

Comparative Analysis of Superhydrophobic Elastic Polymethylsilsesquioxane Aerogels Prepared at Atmospheric Pressure and under Supercritical Conditions

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Abstract—The influence of the conditions of drying elastic polymethylsilsesquioxane aerogels (at atmospheric pressure, during supercritical drying in CO₂ or methanol) on their structure (including porosity and specific surface area), hydrophobicity, and mechanical compressive properties is investigated. The structure and physicochemical properties of the aerogels prepared under different conditions are found to differ insignificantly, which demonstrates the possibility of producing elastic highly porous superhydrophobic materials without the use of expensive equipment for supercritical drying. The polymethylsilsesquioxane aerogels synthesized by supercritical drying in methanol have the best mechanical characteristics under compression (ultimate strength, relative deformation).

Keywords: methyltrimethoxysilane, polymethylsilsesquioxane aerogels, supercritical drying, mechanical properties, hydrophobicity, elasticity, fracture

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INTRODUCTION

Aerogels are the gels in which a liquid phase is replaced by a gas as a result of drying in a supercritical fluid. When the critical values of temperature and pressure are reached, the liquid–vapor interface disappears and the removal of a solvent does not lead to failure of a gel structure under the action of surface tension forces [1]. This approach makes it possible to preserve the hierarchical open highly porous structure of the gel, which consists of micro- and mesopores [1] and determines a unique set of properties (low density, high specific surface area, high porosity, low sound conductivity and thermal conductivity) that are important for the use of aerogels as insulating materials [1, 2].

Silica-based aerogels, which have a low thermal conductivity (on the order of 0.01 W/(m K)) and retain high textural and functional properties over a wide temperature range from –273 to 600°C [3], have received the most attention. Tetramethoxysilane and tetraethoxysilane are usually used as precursors for the production of silica aerogels; however, such gels are characterized by hydrophilicity and a low mechanical strength [4, 5]. In turn, aerogels synthesized on the

basis of methyltrimethoxysilane (MTMS), or polymethylsilsesquioxane (PMSQ) aerogels, are characterized not only by pronounced hydrophobic and superhydrophobic properties, but also by reversible deformation ability (elasticity) [6–11]. The conditional chemical formula of PMSQ aerogels is SiO_{1.5}(CH₃) [7–12].

The hydrophobicity and elasticity of PMSQ aerogels are caused by the presence of hydrolytically stable Si–CH₃ fragments, which provide a relatively low degree of cross-linking of silicon–oxygen polymer structures, in their composition. Depending on the method of preparation, the contact angle between water and PMSQ aerogels is 135°–175°. In addition, such aerogels are characterized by high chemical resistance to acids and organic solvents [12]. An important feature is the possibility of synthesis of PMSQ aerogels at atmospheric pressure, which eliminates the need to use expensive installations required for supercritical drying [6].

As compared to other silicon-containing aerogels, the structure of PMSQ aerogels is more stable during uniaxial compression and bending. The high mechanical properties of these materials are due to the low

degree of cross-linking of the polymer network, which allows some structural elements to move freely relative to others during mechanical deformation [13]. The high content of methyl groups and the low content of hydroxyl groups in the polymer composition decrease the degree of its irreversible shrinkage during compression and promotes geometric shape recovery after removal of the load. In addition, a significant role is played by the high porosity of the material, which prevents destruction during compression [13]. Young's modulus of PMSQ aerogels usually varies in the range from 3 to 140 kPa and is determined, among other things, by the geometric density of the material. The ultimate compressive strength of such aerogels is $\sigma_u^{\text{com}} = 0.05\text{--}0.2$ MPa [14–19].

Prospects for the use of PMSQ aerogels are associated, first of all, with the production of heat-insulating materials, including moisture-resistant ones. At the same time, they are considered as highly effective sound-absorbing fillers and of vibrated insulating system components [20–22]. Moreover, the excellent sorption properties of such aerogels with respect to nonpolar organic compounds allow them to be used to eliminate the spills of lubricating oils and petroleum products [21].

Despite the wide possibilities of practical application of PMSQ aerogels, their mechanical behavior, especially under cyclic load conditions, was studied in a few works [23]. The problem of the influence of a drying method on the mechanical properties of such aerogels is of considerable interest. Therefore, the purpose of this work is to analyze the influence of the conditions of drying PMSQ aerogels on their structure, mechanical compressive properties, and textural and functional characteristics (including specific surface area, porosity, hydrophobicity).

EXPERIMENTAL

The aerogels were prepared as follows. MTMS ($\geq 98\%$) in an amount of 1.018 mL (0.007 mol) was dissolved in 1.605 mL (0.021 mol) special purity grade methanol, and a mixture of 0.022 mL (0.0005 mol) 40% aqueous solution of special purity grade HF acid and 0.5 mL (0.028 mol) distilled water was added to the resulting solution and stirred for 1 h. Then, 0.064 g (0.00067 mol) reagent grade ammonium carbonate preliminarily dissolved in 0.6 mL (0.034 mol) distilled water was added to the mixture and additionally stirred for 1 min. The resulting sols (3–5 mL) were moved to cylindrical polypropylene containers to produce gels. Gel formation took ≈ 40 min. The gels were stored at room temperature for 24 h and then washed with isopropanol once a day for 5 days. The resulting gels were dried according to the following three methods: at atmospheric pressure and 23°C and under supercritical conditions, namely, in CO₂ at a pressure of 15 MPa

and 55°C or in methanol at a pressure of 9–10 MPa and 255–265°C.

A microstructure was studied by scanning electron microscopy (SEM) using a Carl Zeiss NVision 40 microscope at an accelerating voltage of 1 kV.

The specific surface area S_{sp} of the aerogels was determined using low-temperature nitrogen adsorption, an ATX-06 analyzer (KATAKON, Russia), and the Brunauer–Emmett–Teller model at six points in the partial nitrogen pressure range 0.05–0.25. Before measurements, samples were degassed in a dry helium flow at 200°C for 30 min. Bulk density ρ was determined by measuring the geometric dimensions of cylindrical samples accurate to 0.1 mm and by weighing accurate to 0.1 mg. The skeletal density of the aerogels ρ_s was determined with a Quantachrome Ultrapycnometer 1000 helium pycnometer. The porosity was calculated by the relationship $\Pi = (1 - (\rho/\rho_s)) \times 100, \%$.

The contact angle of wetting with water θ was determined on an FTÅ200 device (First Ten Angstroms Inc., United States) using water droplets with a volume of 20 μL .

The mechanical properties of the aerogels (ultimate compressive strength σ_u^{com} , relative deformation ϵ_{com}) were studied during compression on a universal Instron 3382 testing machine at a loading rate of 5 mm/min in accordance with GOST 4651–2014 (ISO 604:2002). Aerogel specimens in the form of cylinders 5 mm in diameter and 25 mm in height were installed between the support areas so that the vertical axis of the specimen coincided with the loading direction. The results were processed with the Instron Bluehill 2.0 software. The relative error of stress measurements did not exceed 0.5%. Each value was obtained by averaging the results of testing five samples.

Cyclic compression tests were carried out in under loading–unloading conditions at constant strain from 0 to 30% in 10 cycles at a speed of 5 mm/min using the universal Instron 3382 testing machine.

RESULTS

According to SEM data, the microstructures of all aerogels are almost similar regardless of the drying conditions. The aerogels consist of chains of cross-linked particles up to several μm in size (Fig. 1), which is typical of the materials produced by hydrolysis of MTMS [24–26]. The porous structure of the materials under study is open and has a hierarchical character. The sizes of the pores located between chains of particles and between chain pileups are 0.02–3 and 2–10 μm , respectively. This is slightly larger compared to the results from [24, 25], where a particle size of 50–400 nm and a pore size of 0.1–3 μm were reported. Such differences can be related to the peculiarities of the technology of producing aerogels (gel formation

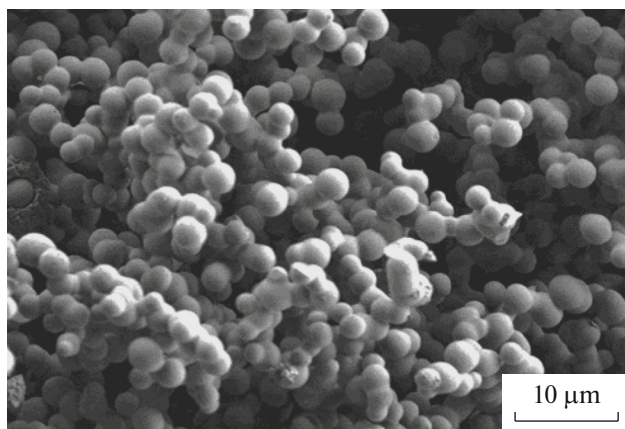


Fig. 1. Typical microstructure of PMSQ aerogels.

catalyst, the ratio of solvent to methyltrimetoxysilane, use of surfactants, etc.).

The physicochemical properties of the aerogels prepared under different drying conditions are comparable: the geometric density is $\approx 0.2 \text{ g/cm}^3$, the specific surface area is 460–480 m^2/g , and the porosity is 86–88% (Table 1). It should be noted that the obtained texture characteristics correspond satisfactorily to those given in [17, 23, 24, 27].

For all samples, the water contact angle is approximately the same, $\theta = 152^\circ\text{--}155^\circ$ (Fig. 2), which makes it possible to attribute the fabricated aerogels to superhydrophobic ones [8, 10, 24]. The identical high hydrophobic properties of the synthesized materials are caused by both their close chemical compositions, namely, the presence of methyl groups on the surface, and similar microstructures [6–11, 24, 25].

The stress–strain curves of all aerogels under static compression are also almost identical (Fig. 3). The strength and plastic characteristics of the aerogels prepared by drying both at atmospheric pressure and under supercritical conditions are quite close to each other (see Table 1) and are comparable with those in [13–19, 28, 29]. The aerogels fabricated by drying in supercritical methanol have the highest mechanical properties, $\sigma_u^{\text{com}} \approx 0.35 \text{ MPa}$ at $\epsilon_{\text{com}} \approx 60\%$. This finding can be related to lower porosity, smaller specific surface area, and higher density of the material. This difference is obviously due to the high solubility of silicon oxide gels in organic solvents at elevated tempera-

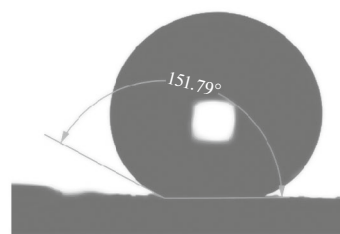


Fig. 2. Contact angle of PMSQ aerogel.

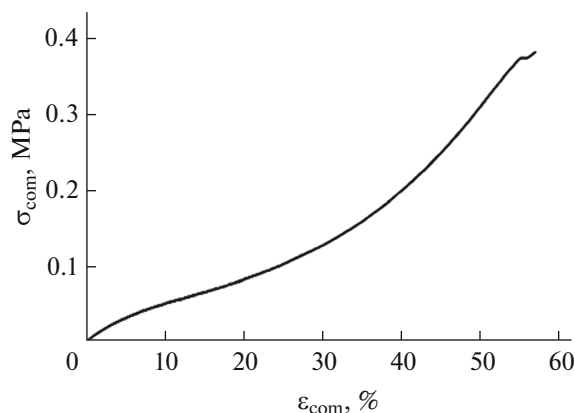


Fig. 3. Typical stress–strain curve of PMSQ aerogels (drying under supercritical conditions in methanol).

tures, which leads to hardening as a result of aging processes and chemical modification of a structure [30–32].

The data of cyclic tests of the aerogels are very encouraging (Fig. 4). After the first cycle, the irreversible deformation of a specimen was 3.7%, and the sample acquired a barrel shape. In the next cycles, there was only a slight increase in the degree of irreversible deformation; after 10 loading–unloading cycles, it reached 6% at the corresponding increase in the diameter. Note that the storage of the specimen subjected to 10 loading–unloading cycles for 3 days at completely removed stresses led to deformation relaxation: the relative residual deformation decreased to 2%, and the initial cylindrical shape of the specimen was largely recovered. Such high elasticity and reversible deformation ability are characteristic of MTMS-based aerogels, which contain hydrolytically stable

Table 1. Properties of the PMSQ aerogels

Drying conditions	ρ , g/cm^3	ρ_s , g/cm^3	S_{sp} , m^2/g	Π , %	σ_u^{com} , MPa	ϵ_{com} , %
Atmospheric pressure	0.191 ± 0.001	1.50 ± 0.01	468 ± 15	87.2 ± 0.5	0.20 ± 0.02	47 ± 2
Supercritical CO_2	0.193 ± 0.003	1.50 ± 0.02	479 ± 20	88.1 ± 0.6	0.30 ± 0.02	53 ± 2
Supercritical methanol	0.202 ± 0.002	1.50 ± 0.01	460 ± 20	86.5 ± 0.3	0.35 ± 0.02	60 ± 2

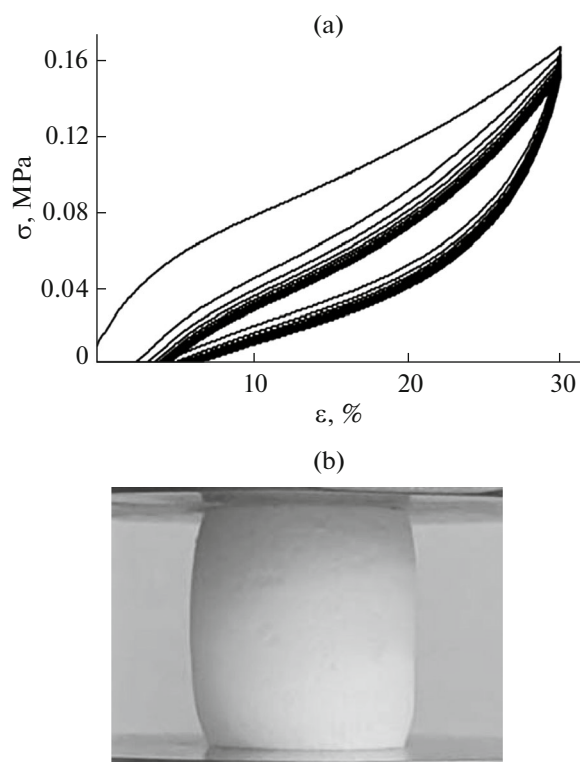


Fig. 4. (a) Stress–strain curves for 10 loading–unloading cycles for PMSQ aerogel at a given compressive strain of 30% and (b) specimen after deformation by 30%.

Si–CH₃ bonds, and are generally atypical of other silicon oxide aerogels [20].

After significant deformation (up to 60%), the specimens undergo fracture with the formation of a shear macrocrack at an angle of 45°, and the fracture surface has a typical river pattern (Fig. 5). An analysis of the microstructure of the fracture surface allows us to conclude that the predominant mechanism of microfracture is the separation of particles from each other at the sites of their connection into chains (Fig. 6a). However, some interparticle contacts can be very strong, and fracture in this case can occur throughout the particle volume (Fig. 6b).

DISCUSSION

The mechanical properties and deformation behavior of aerogels substantially depend on their microstructure and the composition of an organosilicon polymer. From the chemical point of view, the closest analog of the aerogels under study are cross-linked polymers, the mechanical properties of which are determined by the degree of cross-linking. Due to the flexibility of macromolecules, polymers with a low degree of cross-linking have high elastic properties and the possibility of shape change under external forces. In turn, when the degree of cross-linking increases, the stiffness of the material increases correspondingly.

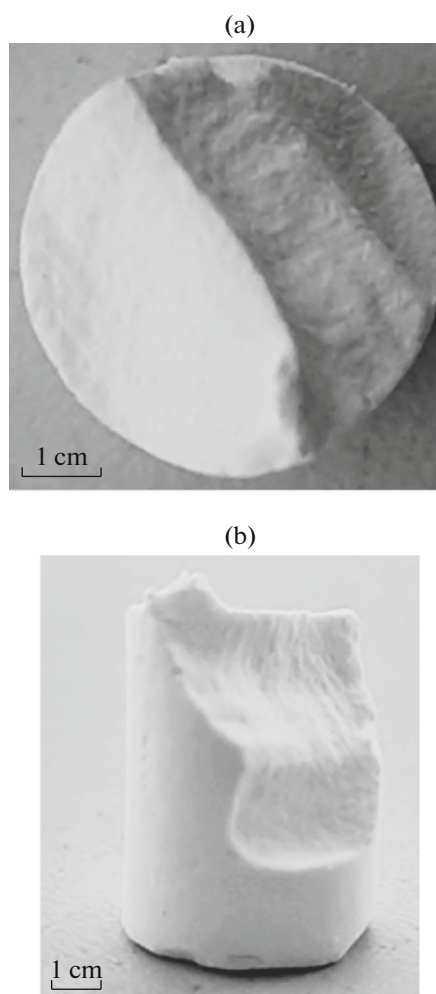


Fig. 5. Fracture surface of a PMSQ aerogel specimen.

The general shape of the stress–strain curves of aerogels during compression (see Fig. 3) is very close to that of the curves typical of cross-linked polymers [33, 34], and their macrofracture patterns are also similar. On the other hand, another class of ultralight materials, namely, polymer foams with open porosity, in particular, silicone foams, have a microstructure similar to that of PMSQ aerogels [35]. Indeed, the stress–strain curves of aerogels during compression are similar to those of polymer foams [35–39]. By analogy with polymer foams, macroscopic plastic deformation appears in aerogels when the maximum stress is reached, and it is accompanied by loss of stability of the cage and its fracture [40]. However, the walls of polymer foams are much thicker, pores are larger, and the geometric density is about twice as high (0.4 g/cm³) [40]. As polymer foams, aerogels are characterized by a positive correlation between the geometric density and the mechanical properties [15, 17, 19, 41, 42], which explains the lower (as compared to foams [43]) strength and residual strain of PMSQ aerogels.

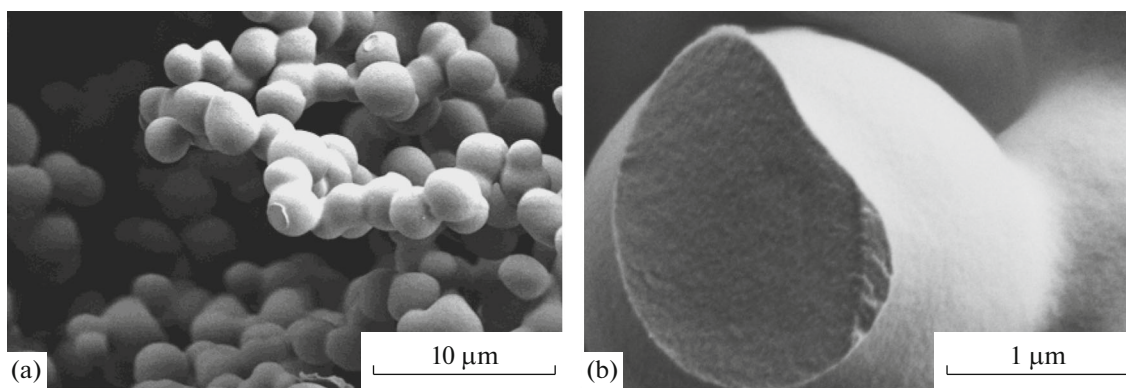


Fig. 6. (a) Broken chain of particles and (b) fractured particle in PMSQ aerogel.

CONCLUSIONS

(1) PMSQ aerogels characterized by geometric density of $\approx 0.2 \text{ g/cm}^3$, a specific surface area of 460–480 m^2/g , and a porosity of 86–88% were prepared by the hydrolysis of MTMS and subsequent drying of the formed gels at atmospheric pressure and in supercritical methanol or carbon dioxide. The synthesized aerogels exhibit pronounced superhydrophobic properties: the water contact angle reaches 154° .

(2) The ultimate compressive strength of the aerogels reaches 0.35 MPa at a relative deformation of 60%. Aerogels have a high elasticity, the residual strain after 10 cycles of loading to a relative deformation of 30% and unloading reaches 6%, and it relaxes to 2% three days after complete unloading.

(3) The physicochemical characteristics of the aerogels weakly depend on the method of drying, which demonstrates the advantages of the method of drying at atmospheric pressure as compared to more expensive and dangerous methods of drying in supercritical CO_2 or aliphatic alcohols.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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