



Preparation of Hydrophobic Silica Aerogel

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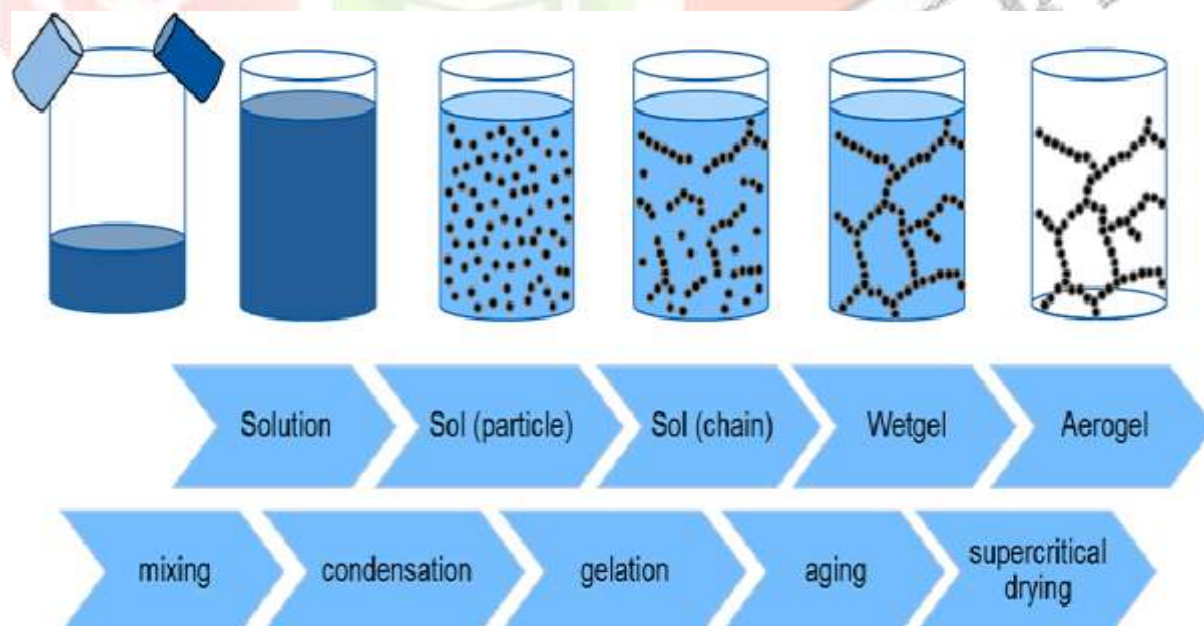
Abstract

Hydrophobic silica aerogel is known as “Frozen Smoke”. Its lightweight porous materials exhibit exceptional properties. This abstract provides an overview of hydrophobic silica aerogel. It's synthesized through a sol-gel process where it's modified to hydrophobic properties by replacing $-OH$ groups. This hydrophobicity also preserves the aerogel's low density, high porosity, and thermal insulating properties.

These aerogels have multiple applications due to their extraordinary properties. In aerogel properties low density and high porosity are applicable in lightweight materials such as in spacecraft technology.

In this abstract, we get the details of the preparation method, characteristics, hydrophobicity of silica aerogel, Chemical Reaction, and their application in the future perspective research technology.

Graphical Abstract



[Fig.1. Whole Process of Synthesis of Silia aerogel]

Introduction

Silica aerogel was first evolved during the 1930s by Samuel Kistler who utilized the Sol Gel strategy to supplant the fluid part of silica aerogel with Air. His hydrophobicity is accomplished by changing the hydrophobic gatherings, for example, alkyl chains (-OH). Hydrophobic silica aerogels are a subtype of silica aerogel. It acquired consideration because of its water-repellent qualities and wide-reach application later on innovation. This presentation will outline hydrophobic silica aerogel highlights, which portray the silica aerogel properties. Silica aerogels, frequently hailed as "frozen smoke" because of their low thickness and striking gentility, stand out in material science and design. Among the different kinds of aerogels, hydrophobic silica aerogels stand apart as an unmistakable class of materials with interesting properties that make them especially encouraging for a scope of uses. These materials can captivate to repulse water, opening up roads for imaginative purposes in protection, oil slick remediation, and then some. Aerogels, by and large, are exceptionally permeable materials obtained from a gel where the fluid part has been supplanted with a gas, bringing about a strong design made out of an organization of interconnected nanoscale pores. The hydrophobic idea of silica aerogels is accomplished through surface alterations, regularly including the presentation of hydrophobic moieties, for example, organosilanes, onto the silica structure. This modification confers water-repellent qualities, making hydrophobic silica aerogels profoundly significant in applications where water opposition is fundamental. The extraordinary mix of properties showed by hydrophobic silica aerogels, including their super low thickness, brilliant warm protection, and hydrophobicity, positions them as promising contenders for applications in cruel conditions, where customary materials might miss the mark. This paper digs into an exhaustive investigation of the blend strategies, portrayal methods, and expected utilization of hydrophobic silica aerogels, meaning to add to the developing group of information in this thrilling and quickly advancing field.

Goals of the Review:

1. **Synthesis Strategies:** Research and think about different techniques for blending hydrophobic silica aerogels, investigating the impact of various boundaries on their design and properties.

2. **Characterization Strategies:** Utilize progressed scientific procedures to portray the morphology, porosity, and surface properties of hydrophobic silica aerogels, giving an itemized comprehension of their design.

3. **Applications:** Investigate possible utilizations of hydrophobic silica aerogels, with an emphasis on regions, for example, warm protection, oil slick

cleanup, and ecological remediation. By tending to these targets, this study means to contribute significant experiences into the union, properties, and uses of hydrophobic silica aerogels, preparing

for their proceeded with progression in different modern and logical fields. As a consequence of this exploration, the hydrophobic silica aerogel structure isn't promptly wet with water. It addresses a wonderful class of materials with unmatched properties like low thickness, high porosity, and water-repellent qualities. Its utilization to understand the importance and likely uses of hydrophobic silica aerogel in different fields.

Silica aerogel properties

Properties of Silica Aerogel		
Density	Low Density	0.01 g/cm ³ to 0.3 g/cm ³
Porosity	High Porosity	90 to 99%
Thermal insulation	Low Thermal insulation	0.010 to 0.030 W/m.k
Surface Area	High Surface Area	500 to 1000 m ² /gm
Sound Insulation	Good	Good
Mechanical Strength	Low	Easily fracture under stress

[Table 1. Properties of Silica Aerogel]

Types of Precursor used in Silica Aerogel:

In the silica aerogel readiness, we can utilize forerunners like TEOS (Tetraethyl Orthosilicate), MTMS (Methyl Tri Methoxy silicate), Sodium Silicate, Ethyl Silicate, MTES (Methyl Tri Ethyl Silicate), Hexamethyl disiloxane (HMDSO), etc. we need to create naturally adjusted aerogel that is the reason we favored MTMS substance in this exploration.

Precursor Name	Details
TEOS	Most common precursor
MTMS	Use To prepare organically modified aerogel
Sodium silicate	Economically precursor
HMDSO (Hexa methyl disiloxane)	For creating hybrid material

[Table 2. Types of Precursor use]

Chemical Properties of Silica Aerogel:

The Amalgamation of silica aerogel by a mix of Methyl Tri Ethyl Methyl silane (MTMS), Calcium Hydroxide [CA(OH)₂], Oxalic Corrosive, and Methanol to deep-rooted and produce silica aerogel. These all assume a basic part in accomplishing the ideal aerogel qualities structure. Here a few brief properties of these synthetic substances are displayed in the table.

Number	1	2	3	4	5
Chemical Name	MTMS (Methyl Tri Methoxy silane)	Methanol	Oxalic Acid	CaOH	NaOH
Molar Mass	136.222 g/mol	32 g/mol	90 g/mol	74 g/mol	40 g/mol
Density	0.955 g/cm ³	0.786 g/cm ³	1.90 g/cm ³	2.21 g/cm ³	2.13 g/cm ³
Boiling Point	102-104 °C	64.7 °C	100° C	-	1.38° C
Solubility in water	Hydrolysis	Miscible in Water	Soluble	Poorly Soluble	-
Flash Point	11 °C	11°C	166° C	-	-
Melting Point	60 °C	- 97.6°C	189.5 °C	580 °C	323° C
Freezing Point	- 97.6 °C	-	101° C	-	-

[Table 3. Properties of Chemicals]

Underneath this table depict the Jobs of the Synthetics during the time spent Readiness silica aerogel. Here brief survey of the critical parts and the union cycle

No.	Chemical Name	The main role of Process
1.	MTMS (Methyl Tri Methoxy Silane)	Main Important Precursor For Silica Aerogel
2.	Oxalic Acid	Hydrolysis and Condensation process
3.	CaOH	Maintaining p ^H
4.	Methanol	For the gelation and Solvent Exchange Process

[Table 4. Roles of Chemicals in Synthesis]

Parameters of Silica Aerogel

Silica aerogels are recognized for their extraordinary properties, which make them reasonable for a great many applications, from warm protection in structures and space apparatus to use in optics, sensors, and medication

conveyance frameworks. The boundaries that characterize the exhibition and appropriateness of silica aerogels include:

1. Porosity

Silica aerogels are exceptionally permeable materials, with porosities ordinarily surpassing 90%. This elevated degree of porosity is liable for their low thickness and adds to their incredible warm protection properties. Silica aerogel's pore size and porosity rely upon the premise of the creation interaction and it will be a scope of 5 - 50 nm.

$$POROSITY = \left(\frac{1}{\rho_b} - \frac{1}{\rho_s} \right)$$

Where,

P_b = Mass Thickness

P_s = Skeletal Thickness

2. Thickness

One of the most striking properties of silica aerogels is their low thickness, which can be in the scope of 0.003 to 0.35 g/cm³. This spreads the word.

3. Surface Region

Silica aerogels have very high surface regions, frequently going from 500 to 1000 m²/g, which makes them exceptionally viable as impetus upholds, in adsorption applications, and sensors. Surface change relies upon the various properties of the synthetics that we use in the technique for the cycle. It all changes the information like thickness, porosity, and so on.

$$\%V_s = \left(1 - \frac{V_a}{V_g} \right) * 100$$

Where,

V_s = Pore shrinkage volume

V_a = Volume of aerogel

V_g = Volume of alcogel

4. Warm Conductivity

The warm conductivity of silica aerogel is particularly low, ordinarily in the scope of 0.012 to 0.020 W/m-K at environmental tension, which makes it a fantastic protecting material, in any event, beating customary protection now and again.

5. Optical Straightforwardness

Silica aerogels can be exceptionally straightforward to notice light, with specific definitions sending more than 90% of episode light while at the same time giving warm protection. This extraordinary mix is important in applications like protecting windows and sun-powered energy gatherers.

6. Mechanical Properties

The mechanical strength of silica aerogels is by and large low because of their high porosity and fragile microstructure, yet adjustments and composite details can work on their compressive and elastic qualities for applications requiring more primary respectability.

Silica aerogel is a very lightweight material due to its low density and high porosity. Its density range is 0.1 to 0.4 g/cm³.

$$Density = Aerogel's \frac{mass}{Volume}$$

7. Acoustic Properties

Silica aerogels likewise show great acoustic protection properties because of their permeable construction, which can disperse and assimilate sound waves successfully.

8. Compound Solidness

While unadulterated silica aerogels are moderately steady synthetically, they are hydrophilic and can ingest dampness, which can influence their properties. Surface alteration can work on their hydrophobicity and compound obstruction, making them appropriate for additional assorted conditions and applications.

9. Hydrophobicity/Hydrophilicity

The surface science of silica aerogels can be changed by making them either hydrophobic or hydrophilic. Hydrophobic aerogels repulse water and are valuable in applications where dampness obstruction is basic, while hydrophilic aerogels can retain dampness and track down use in controlled delivery and adsorption applications.

We can find the contact point of hydrophobicity of silica aerogel by the situation of contact point addressed by Youthful's situation,

$$\sigma_L \cos \theta = \sigma_S - \sigma_{SL}$$

σ_S is the surface energy,

σ_{SL} is strong fluid interfacial energy,

σ_L is a surface strain

The mix of these properties, especially the low thickness, low warm conductivity, and high surface region, pursues silica aerogels as a material of decision for the vast majority of progressed innovative applications, showing an entrancing mix of physical and substance qualities.

Silica Aerogel Synthesis Process

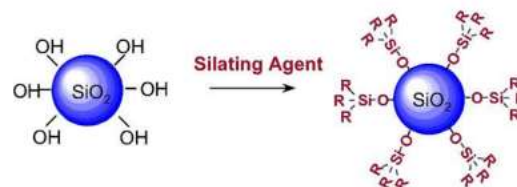
The blend of silica aerogel includes a few key stages, each basic to accomplishing the novel properties of the eventual outcome. Here is a worked-on outline of the interaction:

1. Sol-Gel Cycle

The excursion starts with the sol-gel process, which establishes the groundwork for making the aerogel's design. Silica Aerogel is produced by the Forerunner, Impetus, and the Solvents Combination. In the Blend of Hydrophobic Silica Aerogel process is Hydrolysis, Buildup, Gelation, Maturing, Dissolvable Trade, and last supercritical drying which converts the type of silica aerogel into semi-strong to aerogel structure.

•**Silica Forerunner:** The interaction begins with a silica source, normally silicon alkoxides, for example, tetraethyl orthosilicate (TEOS) or tetramethyl orthosilicate (TMOS). These mixtures are dissolvable in liquor and respond to water in a controlled way.

•**Hydrolysis and Polymerization:** within the sight of an impetus (acidic or fundamental), the silica forerunners go through hydrolysis followed by buildup responses. Hydrolysis parts the alkoxide gatherings (OR) from the silicon, supplanting them with hydroxyl (Gracious) gatherings, while buildup interfaces these silica particles together into a three-layered network, shaping a colloidal suspension known as a "sol."



[Fig.2. Synthesis Process of Silica Aerogel]

•**Gelation:** As the buildup response continues, the thickness of the sol increments until the silica particles interconnect to frame a gel. This gel is a strong organization loaded up with fluid (typically the dissolvable and unreacted water).

2. Aging

After gelation, the silica gel is aged, permitting the gel organization to fortify and settle. This cycle includes keeping the gel in its dissolvable, which frequently keeps on responding gradually, further connecting the silica organization. aging can work on the mechanical properties of the last aerogel.

3. Dissolvable Trade

At first, the pores of the gel are loaded up with the first dissolvable (e.g., ethanol). This dissolvable is frequently supplanted with a fluid that has a lower surface strain and is simpler to eliminate in later strides without falling the gel's permeable design. A typical decision is to trade the first dissolvable with supercritical CO₂ in later advances.

4. Drying

The basic stage in the creation of aerogel is drying, which should be done cautiously to safeguard the gel's nanostructure.

•**Supercritical Drying:** The most widely recognized technique includes supplanting the pore fluid with a liquid that can be eliminated under supercritical circumstances. For CO₂, this includes warming and compressing the gel in an autoclave past the basic mark of CO₂ (31.1°C and 73.8 bar). At supercritical circumstances, the fluid and gas stages become unclear, and the liquid can be taken out without causing hairlike pressure that would some way or another break down the gel's design.

•**Surrounding Tension Drying:** An elective technique that maintains a strategic distance from the high tensions and temperatures of supercritical drying. This cycle includes cautious dissolvable trade and surface change to forestall the gel structure from falling when the dissolvable is dissipated under surrounding conditions. It's more affordable yet considered normal outcomes in aerogels with lower straightforwardness and higher densities.

5. Surface Modification (Discretionary)

To improve the properties of silica aerogel for explicit applications, surface modification can be performed. This generally includes delivering the aerogel hydrophobic to work on its protection from dampness. Silane coupling specialists (e.g., trimethylchlorosilane) can be utilized to supplant surface hydroxyl bunches with non-polar gatherings.

6. Result

The outcome is a silica aerogel that is incredibly lightweight and permeable, with low warm conductivity, and high surface region, and can be designed to have explicit optical, mechanical, and synthetic properties.

This combination cycle features the flexibility and intricacy of making silica aerogels, taking into consideration customization custom-made to many applications.

It all relies upon the cycle on the off chance that we utilize a Supercritical drying process, we will get an Aerogel kind of arrangement of silica gel, if we use a Freeze drying process, it will create the Cryogel sort of development and if we utilize the Vanishing system, it will change over the gel into the Xerogel arrangement.

Synthesis Process of Silica Aerogel

1. MTMS Weakened with Methanol and blended for 10 min.
2. Oxalic corrosive as an impetus and Methanol blended well until it became homogeneous and mixed for 10 min.

Aerogel Precursors Synthesis Methods	
Sol-Gel Process	When Silica Aerogel goes through Hydrolysis And Buildup to shape gel
Chemical Vapor Deposition (CVD)	Decomposition of antecedent gases to frame strong aerogel structure

[Table 5. Synthesis Methods]

Acid methanol combination added with (MTMS+ Methanol) Blend and mixed for 15 min.

4. Addition the added substance of Stake according to the Prerequisite
5. NaOH included the combination following 2 hours for the build-up cycle of silanols to shape sols.



[Fig.3. Practical work]

6. After the sols structure it will be placed in one more dish for gel arrangement at room temperature (28 - 35 °C).

7. After maturing the arrangement the hardening was vacuum at 45 °C.

In the drying system, narrow tension is a more significant variable for eliminating fluid. drying is administered by fine tension which can be addressed by situations like,

$$P_c = - \frac{\gamma l v}{(r_p - \delta)}$$

Where,

P_c = Capillary Pressure

$\gamma l v$ = Surface tension of pore liquid

R_p = Pore radius

δ = Thickness of surface absorbed layer.

Step	Process Name	Role of Process
1.	Hydrolysis and Condensation process	MTMS Hydrolysed by Water
2.	Gelation process	CaOH maintains the p ^H of the gelation form
3.	Aging process	Further Condensation for straitening silica gel
4.	Solvent Exchange process	Methanol used to exchange the liquid within the gel with methanol
5.	Supercritical Drying processes	It is used to remove liquid from the gel and replace the liquid with gas.

[Table 6. Process Name and role]

$$r_p = 2V_p / S_p$$

Where

R_p = pore radius

V_p = pore volume

S_p = Pore surface area

The bulk density of the samples was calculated as

Bulk density = mass of the sample (g) / volume of the sample (cm³)

Bulk density = mass of the sample / volume of the sample (cm³)

In the drying system, the slim tension might reach 90 to 190 MPa with subsequent shrinkage and breaking, tracking down by its situation.

Presently here we can see the sorts of drying strategies which is valuable to plan silica gel to type of silica aerogel.

Drying Methods		
Supercritical drying	Utilizing CO2 to supplant fluid in the gel	For high permeable, At High Temperature and Strain
Ambient Pressure Drying	By dissolvable trade at air pressure	Some Shrinkage and Lessen porosity
Freezing dryer	Freezing gel and eliminating ice through sublimation	The result will called Cryogel same as aerogel however disparate in thickness

[Table 7. Drying Methods]

Remove fluid and supplant it with the gas, the most favored technique is Supercritical drying yet it all relies upon the development of silica aerogel properties.



[Fig.4. Drying Silica Aerogel]



[Fig.5. Silica Aerogel Slab]

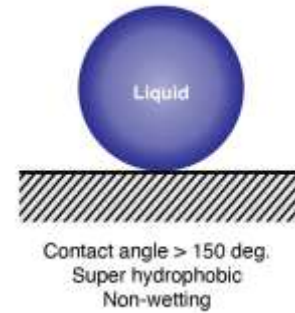
Now it's prepared for the trial of Hydrophobicity by chromatography

Find Its Hydrophilic and Hydrophobicity.

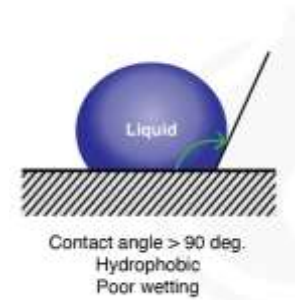
Hydrophobic and Hydrophilic

If the silica aerogel film contacts with water at more than 90 degrees on the surface response by chromatography technique then, at that point, it's called Hydrophobic.

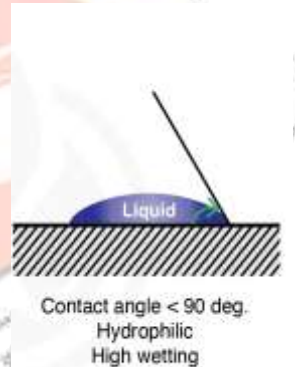
If silica aerogel film contact with water at under 90 degrees on the surface response by chromatography technique then, at that point, it's called Hydrophilic.



[Fig.6. Super Hydrophobic Angle]



[Fig.7. Hydrophobic Angle]



[Fig.8. Hydrophilic Angle]

Contact point estimation by the sessile drop contact point geometry machine.

Deionized water is used to decide the hydrophobicity of silica aerogel.

A bead of aerogel on the outer layer of the silica aerogel with the accuracy needle of the micro syringe.



[Fig.9. Genio-meter]

The volume of drop size is 2-3 μL.

Estimations will take rehash threefold of the test.

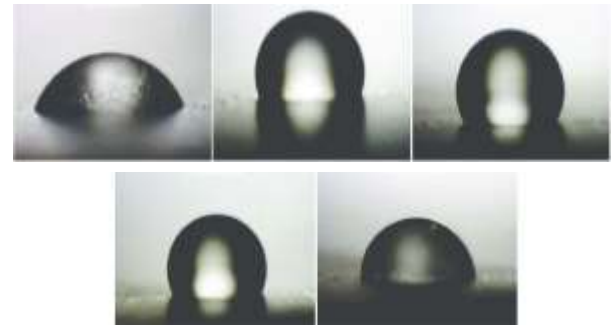
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σ_S is the surface energy,

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σ_L is a surface strain



[Fig.10. Drop of water on silica aerogel]

Different Types of Methods of Hydrophilic and Hydrophobicity find:

Types of Methods of Hydrophilic and Hydrophobic		
No.	Methods Name	Details
1.	Water Contact Angle Measurement	A low point implies under 90 degrees it will be Hydrophilic and at a high point of 90 degrees it will be hydrophobic.
2.	Wilhelmy Plate Method	Lower a piece of the aerogel in water and measure the power confine from the fluid, Hydrophilic materials have higher power, er and Hydrophilic materials have less power to segregate.
3.	Surface Energy Calculation	Compute the surface energy with various fluids, Higher surface energy is called hydrophilic, and Lower surface energy is called Hydrophobic..
4.	Chemical Function Groups	Hydrophilic: Polar capability gatherings (Hydroxyl bunch and so forth.) Hydrophobic: Nonpolar capability bunches like alkyl chain gatherings.

[Table 8. Methods Of Angle]

The above table portrays the techniques for the Hydrophobicity test however it all relies upon the natural condition and the properties of the silica aerogel. It will change at various properties of silica aerogel.

Contact Angle of Silica Aerogel

Sample	Contact Angle(°)	Bulk Density(g/cm³)
MTMS S-1	140.2	0.0770
MTMS S-2	133.3	0.0720
MTMS S-3	145.2	0.0885
MTMS S-4	137.5	0.0824
MTMS S-5	131.4	0.0755
MTMS S-6	140.9	0.0835
MTMS S-7	136.0	0.0800

[Table 9. Result Angle of Silica Aerogel]

Utilization of Silica Aerogel

•Warm Protection:

Silica aerogel has great protective properties because of its low warm conductivity which is the reason it's valuable for pipeline covering, building, and modern materials.

•Oil slick tidy up:

Silica aerogel has hydrophobicity properties which are the reason it does not retain water yet it ingests other fluids like oil or some other fluids. So it's valuable to eliminate oil from the water at whatever point it's needed. Its retention fluid is close to 15 seasons of its mass.

•Defensive Garments:

Silica aerogel has great warm protection properties and lightweight material because of its low thickness and high porosity so it is used in garments to safeguard against cold or intensity. Firemen likewise utilize these kinds of garments to defend themselves in crisis circumstances ablaze.

•Nursery Protection:

Silica aerogel is utilized in the nursery impact to keep up with the temperature in the nursery structure because of its low warm conductivity.

•Oil and Gas Ventures:

In the oil and gas ventures heat protection is vital for transportation to happen, that is the reason silica aerogel is used to cover the pipeline by the silica aerogel cover so the natural intensity impact doesn't impact the internal

temperature of the pipeline and it's helpful to not make wax in the oil transportation.

•Ecological remediation:

Silica aerogel is used in the climate to eliminate contaminations from the air and water by the retention limit.

•Space apparatus innovation:

Silica aerogel has lightweight material properties which is the reason it will be used in making a lightweight material for space innovation.

•Sound Protection:

Silica aerogel has properties that it doesn't move the sound means it protects the sound so it's material in the cinema.

Conclusion

As a consequence of this trial research, the Antecedent job is the principal for hydrophobicity. Silica aerogel's Hydrophobicity is subject to the adjustment of the outer layer of silica aerogel. Hydrophobicity repulses the water on the outer layer of the silica aerogel. It's all subject to the properties of the synthetics which is utilized for the planning of the silica aerogel. Silica aerogel surface changes are additionally impacted by the low thickness, warm Protection, High porosity, and Surface region of the silica aerogel properties. Its Hydrophobicity Properties are Generally Applied in store for the examination innovation.

Taking everything into account, hydrophobic silica aerogels offer striking properties and possible applications because of their extraordinary construction and surface science. These materials have earned huge premiums in different fields, going from protection and ecological remediation to biomedical and optical applications. Here are a few central issues to sum up the meaning of hydrophobic silica aerogels:

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