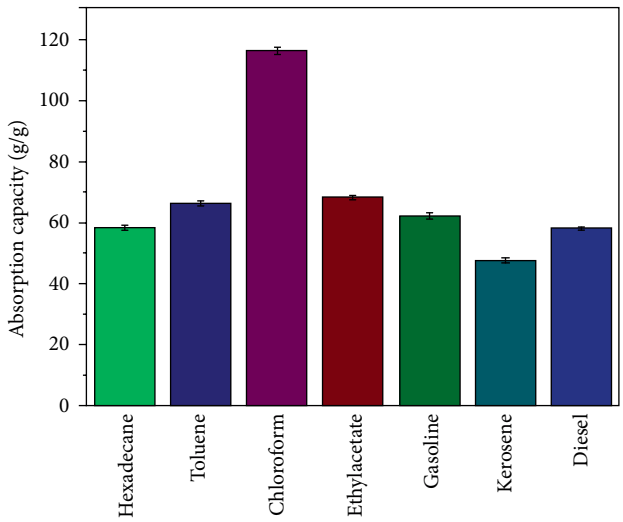

Figure 7: Absorption capacities of the PDMS-SP sponge for different organic solvents and oils.

contacted with water. To further demonstrate the wettability of the raw sponge, water contact angle (CA) is performed by placing droplet of deionized water on PDMS-SP surface. As shown in Figure 5(e), the WCA of PDMS-SP sponge notably increased from 0° for the raw SM sponge to 154.5 ± 1.1°. After UV irradiation, not only the color of sponge changed, but also WCA has a small amount of reducing to 145.0 ± 1.8° showed in Figure 5(f), OCA was still about 0°.

The superhydrophobic surface and porous structure make PDMS-SP coated melamine sponge a viable material for the rapid removal of various organic solvents and oils from water. Selective sorption is a crucial property of oil/water separation materials. The high selective sorption capability of PDMS-SP sponge is showed in Figure 6, both oil float hexadecane (dyed with Sudan I) and sank chloroform (dyed with Sudan I) are removed quickly and completely when the purple color PDMS-SP coated melamine sponge was in contact with oils, then store it in the porous of sponge. The PDMS-SP coated melamine sponge has different sorption performances with different oils and organic solvents. In this test, some common pollutants in industry, such as gasoline, diesel, and toluene etc., are collected.

Figure 7 shows the maximum sorption capacity for oils and solvents in oil/water mixtures, it exhibits excellent absorption capacity in the range 48–116 times of itself weight. The excellent oil/solvent separation capacity is attributed to the highly porous structure as well as the oleophilic properties. In addition to oil absorption, PDMS-SP coated sponge also has photochromic properties. It is well-known that UV curing of three-dimensional porous structure materials is a confusing problem, and the chromic properties can provide intuitive tracking and visualized proof for the research of curing depth.

4. Conclusions

In summary, a superhydrophobic PDMS-SP coated melamine sponge is fabricated with a facile method of dip-coating and thermocuring. The developed PDMS-SP sponge not only absorbs various oil in oil/water mixture with high absorption capacity and selectively, but also changes its color from colorless to purple upon exposure to UV irradiation. These results suggest that the superhydrophobic PDMS-SP sponge may not only provide great potential application for oil recovery, but it also shows potential applications in construction of UV curing three-dimensional porous materials.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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