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Eco-friendly approach for preparation of water-based superhydrophobic silica aerogels via ambient pressure drying

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Abstract

In this work, superhydrophobic methyltrimethoxysilane (MTMS)-based silica aerogels were fabricated via water-based sol–gel reaction by ambient pressure drying (APD) method in the presence of surfactant. The structure, morphology, and hydrophobic properties of the obtained silica aerogel were characterized by scanning electron microscopy (SEM), Brunauer–Emmett–Teller (BET) methods, X-ray diffractometer (XRD), and water contact angle measurement. The effects of the concentration of MTMS, the pH value of the solution, and the amount of surfactant cetyltrimethylammonium bromide (CTAB) on properties of silica aerogels were investigated, respectively. Increasing the concentration of MTMS to a great extent resulted in higher density and lower porosity of silica aerogel. Alkali catalyst was an extremely crucial factor for preparing silica aerogels. With the increase of the pH value from 7.5 to 10 the gelation time of the prepared aerogels was observed to decrease from 40 min to 2 min, and the shrinkage of aerogel sample decreased from 15.5% to 3.5%. The skeleton structure of the prepared silica aerogel gradually transformed from coarsened spherical to continuous irregularity finer structures with the increased concentration of CTAB. In addition, the prepared silica aerogel showed outstanding anti-adhesion property and superhydrophobic ability with a water contact angle (WCA) of 160.6 ± 1.3°.

1. Introduction

Silica aerogel is considered to be a nano-porous material with unique properties such as low density, high porosity, high specific surface area, etc [1, 2], which shows broad prospects of application in multiple fields, including thermal insulation [3, 4], sound insulation noise reduction [5, 6], intelligent textile [7], and drug carrier [8, 9]. Great progress has been made in this field, but most of the researchers focused on the application of silica aerogel currently. Few of them solved the problem of environmental pollution and economic costs during the preparation of aerogel [10]. At present, most of approaches for preparing silica aerogels based on the Stöber sol–gel method involved a large amount of organic solvent like alcohol as the reaction medium. This process increased the cost of production, causing pollution to the environment. Meanwhile supercritical drying technology (SCD) was generally required for preventing wet gel from shrinking and collapsing during drying [11]. However, SCD is a technology with strong pressure, high costs and complicated operation. These factors have hindered the commercial production of aerogels and the extensive development of potential applications to a considerable extent [12].

Compared with SCD, ambient pressure drying (APD) is found to be a more practical method for preparing aerogels owing to the simple operation, high security, and mass production [13]. At present, the preparation of silica aerogels requires complicated steps such as solvent exchange and hydrophobic modification by using APD. Xu et al [14, 15] used methyltrimethoxysilane (MTMS) as precursor and water as solvent, and adopted APD to prepare silica aerogels by greatly increasing the aging time of wet gel. For some researches, the silica aerogel was prepared using MTMS as silicon source and trimethyl chlorosilane (TMCS) as modified agents by APD. Nevertheless, a large amount of organic solvents such as methanol, isopropanol, hexane and N,
N-dimethylformamide (DMF) were applied in the sol-gel process \[16\]. For the above approaches, lots of organic solvents were employed for preparing aerogels, while bringing troubles such as environmental pollution, long time aging or modification, and high cost of production. Therefore, it is of great significance to develop a facile and eco-friendly method for preparing silica aerogels using water as solvent.

It has been reported that organosilane compounds of the type \(\text{RSi(OR)}_3\) (where \(\text{R} \) is alkyl), such as MTMS \[17\], methyltriethoxysilane (MTES) \[18\], were better silicon source as the precursor rather than the type \(\text{Si(OR)}_4\), like tetraethyl orthosilicate (TEOS) \[19\] and tetramethoxysilane (TMOS) \[20\]. Because of the existence of alkyl, the mechanical properties and hydrophobicity of the prepared \(\text{SiO}_2\) aerogels with \(\text{RSi(OR)}_3\) as the precursor have been improved. Furthermore, the modifier and other organic solvents could be avoided before ambient pressure drying. However, organosilane cannot be miscible with water. It is extremely important to develop a surfactant for mixing miscible organic solvents with water. Cationic surfactant cetyltrimethylammonium (CTAB) could indeed be an outstanding alternative. When the aerogels possess superhydrophobic properties, the collapse of aerogel can be effectively avoided, showing wide application prospects. The superhydrophobic aerogel is an excellent material for the preparation of super-hydrophobic coating, which can be used in various substrate surfaces (like textiles, metal, wood, etc.) to realize the functions of oil-water separation, self-cleaning, anti-corrosion and so on.

In this paper, water solvent-based superhydrophobic silica aerogels were prepared using MTMS as precursor and ammonia (\(\text{NH}_3\cdot\text{H}_2\text{O}\)) as catalyst by APD via sol–gel reaction in the presence of surfactant CTAB. The CTAB was applied to promote the miscibility of MTMS with water and prevent phase separation. The effects of the concentration of reactants (MTMS), alkali catalyst dosage (\(\text{NH}_3\cdot\text{H}_2\text{O}\)) and the content of surfactant (CTAB) on aerogel parameters, such as the gelation time and density, specific surface area and wettability were investigated in detail. The obtained silica aerogels were characterized by SEM, BET, Fourier-transform infrared spectrometry (FTIR) and X-ray diffractometer (XRD). The superhydrophobicity of silica aerogels was also evaluated.

2. Materials and methods

2.1. Materials

Methyltrimethoxysilane (MTMS), ammonium hydroxide (\(\text{NH}_3\cdot\text{H}_2\text{O}\)), cetyltrimethylammonium bromide (CTAB) and deionized water were purchased from Sinopharm Chemical Reagent Co. (China).

2.2. Preparation of MTMS-based silica aerogels

The silica aerogels were prepared via a one-step, base catalyzed sol–gel process by APD. Firstly, CTAB was dissolved into 25 mL \(\text{H}_2\text{O}\) at various dosage from 1 mmol l\(^{-1}\) to 28 mmol l\(^{-1}\) (The mass concentration of MTMS was maintained at 28wt.% and the pH value of the solution is 9). After stirring for 10 min, the MTMS precursor was added and stirred at room temperature for 1 h at different MTMS/\(\text{H}_2\text{O}\) mass concentration from 16% to 36%. Then, the 1 M \(\text{NH}_3\cdot\text{H}_2\text{O}\) aqueous solution, was added dropwise to regulate the pH of the as-prepared solution from 7.5 to 10.0 while stirring. The generated gels were aged in distilled water at 60 °C for 12 h. Lastly, the aged gels were dried under ambient pressure at 80 °C, 100 °C, 120 °C for 2 h, respectively, and finally at 150 °C for 1 h. A diagrammatic drawing of the fabrication process is shown in figure 1.

2.3. Characterization

The bulk density (\(\rho\)) of the silica aerogels was measured from the mass to volume ratio of the sample after it was molded into a regular figure. The skeletal density (2.2 Kg m\(^{-3}\)) was measured by helium pycnometry and the porosity was then calculated by equation (1) \[21\]:

![Figure 1. Fabrication process of water-based superhydrophobic silica aerogels.](image-url)
Increased MTMS concentration is obvious that the gelation time and volume shrinkage marked decline. The parameters of aerogels were investigated by increasing the concentrations of MTMS. As shown in figure 2, it is clear that the uneven network skeletons and pore are found at lower concentrations of MTMS and (a) indicates that the diameter of aerogel skeleton is expanded from 200 nm to 500 nm, and the pore diameter slightly decreases with the concentration of MTMS increasing from 16 wt.% to 36 wt.%. This result confirmed that the density of aerogel was increased with increasing MTMS concentration.

### 3. Results and discussion

#### 3.1. The effects of mass concentration of MTMS on silica aerogel parameters

The parameters of aerogels were investigated by increasing the concentrations of MTMS. As shown in figure 2, it is obvious that the gelation time and volume shrinkage marked decline first and then gradually stabilize with increased MTMS concentration (figure 2(a)). The density is increased from 80 to 220 Kg m\(^{-3}\) and the porosity is decreased from 95.3% to 87% with increasing concentration of MTMS (figure 2(b)). These changes were attributed to the fact that higher concentration promoted the hydrolysis and condensation of the precursors before ageing, leading to more intensive three-dimensional porous network as showed by the FE-SEM images in figure 3. Compared with reported researches, in our study, the shrinkage of the silica aerogel samples was lower via APD [14], which was mainly owed to the methyl groups of the MTMS lessening the condensations of the surface silanol groups (Si-OH) to avoid continued shrinkage. Furthermore, the mutual exclusion between hydrophobic methyl also resulted in a decrease in shrinkage of the wet gel [23]. Figure 2(c) shows that increasing the concentrations of MTMS precursor leads to a slight fall in BET surface area and the average pore size of the aerogels.

The morphology of the silica aerogels synthesized with different concentrations of MTMS was characterized by SEM. As shown in figures 3(a)–(c), the obtained aerogel network skeletons are stacked by silica nanoparticle. It is clear that the uneven network skeletons and pore are found at lower concentrations of MTMS (figures 3(a) and (d)), while the relatively densest skeletons are observed from SEM images of silica aerogels with high concentrations of MTMS (figures 3(c) and (f)). A closer observation of these sample structures at a higher magnification figures 3(d) and (f)) indicates that the diameter of aerogel skeleton is expanded from 200 nm to 500 nm, and the pore diameter slightly decreases with the concentration of MTMS increasing from 16 wt.% to 36 wt.%. This result confirmed that the density of aerogel was increased with increasing MTMS concentration.

#### 3.2. The effects of the pH on silica aerogel parameters

The pH value of the solution is one of crucial factors for preparation of the MTMS-based silica aerogels. In this study, the gelation time of the wet sol was extremely affected by using ammonium hydroxide as the catalyst. The silica aerogels properties were investigated with different pH by changing the amount of NH\(_3\)·H\(_2\)O while the concentration of MTMS remained 28 wt.% and the dosage of CTAB was 10 mmol l\(^{-1}\). The wet sol could not...
accomplish the gel formation within 48 h at pH lower than 7.5. Because the condensation of MTMS was exceptionally slow at neutral and acid, generating linear and low branched particles. So that it is unable to gel [6].

As the pH value increases from 7.5 to 10, the gelation time of the wet gel steeply decreases from 40 min to 2 min, and the shrinkage of aerogel decreases from 15.5% to 3.5% (figure 4(a)), resulting in a slight fall in density from 190 Kg m⁻³ to 126 Kg m⁻³ and increase of the porosity from 88.8% to 92.5% (figure 4(b)). Higher pH of the solution accelerates the condensation reaction, and forms larger silica particles and pore, which leads to a decrease in BET-specific surface area from 450 m² g⁻¹ to 100 m² g⁻¹ (figure 4(c)).

Figure 5 shows the morphologies of silica aerogels prepared at different pH values and the corresponding silica aerogel images. As shown in figures 5(a) and (b), when the pH of solution is at 8, the particle size of aerogel ranges from 20 nm to 40 nm, and the particles are packed tightly together so that three-dimensional porous network structure could not be formed. As the pH is further increased, the particle size of aerogel increases dramatically, resulting in larger pores, which confirms the results in figures 5(b) and (c). As can be seen from figures 5(a)–(c), the aerogel is fragmented into small pieces with a lower pH value of 8, while the crackless aerogels could be prepared with higher pH values at 9 and 10. These appearances were attributed to the generation of larger pores at a higher pH value, which led to reduce of the capillary pressure (Pc) of the wet gel, preventing the gel from collapsing and splitting during the APD [24]. As a result, in order to prepare the crackless aerogels with low gelation time and density, the reasonable pH value should be 9–10.

3.3. The effects of the amount of CTAB on silica aerogel parameters

In our study, the precursor MTMS is nonpolar and insoluble in aqueous solvents. Therefore, surfactant (CTAB) is the crucial factor for the preparation of silica aerogels by using water as solvent. CTAB possesses both hydrophobic and hydrophilic functional groups. Once its concentration exceeds the first critical micelle concentration (0.89 mmol 1⁻¹) [25], CTAB can form micelles spontaneously, leading to a steep reduce in the
surface tension of the aqueous solution [26], so that it could be mixable with MTMS. As shown in figure 6(a), the gelation time and volume shrinkage decrease first and then slowly rise up with increased CTAB. This result may be due to the superfluous CTAB which interferes with the formation of gel networks. The density of silica aerogel samples decreases from 174 Kg m$^{-3}$ to 133 Kg m$^{-3}$, while the porosity increases from 89.7% to 92.1%, as the concentration of CTAB varies from 1 mmol l$^{-1}$ to 28 mmol l$^{-1}$ (figure 6(b)).

As shown in the figure 7, with the increase of the concentration of the surfactant, the porous structure of aerogels is closely cross-linked. Meanwhile, the particle size of silica aerogel decreases significantly, resulting in increase of BET-specific surface area from 140 m$^2$ g$^{-1}$ to 301 m$^2$ g$^{-1}$ (figure 6(c)). When the concentration of CTAB slightly exceeds the first critical micelle concentration, the micelles are globular and scattered in the solution, leading to the formed spherical skeleton of aerogel (figures 7(a) and (d)). As the CTAB concentration further increases, the skeleton of the prepared silica aerogel gradually transforms from coarsened spherical to continuous irregularity finer structures. In addition, the finer structures and the smaller pore of aerogel were derived from the excessive surfactant, forming the higher capillary forces, and the wet gels eventually could crack and shrink during ambient pressure drying. The capillary pressure inflicted on the wet gel network enhances with the diminishing of pore sizes in terms of the following equation (3):

$$P_r = -2\gamma_{LV}/r_m$$  \hspace{1cm} (3)

where $\gamma_{LV}$ is the surface tension of water in the pores, $r_m$ is the pore radius.

3.4. Characterization of MTMS-based silica aerogel

FTIR characterization of silica aerogel was carried out (figure 8(a)). It is clearly shown that there are no obvious stretching vibration peak and bending vibration peak of hydroxyl (–OH) at 3435 cm$^{-1}$ and 1618 cm$^{-1}$, respectively, revealing that there is hardly any –OH groups for the prepared aerogel. Two peaks observed at 2971

Figure 5. FE-SEM images of silica aerogels prepared at different pH, (a) pH 8, (b) pH 9, (c) pH 10. (d)–(f) are high magnification images of (a)–(c), respectively.

Figure 6. Properties of silica aerogels synthesized with different concentration of CTAB: (a) gelation time and volume shrinkage; (b) density and porosity; and (c) BET surface area and average pore size.
and 1472 cm$^{-1}$ are assigned to $\ce{-CH_3}$ stretch vibrations and the C–H absorption peak, respectively, which is consistent with the previous research results [27]. Strong peak at nearly 1271 cm$^{-1}$ corresponds to the Si–C stretching vibration. The peaks at 1136 cm$^{-1}$ and 1036 cm$^{-1}$ are attributed to the asymmetrical stretch vibration of Si–O–Si, and the peaks at 784 cm$^{-1}$, and 442 cm$^{-1}$ are ascribed to the stretching and bending vibration absorption of Si–O. Therefore, the obtained silica aerogel is composed of silica and contains large
amounts of methyl groups. XRD was used to characterize crystal structure of prepared silica aerogel. As can be observed from figure 8(b), there is a wide peak with relatively lower peak intensity at the range of $2\theta = 20^\circ \sim 25^\circ$, which is regarded as an amorphous diffraction peak of the silica. In addition, there is inexistence characteristic diffraction peaks at other locations. Consequently, the obtained silica aerogel possesses a typical amorphous structure.

The typical nitrogen adsorption desorption isotherms of the prepared silica aerogel at optimal process are displayed in figure 8(c), and its pore size distribution is shown in figure 8(d). As is shown in figure 8(c), the adsorption isotherms increase slightly at $P/P_0 < 0.8$ and increase sharply at $P/P_0 > 0.8$, revealing the micropore and mesoporous formed in the silica aerogel structure. According to International Union of Pure and Applied Chemistry (IUPAC) [28, 29], the aerogel reveals feature of type-IV nitrogen adsorption–desorption isotherms and H4 hysteresis loop, reflecting a layered pore structure of the sample. Figure 8(d) shows that the pore size distribution of aerogel is uniform, and the average pore size is about 5.2 nm by using the BJH method within the detectable range of pore sizes (1.5–40 nm).

### 3.5. Wettability of MTMS-based silica aerogel

It is generally known that the appropriate surface roughness in combination with low surface energy to fabricate superhydrophobic surfaces [30, 31]. In our study, silica aerogels were prepared using MTMS as precursor. The superhydrophobic properties of the prepared silica aerogel was investigated. Due to the silica aerogel is composed of silica nanoparticles, and the surface of aerogel has a host of methyl groups, resulting in the excellent superhydrophobic properties of aerogel. As shown in figure 9(a), the water droplets exhibit a stable sphere-like shape on the silica aerogel samples, which displays a water contact angle (WCA) of $160.6 \pm 1.3^\circ$. Furthermore, when the silica aerogel was immersed in water, the surface of the sample obviously shows air layer, producing a ‘silver mirror’ phenomenon and displaying the high-performance suppress of water penetration (figure 9(b)). In addition, the anti-adhesion test of silica aerogel is shown in figure 9(c). When the silica aerogel sample is removed up and down, the water drops could entirely leave from the surface of aerogel even after repeated contact and transformation procedures. As illustrated in figure 9(d), liquid droplets like cola, black tea, and blue colored water are dropped on the surface of aerogel and could keep perfectly spherical shapes. The above results manifested that the aerogel possessed excellent superhydrophobic properties. The prepared bulk silica aerogel with high porosity and rough porous structure had lots of hydrophobic methyl groups in the siloxane backbone, lowering the surface energy. The large fraction of air trapped between mesopores of silica aerogel was more favorable to superhydrophobic surfaces. As a result, superhydrophobic silica aerogels were fabricated via the combination of porous rough micro structures and high specific surface area and methyl groups with low surface energy.

### 4. Conclusion

In summary, MTMS-based silica aerogels with excellent superhydrophobic performance were fabricated using water as the solvent via APD method in the presence of surfactant CTAB. The effects of the concentration of MTMS, the pH value, and the amount of surfactant (CTAB) on silica aerogels were investigated, respectively. The density and volume shrinkage of silica aerogels were greatly influenced by increasing the concentration of MTMS. Alkali catalyst was an extremely crucial factor for aerogels, and the gelation time of the wet gel steeply
decreased from 40 min to 2 min with the increase of the pH value from 7.5 to 10. The skeleton structures of aerogels formed were highly dependent on the amount of CTAB. As the concentration of CTAB increased, the skeleton of the prepared aerogel gradually transformed from coarsened spherical to continuous irregularity finer structures, leading to a sharp increase on BET-specific surface area. Furthermore, the silica aerogel showed outstanding anti-adhesion property and superhydrophobic ability with a WCA of 160.6 ± 1.3°. This study provided an eco-friendly approach to prepare low density, high specific surface area, and porous superhydrophobic silica aerogel, and the fabrication method could be highly beneficial for wide applications.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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