

Research Article

Preparation of Silica Aerogel and Its Adsorption Performance to Organic Molecule

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Hydrophobic and lipophilic silica aerogel was prepared from water-glass by gelling, aging, silylation, and drying under atmospheric pressure and characterized by FT-IR and SEM. The effect of preparation process on aerogel density and the aerogel density on contact angle of water on it were investigated in detail. pH 6 is most beneficial to shorten gelling time and to obtain the lowest density of silica aerogel. Increasing TEOS concentration of aging solution to 25 v% could decrease aerogel density to 0.093 g/cm³. The silica aerogel exhibits good hydrophobicity even though its density is 0.30 g/cm³. There are few changes in their adsorption capacities after 3 cycles of adsorption-desorption. The adsorption performance of the silica aerogel to organic solvent in water is different from in pure solvents. The critical surface tension (γ_C) of the silica aerogel prepared here is about 30.8 mN/m. If the surface tension of aqueous solvent solution (γ) is greater than γ_C , it will wet the aerogel surface partially. If $\gamma \leq \gamma_C$, the solution will wet all aerogel surface and be adsorbed well. This work delivers us a method to adsorb solvents from their waste water by adjusting the surface tension of the waste water to lower than γ_C of the adsorbent.

1. Introduction

Silica aerogel [1–4] is a nanostructured material with open foam-like structures having very low density (as low as 3 kg/m³), high specific surface area (700–1000 m²/g), and extremely high porosity (98%–99%) [5]. These features lead to unique properties due to excellent accessibility of the inner surface through open porous network. The most interesting feature of silica aerogel is the controllability of the surface polarity between hydrophobicity and hydrophilicity. Various applications such as thermal super insulation, catalyst support, microfilters, controlled release of drugs, and inertial confinement fusion targets have already been investigated and reported [6, 7]. Because of their very high specific surface area and porosity as well as hydrophobicity, silica aerogel is an ideal adsorbent for organic compounds.

Rao et al. investigated the adsorption and desorption of organic liquids on silica aerogel and found that the aerogel could adsorb the organic liquids and oils by nearly 15 times

of its own mass [8]. Wang et al. studied the equilibrium and kinetics of sorption of generic vegetable cooking oil, motor oil, and crude oil from the liquid phase and from an oil-in-water emulsion on two different particle-size hydrophobic silica aerogels and found that the aerogels had a very high capacity (up to 15.1 g oil/g aerogel) and rate (uptake time of 25–1200 s) for adsorption of liquid oils; thus they regarded the aerogels as a very attractive adsorbent for oil spill clean-up [9]. Wang et al. investigated the adsorption of six volatile organic compounds in vapor, liquid, and aqueous solution on hydrophobic aerogels and found that the rate for adsorption are very different, with the uptake time in the order of about 100 min for vapor, 10 min for solution, and 10 s for liquid. The slowest rate of adsorption for vapor is due to the fact that the aerogel has an extremely low thermal conductivity [10]. Štandeker et al. synthesized some silica aerogels and used them to adsorb toxic organic compounds from water. They found that the adsorption capacity of both hydrophobic silica aerogel adsorbents M9 and E6 exceeded the capacity of

comparable granular activated carbon, for all toxic organic compounds tested in ranges of factors from 15 to 400 [3]. Štandeker et al. assessed the ability of modified silica aerogel with mercapto group to remove Cu(II) and Hg(II) from aqueous solutions using batch adsorption technique under equilibrium conditions. They found the adsorbent exhibited very high adsorption potential for Cu(II) and Hg(II) and more than 99.0% removal was achieved in the pH range of 4–6 [11].

The common preparation of silica aerogel includes supercritical drying and expensive raw materials like tetraethoxysilane which prohibit its commercialization. There are capillary forces in the gel pores which make the gel shrink and even lead to the gel collapse when aerogel is dried [12]. Supercritical drying process can avoid the shrinkage and the collapse of aerogel completely, but its high cost makes it difficult to prepare aerogel in large scale. Schwertfeger et al. used water-glass combined with silylation and drying of the lyogel under atmospheric pressure to produce durably hydrophobic aerogel [13]. Their new process makes the aerogel production become cost-effective and more commercial.

Silica aerogel shows a very high uptake capacity for pure organic compounds. It adsorbs about 14.3 g benzene, 13.6 g toluene, or 14.4 g *p*-xylene per gram silica aerogel [10]. But its uptake capacity for organics from their aqueous solution cuts down sharply, for example, 87 mg benzene, 223 mg toluene, or 1064 mg *p*-xylene per gram silica aerogel, respectively [10]. It is the hydrophobicity of silica aerogel that prohibits the aqueous solution of organic compounds entering into the interior pores of silica aerogel, and only external space but not internal space of the aerogel adsorbs the organic compounds. This is the main cause of the decreased uptake capacity of organic compounds on silica aerogels. In the present study, we used water-glass as precursor, combined with silylation and drying of the lyogel under atmospheric pressure to prepare silica aerogel. The performances to adsorb organic compounds from their aqueous solutions on the aerogel were studied. The uptake capacity of these adsorbent will increase greatly in the presence of penetrating agent.

2. Experimental

2.1. Preparation of the Silica Hydrogel. The silica hydrogels were prepared through Shen's process with water-glass as starting material [14, 15]. One L water-glass solution (SiO₂ content 8 wt%, $n(\text{Na}_2\text{O}) : n(\text{SiO}_2) = 1 : 3.1$) was filtrated to remove solid impurities. 81 g water-glass solution was dropped into 4.5 g 35% hydrochloric acid with agitation at ambient temperature. After dropping, the mixture was continued agitation for 7 h to hydrolyze the water-glass. Then, some aqueous ammonia solution was added to adjust the pH value of the mixture. The hydrogel obtained was aged in distilled water for 2 h and in anhydrous alcohol for several hours at ambient temperature, respectively.

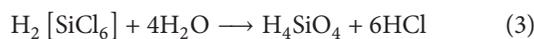
2.2. Silylation and Drying of the Silica Hydrogels. For the silylation, 5 g of the aged hydrogel sticks was placed together with 20 g hexamethyldisiloxane (HMDSO), 15 g trimethylchlorosilane (TMCS), and 50 mL hexane in a beaker at ambient temperature for 24 h. The resulted silylated lyogel sticks were dried at ambient temperature for 2 h, then elevated temperature at 1°C/min to 60°C, and stayed at 60°C for 2 h. After cooling to ambient temperature, the silica aerogel was obtained.

2.3. Characterization of the Silica Aerogels. The microstructure of the silica aerogel was observed by Hitachi S-4700 field emission scanning electron microscope (Hitachi). Specific surface and pore radius were measured by SP 1900 Physisorption Analyzers. Hydrophobicity was presented as water contact angle on the aerogel which was measured by OCA-20 optical contact angle measuring system (Dataphysics). Silylation efficiency was measured by NEXUS Fourier transform infrared spectroscopy (Thermo Nicolet). The bulk density (ρ_b) of the aerogel was measured using a known volume of the aerogel and its weight was measured with a microbalance, 10⁻⁵ g accuracy.

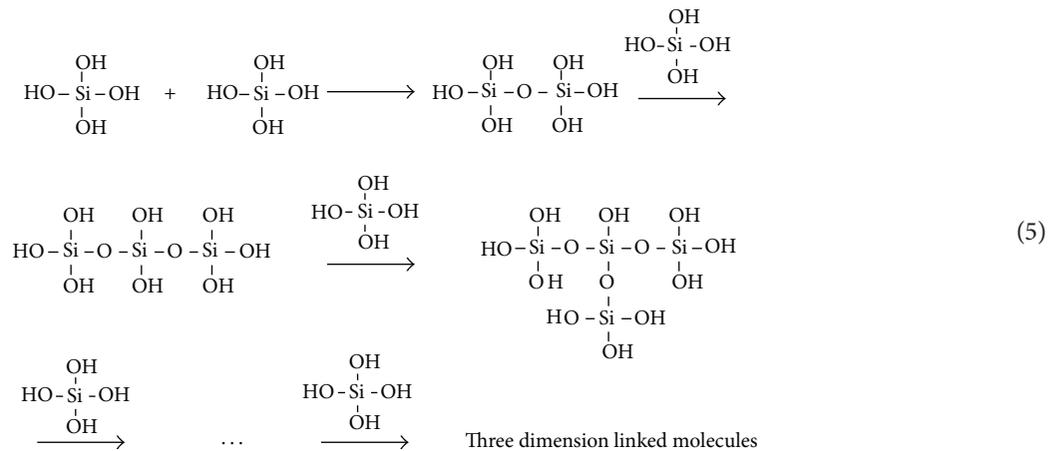
2.4. Adsorption Capacity Determination. The aerogel sample was kept in an organic solvent until it was completely wetted by the liquid. A conventional batch equilibration procedure was used as isotherm testing [16]. To achieve accurate results, the adsorption measurements were accomplished under equilibrium conditions.

3. Results and Discussion

Using low-cost industrial water-glass as silicon source to produce silicon aerogel, complete ionic exchange and water washing are necessary steps. But the process using water-glass with the two steps of acid-base catalysts (HNO₃-NaOH), without ionic exchange, and possessing surface modification under atmospheric condition to produce nanoporous silica aerogels was reported [14]. We used hydrochloric acid and aqueous ammonia solution (HCl-NH₄OH) to substitute HNO₃-NaOH. The hydrolyzation is presented as the reactions (1)~(3). Reactions (2) and (3) are the same as reaction (4):



After neutralizing with NH₄OH, the mixture gelled and polycondensed to three-dimension molecules (5).



The relation of gelling time to the pH value of the mixture was shown in Figure 1. When the mixture was acidic, for example, pH 4, hydrolyzation was fast, and the polycondensation of the orthosilicic acid (H_4SiO_4) was slow, the mixture gelled slowly and took about 260 min. The gelling time declined sharply with increased pH value and was 20 min at pH 6. When the pH value increased from pH 6 to pH 8, the gelling time declined very slowly, but white floccule appeared at pH 7 and white deposit occurred quickly at pH 8. The polycondensation rate increased with the increase of the pH value of the mixture, and when pH value is greater than 6, H_4SiO_4 condenses so quickly that it was easy to form gel particles, gathered to form white floccules (pH 7) and even a dense white deposit (pH 8). So, pH 6 is most proper for gelling. Good aerogel generally has very high porosity which produces its low density and high specific surface area. Shen has found that two steps of HNO_3 -NaOH catalysts with water-glass could result in lower aerogel density ($0.068\sim 0.096 \text{ g/cm}^3$) than that of quick step of HF ($0.327\sim 0.354 \text{ g/cm}^3$). We found that adding NH_4OH to the mixture could influence the aerogel density obtained. The effect of the pH value of the mixture on the aerogel density was shown in Figure 2. Figure 2 shows that gelling at pH 6 could prepare aerogel with the smallest density. Elevating or decreasing the pH value of the mixture made the aerogel density increase. It is clear that pH 6 is the best pH value in gelling. The hydrolyzation rate may be equal to the polycondensation rate at pH 6; the aerogel grew properly in three dimensions and formed some internal pores which made the aerogel density decline. If the pH value was smaller than 6, the hydrolyzation rate may be greater than the polycondensation rate; many aerogel particles appeared and grew at the same time which made aerogel particle become smaller and made internal pores reduce, so that the aerogel density increased. If the pH value was great than 6, for example, pH 8, the hydrolyzation rate may be smaller than the polycondensation rate, H_4SiO_4 polycondensed very quickly as soon as it produced from hydrolyzation, precipitation but

not aerogel growth took place, and few pores formed so that the aerogel density also increased.

Aging of hydrogel is very important operation for preparing silica aerogel. The polycondensation inside hydrogel does not finish completely without aging; then the hydrogel network will be not strong enough to endure the capillary force added by the surface tension of solvent during drying stage, which makes the aerogel shrink and dilapidate. Aging of hydrogel is a necessary step for aerogel to avoid shrinkage and dilapidation. In aging operation, hydrogel, were dipped into 0%, 5%, 15%, 25%, 35%, and 40% tetraethoxysilane (C_{TEOS} v%) solution in ethanol, respectively. The effect of C_{TEOS} v% of aging solution on the aerogel density was shown in Figure 3.

When pure ethanol was used, $C_{\text{TEOS}} = 0$ v%, the density was 0.15 g/cm^3 ; adding TEOS gradually, the density declined firstly and then increased; when $C_{\text{TEOS}} = 25$ v%, the density reached the smallest value, 0.093 g/cm^3 ; when $C_{\text{TEOS}} > 25$ v%, the density turned to increase. This is because there was water in hydrogel which made TEOS hydrolyze to $\equiv\text{Si}-\text{OH}$, $=\text{Si}(\text{OH})_2$, and $-\text{Si}(\text{OH})_3$; they reacted with the hydroxy groups of hydrogel to strengthen hydrogel network, so that to delimit the aerogel shrinkage during drying. Before aging, there were many interpores full of water in the hydrogel. During aging, the water in the pores could be substituted by ethanol. This substitution should be finished enough so that the aerogel could avoid shrinkage and collapse; then smaller density of aerogel could be obtained. When C_{TEOS} was lower, the water was substituted completely. When C_{TEOS} was higher, or C_{ethanol} was lower, the water could not be substituted completely. The higher the C_{TEOS} , the more the water remained in the pores and the more shrinkage and collapse of aerogel, then the greater the aerogel density.

The silica aerogel prepared is Cambridge blue solid with BET surface area of $838.6 \text{ m}^2/\text{g}$, pore volume of $1.072 \text{ cm}^3/\text{g}$, and average pore width of 5.11 nm . SEM image of the silica aerogel was shown in Figure 4. It is very clear that the silica aerogel is multiporous nanomaterial with concatenate

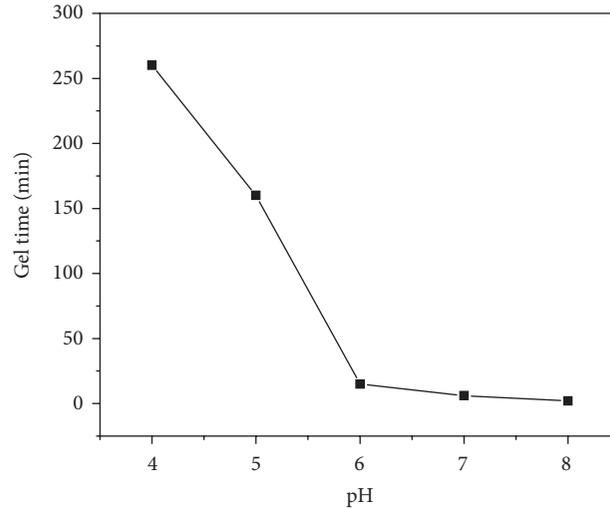


FIGURE 1: The relation of gelling time to the pH value of the mixture.

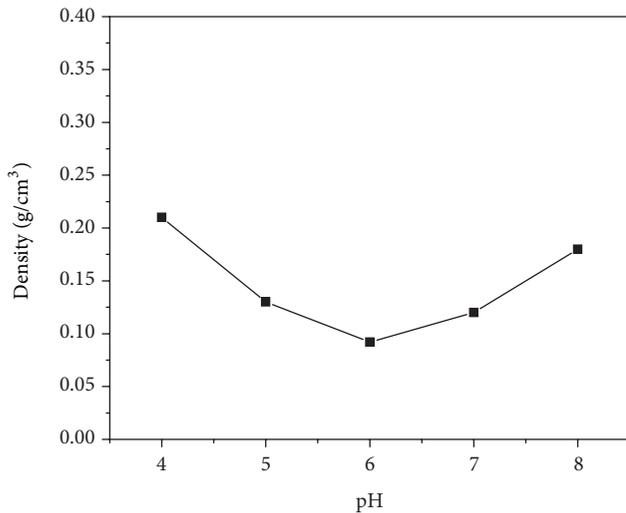


FIGURE 2: The relation of aerogel density to the pH value of the mixture.

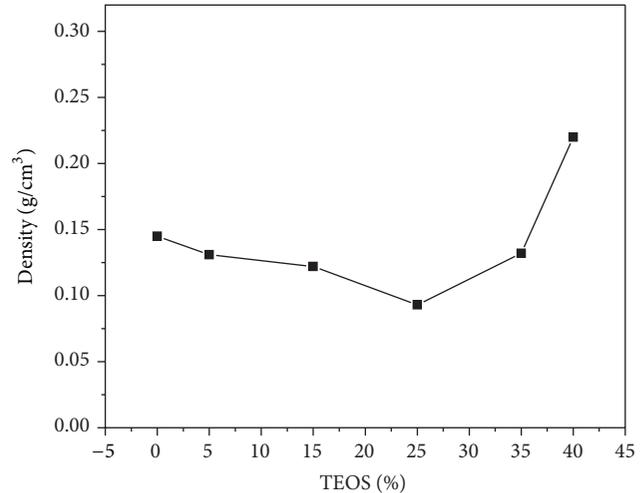


FIGURE 3: The relation of aerogel density to the TEOS concentration in aging solution.

network structure. Because the external and internal surfaces of the aerogel were treated with hexamethyldisiloxane (HMDSO) and trimethylchlorosilane (TMCS), it exhibited hydrophobicity. The image of water on the silica aerogel prepared was shown in Figure 5. The water contact angle on the aerogel was about 138°.

The water contact angle on aerogel is related to the preparation process containing silicon source, gelling, aging, silylation, and drying; all of that can influence the aerogel density, which determines the water contact angle on the aerogel and its hydrophobicity. We selected some silica aerogels with different densities ranged from 0.093 g/cm³ to 0.30 g/cm³ and measured their water contact angles which ranging from 138° to 110°. The relation of the water contact angle to aerogel density was shown in Figure 6. It is very clear that the water contact angle on the silica aerogel declines

gradually with the increase of its density. The lower the aerogel density, the greater the water contact angle. The silica aerogel presents good hydrophobicity even though its density is 0.30 g/cm³. This result tells us that controlling silica aerogel density can adjust its hydrophobicity.

Hydrophobic silica aerogel is lipophilic and can adsorb organic compounds. One g powdery active carbon, silica gel, and silica aerogel (0.084 g/cm³) prepared were added into pure toluene, xylene, chlorobenzene, DMSO, and MeOH at ambient temperature for 1h, respectively. After filtration in vacuum, the wet solid adsorbents were weighted and the adsorption capacities of each adsorbents were calculated, which were shown in Figure 7. For adsorbates, the adsorption capacity order is silica aerogel \gg silica gel > active carbon except for MeOH. Active carbon is a common adsorbent for organic adsorbates; its adsorption capacity is smaller than

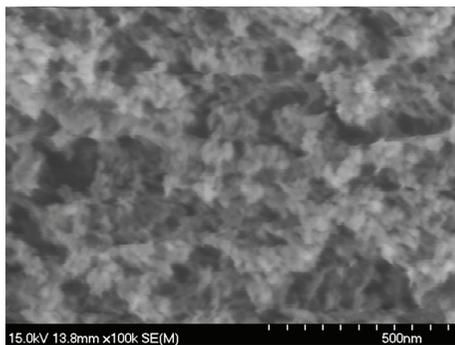


FIGURE 4: SEM micrograph of silica aerogel.



FIGURE 5: Image of water on the silica aerogel.

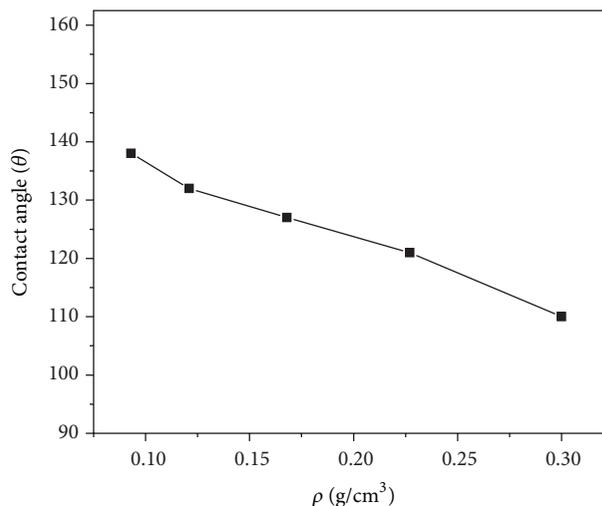


FIGURE 6: Water contact angle versus aerogel density.

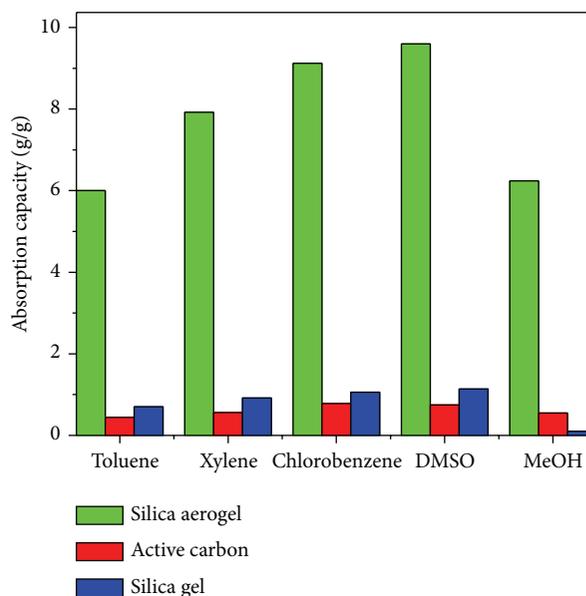


FIGURE 7: Adsorption capacities of various adsorbents.

1 g/g for organic compounds tested. But the corresponding adsorption capacity of silica aerogel is equal to or greater than 7 g/g. The aerogel can especially adsorb about 10 times of chlorobenzene compared to 0.4 time for active carbon. Silica aerogel exhibits excellent adsorption performance.

After adsorption, organic adsorbates were desorbed from silica aerogel at ambient temperature. Elevated temperature will quicken desorption. For example, it takes about 220 min (at 25°C) and 10 min (at 100°C) for toluene desorption, respectively. The images of silica aerogel in different states of adsorbing and desorbing toluene were shown in Figure 8. After absorbing toluene, the volume of aerogel became a little bigger and its color looked gloomy. After desorption, the appearance of aerogel seemed to return to its original state.

After desorption, the silica aerogel was applied to adsorb organic compounds again and their adsorption capacities were listed in Table 1. There are few changes in their adsorption capacities among the first, the second, and the third cycles.

Various aqueous solutions of THF, DMF, acetone, CH₃CN, ethanol, AcOH, and MeOH were prepared. 0.05 g silica aerogel was added into 20 mL solutions at ambient temperature for sufficient time, respectively, and the adsorption capacities were calculated. The capacities of silica aerogel to adsorb solvent from their aqueous solution in different volume concentrations (C_{Solvent}) were recorded in Figure 9.

It is shown that when C_{Solvent} is lower than 30 v%, the adsorption capacities are similar and smaller than 1 g/g. Increasing C_{Solvent} , especially from 30 v% to 50 v%, the adsorption capacities increase sharply. If C_{Solvent} continues to increase, the adsorption capacities continue to increase slowly. The performances of aerogel in adsorbing pure solvents and aqueous solvent solutions are very different. In adsorption equilibrium, the adsorbing capacities in aqueous solvent solutions are very lower than those of corresponding pure solvents. For example, the capacity to adsorb 30 v% ethanol solution is about 0.85 g/g, but 7.00 g/g for pure ethanol. The adsorbing performance of aerogel to DMF solution is very interesting. When $C_{\text{DMF}} < 75$ v%, little adsorption takes place, but when C_{DMF} increases a little, the adsorption capacity increases sharply. When $C_{\text{DMF}} = 80$ v%, the adsorption



FIGURE 8: Images of aerogel adsorbing and desorbing toluene.

TABLE 1: The adsorption capacities of silica aerogel.

No	Adsorbate	Adsorption capacities (g/g)		
		First cycle	Second cycle	Third cycle
1	Chlorobenzene	10.12	10.06	10.01
2	DMSO	9.60	9.51	9.44
3	Xylene	8.92	8.89	8.82
4	Methanol	7.24	7.19	7.11
5	Toluene	7.00	6.94	6.95

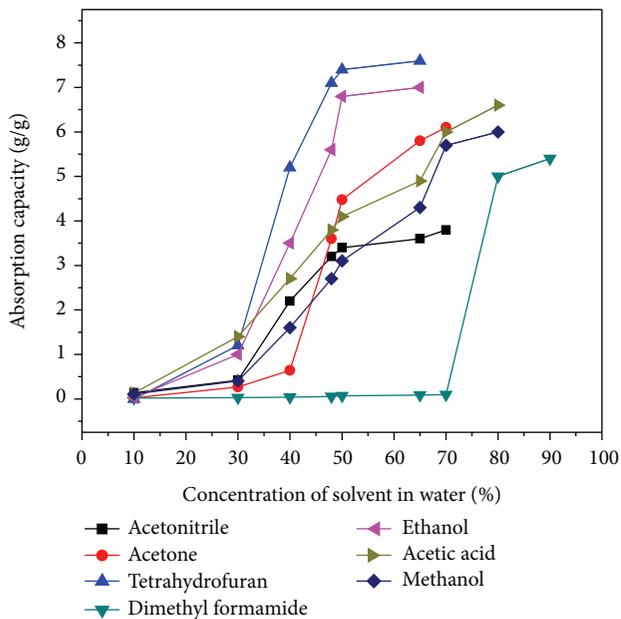


FIGURE 9: Capacities of silica aerogel adsorbing solvents from their aqueous solution.

capacity reaches to 5.0 g/g. The differences of adsorption capacities of aerogel between pure solvents and aqueous solvent solutions may be caused by surface hydrophobicity and lipophilicity of silica aerogel.

Figure 10 presents the balance of forces at the solid-vapor, solid-liquid, and liquid-vapor interface. If the contact angle is above 90° , there is no spreading of the liquid and the aerogel is said to be impervious. When the contact angle is less than 90° , the liquid spreads on the aerogel surface and when the

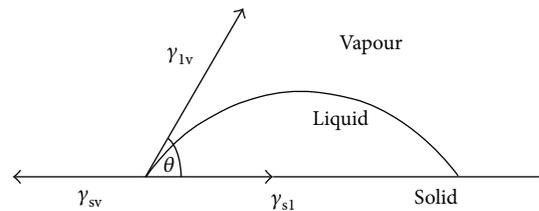


FIGURE 10: Balance of forces at the contact line solid-liquid-vapor system.

liquid is completely adsorbed by the aerogel, the contact angle is equal to zero. The imperviousness of the aerogels against solvents in water observed in various concentrations was researched by the contact angle measurements. Table 2 shows the contact angle (θ) of aqueous solution with different percentage of solvents (C_{Solvent} , v%). It is very clear from Table 2 that, with the increase of C_{Solvent} , the contact angle decreases. The aerogel is impervious against the solvents in water, the concentrations of which are up to 37 v% (THF), 48 v% (acetone), 34 v% (CH_3CN), 74 v% (DMF), 35 v% (ethanol), 26 v% (acetic acid), and 39 v% (methanol), respectively.

The mass of the organic liquid adsorbed by aerogel depends upon the surface tension (γ) of the corresponding liquid. From Young's equation [17],

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos \theta, \quad (6)$$

where γ_{sv} , γ_{sl} , and γ_{lv} are solid-vapor, solid-liquid, and liquid-vapor interfacial energies, respectively (Figure 10). If the material has a low energy surface ($\gamma_{sv} < \gamma_{sl}$), the contact angle (θ) will be $>90^\circ$ and the liquid cannot spread on the surface but remain in the form of a drop. This phenomenon

TABLE 2: Contact angle measurements for silica aerogel with different solvents in water.

Solvent	C_{Solvent} (v%)	Contact angle ($^{\circ}$)
THF	0	153
	10	150
	30	137
	37	90
	40	82
	48	Adsorption
Acetone	10	147
	30	145
	40	137
	48	90
	50	85
	65	Adsorption
CH ₃ OH	10	147
	30	126
	39	90
	40	86
	50	69
	70	34
CH ₃ CN	73	Adsorption
	10	150
	30	130
	34	90
	40	69
C ₂ H ₅ OH	50	Adsorption
	10	148
	30	110
	35	90
	40	70
	50	40
DMF	53	Adsorption
	10	148
	50	141
	70	138
	74	90
	75	80
CH ₃ COOH	90	Adsorption
	10	143
	26	90
	30	82
	40	63
	50	41
	70	21
	78	Adsorption

is observed when C_{Solvent} is low, for example, 10 v%. If the material has a high energy surface ($\gamma_{\text{sv}} > \gamma_{\text{sl}}$), the contact angle (θ) will be less than 90° and the liquid can spread on the surface. For example, when $C_{\text{Methanol}} = 70$ v%, the aqueous methanol solution spreads on the aerogel. If the material has a very high energy surface ($\gamma_{\text{sv}} \gg \gamma_{\text{sl}}$), the contact angle (θ) will

TABLE 3: The lowest concentration of solvents solution in which $\theta = 0$ and their surface tensions.

Solvent	Ethanol	Methanol	THF	Acetone
C (v%)	53	73	48	65
γ (mN/m)	29.6	31.0	31.9	30.9

be zero and the liquid is fully adsorbed by the material. For example, when $C_{\text{Methanol}} = 73$ v%, methanol was adsorbed completely by the aerogel.

Table 3 shows the lowest concentrations of ethanol, methanol, THF, and acetone in water in which the solvent solutions can be adsorbed by the silica aerogel (i.e., $\theta = 0$). We measured the surface tensions of these aqueous solutions and listed these results in Table 3. From the data, we found that the average surface tension is about 30.9 mN/m which equals the critical surface tension (γ_C) of the silica aerogel according to Zisman's rule [18]. If the surface tension of aqueous solvent solution $\gamma > \gamma_C$, the solution will wet the aerogel surface partially. If $\gamma \leq \gamma_C$, the solution will wet all of the aerogel surface. If $\gamma \gg \gamma_C$, the solution hardly enters inside aerogel and wets few aerogel surface, and the contact angle of the solution drop is greater than 90° , so aerogel adsorbs little solvent from the solvent solution in water. We prepared 20 v% solvent solutions in water, using penetrating agent A (delivered by Taizu New Materials Co., Ltd) to adjust the surface tensions of these solutions and measured the capacities of aerogel adsorbing the solvents in water before and after adjustment, respectively. The results were listed in Table 4 and show that the surface tensions of aqueous 20 v% solvent solutions are greater than γ_C , so their adsorption capacities are very small; once adding penetrating agent A in the solvent solutions, the surface tensions decline sharply to much smaller than γ_C , and the adsorption capacities are increased sharply. This deliver us a process to adsorb solvents from their waste water by adjusting the surface tension of the waste water to much lower than γ_C of the adsorbent. This will be very useful and practical to deal with the waste water containing some organic compounds.

4. Conclusion

Hydrophobic and lipophilic silica aerogel was prepared from water-glass by gelling, aging, silylation and drying at atmospheric pressure. The pH value influence the gelling time of water-glass mixture. pH 6 is the best value to shorten gelling time and to obtain the lowest density of silica aerogel. Increasing TEOS concentration of aging solution to 25 v% could decrease aerogel density to 0.093 g/cm^3 . The preparation process will influence the aerogel density which determines the water contact angle on the aerogel, that is, its hydrophobicity. The water contact angle on the silica aerogel declines gradually with the increase of its density. The silica aerogel exhibits good hydrophobicity even though its density is 0.30 g/cm^3 . The adsorption capacities of silica aerogel are equal to or greater than 7 g organics tested/g adsorbent. There are few changes in their adsorption capacities after 3 cycles of adsorption-desorption. The performances of aerogel

TABLE 4: The adsorption capacities of 20 v% solvent solutions in water in different surface tensions.

Solutions	20 v% solvent		20 v% solvent—6 g/L penetrating agent	
	γ (mN/m)	Adsorption capacity (g/g)	γ (mN/m)	Adsorption capacity (g/g)
CH ₃ CN	33.67	0.3	20.14	4.0
Acetone	35.54	0.2	21.22	6.2
THF	32.21	0.7	19.94	7.4
DMF	48.98	0	23.20	5.2
Ethanol	38.65	0.5	22.12	6.3
CH ₃ COOH	41.72	0.8	22.47	6.4
Methanol	45.83	0.3	23.11	5.8

adsorbing to solvent solutions in water are different from those in the pure solvents. When C_{Solvent} is lower than 30 v%, the adsorption capacities are similar and smaller than 1 g solvent/g adsorbent. The critical surface tension (γ_C) of the silica aerogel prepared is about 30.8 mN/m. If the surface tension of aqueous solvent solution (γ) is greater than γ_C , it will wet the aerogel surface partially. If $\gamma \leq \gamma_C$, the solution will wet all the aerogel surface. Adjusting surface tension of the solvent solution in water to much smaller than γ_C of aerogel, the aerogel will adsorb solvent efficiently from its aqueous solution.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgment

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