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CHAPTER 17

Fabrication and Processing Techniques of Silica Aerogels

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Abstract: Silica aerogels are highly porous, lightweight materials renowned for their exceptional thermal insulation, low density, and high surface area, making them valuable across diverse scientific and industrial applications. Their fabrication typically involves a sol–gel process followed by controlled drying to preserve the delicate nanoporous network. The choice of precursor—commonly tetraethyl orthosilicate (TEOS) or sodium silicate—along with catalyst type and processing parameters, significantly influences the aerogel's structural and functional properties. Drying techniques such as supercritical drying, ambient pressure drying, and freeze-drying each present distinct advantages and limitations in terms of cost, scalability, pore retention, and environmental impact. Advances in surface modification, composite reinforcement, and hybrid material synthesis have further expanded the performance and application range of silica aerogels. This review outlines the fundamental steps of silica aerogel

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fabrication, compares various processing methods, and highlights recent innovations aimed at achieving an optimal balance between performance, economic feasibility, and sustainability.

Keywords: Silica aerogel, sol gel, aging, surface modification, drying techniques.

1. Introduction to Silica Aerogels

Silica aerogels are a unique class of advanced porous materials known for their ultra-low density, high surface area, and excellent thermal insulation properties. Often referred to as “frozen smoke” due to their translucent appearance and light weight, silica aerogels are primarily composed of silica (SiO_2) and contain up to 99.8% air by volume. These materials are synthesized through the sol-gel process, followed by careful drying methods that preserve their highly porous three-dimensional network.

Originally developed in the 1930s by Samuel Kistler, silica aerogels have gained renewed interest in recent decades due to advancements in processing techniques and a growing demand for lightweight, high-performance materials across various industries. Their applications span across aerospace, construction, thermal insulation, oil spill cleanup, sensors, and even drug delivery systems.

Silica aerogels are the most extensively studied type of aerogels, largely due to silica’s abundance, low cost, and suitability for scalable production in commercial applications.[1] Structurally, silica aerogels consist of a web-like network formed by interconnected chains of spherical nanoparticles.[2] Their synthesis typically involves the sol-gel method, which begins with the formation of a sol containing a silica precursor, water, and a catalyst.[3] Commonly used silica precursors include sodium silicate (Na_2SiO_3), tetramethoxysilane (TMOS) and tetraethoxysilane (TEOS).[4-6] However, due to concerns over the toxicity of some synthetic precursors, there is a growing interest in using natural, environmentally friendly, and cost-effective biomaterials instead.

This chapter explores the synthesis and processing techniques of silica aerogels, outlining key steps such as sol-gel chemistry, gel aging, and various drying methods including supercritical, ambient, and freeze-drying. Additionally, the influence of processing parameters on the final properties of the aerogels is discussed in detail.

2. Development of Silica Aerogels

The preparation of silica aerogels involves a multi-step process designed to create and preserve a highly porous silica network. The general procedure includes **sol–gel synthesis**, **aging**, and **drying**. Each step is crucial for determining the final properties of the aerogel, such as porosity, density, surface area, and mechanical strength. The transformation from sol to gel involves two main stages: the formation of a wet gel, followed by drying, with an aging phase in between. The drying process is particularly crucial, as it significantly impacts the structural and physical characteristics of the resulting aerogel.[7] Depending on the method used, different types of porous materials can be produced from hydrogels: **aerogels** through supercritical drying, **xerogels** via conventional evaporative drying, and **cryogels** through freeze-drying.

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It's important to distinguish that cryogels made by freeze-drying hydrogels differ from those produced through **cryogelation**, a process carried out at temperatures below the system's freezing point.[8]

The production of silica aerogels typically follows three main stages [9]:

1. **Gel Formation**: A sol–gel process is used to create the silica gel. This involves preparing a sol from a silica-containing solution, followed by the addition of a catalyst to initiate gelation. The resulting gels are categorized based on the liquid medium used during their formation, such as hydrogels (water-based), alcogels (alcohol-based), or aerogels (air-dried).
2. **Gel Aging**: After formation, the gel is left to age in its original solution. This step enhances the strength and stability of the gel network, helping to minimize shrinkage during the subsequent drying process.
3. **Gel Drying**: The final step involves removing the liquid from the gel's pores. To preserve the delicate porous structure and prevent it from collapsing, the drying process is carried out under carefully controlled conditions.

Figure 1 presents a flowchart outlining the steps involved in the preparation of silica aerogel.

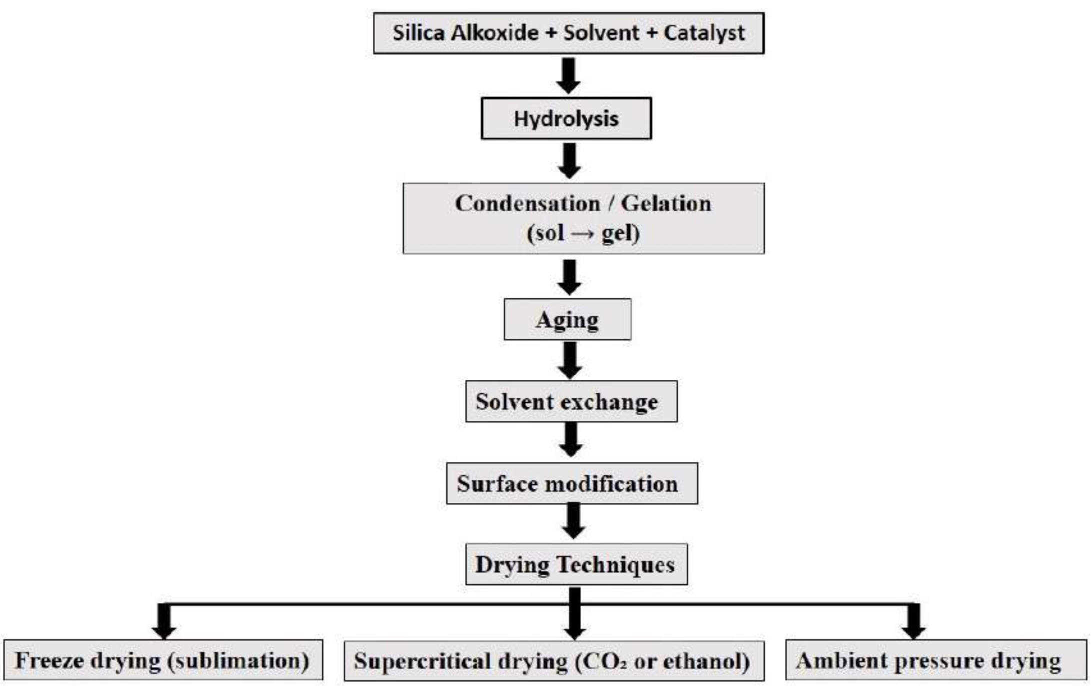

Fig. 1. Flowchart of the preparation of silica aerogel.

While all aerogel synthesis methods follow these fundamental steps, additional modifications may be applied to tailor the final material's properties.

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2.1 Sol–Gel Synthesis

The formation of silica aerogels begins with the **sol–gel process**, a chemical method that converts a liquid sol into a solid gel. This involves the hydrolysis and condensation of silica precursors in the presence of a catalyst.

Sol Preparation is the initial step in the synthesis of silica aerogels and involves forming a homogeneous solution by mixing a silica precursor with water and an organic solvent, usually ethanol or methanol. The choice of solvent helps regulate the rates of hydrolysis and condensation reactions, which are essential for forming the gel network. To initiate the sol–gel transition, a catalyst is introduced into the solution. The type of catalyst used significantly influences the structure of the resulting gel. Acidic catalysts, such as hydrochloric acid (HCl), typically lead to the formation of more linear, chain-like structures, whereas basic catalysts, like ammonium hydroxide (NH_4OH), promote the development of more branched and cross-linked networks. This step lays the foundation for the final gel's porosity, mechanical properties, and overall structure.

Hydrolysis and Condensation

Hydrolysis: $Si(OR)_4 + H_2O \rightarrow Si(OH)_4 + ROH$

Condensation: $Si(OH)_4 \rightarrow Si\text{–}O\text{–}Si + H_2O$ or ROH

The sol eventually transforms into a gel—a semi-solid network filled with solvent—called a **wet gel**.

2.2 Gel Aging

After gelation, the wet gel is **aged** in its mother liquor or in fresh solvent. Aging allows further condensation reactions to strengthen the gel network, reduce shrinkage during drying, and improve mechanical integrity.

Aging is a crucial step in the preparation of silica aerogels, carried out after gelation to enhance the structural integrity of the wet gel. The primary purpose of aging is to **improve the gel's stability,** making the network more robust and less susceptible to **pore collapse during the drying process**. Additionally, aging helps to **enhance pore connectivity and increase surface area**, both of which are important for the aerogel's performance in various applications. The **conditions for aging** typically involve maintaining the gel in its mother liquor or a fresh solvent for a period ranging from **a few hours to several days**. This process is usually conducted at **room temperature or at slightly elevated temperatures**, generally between **30°C and 60°C**, depending on the desired properties of the final product.

2.3 Solvent Exchange

Before drying, the water or original solvent in the gel pores is often replaced with a low-surface-tension solvent such as ethanol or liquid CO_2 to minimize capillary stress during drying. This step is especially important for supercritical or freeze-drying.

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2.4 Drying Methods

Drying is the final and most critical stage in gel production, as it directly influences the type and characteristics of the resulting material. The three primary gel types—aerogels, xerogels, and cryogels—are mainly distinguished by the drying method employed. Aerogels are typically formed through supercritical drying, xerogels via ambient pressure drying, and cryogels by freeze-drying. These drying techniques lead to significant differences in physical properties such as porosity and surface area.

However, it's not just the drying method that determines these characteristics—the choice of precursor used to form the initial gel network (e.g., silica, carbon, or polymer) also plays a major role. For example, silica aerogels often exhibit average pore sizes between 20–40 nm and surface areas ranging from 600 to 1000 m^2/g. In comparison, organic and carbon xerogels tend to have smaller pore sizes (around 2–4 nm) and surface areas in the range of 300–400 m^2/g.[10] Cryogels vary more widely; some, like carbon cryogels, can be mesoporous with surface areas exceeding 800 m^2/g, while others are supra-macroporous with very low BET surface areas.[11]

When comparing gels made from the same base material, such as cellulose, aerogels generally have surface areas that are ten times greater than those of cryogels, while xerogels may have negligible surface areas in comparison.

Drying is the most critical step, as it determines whether the gel retains its porous structure or collapses. Three main drying techniques are used:

2.4.1 Ambient Pressure Drying (APD)

It is a widely used method for producing silica aerogels due to its simplicity, scalability, and lower operational risk compared to supercritical drying. This technique involves drying the gel at atmospheric pressure and moderate temperatures. However, because drying under ambient conditions can lead to significant capillary forces that collapse the delicate pore structure, **surface modification** is typically required beforehand. Agents like **trimethylchlorosilane (TMCS)** are used to modify the surface of the gel, reducing capillary stress and helping to preserve its porous network. While APD is generally safer and more cost-effective than supercritical methods, it often results in the formation of **xerogels**—materials with lower porosity—unless the drying parameters and surface treatments are carefully optimized.

2.4.2 Supercritical Drying

Supercritical Drying is a key technique in the production of high-quality silica aerogels, as it allows for the removal of the pore liquid without collapsing the fragile gel structure. This method can be categorized into two types: High-Temperature Supercritical Drying (HTSCD) and Low-Temperature Supercritical Drying (LTSCD).

HTSCD is performed in a high-pressure autoclave using organic solvents such as methanol or ethanol. During the process, the system is heated above the critical temperature and pressure of the chosen solvent, allowing the solvent to transition into a supercritical fluid. In this state, the solvent has no surface

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tension, which prevents the collapse of the gel's porous network. The supercritical fluid is then slowly vented while maintaining a constant temperature, and the system is gradually returned to ambient pressure and temperature. High-temperature supercritical drying (HTSCD), first introduced by Kistler in 1931, remains a widely used method for producing silica aerogels.[12] This process takes place in an autoclave, where organic solvents like methanol or ethanol are used. The system is heated and pressurized above the solvent's critical point, and the solvent is then gradually released while maintaining a constant temperature. Once the pressure is reduced to ambient levels, the system is cooled back to room temperature.[13] Despite its effectiveness, HTSCD has significant safety concerns due to the high temperatures involved, which present a fire risk.[14]

Alternatively, LTSCD utilizes liquid carbon dioxide (CO_2) as the drying medium. CO_2 has a relatively low critical point (31.1 °C and 7.38 MPa), making this method safer and more energy-efficient compared to HTSCD. While LTSCD generally results in hydrophilic silica aerogels, their surface characteristics can be altered through post-synthesis surface modification to achieve hydrophobicity, which enhances their performance in moisture-sensitive applications. As a safer alternative, **low-temperature supercritical drying (LTSCD)** uses **liquid carbon dioxide (CO_2)**, which has a critical point much closer to ambient conditions.[15] This method is generally more cost-effective and results in silica aerogels that are naturally **hydrophilic**. However, the surface properties can be adjusted through chemical modification to make the aerogels **hydrophobic** if desired.[16]

2.4.3 Freeze drying (Lyophilization)

Freeze-drying typically involves freezing the material at low temperatures (typically –50 to –80 °C) and the solvent is removed by **sublimation** of ice under vacuum. Prevents shrinkage but may form cracks due to **ice crystal formation** within the pores This method has been successfully used to produce aerogels made from materials like silica, cellulose, clay, graphene, and carbon.[17] In the case of silica and composite aerogels, freeze-drying is often carried out at extremely low temperatures ranging from –50 °C to –83 °C under vacuum conditions of 5–30 Pa.[18]

Although freeze-drying is more cost-effective than supercritical drying and helps minimize shrinkage, its application is limited. This is primarily because solvent crystallization within the gel's pores during freezing can cause the formation of cracks. Despite this drawback, freeze-drying remains in use for the fabrication of silica-based composite aerogels.[19]

It's important to note that when hydrogels are freeze-dried, the resulting materials do not possess the true characteristics of cryogels. This is because ice crystals form within the gel structure, not in a liquid solution. In contrast, cryogels formed through cryogelation—performed at sub-zero temperatures - tend to demonstrate superior properties compared to those derived from freeze-dried hydrogels. In **Table 1** summarizes the advantages and disadvantages of all drying techniques

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Table 1: Summery of advantages and disadvantages of Ambient pressure, Supercritical and Freeze drying techniques.

Drying Method	Advantages	Disadvantages
Ambient Pressure Drying (APD)	Low cost and simple equipment	High capillary stresses can cause shrinkage and cracking
	Easy to operate	Loss of fine structure in delicate materials
	Suitable for large-scale drying	Slower drying rate for dense samples
	No need for high-pressure systems	
Supercritical Drying (SCD)	Eliminates liquid–gas interface, avoiding capillary stresses	Requires expensive high-pressure equipment
	Preserves delicate porous structures (e.g., aerogels)	Safety concerns with high-pressure CO_2 or other fluids
	High quality and uniformity of dried product	High operational cost
		Complex process control
Freeze Drying (Lyophilization)	Best for preserving biological activity and heat	Very slow process
	sensitive materials	High energy consumption
	Minimal shrinkage and structural damage	Requires vacuum and refrigeration systems
	Good for long-term stability of products	Higher operating cost compared to APD

2.4.4 Surface Modification

Surface modification is an important step in enhancing the durability and functionality of silica aerogels. Naturally, silica aerogels are **hydrophilic**, meaning they readily absorb moisture from the environment, which can compromise their structure and performance. To overcome this limitation, surface modification is carried out to render the aerogels **hydrophobic**, significantly improving their **stability** and making them suitable for use in a wider range of applications. This modification is typically achieved using reagents such as silylating agents (e.g., trimethylchlorosilane TMCS, hexamethyldisilazane HMDS) react with the surface silanol groups replace surface –OH with non-polar groups (–Si $(CH_3)_3$) to make gel hydrophobic

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nature.[20] The resulting **hydrophobic aerogels** exhibit enhanced resistance to moisture, improved shelf life, and are particularly effective in applications such as **thermal insulation** and **oil–water separation**.

3. Applications of Silica Aerogel

I. Thermal Insulation & Energy Efficiency

- Owing to the extremely low thermal conductivity and light weight, silica aerogel is used in high-performance insulation solutions like skylights, roofs, and retrofit systems.[21]
- Silica aerogels (including silica or related nanofibrous aerogels) can enhance passive daytime radiates cooling by reducing solar absorption and improving thermal emittance.[22]

II. Optical Applications & High-Energy Physics

- Transparent silica aerogel tiles with controllable refractive indices are deployed in Cherenkov detectors to identify high-energy subatomic particles in modern physics experiments such as Cherenkov Radiators.[23]
- Silica aerogel thin films used as antireflective coatings of Solar Cells to improve photovoltaic efficiency by boosting light absorption.[24]

III. Environmental Remediation & Energy Storage

Silica aerogels exhibit promise in adsorbing contaminants (e.g., for water treatment), and composites with graphene show synergy in pollution cleanup and energy conservation.[25]

IV. Acoustic Insulation

Silica aerogel and its composites (nonwoven blankets, glazing) as effective materials for sound absorption in acoustic insulation, though standardization and deeper mechanistic studies are still needed.[26]

V. Biomedical & Drug Delivery

Aerogels act as drug delivery matrices across administration routes (oral, pulmonary, topical), enhancing bioavailability and controlled release. They also serve as scaffolds for wound healing and regenerative medicine.[27]

VI. Catalysis & Electronic Applications

Due to high surface area and porosity, silica aerogels support catalysts in fuel cells and serve in energy storage applications like supercapacitors and as dielectric materials.[28]

VII. Cosmic Dust Capture & Space Applications

Silica aerogel was utilized in NASA's Stardust Space mission to gently capture cosmic dust particles at high velocities with minimal thermal alteration.[29]

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Conclusion

The preparation of silica aerogels is a highly sensitive and controlled process in which each stage—from sol–gel synthesis, through aging and surface modification, to final drying—has a significant influence on the structural, mechanical, and thermal characteristics of the end product. Supercritical drying, although still considered the benchmark for producing aerogels with superior porosity and minimal shrinkage, often faces limitations in cost, equipment complexity, and safety. Consequently, alternative approaches such as ambient pressure drying and freeze-drying are increasingly being explored for their potential in large-scale and environmentally friendly production. The selection of suitable silica precursors (e.g., TEOS, sodium silicate), catalysts (acidic or basic), and drying techniques must be strategically optimized in alignment with the intended application—be it ultralight thermal insulation, advanced filtration media, catalyst supports, or structural composites. By fine-tuning these parameters, it is possible to achieve a balance between performance, durability, and economic feasibility, paving the way for the broader commercial adoption of silica aerogel technology across diverse industries.

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