

Water vapor sorption on surfactant-templated porous silica xerogels

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ABSTRACT

Porous silica xerogels are prepared by sol–gel syntheses templated with Brij56 or F127 surfactants. The porous properties of the obtained silica xerogels are characterized by N₂ sorption measurements. The F127 surfactant-templated silica xerogel exhibits a pore size distribution peaking at 4.2 nm in the mesopore region, a pore surface area of 510 m² g⁻¹, and a pore volume of 0.545 mL g⁻¹, while the Brij56 surfactant-templated silica xerogel has a bimodal pore size distribution with maxima at 0.65 nm and near 2 nm in the micropore region, a pore surface area of 739 m² g⁻¹, and a pore volume of 0.354 mL g⁻¹. Their water vapor sorption properties are evaluated under various relative humidity at 25 and 35 °C using gravimetric technique. The results show that the F127 surfactant-templated xerogel with larger pore size and higher volume could absorb a large amount of water molecules at high humidity and efficiently release it at low humidity, such that it has a large water adsorption–desorption capacity, whilst the Brij56 surfactant-templated xerogel with smaller pore size and larger pore surface area could maintain a high water content even at low humidity levels.

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1. Introduction

Water vapor sorption properties in porous materials have attracted much attention, because they relate to a number of applications, such as gas drying, humidity control, adsorption heat pumps, and the production of fresh water from the air [1–5]. Among porous materials, activated carbon has been widely utilized [1,6], but it tends to be flammable and nondurable. Inorganic compounds, such as halides and sulfates forming hydrates with water molecules [7], display good water adsorption capabilities, but these materials are incompatible with the human body. Hence, considering practical applications, especially those concerning the living environment, foods, and cosmetics, there is a strong need to develop harmless, low-cost porous inorganic materials with controllable water sorption capacities.

The primary consideration in setting up any porous materials for adsorbent and humectant applications should be able to control the pore features including the pore size, the surface area and the pore volume, which determine the water vapor adsorption–desorption properties. Many researchers have analyzed the water vapor sorption of various porous materials, such as activated carbon [1,6], silica gel [4], Vycor glass [8], and γ -almina [9], but the pore feature of the porous materials was hard to control. Many studies have been devoted to the development of sol–gel porous materials templated by the self-assembly of surfactant molecules

[10–13]. It has been confirmed that surfactant-templated approach for the production of the porous materials could tailor very precisely the pore feature by tuning the length of the surfactant molecules as pore-forming agents [3,11–13]. Such porous materials have been reported to be usable for catalysis, drug delivery, separation, and in sensors and fuel cells [14–18]. However, there have been few studies concerning the water vapor sorption properties of the surfactant-templated porous silica materials [3].

In the work described herein, we have prepared silica xerogels with different porous configurations, in which two kinds of low-cost commercially available non-ionic surfactants, namely alkyl poly(ethylene oxide) oligomeric Brij56 (C₁₆H₃₃(OC₂H₄)₁₀OH, Aldrich) and poly(ethylene oxide)–poly(propylene oxide)–poly(ethylene oxide) triblock copolymer F127 (HO(OC₂H₄)₁₀₆(OC₃H₆)₇₀(OC₂H₄)₁₀₆OH, BASF), were used as templates. The relationship between the porous structures and the water vapor sorption properties of the prepared silica xerogels has been investigated. As a common feature derived from the selective both surfactants, Brij56 and F127, the resultant porous silica materials possess ordered pore structures that are connected in a three-dimensional network [11], so as water molecules from ambient moisture could enter the interior through accessible pore channels [19].

2. Experimental section

2.1. Preparation

The surfactants used as templates were F127 and Brij56. An F127 surfactant-templated silica sol solution was prepared by

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mixing tetraethoxysilane (TEOS, Aldrich), distilled water, hydrochloric acid, and F127 surfactant. The molar ratio was 1 TEOS:74 H₂O:0.13 hydrochloric acid:0.01 F127. The mixture was stirred vigorously for 2 h at room temperature to obtain a clear sol. A Brij56 surfactant-templated silica sol solution was prepared as follows. First, TEOS, propanol, water, and HCl were mixed in a 1:3.8:1:8 × 10⁻⁵ molar ratio. Then, a solution of Brij56 surfactant in propanol was added to the prepared sol. The mixture was stirred for 1 h at room temperature. The final molar ratio was 1 TEOS:11.4 propanol:5 H₂O:0.004 hydrochloric acid:0.1 Brij56. In both cases, the precursor solution was poured onto a hot-plate at 100 °C to form a wet-gel. Finally, the porous silica xerogels were obtained after calcination of the wet-gels at 500 °C for 1 h under ambient conditions to remove the surfactants.

2.2. Characterization

The infrared (IR) spectra of the silica xerogels were recorded on an EQUINOX 55 FTIR spectrometer (Bruker Co.). The porous structures of the samples were characterized by means of a nitrogen adsorption–desorption apparatus (ASAP 2010 M+C, Micromeritics Inc.). The samples used were pre-treated at 150 °C under vacuum until completely degassed. BET specific surface areas were determined from N₂ adsorption branch. Pore size distributions and pore volumes were evaluated using BJH model. In addition, Horvath–Kawazoe sorption model was used to characterize the micropores of the Brij56-templated silica xerogel.

To examine the water vapor adsorption–desorption properties of the silica xerogels, the weight changes of the samples at various relative humidity levels were monitored. Before the water sorption measurements, the silica xerogels were slowly dried at 90 °C under vacuum conditions. The dried samples were placed in enclosed glass boxes containing saturated solutions of appropriate salts (MgCl₂·6H₂O for 33% RH; Ca(NO₃)₂ for 51% RH; KCl for 84% RH), within a thermostatically controlled chamber at constant temperatures (25 and 35 °C). The weights of the dried samples and those taken from the humidified glass box at intervals were measured on an FA 2104S analytical balance with a sensitivity of 0.1 mg, so that the actual water content within the silica xerogels with exposure time could be obtained. After saturating with water vapor of 100% RH for 2 days, desorption measurements on the samples were carried out in a similar manner as the adsorption measurements. Three replicate samples are evaluated for each water vapor sorption experiment. The water sorption properties were evaluated by calculation according to the following formula.

$$\text{Water uptake(or retention)} = (W_{\text{wet}} - W_{\text{dry}})/W_{\text{dry}} \text{ (g/g)} \quad (1)$$

where W_{wet} is the weight of samples sorbed under various humidity conditions, and W_{dry} is the weight of samples dried at 90 °C under vacuum conditions.

3. Results and discussion

3.1. Synthesis of silica xerogels

Non-ionic surfactants can serve as useful templates for preparing porous silica materials [11–13]. In the present study, we used oligomeric Brij56 and triblock copolymer F127 non-ionic surfactants as templates. The silica xerogels were prepared by sol–gel synthesis from TEOS and surfactant in solution, with subsequent annealing on a hot-plate at 100 °C and final calcination at 500 °C under ambient conditions to remove the surfactant.

In the sol–gel process, TEOS is converted to various hydrophilic oligomers (Si(OR)_x(OH)_{4-x}, $x = 0-4$) by partial hydrolysis. In the annealing stage, polymerization and condensation between the hydrophilic oligomers causes cross-linking, and ordered silica/surfactant mesophases are formed by evaporation-induced self-assembly [20]. The porous configuration of the silica xerogels was created by elimination of the organic surfactant template upon calcination at 500 °C.

3.2. Structural characterization

The formation of the silica skeleton structure and complete elimination of surfactant molecules from the silica xerogels was confirmed by IR spectroscopy. Fig. 1a and b shows the IR spectra of the wet gels and the corresponding xerogels after calcination, obtained using Brij56 and F127 surfactants as templates, respectively. Characteristic peaks of the Si–O–Si bond at around 1080 and 800 cm⁻¹ appear in each of the spectra, indicating the formation of a silica skeleton structure [21]. An absorption shoulder at 960 cm⁻¹ arises from the Si–O stretch of silanol groups, Si–OH [22]. An absorption peak at 1640 cm⁻¹ can be assigned to the bending mode of adsorbed water molecules, H–O–H [23]. For both of the wet gel samples, absorption bands are seen at around 2800–3000 cm⁻¹, which can be assigned to the C–H stretching vibrations of the surfactant template and unhydrolyzed ethoxy groups (OC₂H₅). After calcination, these peaks disappear completely. This confirms that the surfactants bearing organic moieties are

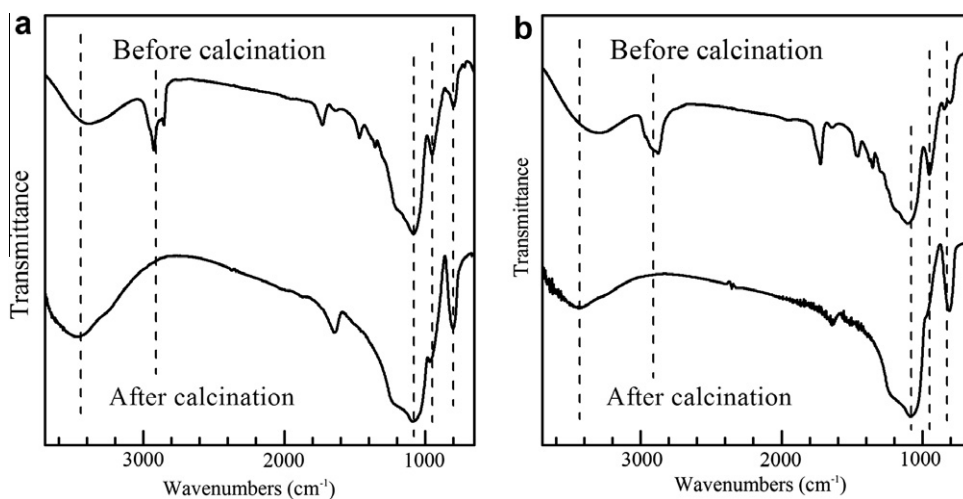


Fig. 1. IR spectra of silica wet gels and corresponding xerogels templated using Brij56 (a) and F127 (b) surfactants, respectively.

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