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Preparation of flexible, hydrophobic, and oleophilic silica aerogels based on a methyltriethoxysilane precursor

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Abstract We report the synthesis of flexible, hydrophobic, and oleophilic silica aerogels through a two-step acid–base sol–gel reaction followed by supercritical drying, in which methyltriethoxysilane (MTES) is used as a precursor, ethanol (EtOH) as a solvent, and hydrochloric acid (HCl) and ammonia ($NH_3{\cdot}H_2O$) as catalysts. At the optimal molar ratio of MTES:EtOH:H_2O:HCl:$NH_3{\cdot}H_2O$ is 1:18:3.5:1.44 × 10^{-4}:1.2, MTES-based silica aerogels show the minimum density of 0.046 g/cm^3 and the maximum compression ratio of 80 % with 15.09 kPa stress. They are superhydrophobic with a water contact angle of 157° and thermally stable up to 350 °C. We also find that they show the excellent adsorption for ethanol with a ratio of 1400 %.

Introduction

Silica aerogels are a highly porous material derived from a silica gel, in which the liquid phase confined in three-dimensional (3D) porous silica networks is replaced with a gas phase [1, 2]. Due to their low density, low thermal conductivity, high porosity, and large surface area, silica aerogels have gained tremendous interests as catalyst supports [3], light-weight thermal insulators [4], absorbents [5], and drug delivery systems [6]. Tetraethylorthosilane (TEOS) [7, 8] is a traditional precursor for the synthesis of silica aerogels through sol–gel process. However, the TEOS-based silica aerogels are fragile and show strong moisture absorption due to the presence of hydrophilic Si–OH groups on the silica networks [9], which limit their potential applications in a number of fields [10]. The flexible and hydrophobic properties of silica aerogels are extremely important for their long-term applications.

Recently, progresses have been made in improving the strength and toughness of silica aerogels by integrating high-strength fibers in the silica aerogels [11] or cross-linking of silica aerogels with polymers [12]. Although these methods are effective in improving the strength and toughness of silica aerogels, further surface modification is required to improve their hydrophobic property [13]. It has been shown that silica aerogels made of methyltrimethoxysilane (MTMS) [14, 15] and methyltriethoxysilane (MTES) [16, 17] precursors are flexible and hydrophobic due to the presence of non-polar methyl terminated groups on the three-dimensional (3D) porous silica networks. Hydrophobic aerogels are great promise in the removal of oil from water for oil spill cleanup [18].

Herein, we study the effect of solvent and catalysts on the flexibility and hydrophobicity of silica aerogels made of MTES precursor through a two-step acid–base sol–gel reaction [19], in which ethanol (EtOH) is used as a solvent, hydrochloric acid (HCl) and ammonia ($NH_3{\cdot}H_2O$) are as catalysts. At the optimal molar ratio of MTES:EtOH:H_2O:HCl:$NH_3{\cdot}H_2O$ is 1:18:3.5:1.44 × 10^{-4}:1.2, the MTES-based silica aerogels show the minimum density of 0.046 g/cm^3 and the maximum compression ratio of 80 %. The MTES-based silica aerogels are superhydrophobic

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with a water contact angle of 157° and show the excellent uptaking capacity of ethanol with an adsorption ratio of 1400 %. The flexible, hydrophobic, and oleophilic MTES-based silica aerogels are thermally stable up to 350 °C.

Experimental

MTES, EtOH, HCl, and $NH_3 \cdot H_2O$ were obtained from Sinopharm Chemical Reagent (Shanghai, China). All chemicals were used without further purification. In our experiments, the silica gels were synthesized by MTES as a precursor, EtOH as a solvent, and HCl and $NH_3 \cdot H_2O$ as acid–base catalysts in two steps. Firstly, MTES precursor was diluted with EtOH and H_2O, followed by the addition of 0.01 mol/L HCl alcohol solution (acidic catalyst). The mixed solution was placed at room temperature for the hydrolysis of MTES. Secondly, after 18 h, 10 mol/L $NH_3 \cdot H_2O$ alcohol solution (basic catalyst) was added to the mixed solution to accelerate condensation reactions. Table 1 shows the specific molar ratios of MTES:EtOH:H_2O:HCl:$NH_3 \cdot H_2O$ to create various samples in this work. The resultant sol was stirred for 20 min and transferred to airtight glass tubes. The gelation took place after 5–16 h. The gels were then aged in an EtOH bath for 2 days to remove water confined in gel networks. Finally, the MTES-based aerogels were obtained by the supercritical drying (at 260 °C and 10 MPa).

The bulk density ($\rho = m/V$) of MTES-based silica aerogels was calculated from the mass (m) to volume ratio (V). The mass was measured by an electronic balance with 0.01 mg accuracy. The dimension was measured using calipers with 0.01 mm accuracy. The flexibility of MTES-based silica aerogels was defined by the compression ratio ($S = \Delta L/L$), where ΔL is the largest length change with stress and L is the original length. The uniaxial compression tests were performed by KDII-0.01 testing machine (Shenzhen Kaiqiangli Testing Instruments, Shenzhen, China), equipped with two flat-surface compression stages and 100 N load cells. The cylindrical samples with 12 × 12 × 20 mm size were used. The contact angle of water droplets on MTES-based silica aerogels was measured with a JC2000A contact angle analyzer (Shanghai Advanced Photoelectric Technology, Shanghai, China). Thermo gravimetric and differential thermal analyses (TGA/DTA, STA 409 EP/DIL 404, Netzsch, Selb, Germany) were conducted from room temperature to 800 °C at a rate of 10 °C/min under air flow. The chemical composition of MTES-based silica aerogels was characterized with Fourier transform-infrared spectroscopy (FT-IR, Avatar 360, Thermo Nicolet, Madison, WI, USA). The microstructure of MTES-based aerogels was characterized using scanning electron microscopy (SEM, LEO1530, Germany LEO electron, Oberkochen, Germany).

Results and discussion

Figure 1 shows a two-step acid–base catalyzed process for preparing flexible and hydrophobic silica gels. MTES was partially hydrolyzed in water under acidic condition, and then condensed into silica network under basic condition. Compared to TEOS-based aerogels, the MTES-based aerogels are hydrophobic with a lower cross-linking density and higher deformability due to one non-hydrolysable, hydrophobic methyl group present. It has been known that

Table 1 Arrangement and results of L_9 (3^4) orthogonal test

	H_2O (A)	HCl (B)	$NH_3 \cdot H_2O$ (C)	None	MTES	EtOH	Compression ratio (S %)
A1B1C1	4	1.44×10^{-4}	1.2	–	1	16	71.43
A1B2C2	4	7.2×10^{-4}	1	–			50.00
A1B3C3	4	3.6×10^{-4}	0.72	–			40.54
A2B1C2	3.5	1.44×10^{-4}	0.72	–			63.33
A2B2C3	3.5	7.2×10^{-4}	1.2	–			67.86
A2B3C1	3.5	3.6×10^{-4}	1	–			40.00
A3B1C3	3	1.44×10^{-4}	1	–			50.00
A3B2C1	3	7.2×10^{-4}	0.72	–			62.16
A3B3C2	3	3.6×10^{-4}	1.2	–			56.25
$\bar{\kappa}_1$	53.990	61.587	57.863	65.180			
$\bar{\kappa}_2$	57.063	60.007	56.527	46.667			
$\bar{\kappa}_3$	56.137	45.597	52.800	55.343			
R	3.073	15.990	18.513	5.063			
Optimal result A2B1C1	3.5	1.44×10^{-4}	1.2	–	1	16	74.36

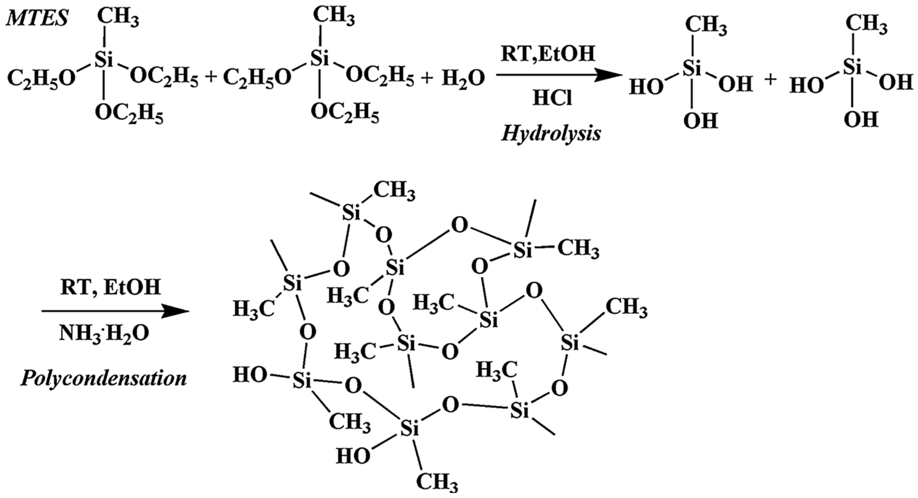

Fig. 1 Two-step acid–base sol–gel synthesis for MTES-based silica gels

the microstructures of silica aerogels are primarily controlled by the molar ratio of reactants [15]. MTES-based silica aerogels were synthesized at three molar ratios of H_2O, HCl, and $NH_3{\cdot}H_2O$. Orthogonal tests were used to assess their flexibilities. Table 1 shows the result of a L_9 (3^4) orthogonal array with nine combinations was applied. The optimal result via the ANOVA analysis [20] reveals that MTES-based silica aerogels synthesized at the MTES:H_2O:HCl:$NH_3{\cdot}H_2O$ molar ratio of 1:3.5:1.44 × 10^{-4}:1.2 have the maximum flexibility.

Furthermore, we studied the effect of EtOH solvent on the flexibility of MTES-based silica aerogels, in which the molar ratio of EtOH and MTES was varied from 8 to 20, while the molar ratio of MTES:H_2O:HCl:$NH_3{\cdot}H_2O$ was kept at 1:3.5:1.44 × 10^{-4}:1.2. We find that the gelation time increases with the increase of the molar ratio of EtOH and MTES from 8 to 20. This is due to the fact that the high dilution of MTES molecules in EtOH solvent would slow down the hydrolysis and condensation reaction. Simultaneously, the high dilution of MTES molecules in EtOH solvent reduces silica contents, leading to the lower density of the silica aerogels. As can be seen in Fig. 2, the densities of MTES-based silica aerogels decrease from 0.082 to 0.046 g/cm^3 with the increase of the molar ratio of EtOH and MTES from 8 to 18. However, a slight increase in the density of MTES-based silica aerogels is observed when the molar ratio increase from 18 to 20. In this molar ratio range, the resultant silica network becomes weaker, resulting in the increase of volume shrinkages during drying. When the molar ratio is increased to 22, there is no gel formation observed. In addition, the compression ratio of MTES-based silica aerogels increases from 15.40 to 80.00 % as the increase of the molar ratio from 8 to 18 (Fig. 3). When the molar ratio is increased to 20, a slight decrease in the compression ratio is noticed. From Figs. 2 and 3, we conclude that the minimum density (0.046 g/cm^3) and the maximum flexibility (80.00 %) of MTES-based silica aerogels can be obtained at the MTES:EtOH:H_2O:HCl:$NH_3{\cdot}H_2O$ molar ratio of 1:18:3.5:1.44 × 10^{-4}:1.2.

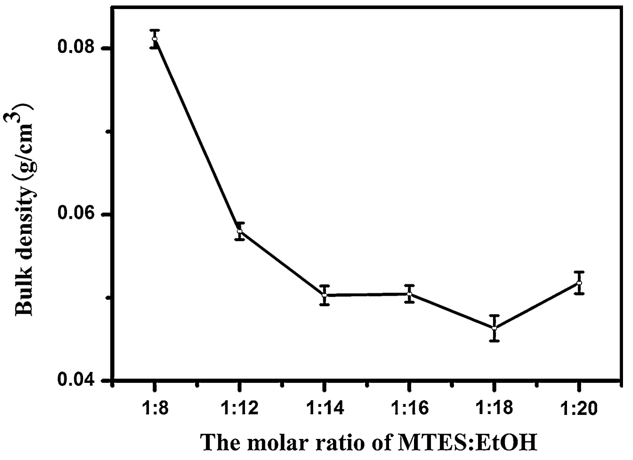

Fig. 2 The bulk density of the MTES-based silica aerogels synthesized at different molar ratios of MTES:EtOH

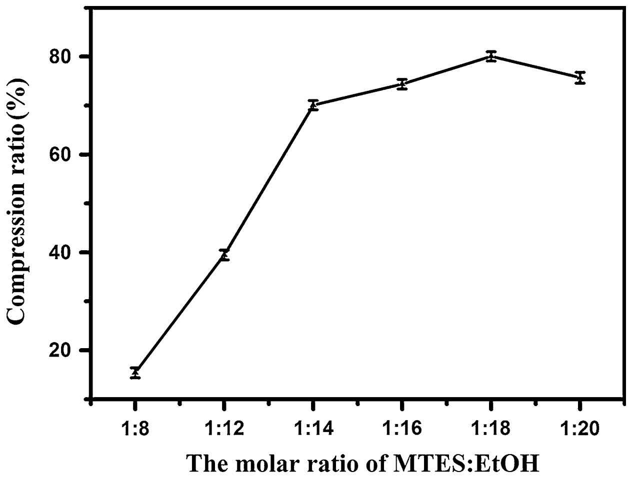

Fig. 3 The compression ratio of the MTES-based silica aerogels synthesized at different molar ratios of MTES:EtOH

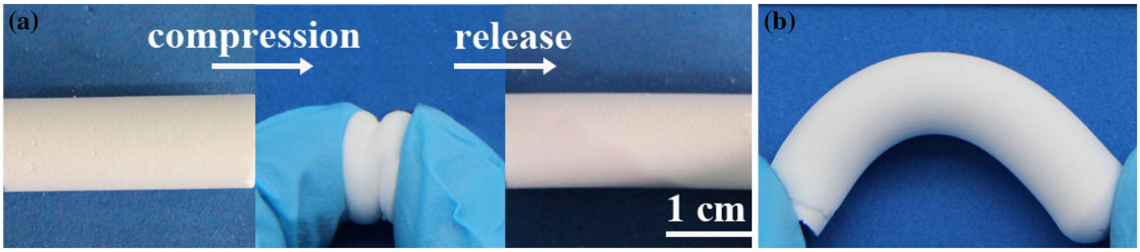

Fig. 4 Images of **a** the compressed and uncompressed and **b** the maximum bending of MTES-based silica aerogels formed at the MTES:EtOH molar ratio of 1:18

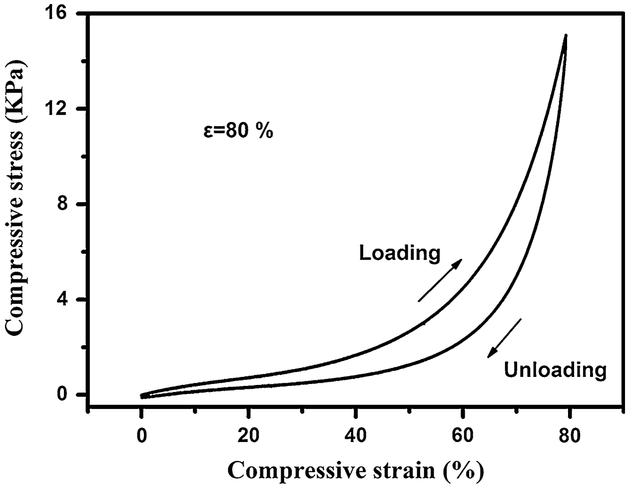

Fig. 5 The compressive strain–stress curve of MTES-based silica aerogels formed at the MTES:EtOH molar ratio of 1:18 with set strains ε of 80 %

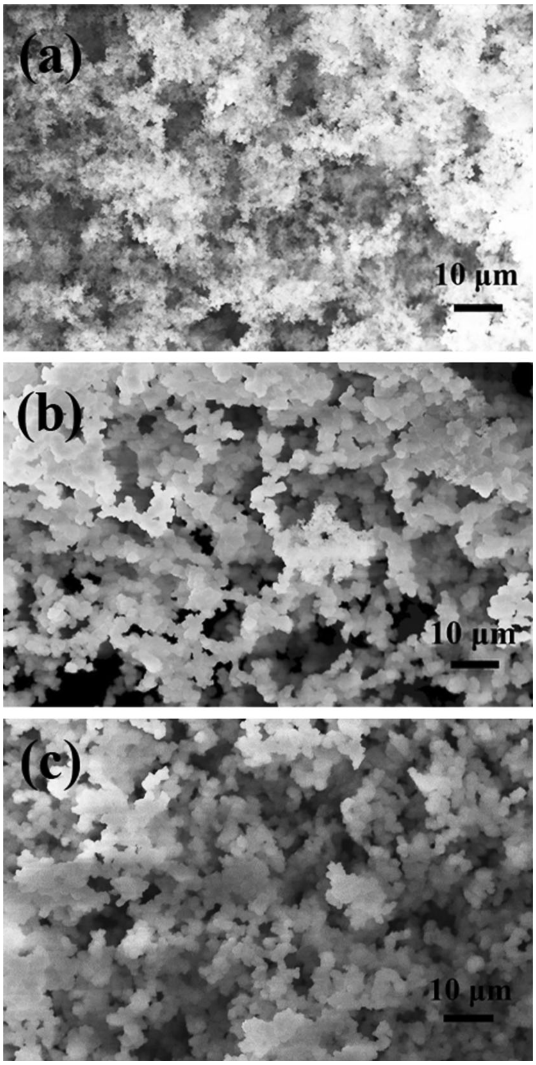

Fig. 6 SEM images of MTES-based silica aerogels formed at the MTES:EtOH molar ratios of **a** 1:14, **b** 1:18, **c** 1:20, respectively

Figure 4a shows the pictures of MTES-based silica aerogels which is compressed to 80 % of their original length. After releasing the stress, the silica aerogels recover completely. The compression and recovery process of MTES-based silica aerogels can be repeated many times. Figure 4b shows the maximum possible bending of MTES-based silica without cracks. Further, exact mechanical properties of MTES-based silica aerogels were measured by a universal testing machine. Figure 5 shows plots of compressive strain–stress for the aerogels at the set strain (ε) maxima of 80 % and shows a linear elastic region at $\varepsilon < 20$ %, followed by densification regions at $\varepsilon > 20$ % [21]. At $\varepsilon = 80$ %, the maximal stress was measured to be 15.09 kPa, illustrating that the aerogels are quite soft material. Moreover, the aerogels recovered their original size rapidly and almost completely, while the unloading curves return to zero at $\varepsilon = 0$ of all strains, corresponding to the experimental observation in Fig. 4. The flexibility of MTES-based silica aerogels is a result of the formation of 3D silica networks with the CH_3 terminal group. Figure 6 shows SEM images of MTES-based silica aerogels formed at the molar ratio of MTES:EtOH is 1:14, 1:18, and 1:20, respectively. The MTES-based silica aerogels have branched microstructures, in which silica aggregates interconnect to form a porous network. We noticed that the pore size in the network structure becomes larger when the molar ratio of MTES:EtOH increases from 1:14 to 1:18, while pore sizes have slightly reduced when the molar ratio increases from 1:18 to 1:20. The network structure with large pore size and strong skeleton could avoid the easily

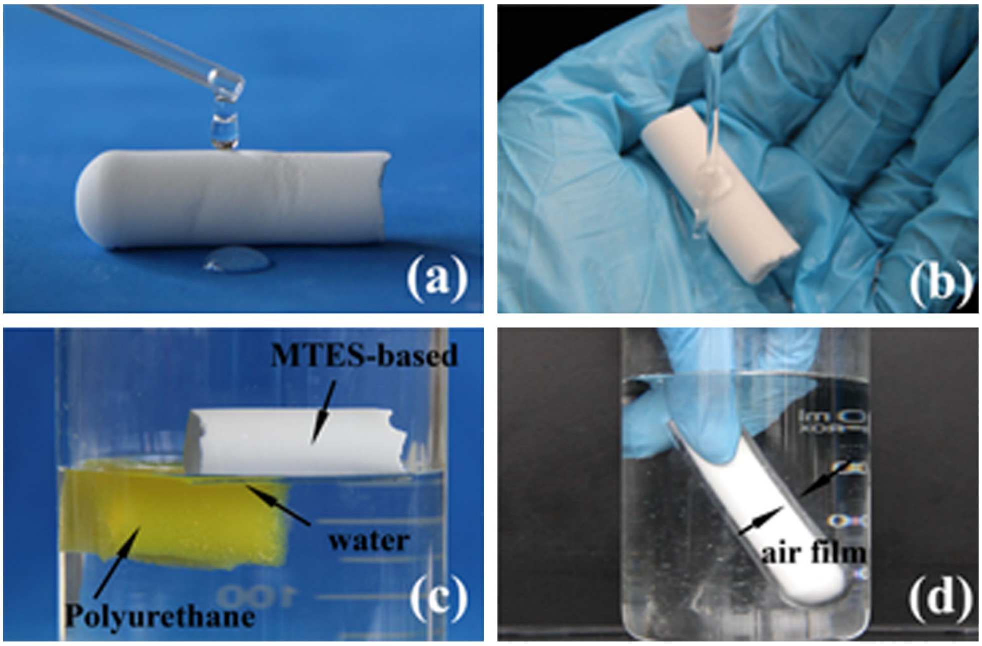

Fig. 7 Hydrophobic behaviors of MTES-based silica aerogels formed at the MTES:EtOH molar ratio of 1:18

damage under stress and hence more flexible, as well as the dense network with smaller particles and pore sizes diminished the flexibility [16]. The SEM images conform to the regularity of compression with the increase of ethanol solvent.

MTES-based silica aerogels are hydrophobic. Like sacred lotus leaves and water strider's legs, water droplets can roll off the surface of the MTES-based silica aerogels without wetting (Fig. 7a). The hydrophobicity of the MTES-based silica aerogels remains unchanged after being washed with running tap water for several minutes (Fig. 7b). Compared to the conventional cleaning sponge made from polyurethane microfibers which sinks in water, the MTES-based aerogels can float on the surface of water due to their low density and hydrophobic nature (Fig. 7c). If we push the MTES-based silica aerogels in water, the interface between the aerogels and the water is formed due to the formation of an air film on the surface of hydrophobic silica aerogels (Fig. 7d). The thickness of the air film is about 1.3 mm.

The water contact angle of MTES-based silica aerogels was measured by a static method, in which a water droplet is ejected from a syringe and a MTES-based silica aerogel film is then moved up to make the contact with the water droplet (Fig. 8). We find that a 3 μL water droplet cannot be released to the surface of the silica aerogel film even when it is squeezed into an ellipse. Until the water volume increasing to 10 μL, the successful release of water droplets could be observed, however, the water droplets roll away on the surface of silica aerogel films immediately. After several attempts, the static water contact angle of 10 μL water droplet is ~157°, indicating the superhydrophobicity of the MTES-based silica aerogels. It is known that the wettability of a surface is determined by its chemical composition and geometrical structures [22]. The peaks of 1270 and 849 cm^{-1} in FT-IR spectrum (Fig. 12) are associated with the stretching of the Si-CH_3 bond and the presence of CH_3 groups at the surface of the silica networks lowers the surface energy making the MTES-based silica aerogels exhibit fairly good water repellency. Moreover, the surface roughness could be obtained by tuning the microstructures of the sol–gels through control of hydrolysis and condensation reactions [23]. The MTES-based silica aerogels via sol–gel method are shown 3D porous network structure in Fig. 6.

The oleophilicity of MTES-based silica aerogels is also studied. Water droplets on MTES-based silica aerogels maintain a spherical shape, while ethanol droplets are absorbed totally [24]. Thus they can only allow organic compounds entering their pores and inhibit the entry of water, showing selectivity [25]. By placing a small piece of MTES-based aerogels on the surface of hexane-water mixtures, as expected, the hydrophobic and oleophilic aerogels separated hexane (labeled with Oil Red O) from the water surface completely after a few seconds, and then floating on the water (Fig. 9). The hexane was removed from the mixture after putting the aerogels out. The absorption efficiency of MTES-based aerogels for ethanol was estimated by measuring the adsorption ratio, which is defined as the ratio of the absorbed ethanol weight and the

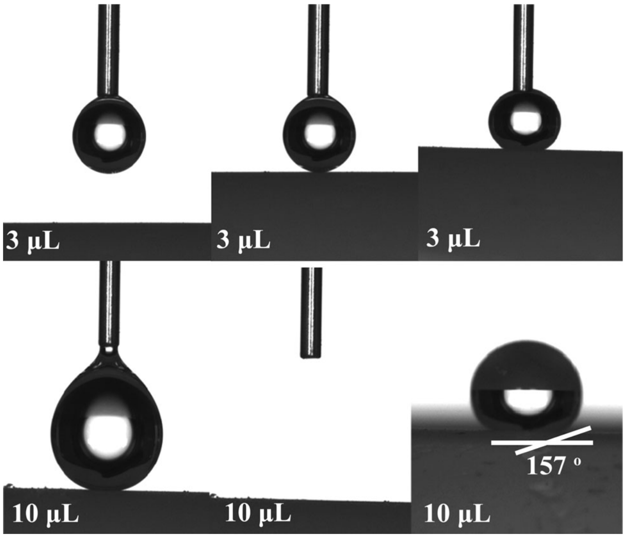

Fig. 8 Water contact angle of MTES-based silica aerogels formed at the MTES:EtOH molar ratio of 1:18

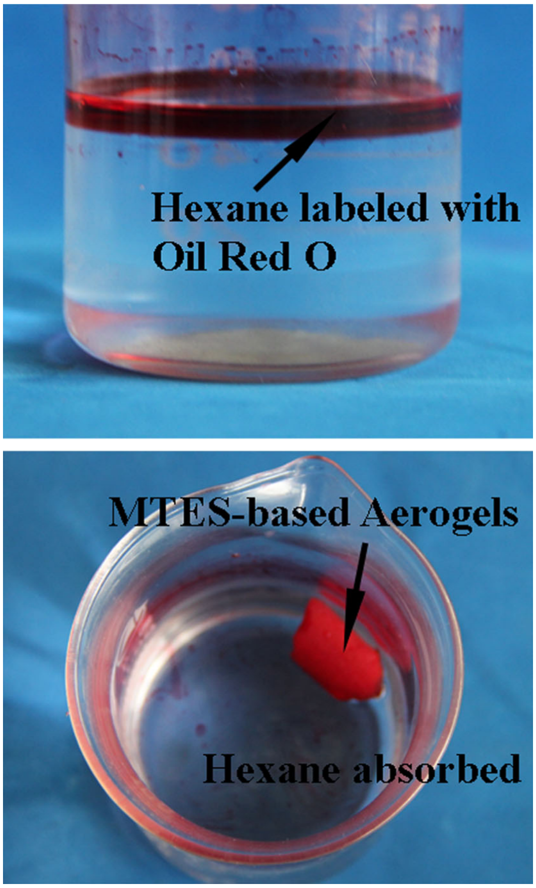

Fig. 9 FT-IR spectra of MTES-based silica aerogels formed at MTES:EtOH the molar ratio of 1:18 after being treated at different temperatures

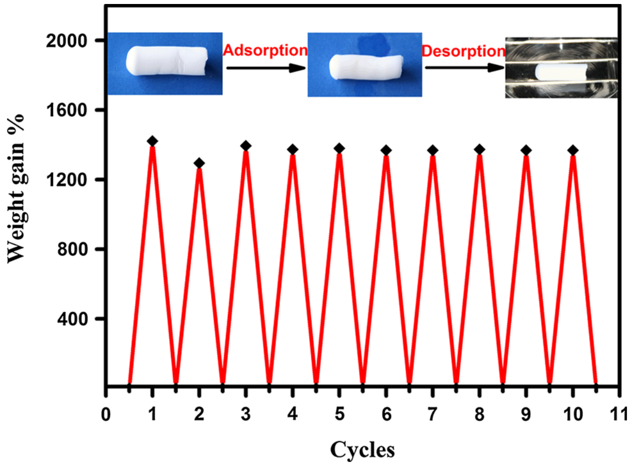

Fig. 10 Removal and collection hexane from the water surface by MTES-based silica aerogels formed at the MTES:EtOH molar ratio of 1:18. Hexane was labeled with Oil Red O

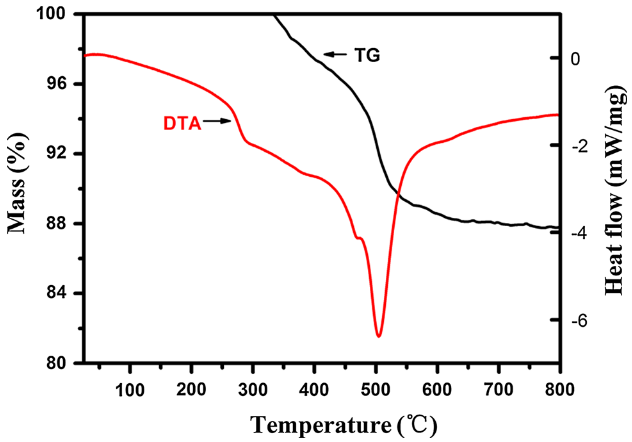

Fig. 11 Weight gain of MTES-based silica aerogels formed at the MTES:EtOH molar ratio of 1:18 during absorption and desorption ethanol cycles. The inset images show the sample state during adsorption–desorption process

pristine aerogels weight. We found that the ethanol adsorption ratio can reach to as high as 1400 % and remain unchanged even after 10 adsorption–desorption process cycles (Fig. 10), in which desorption was carried out by drying the MTES-based silica aerogels in an oven at 60 °C for 2 h. With the advantages of high adsorption efficiency, selectivity, and excellent recyclability, the MTES-based silica aerogels could be a promising absorbent material.

TG–DTA experiments [26] were performed to investigate the thermal stability of MTES-based silica aerogels. Figure 11 shows that the TG–DTA curves of MTES-based aerogels obtained in the temperature range from 25 to 800 °C. There is a small amount of heat loss in the temperature range from 25 to 350 °C in the DTA curve and hardly any weight loss in the TG curve. Then a sharp decline in the weight is observed in the temperature range from 350 to 550 °C in the TG cure, the weight loss is about 12 %, with an exothermic peak at 500 °C in the DTA curve. To better understand the effect of temperature on the

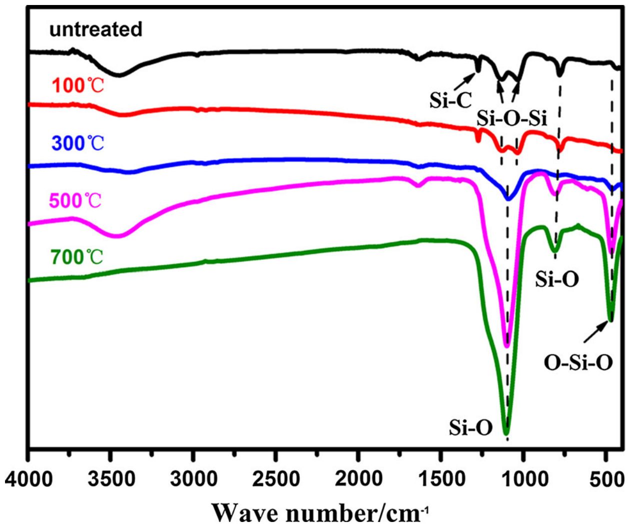

Fig. 12 TG–DTA curves of MTES-based silica aerogels formed at the MTES:EtOH molar ratio of 1:18

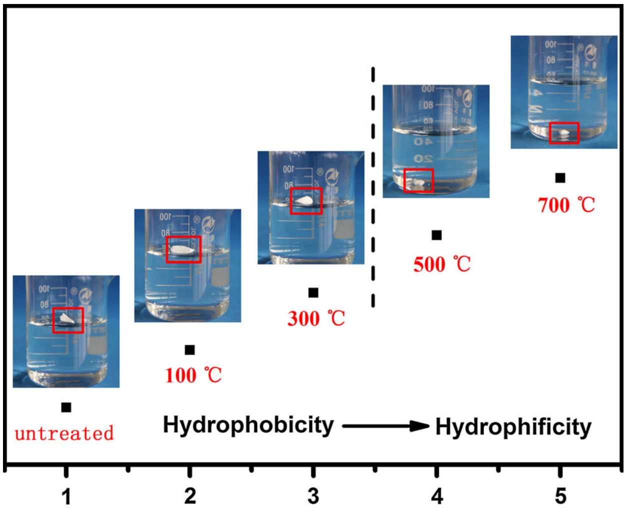

Fig. 13 Images depicting the hydrophobicity of MTES-based silica aerogels formed at the MTES:EtOH molar ratio of 1:18 after being treated at different temperatures

structure or properties of MTES-based aerogels, the aerogels were heated in a tube furnace at 100, 300, 500, and 700 °C for 8 h, respectively. We observed that the samples treated at 500 and 700 °C can be broken by hand easily, indicating that the aerogels lose their elastic nature at high temperature. Chemical reactions are responsible for this change, which is evidence from the FT-IR spectra of MTES-based silica aerogels treated at different temperatures (Fig. 12). It can be also seen that the Si–O–Si bonds indicated by the double peaks located at 1134 and 1035 cm^{-1}disappears when temperature is above 300 °C and the Si–O stretching at 1079 $\text{cm}^{-1}$and the O–Si–O stretching at 500 cm^{-1} were observed. In addition, the peak at $\sim$1270 cm^{-1}, which represents the stretching of Si-CH_3 groups, gradually decreases with the increase of temperature and finally disappears at 500 °C that correlate well with the mass loss data shown in the TG curve. We place the silica aerogels treated at different temperatures into water to confirm the thermal stability of hydrophobic MTES-based silica aerogels (Fig. 13). The silica aerogels heated at temperatures below 300 °C float on the surface of water. While the aerogels treated at 500 and 700 °C sink to the bottom of water. This suggests that the aerogels treated at temperatures above 500 °C become hydrophilic due to the oxidation of the terminated CH_3 groups on the silica networks.

Conclusions

Light, flexible, hydrophobic, and oleophilic silica aerogels have been synthesized through a two-step acid–base sol–gel reaction followed by supercritical drying, in which MTES is used as a precursor, EtOH as a solvent, HCl and $NH_3{\cdot}H_2O$ as catalysts. Under the optimal MTES:EtOH:H_2O:HCl:$NH_3{\cdot}H_2O$ molar ratio of 1:18:3.5:1.44 $\times$ 10^{-4}:1.2, MTES-based silica aerogels show the minimum density of 0.046 g/cm^3 and the maximal stress was measured to be 15.09 kPa at the set strain (ε) of 80 %. They are superhydrophobic with a water contact angle of 157° and oleophilic with an ethanol adsorption ratio of 1400 %. The TGA and FT-IR measurements show that the MTES-based silica aerogels are stable up to 350 °C. The MTES-based aerogels have potential for the efficient removal for oil from water.

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