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Emulsion-templated, polymerization-free, ultralight sponges for oil spills reclamation

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ABSTRACT

We report the fabrication of monolithic, macroporous, hydrophobic polymer sponges from a water-in-oil (w/o) high internal phase emulsion (HIPE) without using polymerization process. They were formed from sulfonated polystyrene-*block*-poly(ethylene-*ran*-butylene)-*block*-polystyrene (SSEBS) and tetra-functional poly(amidoamine) (PAMAM) dendrimers via ionic interaction within a W/O HIPE. The sponges obtained possessed typical emulsion-templated interconnected macroporous structures, with void size ranging from 1 to 20 μm and a density of 0.08–0.1 g mL^{-1}. The sponges were hydrophobic, with a water contact angle of around 128.1°, enabling highly selective absorption of oil from oil-water mixture with an absorption capacity from 15.4 to 25.8 g g^{-1} for organic solvents and oils. The sponges were recyclable and their absorption capacity was preserved at around 85% after being reused for 30 times. The high selectivity, absorption capacity and reusability enabled these sponges to be an excellent candidate in the reclamation of oils from wastewater and oil leakage.

Keywords: Macroporous materials; Polymer sponges; High internal phase emulsion; Oil spills reclamation

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1. Introduction

Oil spill and organic pollutants discharge become increasingly severe, and their threats to environment and human health are also growing more seriously [1–4]. For example, the Gulf of Mexico oil-spill in 2010 led to an economic and environmental tragedy [5]. Various ways has been attempted to solve the issues, including in-situ burning, mechanical method, biodegradation, solvent extraction and absorption and so on [6–8]. Among those methods, absorption is promising, because it does not generate secondary pollution and the spilled oils can be recovered [9]. Hydrophobic polymers with macroporous structures and low density are one of the most promising absorbent, considering the relatively high viscosity of spilled oils [10–12].

Emulsion-templated macroporous polymers, conventionally known as polyHIPEs, have drawn considerable attention due to their relatively high selectivity, rapid absorption and

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high absorption capacity [13]. PolyHIPEs are macroporous polymers polymerized within high internal phase emulsions (HIPEs), emulsions with the total volume fraction of the dispersed internal phase exceeding 74% [14]. PolyHIPEs are usually prepared by polymerizing the continuous phase of HIPEs to solidify the microstructures of emulsion followed by removing the residue in the continuous phase and the dispersed phase [15,16].

Recently, we developed a novel type of emulsion-templated porous polymers (HIPE xerogels) from polymer organogel-stabilized HIPEs [17]. The polymer organogels were formed from a triblock ionomer, sulfonated polystyrene-*block*-poly(ethylene-*ran*-butylene)-*block*-polystyrene (SSEBS) and oppositely-charged components through ionic interaction-induced assembly [18]. These obtained HIPE xerogels showed excellent properties for oil-water separation, but they were fragile as indicated by the loss of the emulsion-templated structures and monolithic structure. These xerogels tend to break into small pieces during oil absorption, making them hard to be collected. Magnetic nanoparticles were incorporated to HIPEs and thus magnetic composites were formed [19]. Although magnetic composites in small pieces can be collected with a magnet, the fragility has not been improved yet.

Herein, we report the fabrication of monolithic, hydrophobic, emulsion-templated sponges from SSEBS and tetra-functional poly(amidoamine) (PAMAM) dendrimers with one-step process, a simple and feasible approach. The monolithic shape and the presence of the typical emulsion-templated macroporous structures reflected the relatively high strength of the sponges. The sponges exhibited high selectivity to oil, relatively high absorption and high recyclability, making them an excellent candidate for oil reclamation from oil-water mixture.

2. Experimental setup

2.1. Materials

Ethylenediamine (EDA), methyl acrylate (MA), acetic anhydride, polystyrene-*block*-poly(ethylene-*ran*-butylene)-*block*-polystyrene (SEBS) (29 mol% polystyrene blocks), concentrated sulfuric acid (96%), isopropanol, methanol, toluene, dimethylbenzene, *n*-hexane, cyclohexane, 1,2-dichloroethane (DCE), trichloroethane, ethyl acetate (analytical reagents) and engine oil were all purchased from Sigmal-Aldrich and used as received. Crude oil was purchased from PetroChina Southwest Oil and Gas Field Company (Chengdu, China).

2.2. Preparation of SSEBS

SSEBS was prepared according to a reported approach [20–23], and the route is shown in Fig. 1. A typical procedure for SSEBS preparation was briefly described as follows. SEBS was dissolved in a mixture of DCE (60 mL) and cyclohexane (10 mL) at 50°C under nitrogen atmosphere, and then fresh sulfonating agent obtained by mixing concentrated sulfuric acid and acetic anhydride in DCE at 0°C was added. After 5 h, the reaction was stopped by the addition of isopropanol. Solvents were removed through evaporation and extra acid was removed by washing with deionized water. The obtained SSEBS was finally dried at 60°C in a vacuum oven and its sulfonation degree was determined to be 28 mol% according to acid-base titration [21,24].

2.3. Synthesis of PAMAM dendrimers

PAMAM dendrimers were prepared according to a method reported previously [25,26], and the route was shown in Fig. 2. Typically, EDA (1.5 g, 25 mmol) was dissolved in methanol (15 mL) under stirring at about 0°C, and then MA (21.5 g, 250 mmol) was added to the EDA solution dropwise. After the addition of MA, the reaction was conducted at 25°C for 24 h, and then methanol and unreacted MA was removed to obtain an oil. The oil (8.1 g) was dissolved in methanol (25 mL) at around 0°C, and then EDA (12.1 g, 200 mmol) was added dropwise. After reaction for 24 h, the solvents and extra EDA were removed to obtain tetra-functional PAMAM dendrimers.

2.4. Preparation of HIPE organogels

PAMAM dendrimers were dissolved in deionized water to obtain a 4% aqueous solution, and SSEBS was dissolved in a mixture of toluene and methanol (98:2) to obtain a 5% solution. Organogels with different amount of PAMAM dendrimers and SSEBS (n_{amine}: $n_{\text{sulfonate group}}$ = 1.2:1, 1.5:1, 1:1, or 0.8:1) were synthesized, which can be used to optimize the preparation of organogels to obtain better comprehensive properties sponges. Typically, SSEBS solution (1 mL) was added to the PAMAM dendrimers solution (126 μL)

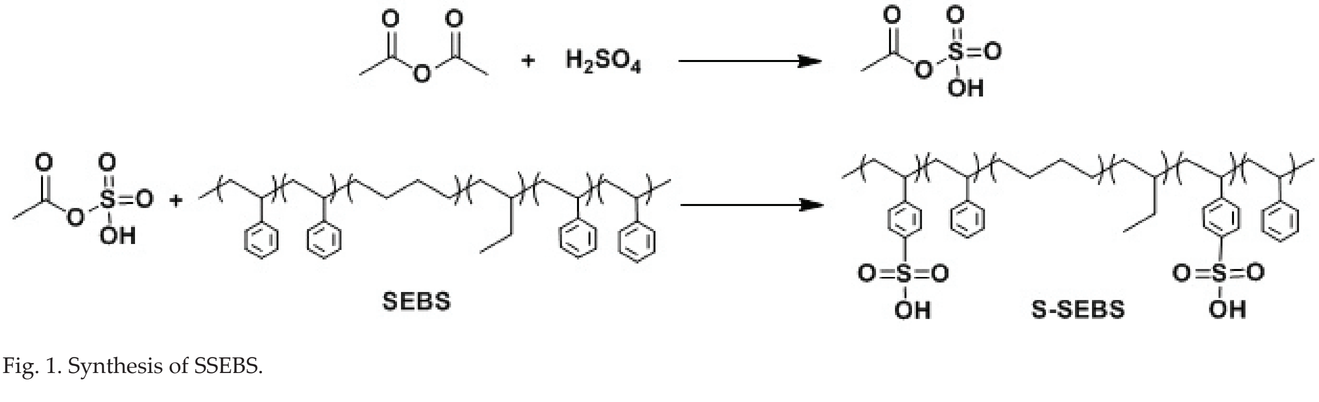

Fig. 1. Synthesis of SSEBS.

(n_{amine}: $n_{sulfonate\ group}$ = 1:1) and mixed via a XH-C Vorter mixer. After 12 h, deionized water (4 mL) were added and then the tube was vibrated to obtain HIPEs. The HIPEs did not flow in an invert tube, indicative of the formation of HIPE gels.

2.5. Preparation of PolyHIPE sponges

HIPE organogels were frozen at −20°C for about 2 h and then were transferred into a frozen-dryer (FD-1, Shanghai Chubai Instrument Co., Ltd., China) at −50°C. After freeze-drying, porous PolyHIPE sponges was obtained. The formation mechanism of the PolyHIPEs is shown in Fig. 3.

2.6. Characterization

2.6.1. Rheological properties

The formation of the as-obtained HIPEs was confirmed by rheological measurements (HAAKE, MARS III, Thermo Fisher Scientific Company, China). Appropriate amount of an HIPE with a beaker were put on the platform of the rheometer. The shear strain was obtained after keeping 30 min at 25°C. Then, 1% strain was a fixed value, and a new sample was measured under the same conditions. Storage modulus and loss modulus of gels were acquired.

2.6.2. Optical microscopy (OM)

HIPEs were evenly coated on a glass slide and then covered with a cover glass. The microstructure of the HIPEs was observed under an optical microscope (XJG-05, Sunny optical technology (group) co., LTD, China).

2.6.3. Fourier transform infrared (FT-IR) spectroscopy

The chemical composition of the SSEBS, PAMAM dendrimers and sponges were verified by FT-IR spectrometer (NICOLET 6700, Thermo Fisher Scientific Company, America) in the range of 4000–500 cm^{-1}. PAMAM dendrimers were uniformly coated on the surface of as-prepared KBr disk and tested; SSEBS with THF solution were dropped on the surface of as-prepared KBr disk and tested after the evaporation of THF at ambient temperature; the sponge was ground with KBr and pressed into discs for analysis.

2.6.4. Scanning electron microscopy (SEM)

The porous morphology of the sponges was observed by SEM (Zeiss EVO MA 15, Germany) at 20 kV, and the fracture surface was obtained from cryogenic fracture coated with a thin gold layer.

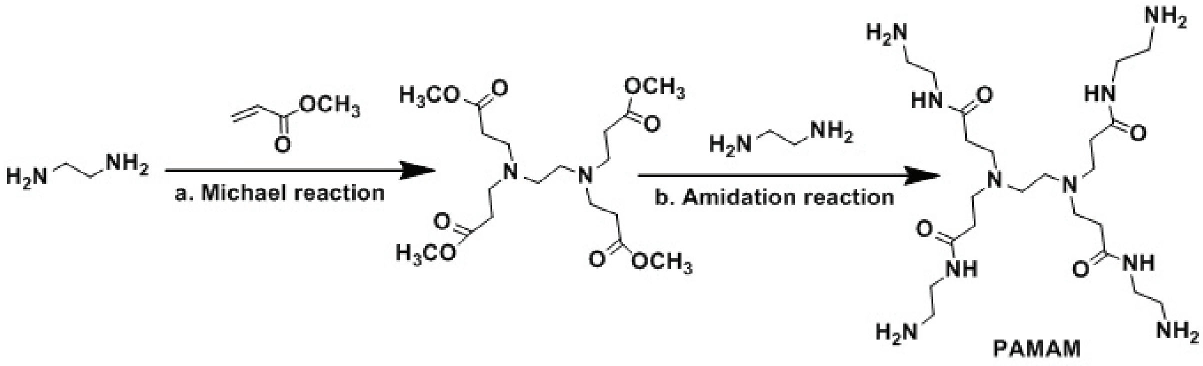

Fig. 2. Synthesis of PAMAM dendrimers.

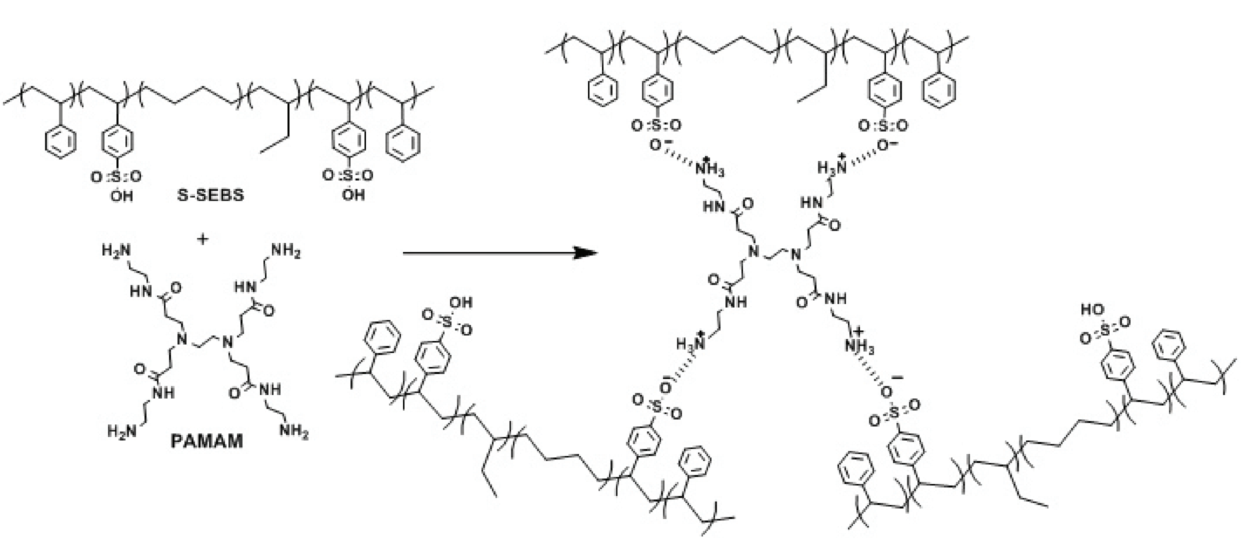

Fig. 3. Formation mechanism of the PolyHIPEs.

2.6.5. Contact angle measurement

The hydrophobicity of the sponges was evaluated by contact angle measurement (JGW-360A, Chengde, China), and about 10 μL of water was dropped on the fracture surface of the sponges.

2.6.6. Oil absorption

The method for the evaluation of the oil absorption (*Q*) has been reported in recent years [27]. A certain amount of dried sample (m_0) was put into a vial with oil or organic solvents such as toluene, dimethylbenzene, *n*-hexane, cyclohexane, 1,2-dichloroethane, trichloroethane, ethyl acetate and engine oil. The weight change of the sponges was recorded every 5 min until it reached new equilibrium. The sponges were taken out to remove the liquid on the surface and then weighed (m_1). The oil absorption capacity was calculated by the following equation:

$$Q = (m_1 - m_0)/m_0 \quad (1)$$

where *Q* is the absorption capacity of the sponges (g g^{-1}), m_0 is the initial weight of the sponges (g); m_1 is the weight of the sponges after absorption of oil (g). The *Q* measurement for very sample was repeated for three times to enhance the accuracy of the results. The reported results were from the average values of three measurements.

2.6.7. The oil absorption rate

The oil absorption rate of sponges was determined according to the time mentioned earlier.

2.6.8. Oil-water separation

Oil-water separation of the sponges was evaluated by absorption of oil from oil-water mixture. Oil-water mixture was formed by adding *n*-hexane (dyed with oily red) into water.

2.6.9. Reusability

A certain amount of sponge was immersed in oil for about 10 min, and then the sponge was weighted. After evaporation of oils within the sponge, the sponge was used to absorb oil again.

3. Results and discussion

Studies to obtain the sponges with excellent comprehensive properties were conducted by controlling the mole ratio of the monomers displayed in Table 1. It can be concluded that the optimal molar ratio of amine and sulfonate group is 1:1, by investigating the hydrophobicity and absorption capacity in different organic solvents. So the sponges prepared under the optimum conditions were thoroughly researched on the structures and relevant properties.

Oscillatory shear tests were adopted to perform the linear viscoelastic properties of the HIPE gels. From the results of oscillatory strain of gels displayed in Fig. 4(a), it can be observed that the gels remain stable due to the linear behavior of the dynamic modulus. When oscillatory strain is over a critical value (ca. 10%), the structure of HIPE gels is destroyed from a reflection of a non-linear behavior of the dynamic modulus. Fig. 4(b) demonstrates that storage modulus G' is always higher than the corresponding loss modulus G''. Moreover, G' and G'' appear independent and they are parallel to each other. These results demonstrate that the 3D network is formed well and a huge benefit of this structure is that it can be used to deal with oil and organic solvents contamination [20].

The as-obtained HIPEs exhibit high viscosity, which prevents flow within an inverted tube (Fig. 5(a)). The morphologies of the HIPE gels from optical microscopy (Fig. 5(b)) are of typical HIPEs with polyhedral structures. The sizes of emulsion droplets range from 1 to 20 μm.

The FT-IR spectra of PAMAM, SSEBS and HIPE sponges (SSEBS/PAMAM) are shown in Fig. 6. The remarkable absorption peaks at about 3,440 cm^{-1} can be ascribed to the stretching vibration absorption of –NH. 2,920 and 2,850 cm^{-1} are the stretching vibration absorption of –CH. The peaks at 1,645, 1,560, 1,500 and 1,450 cm^{-1} are the characteristic peaks for benzene moiety. The remarkable absorption peaks at 1,100 and 700 cm^{-1} can be ascribed to the stretching vibration of O=S=O and –S–O, respectively. It is noteworthy that the differences of raw materials and HIPE sponges are as follows. The symmetric stretching vibration at 1,100 cm^{-1} changes into a doublet at about 1,200 cm^{-1}, which can be verified to the asymmetric vibration of the $–SO_3^-$ [21]. Besides, the appearance of the association peak of –N–H at 3,080 cm^{-1} and the characteristic absorption peak at 1,030 cm^{-1} indicates that ionic crosslinking is formed between SSEBS and PAMAM dendrimers.

The morphologies of sponges were observed with SEM under different magnifucation. And the local enlarged of morphology of sponges can be more clearly presented in

Table 1
The performance comparison of water contact angle and absorption capacity in different organic solvents with various molar ratio of amine and sulfonate group

	Molar ratio	Water contact angle/°	Absorption capacity/g g^{-1}		
			Crude oil	Chloroform	*n*-Hexane
n_{amine}: $n_{\text{sulfonate group}}$	1.5:1.0	116.4	12.8	14.7	25.4
	1.2:1.0	120.8	13.1	21.5	24.8
	1.0:1.0	128.1	14.4	25.8	23.1
	0.8:1.0	94.2	13.7	12.5	18.7

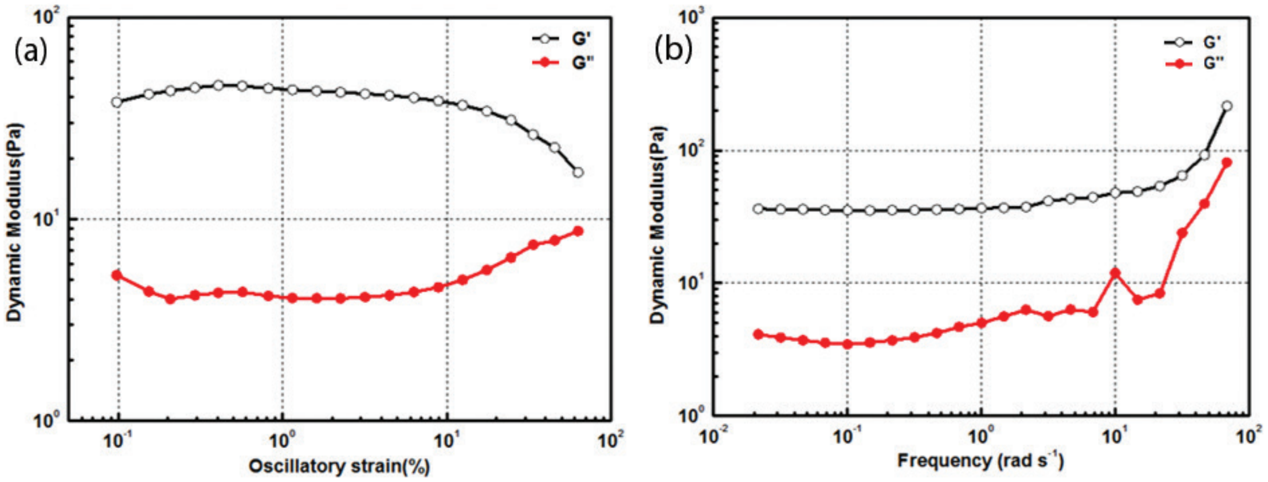

Fig. 4. G' and G" as a function of (a) oscillatory strain and (b) oscillatory frequency of PolyHIPEs gels.

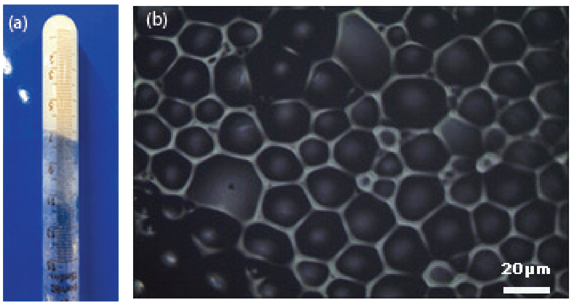

Fig. 5. Images of HIPE gels (a) and their optical micrograph (b).

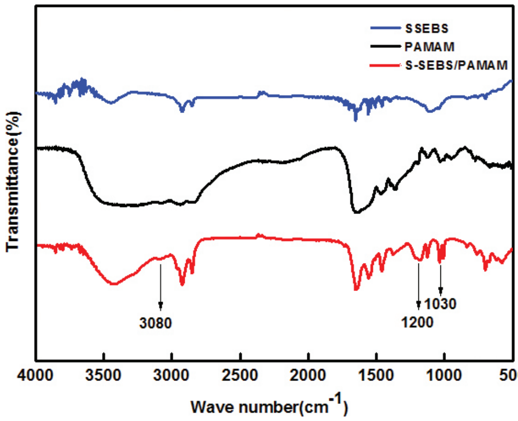

Fig. 6. FT-IR spectra of PAMAM dendrimers, SSEBS and sponges.

Fig. 7b, compared with morphology in Fig. 7a. Interconnected macroporous structures are formed within the sponges and the voids are evenly distributed. The diameter of the voids ranges from 1 to 20 μm, similar to the results observed with OM, indicating that the voids are well-preserved during freeze-drying process. In addition, the volume density (ρ_s) and porosity (P_s) of the sponge were calculated via the following equations [28]:

$$\rho_s = m_s/v_s \tag{2}$$

$$P_s = (1-\rho_s/\rho_0) \times 100\% \tag{3}$$

where m_s denotes the masses of the sample, v_s denotes the volume of the sample, and ρ_0 denotes the density of the polystyrene-block-poly(ethylene-ran-butylene) -block-polystyrene (0.92 g cm^{-3}). Based on the rough calculations, the volume density is close to 0.10 g cm^{-3}, and porosity can be calculated to be 90%.

The as-obtained sponges are hydrophobic, which can be indicated by putting them in water. The sponges floated on the water surface, and they did not absorb water for one day. Besides, when they were immersed into water, bubbles were rising in the meantime (Fig. 8(a)), indicating that the sponges is hydrophobic. Hydrophobicity of the sponges was quantitatively studied through contact angle (CA) measurement, and the sponge exhibits a high water contact angle of 128.1° (Fig. 8(b)).

The absorption of sponge was conducted by using different types of organic solvents and oils. The absorption capacities of the sponges were shown in Fig. 9, and they vary from 15 to 26 g g^{-1} which is higher than other reported HIPE xerogels [29–31]. For example, Lee et al. [30] fabricated 12-Hydroxystearic acid xerogels with lower absorption capacity which increased in weight by 459 ± 33 wt% in diesel and 583 ± 42 wt% in gasoline. Choi et al. [31] presented polydimethylsiloxane sponge with the absorption capacities in the range from 400 to 1,100 wt% for various oils and organic solvents. Especially, based on the principle of the dissolution in the similar material structure, the absorption of *n*-hexane is higher than the solvents less dense than water which is attributed to the alkyl segments in the SSEBS. Because of the higher density of dichloromethane and chloroform, the saturated absorptions are higher (23.4 and 25.8 g g^{-1}). As the viscosity and molecular weight of crude oil are relatively larger, the absorption capacity is lower than the previous several solvents. The above results indicate that the absorption capacity of the sponges is not only related to the structure of themselves, but also to the structure, molecular weight and viscosity of the adsorbed solvents.

The absorption rate of sponges on the organic solvents and oils were investigated by the determination of the

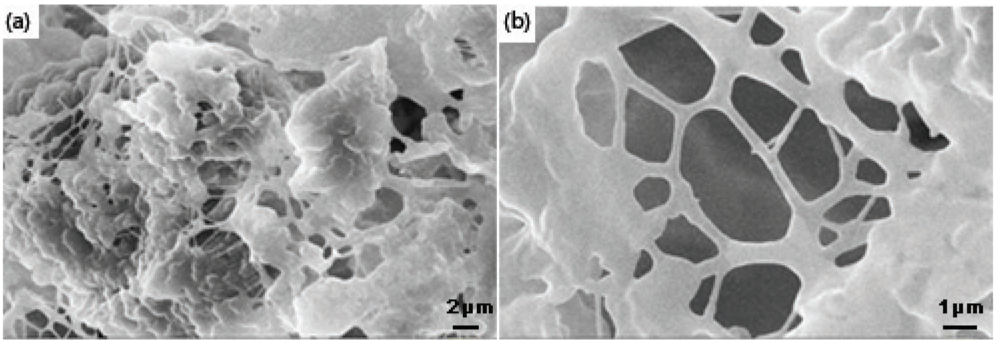

Fig. 7. SEM micrographs of sponges under different magnification (a) Mag = 5.00 K X, (b) Mag = 15.00 K X.

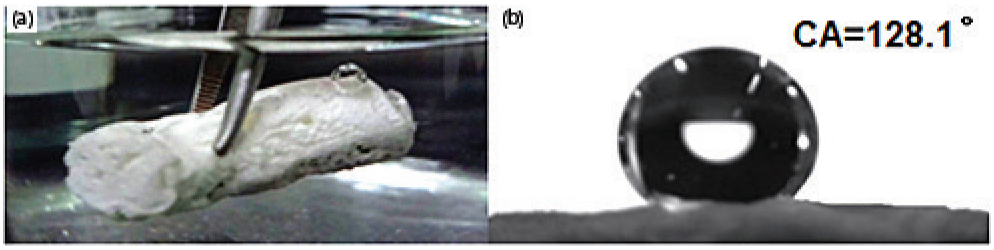

Fig. 8. Infliction photograph underwater (a) and CA image (b) of sponges.

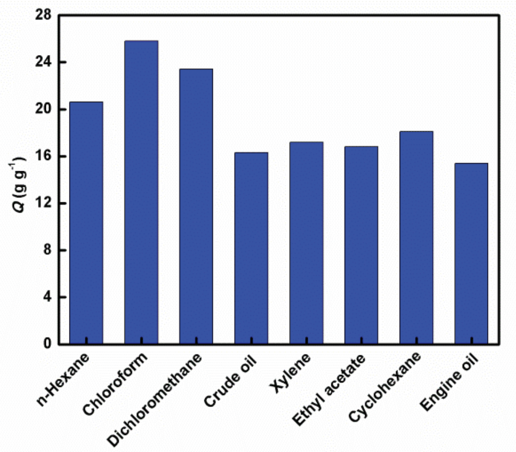

Fig. 9. Absorption capacities of sponges to different organic solvents and oils.

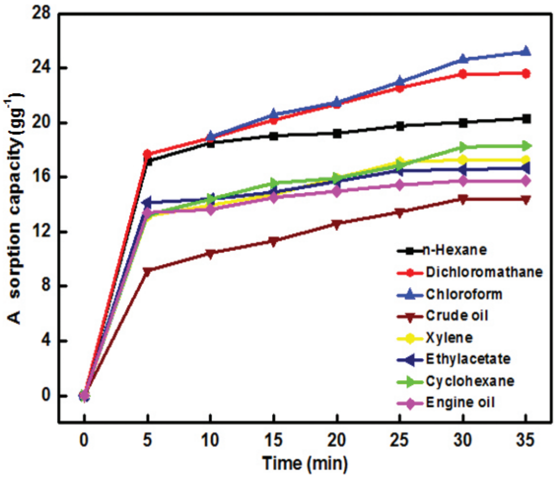

Fig. 10. Absorption rate of the sponges to different organic solvents and oils.

saturated oil absorption. The results in Fig. 10 indicate that saturated time of *n*-hexane and energy oil are fast due to the principle of the dissolution in the similar material structure of them and the large viscidity and molecular weight, respectively. The absorption of various solvents is saturated in about 25 min because of the fact that sponges possess porous structure and rough surface. Fast oil absorption rates make them own a good market prospects in handle the problem of the oil leakage.

It can be seen from Fig. 11 that organic solvents are completely adsorbed by a small piece of sponge in about 20 s, and no solvent drips when the monolith is lifted. These results indicate that the sponges can achieve the oil-water separation rapidly and possess excellent oil binding capacities compared with reported oil-adsorbing materials [29].

Reusability of the sponge was evaluated using *n*-hexane and crude oil as model oils (Fig. 12). These results show that the absorption capacity to *n*-hexane is almost unchanged after 30 times, indicating that the macro-porous structures are well-preserved during the process. Although the amount of crude oil absorption gradually decreases after repeated iterations, the absorption capacity is still up to 12 g g^{-1}. The result suggests that the porous structure of sponges might be sealed by oil so that the absorption of sponges gradually decreases. All these results show that the sponges have an excellent cycling nature because they can be recycled many times without obvious decline of absorption capacity compared with other absorption materials [32].

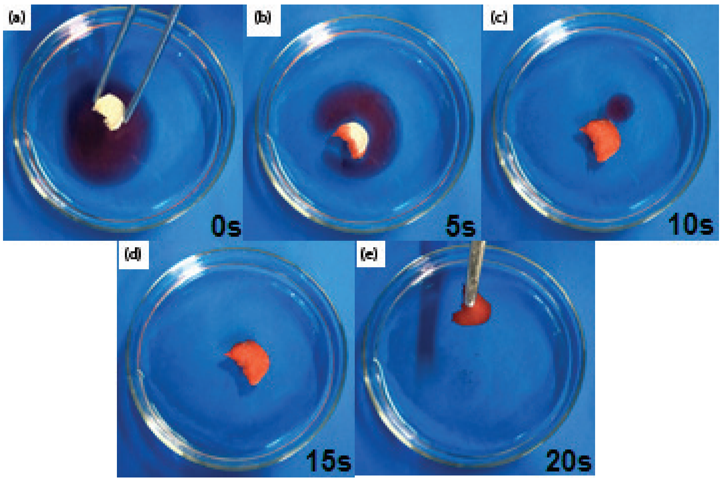

Fig. 11. Oil absorption process from water surface (*n*-hexane dyed with oil red).

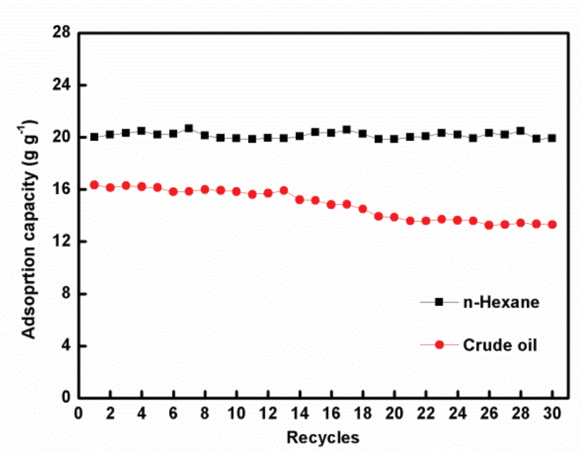

Fig. 12. Reusability of HIPE sponges for *n*-hexane and crude oil.

4. Conclusions

Monolithic HIPE sponges have been synthesized through HIPE templating without using polymerization from SSEBS and PAMAM dendrimers. The HIPE sponges show low densities and typical emulsion-templated interconnected porous structures, ranging from 1 to 20 μm. The sponges are hydrophobic, with water contact angle of 128.1° and exhibit relatively high absorption capacity and high recyclability. Such good properties make these sponges to be an excellent candidate for the reclamation of spilled oils.

Acknowledgements

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