

# **Synthesis and Manufacturing of Silica Aerogel at Supercritical Conditions Using Ordinary CO<sub>2</sub> Gas Cylinder**

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**SONARGAON UNIVERSITY (SU)**

**147/I, GREEN ROAD, PANTHAPATH, TEJGAON, DHAKA**

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“A Graduation Exercise Submitted to the Department of Mechanical Engineering in Partial Fulfillment of the Requirements for the Degree of Bachelor of Science in Mechanical Engineering”

DEPARTMENT OF MECHANICAL ENGINEERING  
SONARGAON UNIVERSITY (SU)  
147/I, GREEN ROAD, PANTHAPATH, TEJGAON, DHAKA

SEPTEMBER 2023

## STUDENT DECLARATION

This is to certify that the thesis entitled “**Synthesis and Manufacturing of Silica Aerogel at Supercritical Conditions Using Ordinary CO<sub>2</sub> Gas Cylinder**” is an outcome of the investigation carried out by the author under the supervision of **Md. Sojib Kaiser** Assistant Professor, Dept. of Mechanical Engineering, Sonargaon University (SU). This thesis or any part of it has not been submitted elsewhere for the award of any other degree or diploma or other similar title or prize.

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## ACKNOWLEDGEMENT

This is to certify that the investigation conducted by the author under the guidance of **Md. Sojib Kaiser**, Assistant Professor, Dept. of Mechanical Engineering, **Sonargaon University (SU)**, resulted in the thesis, “**Synthesis and Manufacturing of Silica Aerogel at Supercritical Conditions Using Ordinary CO<sub>2</sub> Gas Cylinder.**” This thesis or any portion of it has not been submitted to another institution for the granting of another degree, diploma, or any title or reward that is identical as well.

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January 2023

## **Dedication**

To our parents, family and supervisor, with most profound thanks.

## Abstract

Silica aerogels are remarkable materials for their exceptional porosity, low density, and superb insulating properties. Their synthesis typically relies on supercritical drying using siphon CO<sub>2</sub> gas cylinders, which offer precise control over the drying process. However, the limited availability, cost considerations, and environmental impacts of siphon CO<sub>2</sub> cylinders have sparked interest in alternative sources, notably ordinary CO<sub>2</sub> gas cylinders. The study's central aim is to scrutinize the influence of the CO<sub>2</sub> source on critical properties of the aerogel, encompassing density, porosity, thermal conductivity, and structural integrity. The results show that silica aerogels made using regular CO<sub>2</sub> gas cylinders and syphon CO<sub>2</sub> cylinders have very different physical, thermal, and structural characteristics. Ordinary cylinders may allow for cost reductions and more accessibility, but they also make it difficult to preserve the required characteristics of silica aerogels and provide consistent results.. These differences emphasize the importance of careful consideration when selecting the CO<sub>2</sub> source for specific applications. The thesis continues by exploring the effects of these results, offering recommendations for further research, and highlighting areas for optimization in the synthesis process. This research contributes to the evolving field of aerogel technology, providing valuable insights into selecting CO<sub>2</sub> sources for silica aerogel synthesis and guiding future advancements in materials science and engineering.

## Table of Contents

STUDENT DECLARATION .....	3
ACKNOWLEDGEMENT.....	4
Dedication .....	5
<b>Abstract</b> .....	vi
Table of Contents.....	vii
<b>List of Abbreviations</b> .....	xi
Chapter 1 .....	12
1 Introduction.....	12
1.1 Types of Aerogels .....	14
1.2 Unique Properties of Aerogel .....	18
1.3 Limitation of Silica Aerogel.....	18
1.4 Physical Properties of Silica Aerogel: .....	19
1.5 Aerogel Applications .....	20
1.6 Synthesis of Silica Aerogel.....	21
1.7 Aerogel Drying Process .....	23
1.8 Significance of the Research.....	24
1.9 Problem Statement .....	25
1.10 Hypothesis.....	26
1.11 Objectives .....	26
Chapter 2.....	27
2 Literature Review.....	27
Chapter 3.....	34
3 Methodology .....	34
3.1 Common peripheral Equipment's: .....	34
3.1.1 A supercritical dryer or autoclave:.....	34
3.1.2 Variac:.....	39
3.2 Common Chemicals: .....	40
3.2.1 TMOS as precursor: .....	40
3.2.2 Methanol as Solvents and Cleaning Agents: .....	41

3.2.3	Ammonium hydroxide as Catalysts:.....	42
3.2.4	CO <sub>2</sub> as Supercritical Fluids:.....	42
3.2.5	Distilled Water .....	44
3.3	Making sol-gel.....	45
3.3.1	Preparation of Solution-1:.....	45
3.3.2	Preparation of Solution-2:.....	46
3.3.3	Combining Solution-1 and Solution-2:.....	47
3.3.4	Gel Formation: .....	47
3.3.5	Aging: .....	48
3.4	Aerogel Making.....	49
3.4.1	Autoclave purging.....	49
3.4.2	Insertion of the Autoclave Vessel:.....	49
3.4.3	Autoclave Sealing: .....	50
3.4.4	Filling with Supercritical Fluid:.....	50
3.4.5	Solvent Extraction:.....	51
3.4.6	Slow Methanol Draining.....	51
3.4.7	Supercritical drying.....	52
3.4.8	Gradual Reduction of Pressure: .....	54
3.4.9	Aerogel Collection:.....	54
3.4.10	Post-Processing Considerations:.....	54
3.4.11	Safety Precautions:.....	54
	Chapter 4.....	55
4	Results & Discussions.....	55
4.1	Condition of All Data: Result .....	55
4.2	Discussion.....	58
	Cost Analysis .....	59
	Chapter 5.....	61
	Conclusions.....	61
	References.....	62
	Appendix:.....	66



## List of Tables

Table 1: “physical properties of Silica Aerogel” .....	19
Table 2: Accessories of autoclave.....	35
Table 3:Tetramethyl Orthosilicate properties .....	40
Table 4 Methanol properties .....	41
Table 5:ammonium hydroxide properties .....	42
Table 6: CO2 properties .....	43
Table 7: Distilled Water properties .....	45
Table 8: Cost Analysis of chemicals, autoclave and additional.....	59
Table 9: Experiment 1 .....	66
Table 10: Experiment 2.....	69
Table 11: Experiment 3.....	70

## List of Figures

Figure 1.Schematic structure of the solid phase of a silica gel.[1] .....	14		
Figure 2.Types of Aerogels.[2].....	17		
Figure 3.Transition metal oxide aerogels including an iron oxide (rust) aerogel top. www.aerogel.org.....	17		
Figure 4.CO <sub>2</sub> phase diagram. www.engineeringtoolbox.com .....	23		
Figure 5.A Schematic illustration of the conventional synthesis method of aerogel.[3].....	24		
Figure 6:hydraulic connecting hose pipe and gel holder .....	35		
Figure 7: Accessories of autoclave .....	36		
Figure 8: Schematical View of Autoclave .....	36		
Figure 9:Installation of supercritical dryer (autoclave) .....	37		
Figure 10:Tetramethyl orthosilicate.....	40		
Figure 11: Carbon dioxide gas cylinder.....	44		
Figure 12:Preparation of Solution-1 .....	46		
Figure 13:Preparation of Solution-2 .....	46		
Figure 14:Combining Solution-1 and Solution-2 .....	47		
Figure 15: Gel formation .....	48		
Figure 16:Aging .....	48		
Figure 17: Submerged solgel into methanol .....	49		
Figure 18: Threading process of autoclave.....	50		
Figure 19:Filling with Supercritical Fluid .....	51		
Figure 20 : Methanol draining slowly.....	52		
Figure 21: CO <sub>2</sub> getting its supercritical condition.....	53		
Figure 22:Block diagram of process of making solgel into aerogel. ....	54		
Figure 23.graph of experiment 1.....	55		
Figure 24.graph of experiment 2.....	56		
Figure 25.graph of experiment 3.....	57		
Figure 26:Experiment 1	Figure 27:Experiment 2	Figure 28: Experiment 3	58

## List of Abbreviations

<b>Words/Signs</b>	<b>Abbreviation</b>
SAXS	Small Angle X-ray Scattering
TEOS	Tetraethyl orthosilicate
TMOS	Tetramethyl orthosilicate
PEDS	Polyethoxydisiloxane
RF	Resorcinol-formaldehyde
RC	Resorcinol Catalyst
RW	Resorcinol Water
FTIR	Fourier Transform Infrared Spectroscopy
TPP	Thermal Protective Performance
TMCS	Trimethylchlorosilane
Go	Graphene Oxide
Ga	Graphene Aerogel
PPy	Polypyrene
SEM	Scanning Electron Microscope
TGA	Transmission Electron Microscope
PANI	Polymers Polyaniline
Fig	Figure
D. Water	Distilled Water

# Chapter 1

## 1 Introduction

### Aerogel

An aerogel is a remarkable class of materials known for its extraordinary properties, including extremely low density and exceptional thermal insulating capabilities. An aerogel is a highly porous solid substance comprised mainly of air, frequently referred to as "frozen smoke" or "blue smoke" owing to its transparent appearance. Despite its low density, it exhibits a unique nanostructured network of interconnected pores, giving it impressive physical characteristics.

### Silica Aerogel

Silica aerogel, frequently referred to as the "frozen smoke" of materials, is a unique substance with an ethereal look and exceptional characteristics. Composed chiefly of silica, the same element found in sand, silica aerogels are recognized for their unusually high porosity, typically reaching 99%, which makes them one of the lightest solid materials known to science. With a nanostructured porous network, these aerogels have interconnected pores on the nanoscale scale, giving birth to their features. Their low density, typically from 0.001 to 0.5 grams per cubic centimeter, makes them incredibly lightweight. At the same time, their standout feature is their exceptional thermal insulation capabilities, making them invaluable for energy-efficient building insulation and space exploration applications. Despite their delicate and brittle character, silica aerogels find flexible uses across various sectors, displaying their capacity to handle modern difficulties in new ways.

### Early Investigations (1930s-1940s):

The development of aerogels may be traced back to the early 1930s when American scientist Samuel Kistler made pioneering discoveries. He devised a way to make a gel from a silica precursor by substituting the liquid component with a gas while keeping the gel's structure. This unique material was called "aerogel."

### **World War II (1940s):**

During World War II, aerogels found their first practical use. Due to its excellent insulating characteristics, Aerogel was exploited as a thermal insulator for military equipment, such as radar antennas and high-frequency radio equipment.

### **NASA and Space Exploration (1960s-1970s):**

In the 1960s and 1970s, NASA realized the promise of aerogels for space exploration. Aerogels were utilized as thermal insulation in many space missions, notably the Apollo program and the Mars rovers, where they played a significant role in shielding spacecraft from severe temperatures and trapping cosmic dust particles.

### **Commercialization and Expanded Applications (1980s-1990s):**

The 1980s and 1990s saw the commercialization of aerogels. Companies like Cabot Corporation started making and marketing aerogel solutions for numerous uses, including industrial insulation and oil spill cleaning.

### **Wider Range of Materials (2000s-Present):**

In the 21st century, research into aerogels has extended to incorporate more materials beyond silica, including polymers, carbon, and metal oxides. This diversification opened up new opportunities for applications in domains such as energy storage, biomedical engineering, and environmental remediation.

### **Emerging Technologies (Present-Future):**

Today, aerogels continue to be a focus of intense study and innovation. They show potential for new technologies such as supercapacitors, lightweight composites, and better insulating materials for sustainable building. The history of aerogels reflects a path of discovery, invention, and adaptation. From its modest origins as a strange material in a laboratory to its crucial role in space exploration and its rising commercial uses, aerogels have grown into a versatile class of materials with enormous promise for tackling modern difficulties in varied sectors. The future of

aerogels offers intriguing potential as researchers continue to study their unique features and uses.

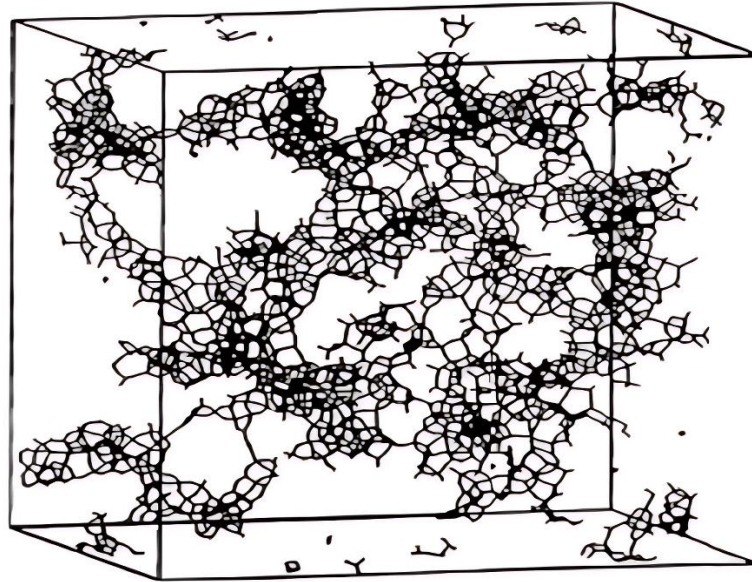


Figure 1. Schematic structure of the solid phase of a silica gel.[1]

## 1.1 Types of Aerogels

Aerogels occur in numerous kinds, each with features and compositions adapted to specific purposes. Here are some of the most popular forms of aero-gels:

### 1. “Silica-Aerogels”:

- **Standard Silica Aerogels:** These are the most well-known aerogels made of amorphous silica networks. They are lightweight, very porous, and have excellent insulating characteristics.
- **Organic-Inorganic Composite Silica Aerogels:** Silica aerogels may be mixed with organic materials to form hybrid aerogels with higher mechanical strength and specialized characteristics.

## 2. Carbon Aerogels:

- **Resorcinol-formaldehyde (RF) Aerogels** are generated from resorcinol and formaldehyde precursors. They have large surface areas and are commonly utilized in energy storage devices and catalysis.
- **Polyacrylonitrile (PAN)-Based Aerogels:** Carbon aerogels generated from PAN are recognized for their electrical conductivity and mechanical strength. They find uses in supercapacitors and sensors.

## 3. Metal Oxide Aerogels:

- **Titanium Dioxide (TiO<sub>2</sub>) Aerogels:** TiO<sub>2</sub> aerogels are recognized for their photocatalytic characteristics and are employed in applications such as water purification and solar cells.
- **Alumina (Al<sub>2</sub>O<sub>3</sub>) Aerogels:** Alumina aerogels have good thermal stability and find utility in high-temperature applications like spaceship insulation.

## 4. Polymer Aerogels:

- **Polyurea Aerogels:** These are polymer-based aerogels noted for their flexibility and mechanical strength. They are employed in applications including oil spill cleaning and thermal insulation.
- **Polyimide Aerogels:** Polyimide-based aerogels demonstrate excellent heat resistance and may be employed in aerospace and fire-resistant materials.

## 5. Chalcogel:

chalcogels are aerogels made of chalcogen elements (sulfur, selenium, or tellurium). They are researched for uses in gas sensing and electronic. Chalcogels are a specialized class of aerogels made using chalcogen elements - sulfur, selenium, and tellurium. Like other aerogels, chalcogels have highly porous structures with extremely low densities and high surface areas. However, the incorporation of chalcogen atoms gives chalcogels unique properties beyond traditional silica-based aerogels. The most common type of chalcogel studied is a sulfur-based polymer gel, which can be prepared through the polymerization of sulfur and an organic monomer. Tellurium-based chalcogels have also been synthesized and explored. The chalcogen atoms interact through van

der Waals forces and reversible covalent bonds, creating 3D networks with robust mechanical strength despite the low density.

A major application area of interest for chalcogels is chemical gas sensing. The porous chalcogel structure allows diffusion of gas molecules. Interactions between the gases and the chalcogel.

#### **6. Biopolymer Aerogels:**

Aerogels may be manufactured from biopolymers like cellulose or chitosan. These biodegradable aerogels are being studied for application in medication delivery, wound dressings, and tissue engineering.

#### **7. Metal Aerogels:**

These aerogels are formed from metal nanoparticles or nanowires. They have potential uses in catalysis, energy storage, and lightweight structural materials.

#### **8. Phenolic Aerogels:**

Phenolic aerogels are made from phenolic resins and have good fire resistance. They are used in thermal insulation and aircraft.

#### **9. Graphene Aerogels:**

These aerogels are constructed of linked graphene sheets. They feature high electrical conductivity and are employed in supercapacitors and energy storage devices.

#### **10. Bio-gels:**

Biodegradable aerogels can be used for controlled drug delivery. The porous structure of the aerogel can be loaded with drug molecules and then implanted or injected into the body. The aerogel slowly degrades over time and releases the drug in a sustained and controlled fashion. This allows for long-term delivery compared to a single bolus injection or oral dose.

Another biomedical use is in tissue engineering scaffolds. The porous network of biodegradable aerogels mimics the natural extracellular matrix of tissues. Cells can infiltrate and grow within this network. The aerogel provides initial structural support and degrades over time as new tissue grows and replaces it. This scaffolding approach helps regenerate or replace damaged tissues and organs.



Beyond biodegradable polymers, aerogels are now being made from an ever-widening array of materials like metals, ceramics, and carbon. Each formulation leads to different physical characteristics and applications. Aerogel physicists and engineers continue to expand the catalog of aerogel varieties.

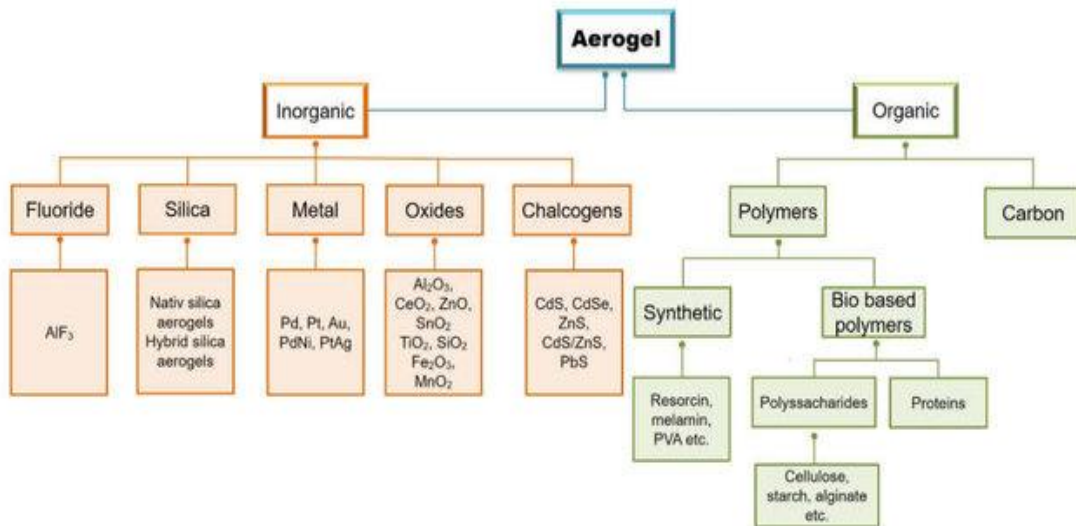


Figure 2.Types of Aerogels.[2]

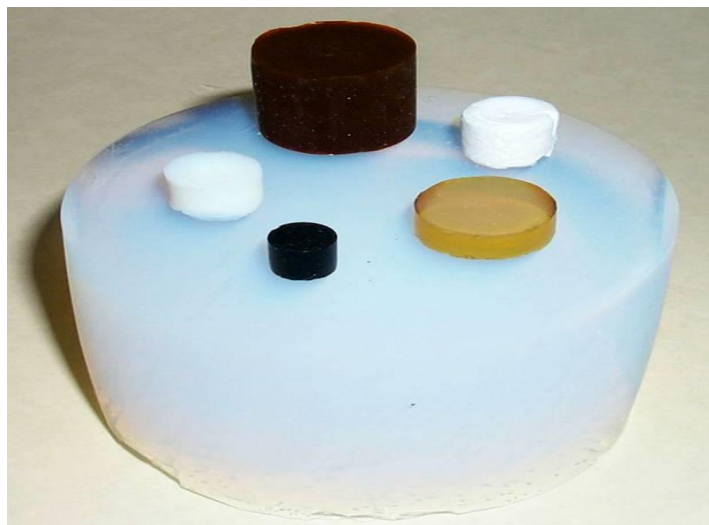


Figure 3.Transition metal oxide aerogels including an iron oxide (rust) aerogel top.  
[www.aerogel.org](http://www.aerogel.org)

## 1.2 Unique Properties of Aerogel

Its density may be as low as 0.001 grams per cubic centimeter ( $\text{g/cm}^3$ ), making it one of the lightest known solids. Regarding thermal insulation, aerogel stands out with its amazingly low thermal conductivity, measuring as low as 0.013 watts per meter-kelvin ( $\text{W/m}\cdot\text{K}$ ). This feature makes it particularly effective at blocking heat transmission. Despite its solid look, aerogel has exceptional transparency, frequently reaching 90% light transmission. It also demonstrates a fantastic absorption ability, absorbing up to 10 times its weight in liquids. Furthermore, aerogel demonstrates astonishing mechanical strength, with certain varieties withstanding compressive stresses of up to 2,000 times their weight. Lastly, it excels as an acoustic insulator, effectively damping sound owing to its low density and open structure.

These unusual features make aerogel a material of tremendous interest and promise in numerous science and engineering disciplines.

## 1.3 Limitation of Silica Aerogel

1. Silica aerogels are remarkable materials with unique properties, but they are not without limitations. Here are some standard rules of silica aerogels:
2. **Fragility:** Silica aerogels are incredibly lightweight and have a low density, making them quite fragile. They can be susceptible to damage from mechanical stress or pressure, limiting their use in load-bearing applications.
3. **Hydrophilic Nature:** Silica aerogels are highly hydrophilic, meaning they readily absorb moisture from the environment. This can lead to a decrease in their insulating properties when exposed to humid conditions.
4. **Transparency:** While the transparency of silica aerogels is advantageous for specific optical applications, it can also be a limitation in situations where opacity or structural integrity is required.
5. **Brittleness:** Silica aerogels can be brittle and may not withstand significant bending or flexing. This can limit their use in applications where flexibility is essential.
6. **High Production Costs:** The production of silica aerogels can be complex and energy-intensive, which can result in relatively high production costs. This cost factor may limit their widespread adoption in some industries.

## 1.4 Physical Properties of Silica Aerogel:

Table 1: “physical properties of Silica Aerogel”

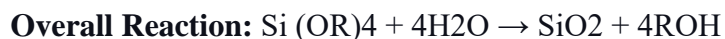
Property	Description
Apparent Density	Typically, around 0.1 g/cm <sup>3</sup> , ranging from 0.003 to 0.5 g/cm <sup>3</sup> .
Inner Surface Area	Varies from 500 to 1500 m <sup>2</sup> /g, comparable to a soccer field within a cubic centimeter.
Solid Volume Percentage	Usually about 5%, with a range of 0.13% to 15% (equivalent to 95% free space).
Mean Pore Diameter	Ranges from 20 to 150 nm and changes with density.
Primary Particle Diameter	Determined using transmission electron microscopy, typically falls between 2 to 5 nm.
Index of Refraction	Quite low for a solid material, typically between 1.007 to 1.24 (Nair = 1.004).
Thermal Tolerance	Can withstand temperatures up to 500°C, with gradual shrinkage beyond 500°C; melting point is around 1200°C.
Poisson’s Ratio	Remains constant at 0.2 regardless of density, similar to dense silica.
Young’s Modulus	Typically ranges from 0.1 to 300 MPa, significantly smaller compared to dense silica.
Tensile Strength	Approximately 16 kPa at a density of 0.1 g/cm <sup>3</sup> .
Fracture Toughness	About 0.8 kPa·m <sup>0.5</sup> for a density of 0.1 g/cm <sup>3</sup> , determined via 3-point bending.
Dielectric Constant	Very low at 1.1 for a density of 0.1 g/cm <sup>3</sup> , exceptionally low for a solid material (k <sub>air</sub> = 1).
Acoustic Impedance	Measured at 104 Kg/m <sup>2</sup> ·s using ultrasonic methods at kHz frequencies.
Sound Velocity	Varies from 20 to 800 m/s, reaching 100 m/s at a density of 0.07 g/cm <sup>3</sup> , which is remarkably low for a solid material.
Optical Property	High transmittance (>90%) at 630nm wavelength, exhibiting transparency with a slight blue haze.
Thermal Conductivity	Very low at 0.02 W/m·K (20°C), providing substantial insulation, equivalent to 30 panes of glass in a 2 cm slab.

## 1.5 Aerogel Applications

1. **Thermal Insulation:** Aerogels are excellent thermal insulators. They are utilized in construction materials, clothes (e.g., insulating coats), and spacecraft to decrease heat transmission.
2. **Oil Spill Cleaning:** Hydrophobic aerogels can absorb and recover oil from water surfaces, making them beneficial for environmental cleaning operations in oil spills.
3. **Aerospace:** Aerogels are utilized in aerospace applications to insulate spacecraft, protect delicate sensors from severe temperatures, and decrease the weight of spacecraft components.
4. **Energy Storage:** Aerogels are employed in energy storage devices, such as supercapacitors and batteries, to increase their performance and energy density.
5. **Cryogenics:** They are employed as insulating materials in cryogenic applications, including storing and transporting liquefied gases like liquid nitrogen and helium.
6. **Environmental Remediation:** Aerogels are utilized in adsorption techniques to remove pollutants and toxins from air and water.
7. **Personal Care Products:** They may be found in cosmetics and skincare products for their light, airy texture and ability to absorb excess oil.
8. **Transparent Insulation:** Silica aerogels may be employed as transparent insulating materials in windows and skylights to offer thermal insulation without compromising natural light.
9. **Scientific Research:** Aerogels are utilized as substrates and support for several scientific studies, including particle detectors and high-energy physics research.
10. **Lightweight Structural Components:** In certain circumstances, aerogels are employed as lightweight structural materials in aerospace and engineering applications.
11. **Soundproofing:** Aerogels may be utilized as sound-absorbing materials in architectural acoustics to minimize noise pollution.
12. **Medical Applications:** They have been examined for drug delivery systems, wound dressings, and tissue engineering because of their large surface area and biocompatibility.
13. **Protective Coatings:** Aerogels may be used as protective coatings for sensitive equipment and electronic devices to protect them from harsh environmental conditions.
14. **Nanotechnology:** Aerogels serve as templates and scaffolds for developing nanomaterials and nanoparticles in nanotechnology research.

## 1.6 Synthesis of Silica Aerogel

Silica aerogels, known for their extraordinary properties, have come a long way since their inception. The original method, developed by S.S. Kistler in 1931, was time-consuming, taking weeks to produce a single aerogel sample. This lengthy procedure posed significant challenges for researchers. A breakthrough occurred in 1962 when a French group led by S.J. Teichner rekindled interest in aerogels. One of Teichner's students working on the project even experienced a nervous breakdown, fearing that it would take years to complete his thesis at that rate. Fortunately, a much faster method emerged, utilizing Tetramethoxysilane (TMOS), dissolved in methanol and hydrolyzed with water to form a silica gel. The gels used in the production of silica aerogels most commonly originate from tetraalkoxysilanes  $\text{Si (OR)}_4$  precursors. These precursors undergo hydrolysis, where water molecules split into  $\text{H}^+$  cations and  $\text{OH}^-$  anions and react with the precursor molecules to form  $\text{Si-OH}$  and  $\text{ROH}$ . Subsequently, condensation reactions occur in a solution containing water, alcohol, and catalysts (acidic or basic). These reactions are pivotal in forming the desired gel structure, leading to the creation of silica aerogels. The complete chemical reaction during silica aerogel formation:



These reactions involve the precursor ( $\text{Si (OR)}_4$ ) reacting with water to form silicic acid ( $\text{Si (OH)}_4$ ), followed by the formation of silicon-oxygen-silicon ( $\text{Si-O-Si}$ ) bonds through condensation. These reactions build silica aerogels' intricate network structure, giving them unique properties.

### Supercritical Drying

Supercritical drying is a method used to create aerogels from gels. The idea is to replace the liquid inside the gel with air carefully. This is important because if the liquid evaporates regularly, it can damage the structure of the gel. This damage happens because of the forces created by the liquid. To avoid this problem, the forces created by the liquid need to be eliminated. This is done by

turning the liquid inside the gel into a supercritical fluid. To do this, the temperature and pressure of the liquid are changed. The exact temperature and pressure depend on what kind of liquid is being used.

### **Supercritical Fluid: A Special State**

Supercritical fluid is a unique state of matter. It occurs when a liquid is pressurized and heated past a certain point. In this distinctive state, the fluid exhibits properties of both a regular liquid and a gas. It can dissolve substances, like a liquid, but lacks the cohesive forces typical of a liquid, resembling a gas.

One interesting aspect of supercritical fluids is their ability to readily mix, akin to different gases. These characteristics hold significance in aerogel production, particularly when dealing with a liquid composed of water and alcohol.

### **In Making Silica Aerogels**

When silica aerogels are made, the liquid in the gel is mostly water and alcohol, like ethanol or methanol. Nevertheless, to make water or alcohol into a supercritical fluid, high temperatures and pressures have to be used. For example, to make water into a supercritical fluid, there need highly high conditions—374°C and 22063.22 Kpa (3200 psi). Ethanol needs 190.9°C and 6140.264 Kpa (890.57 psi), while methanol requires 189.6°C and 8085.758 Kpa (1172.74 psi). These conditions are challenging and can be risky to work with.

### **Challenges and Safety**

Using water as a supercritical fluid requires expensive equipment like autoclaves. On the other hand, making ethanol and methanol supercritical can be dangerous due to the high temperatures and pressures involved. In 1984, a serious incident occurred when a 3000-liter autoclave exploded, showing the dangers of using ethanol-based supercritical drying.

### **A Safer Option: Carbon Dioxide**

To overcome these challenges and safety issues, scientists have turned to carbon dioxide (CO<sub>2</sub>) as a safer alternative to ethanol and methanol. CO<sub>2</sub> can quickly become a supercritical fluid, making the drying process safer and more controllable. Replacing the liquid in the gel with supercritical CO<sub>2</sub> makes the transition from liquid to gas smooth, reducing the risk of damaging the delicate aerogel structure.

Using carbon dioxide instead of traditional liquids for supercritical drying has made aerogel production safer and more efficient and has also opened up new possibilities for aerogel applications. This smart approach shows how science can improve materials and technologies.

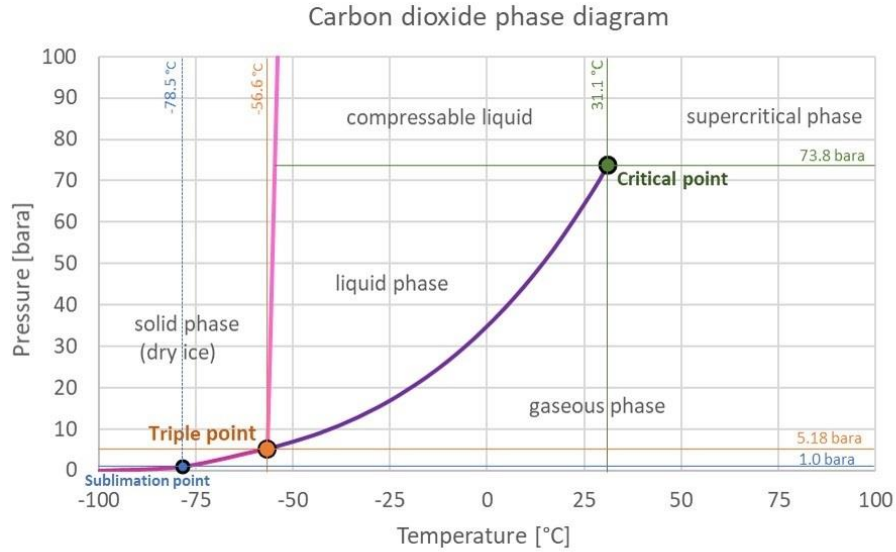


Figure 4.CO<sub>2</sub> phase diagram. [www.engineeringtoolbox.com](http://www.engineeringtoolbox.com)

## 1.7 Aerogel Drying Process

**Supercritical Drying:** Utilizes supercritical fluids to eliminate solvent, keeping a highly porous structure. Ideal for creating low-density, high-porosity aerogels but needs specialist equipment.

**Low Temperature Supercritical Drying:** Using CO<sub>2</sub> but at a low temperature around 40°C. Easier but slower process.

**Ambient Pressure Drying:** Simple and accessible approach where the gel is left to air dry naturally. Easier to install but may result in some structural shrinking.

**Ambient Pressure Drying with Freeze-Drying:** Combines ambient pressure drying with freeze-drying to minimize structural collapse and promote porosity.

**Ultrasonic-Assisted Drying:** Uses ultrasonic waves to expedite solvent removal during drying, increasing porosity and structural integrity.

**Microwave-Assisted Drying:** Microwaves are applied to speed solvent removal, allowing control over drying conditions for quicker outcomes.

**Surface Modification:** Replacing solvent with an organic liquid like hexane that can evaporate at ambient conditions. Can reduce cracking but shrinks pores.

**Freeze Drying:** Freezing the Gel and solvent, then removing the frozen solvent by sublimation. Can work but requires very careful processing to avoid damage.

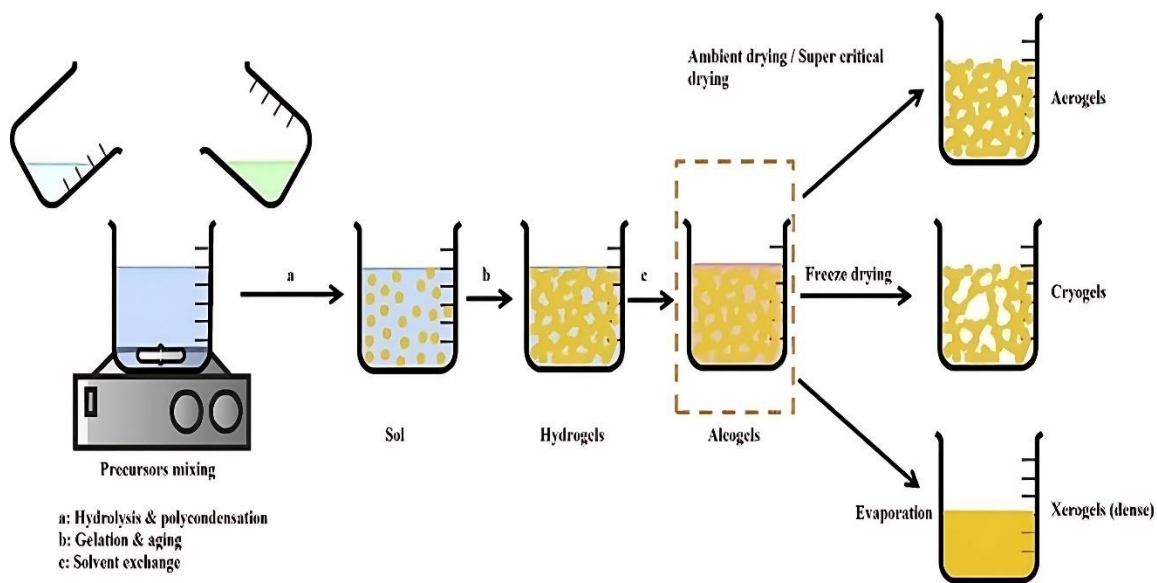


Figure 5.A Schematic illustration of the conventional synthesis method of aerogel.[3]

## 1.8 Significance of the Research

While silica aerogels have demonstrated their potential in various applications, there is an ongoing quest to improve their properties and make their production more cost-effective. One critical aspect of this research is choosing a CO<sub>2</sub> source for supercritical drying. Traditionally, siphon CO<sub>2</sub> gas cylinders have been used for this purpose. “However, there is growing interest in exploring the use of ordinary CO<sub>2</sub> gas cylinders”, which are more readily available and cost-effective. This shift in the choice of CO<sub>2</sub> source presents an opportunity to make the aerogel synthesis process more



accessible and economical. This research is significant because it investigates the feasibility of using ordinary CO<sub>2</sub> gas cylinders for supercritical drying in silica aerogel synthesis. The potential advantages, such as cost reduction and wider availability, could lead to broader adoption of aerogel technology in various industries.

## **1.9 Problem Statement**

The synthesis and manufacturing of silica aerogels by supercritical drying is a well-established technology with several applications in diverse sectors, ranging from thermal insulation to catalysis. Traditionally, siphon CO<sub>2</sub> gas cylinders have been the chosen source for supercritical drying owing to their regulated release of CO<sub>2</sub> and consistent results. However, the availability, expense, and environmental problems related to siphon CO<sub>2</sub> cylinders have motivated the research of alternate sources, including conventional CO<sub>2</sub> gas cylinders. The challenge addressed in this thesis is the necessity to analyze the feasibility and efficacy of employing conventional CO<sub>2</sub> gas cylinders as an alternate source for supercritical drying in the synthesis of silica aerogels. While this technique promises cost reductions and broader accessibility, it raises problems about the influence on aerogel characteristics, repeatability, and overall appropriateness for specific applications. Therefore, a detailed analysis is necessary to understand the degree to which aerogel characteristics are impacted by the choice of CO<sub>2</sub> source, as well as to discover possible benefits, limits, and areas for improvement in the synthesis process. “This research aims to bridge the gap in knowledge regarding the use of ordinary CO<sub>2</sub> gas cylinders for supercritical drying in silica aerogel synthesis, providing valuable insights that can inform future advancements in materials science and engineering and guide the selection of CO<sub>2</sub> sources for various applications.

This research seeks to fill the gap in knowledge regarding use of conventional CO<sub>2</sub> cylinders in silica aerogel synthesis. Through systematic exploration, it aims to elucidate the feasibility, limitations, and potential benefits of this alternate approach. Outcomes will guide selection of appropriate CO<sub>2</sub> sources based on specific production objectives and end-use applications. More broadly, this work intends to advance scientific understanding of aerogel production methods to enable ongoing improvements in these exceptional engineered nano porous materials.

## **1.10 Hypothesis**

This research suggests that using gaseous CO<sub>2</sub> instead of liquid CO<sub>2</sub> for the drying process in aerogel production might lead to superior aerogels. It is also believed that gaseous CO<sub>2</sub> offers advantages such as enhanced safety, excellent suitability for large-scale projects, and improved environmental friendliness. The notion is that aerogels manufactured using this approach should exhibit similar quality while reducing risks and expenses. It is anticipated that these aerogels will possess strength, uniform pore distribution, and a heightened level of safety in handling. Cost savings and environmental benefits may result from these advancements. These ideas are slated for validation through experimental tests and a comprehensive examination of the outcomes. The total capital expenditure for the drying plant capable of producing 525,000 kg/year of 3 cm thick aerogel was projected at \$0.9 million. The yearly operating cost for this facility was calculated at \$0.63 million. The drying cost per unit area reduced exponentially as the thickness decreased, with 1 cm thick aerogel having a drying cost of just \$1.2/m<sup>2</sup>. [4].

The cost-effectiveness of utilizing ordinary CO<sub>2</sub> cylinders for supercritical drying will be evaluated to achieve aerogels in a much more cost-effective manner, with the total production cost, including the Autoclave, being significantly reduced.

## **1.11 Objectives**

The primary objective of this research is to Manufacture the silica aerogels using ordinary CO<sub>2</sub> gas cylinders with the traditional method. The study aims to assess the impact of the CO<sub>2</sub> source on the aerogel's properties, including its density, porosity, thermal conductivity, and structural integrity. Specific research objectives include:

- Manufacturing of silica aerogels synthesized using ordinary CO<sub>2</sub> cylinders.
- Evaluating the cost-effectiveness of using ordinary CO<sub>2</sub> cylinders for supercritical drying.
- Investigating the product quality by choosing CO<sub>2</sub> over siphon type CO<sub>2</sub> cylinder.

## Chapter 2

### 2 Literature Review

#### **Silica aerogels:**

Silica aerogels with large surface area, low density, low thermal conductivity, and strong optical translucency are interesting for numerous applications requiring water resistance, such as transparent insulation systems and drug delivery platforms, and may be manufactured using diverse processes. Aerogels are unusual materials with excellent porosity and low density, frequently referred to as "frozen smoke" or "solid air."

These may be manufactured from numerous materials, including silica, metal oxides, polymers, and carbon-based compounds.[5]

physical characteristics of silica aerogels produced by three distinct precursors (TEOS, PEDS, TMOS) are presented. The monolithicity of the aerogels is significantly reliant on the catalyst utilized for each precursor. Acid catalysts resulted in monolithic aerogels for TEOS and PEDS precursors, whereas base catalysts resulted in monolithic aerogels for TMOS precursors.[6]

The RSCE procedure employs a hydraulic hot press to seal and heat a mold holding aerogel precursors until they reach a supercritical state. After stabilization, the hot press restraining force is lessened, enabling the supercritical fluid to escape and leaving behind an aerogel monolith. The complete procedure takes less than 3 hours. Various factors are regulated, including the restraining force, maximum temperature, heating and cooling rates, pressure release rate, and mold volume fill ratio. Silica aerogels were synthesized using a TMOS-based method, and the impacts of several factors were explored.[7]

Aerogels have great thermal quality owing to their physical and chemical structure, resulting from an improved manufacturing procedure[8]

The resorcinol-formaldehyde (RF) aerogels were discovered to be mesoporous materials with large surface areas and few micropores. The surface area of the RF aerogel was regulated by the mole ratio of resorcinol to primary catalyst (RC). In contrast, the mesopore volume varied using RC or the ratio of resorcinol to water (RW).[9]

A way to create silica aerogel composites with outstanding thermal insulation and mechanical characteristics. It contains MATLAB programs for constructing interactive composition-property charts and gives extensive analysis of experimental data using theoretical models and numerical simulations.[10]

effective impregnation of flexible aerogel onto 4-layer-aligned glass fibers, resulting in nanocomposites with low thermal conductivity and potential for usage in thermal insulation applications. Different configurations of the fiber layers allow for control over the laminated structures of the aerogel composites. Microstructure analysis revealed a homogenous dispersion of nonporous structure and highly aligned fibers inside the aerogel matrix. The fiber-aerogel composites' thermal and mechanical characteristics mainly rely on the fiber layers' laminated design.[11]

Experimental findings on producing clear silica aerogels with low heat conductivity are reported using sodium silicate solution and several solvents. The research focuses on the influence of preparation parameters, such as the number of stages, hydrosol pH, and gel aging temperature, on the characteristics of aerogels. The following parameters were measured: density, thermal conductivity, optical transmission, and contact angle. Fourier Transform Infrared Spectroscopy (FTIR) testing discovered Si-C, C-H, Si-O-Si, and OH bonds. The strength of these connections varied depending on the manufacturing processes, pH, and aging temperature. The optical transmission of the aerogels decreased at aging temperatures of up to 50°C. [12]

The solid silica matrix restricts gas molecules' motion in silica aerogel, leading to changes in mean free path, velocity distribution function, diffusivity, viscosity, and thermal conductivity. The mean free path of gas molecules in aerogel is projected to be roughly the same as in free space for pressures over 10 bar. However, when the pressure is lessened, the mean free path becomes a fixed finite value instead of growing as in free space. Thermal conductivity of a gas in aerogel begins to decline at pressures beyond 10 bar and becomes virtually zero at 0.01 bar. Experimental data support the anticipated thermal conductivity of a gas in aerogel.[13]

Aerogel composite materials offer heat conductivity below 25 mW/mK and high sound absorption, superior to traditional insulators.

Examples include aerogel in bulk form, bound with polymers like PVB, and bonded with vinyl acetate dispersions.[14]

Compared with silica aerogels and activated charcoal, the thermodynamics of azo dye adsorption on a newly synthesized titania-doped silica aerogel.[15]

The research exhibits the potential of aerogel catalysts to convert alcohols to amines via adjustment of composition and reaction conditions.[16]

The research provides a streamlined sulfuric acid approach to describe complicated neutral oils swiftly.[17]

It has been proven possible to create mixed-oxide aerogels using a technique that uses SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and ZrO<sub>2</sub>. The process entails hydrolyzing the suitable alcoholate at room temperature after dissolving it in alcohol or benzene. The solvent is subsequently released in an autoclave under very pressured circumstances. With this method, there is no need to purify the metal oxide that has precipitated or replace water with an organic solvent as is often done when making gels using water. Silica aerogels with parameters, such as the apparent density of 0.05 g cm<sup>-3</sup>, the surface area of 1000 m<sup>2</sup> g<sup>-1</sup>, and the pore volume of 18 cm<sup>3</sup> g<sup>-1</sup>, were successfully created using this process. The resultant aerogels are initially hydrophobic but can be turned into hydrophilic aerogels and made transparent.[18]

A particular manufacturing procedure was used to create silica aerogel for the TASSO Cherenkov detector, which has an index of refraction of  $n = 1.024$ . Various parameters were changed to find out how several factors affected the aerogel samples' form and optical quality.[19]

Exhibits good transparency in the optical area and gives excellent thermal insulation if evacuated. It is ideal as a super-insulating spacer in evacuated window systems. Has a sound velocity of roughly 120 ms at the lowest densities accessible, which is the least yet observed for inorganic solid materials.[20]

The article analyzes how solvent variables like the dielectric constant impact the decomposition kinetics, indicating an ionic transition state. The dioxane-water system permitted systematic modification of solvent characteristics.[21]

The research highlights the ability of silica aerogel as a flattening agent in coatings, demonstrating that it delivers effective flattening while retaining transparency and film characteristics. The ways to optimally integrate aerogel are also discussed.[22]

The research illustrates the insecticidal efficiency of silica aerogel dust for preserving stored agricultural goods against insect damage.[23]

The pore structure of alumina aerogels was examined utilizing nitrogen and carbon tetrachloride adsorption. Pore structure was clarified using gas adsorption, indicating the opening of limited pores in ether aerogels but not alcohol ones.[24]

The structure of porous silica aerogel was examined using small-angle X-ray scattering (SAXS). It clarified the fractal structure of silica aerogel, connecting it to colloidal aggregation during manufacture. This offers insight into the origins of porosity in aerogels.[25]

Ambient temperature CO<sub>2</sub> supercritical drying method as a better, more efficient process for generating high-grade clear silica aerogels. It illustrates the viability and benefits of this innovative technique over previous high-temperature approaches. Drying periods are significantly quicker with the CO<sub>2</sub> process (8-10 hours versus 2-3 days) and more consistent high-grade aerogels.[26]

The Young's modulus of silica aerogels was evaluated using three-point flexural testing. The research demonstrates that synthesis factors impact aerogel structure and elastic characteristics beyond density, connecting structure, and mechanics.[27]

Characterizing the structure of silica in aerogel, which was previously unknown, using Raman spectroscopy and comparing it to fused silica to understand how the silica network varies in the porous aerogel form.[28]

The article's primary goal was to experimentally analyze the possibilities of incorporating aerogel into firefighter apparel to increase its thermal protective properties, analyzing several performance variables. Aerogel has extremely low thermal conductivity; the purpose was to examine whether aerogel incorporation may boost the TPP of firefighter apparel to provide further protection.[29]

The primary goal of this work is to experimentally estimate the diffusion coefficients for methanol in liquid and supercritical CO<sub>2</sub> in silica aerogel. The diffusion dynamics are crucial for improving the supercritical drying process to generate high-quality, clear aerogel monoliths. By evaluating diffusion time influences aerogel drying outcomes and correlating it to diffusion models, the authors intended to quantify the diffusion coefficients required to forecast optimum drying settings. The objective was to understand the diffusion kinetics of the supercritical drying process to provide improved control and optimization for generating precise aerogel products.[30]

This work aimed to create silica aerogel utilizing the sol-gel and supercritical drying procedures. Supercritical drying was selected as the most effective procedure, providing the optimum qualities of the product. They devised and constructed the supercritical dryer stand that enabled drying samples at high pressure and enhanced temperature. The resulting silica aerogels demonstrated excellent transparency, homogeneity, and equivalent density.[31]

To propose and investigate an efficient supercritical drying process as a scalable manufacturing method for silica aerogels at prices permitting commercial viability. Due to slower diffusion, required drying time and expenses grow exponentially with aerogel thickness. Estimated costs are \$1-2 per m<sup>2</sup> for 1cm thick aerogel plates, substantially cheaper than high temperature drying.[32]

The fundamental innovation is the one-step solvent exchange/surface modification of wet gels using an ethanol/TMCS/heptane solution to permit ambient drying while limiting shrinkage and fractures. The research seeks to prove that a unique approach may create high-quality aerogels similar to supercritical drying. The objective is to develop and describe a novel ambient pressure drying technique as an improved approach for synthesizing silica aerogels from waterglass in a simple, low-cost way.[33]

Silica aerogel/glass fiber composites were applied by press forming with additional TiO<sub>2</sub> particles. Glass fibers improved the strength of composites but increased their thermal conductivity. Adding

TiO<sub>2</sub> particles lowered radiative thermal conductivity, particularly at higher temperatures. Thermal conductivity reduced from 0.041 to 0.030 W/mK at 700°C with 20wt% TiO<sub>2</sub> added. A moderate forming pressure of 1.5 MPa produced excellent thermal insulation qualities. Density rose, and pores were more constricted at higher pressures, increasing conductivity.

Mixing silica aerogel with glass fibers and TiO<sub>2</sub> particles enables the production of composites with better strength and decreased heat conductivity compared to pure aerogel. The composition and processing conditions considerably affect the resultant qualities.[34]

Transition metal integrated titania-silica aerogels (M-TiO<sub>2</sub>-SiO<sub>2</sub>, where M = V, Cr, Mn, Fe, Ni) with large surface areas and pore volumes for acetaldehyde oxidation to CO<sub>2</sub> under UV and visible light irradiation. M-TiO<sub>2</sub> samples without silica exhibited apparent light absorption but no photocatalytic activity. M-TiO<sub>2</sub>-SiO<sub>2</sub> samples were active photocatalysts in visible light, with Cr, Co, and Ni being the most active. The presence of silica was needed for visible light action.

Under UV irradiation, M-TiO<sub>2</sub>-SiO<sub>2</sub> also demonstrated better photocatalytic activity than conventional TiO<sub>2</sub>. The authors claim that high oxidation states of transition metals like Cr<sup>6+</sup> Co<sup>3+</sup> are critical for visible light photocatalytic activity. The close mixing of transition metal oxides, TiO<sub>2</sub>, and SiO<sub>2</sub> in the aerogel structure certainly contributes to the photocatalytic activity.[35]

This study proposes a straightforward approach to the synthesis of strong and lightweight carbon aerogels from the phenol-formaldehyde resin by performing the polymerization under hypersaline conditions using zinc chloride (ZnCl<sub>2</sub>) salt without the requirement for specific drying procedures like supercritical drying. ZnCl<sub>2</sub> salt is vital in facilitating this process and producing high porosity. The resultant materials have potential uses in adsorption, separation, and energy storage.[36]

Exploring silica aerogels as prospective carrier materials for medicines, employing supercritical CO<sub>2</sub> for loading the medications by adsorption. Two model medications were tested - ketoprofen and miconazole. Their adsorption onto hydrophilic silica aerogels was evaluated from scCO<sub>2</sub> solutions. Both medicines demonstrated considerable affinity to the aerogel surface. Uptake was up to 1.2 mmol/g for ketoprofen and 1.54 mmol/g for miconazole. No medicines were degraded throughout the adsorption process, as validated by IR, UV-vis spectroscopy, and HPLC. The



adsorption isotherms are essential for regulating the dose in drug-aerogel compositions. The surface chemistry affects the adsorption capacity and may be utilized to modify the release kinetics.[37]

Creating a multifunctional bio-gel covering from gelatin and chitosan may provide flame retardancy to wood in an eco-friendly way. The coating uses self-healing, water retention, and charring behavior to protect wood from fire. The coating displayed outstanding self-healing capacity to recover from damage when heated to 60°C. It might also be recycled by redissolving in water. Bio-gel-coated wood demonstrated better fire-retardant capabilities than uncoated wood, including a 24% lower peak heat release rate, 17.2% lower overall heat release, and six times more incredible ignition delay time in cone tests.[38]

Graphene oxide (GO) was converted to graphene aerogel (GA) using L-ascorbic acid. Polyaniline (PANI) and polypyrrole (PPy), two conductive polymers, are produced in situ inside the pores of the GA to create GA/PANI and GA/PPy composites. SEM, FTIR, and TGA characterized GA, GA/PANI, and GA/PPy. PANI and PPy incorporation was verified.[39]

Stannous chloride ( $\text{SnCl}_2$ ) was used as a dopant during the ultrasonic-assisted sol-gel process to create Sn-TiO<sub>2</sub> aerogel. Amorphous and anatase phases co-existed in the uncalcined Sn-TiO<sub>2</sub> aerogel, referred to as a co-existing phase. The Sn-TiO<sub>2</sub> aerogel co-existing phases showed a high specific surface area of 172.4 m<sup>2</sup>/g, higher than that of other anatase TiO<sub>2</sub> aerogels previously described. The main novelty is the direct production of a mixture of high surface area amorphous and anatase phase TiO<sub>2</sub> aerogels by Sn doping utilizing an ultrasonic sol-gel technique without calcination.[40]

Unlike conventional multi-step modification, the co-precursor technique enables in situ surface modification during aerogel production. Without the requirement for further surface preparation, hydrophobic aerogel was produced from TEOS-TMCS co-precursors. Hydrophobic TEOS-TMCS aerogel has a greater contact angle, rising from a hydrophilic TEOS-only aerogel's 27° to 110°.[41]

Aerogel composite materials offer heat conductivity below 25 mW/mK and high sound absorption, superior to traditional insulators.

# Chapter 3

## 3 Methodology

This thesis paper presents a straightforward yet methodically crafted approach that explores materials science and the complex field of aerogel production. It is firmly based on the basic ideas and methodologies discussed in the previous essay. It meticulously weaves crucial elements, each of which plays a unique and crucial function in research pursuits, into the complicated fabric of the methodological framework. The careful selection and meticulous preparation of materials, the complex alchemy of sol-gel formation, the exact orchestration of autoclave setup and operation, the crucial stage of supercritical drying, the methodical and meticulous collection and analysis of data, and an unwavering dedication to upholding strict safety protocols are all included in these components. This rigorous methodology acts as the compass directing the scientific journey and is comparable to the immovable foundation of research. It guides and drives the research projects, data collection efforts, and analytical procedures, all geared at advancing the exciting and dynamic field of aerogel production and materials science. In the following sections, going in-depth on each element of the carefully created process, illuminating the minute aspects that support the research goals and add to the overall body of knowledge and innovation in materials science and aerogel manufacturing. Here all the experiments were performed with the help of some peripheral support equipment and chemicals. A short description of this support system is given below.

All the experiments were performed with the help of some peripheral support equipment and chemicals. A short description of this support system is given below.

### 3.1 Common peripheral Equipment's:

#### 3.1.1 A supercritical dryer or autoclave:

A supercritical dryer or autoclave is a specialized device used in materials science and engineering to remove solvents from substances, such as gels, without causing structural damage. It employs a supercritical fluid, often carbon dioxide, which exists in a state between a gas and a liquid at specific temperature and pressure conditions. This supercritical fluid replaces the solvent within the material and allows for its removal without collapsing the material's porous structure, making

it particularly valuable in the production of aerogels and other highly porous materials. The accessories of the autoclave are following:

Table 2: Accessories of autoclave

Serial no	Description	Quantity
1.	2"×2" thread T (autoclave)	1
2.	4" plug (threaded)	1
3.	2" plug (threaded)	1
4.	2" nipple	1
5.	2" threaded cap	1
6.	Borosilicate temper glass	1
7.	4" diameter Pressure Gauge	1
8.	½" needle valve	3
9.	½" × ½" bush	1
10.	½" nipple	3
11.	gasket	
12.	Gel holder	1

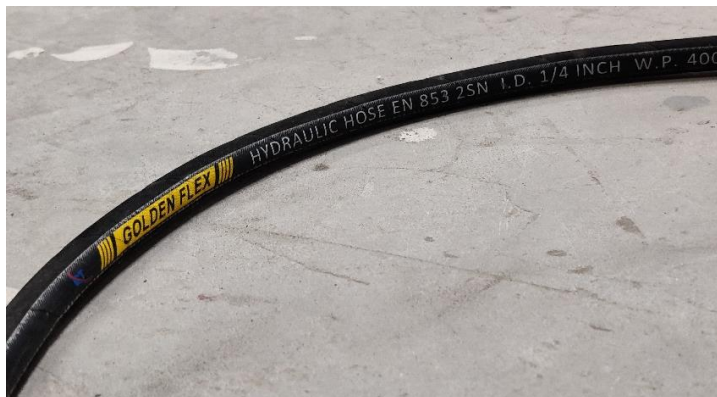


Figure 6:hydraulic connecting hose pipe and gel holder



Figure 7: Accessories of autoclave

**3.1.1.1 Schematical View of Autoclave :**

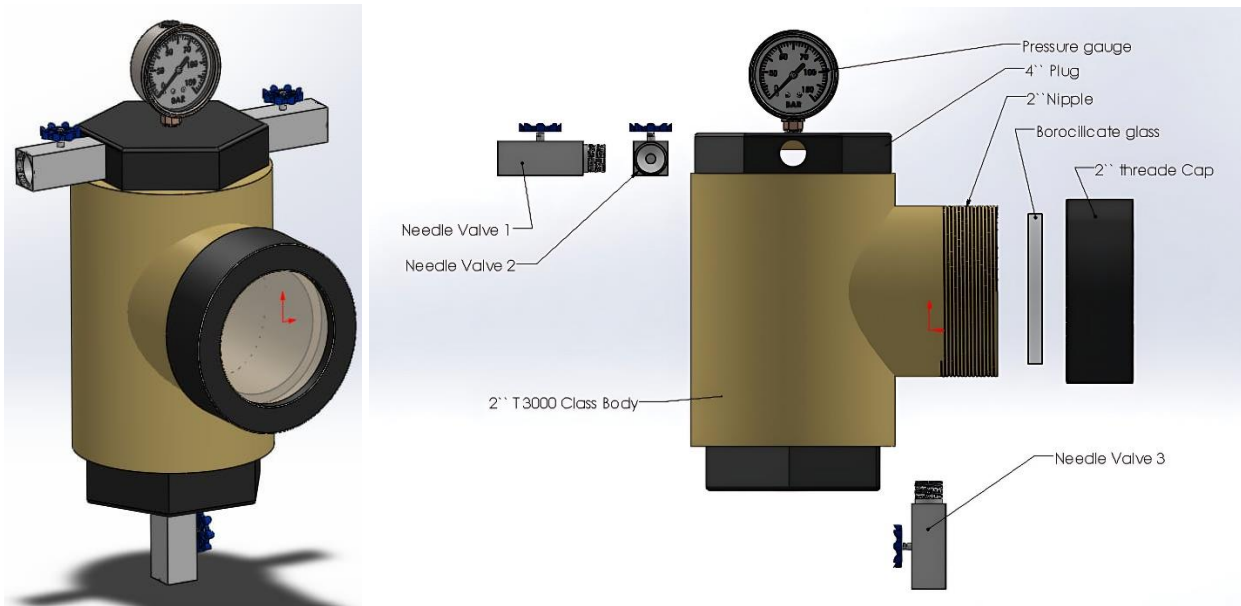


Figure 8: Schematical View of Autoclave

### 3.1.1.2 Installation of supercritical dryer (autoclave):

Before starting an experiment aiming for supercritical conditions, ensure proper autoclave setup:

1. Wrap the autoclave in glass fiber insulation to retain heat and provide electrical insulation.
2. Use nichrome wire as the heating element due to its high-temperature resistance.
3. Connect the wire to a Variac for precise voltage control.
4. Ensure a secure and appropriate electrical supply connection.
5. Gradually increase voltage using the Variac to heat the autoclave.
6. Monitor and maintain the autoclave's temperature for sterilization or experimental conditions.
7. Keep the voltage below 40 volts to prevent damage to the nichrome wire.
8. Continuously observe and adjust as necessary during operation.
9. Safely shut down by reducing voltage and turning off the Variac.
10. Allow the autoclave to cool before opening it for safety.
11. Prioritize safety measures throughout the entire process.



Figure 9: Installation of supercritical dryer (autoclave)

### 3.1.1.3 Explanation of the working process of the autoclave:

#### ▪ **Preparation:**

- The T-shape socket body, made of mild steel, serves as the main vessel where the processes will take place.
- The sight glass, made of borosilicate tempered glass, is attached to one of the ports on the T-shape socket body. This sight glass allows operators to visually monitor the interior of the vessel during the process.
- The inlet needle valve is one of the components and will be used to control the flow of gases and liquids into the vessel.

#### ▪ **Pressure Control:**

- The pressure gauge, with a range of 0 to 250 bars, is installed on the vessel to monitor and control the pressure inside.
- The inlet needle valve is used to introduce gases and liquids into the vessel, allowing precise control over the process.

#### ▪ **Execution:**

- Depending on the specific application, the vessel is loaded with the material or substances to be processed or treated. This could include chemical reactions, sterilization of equipment, or other processes.
- The pressure inside the vessel can be increased to the desired level, which is monitored using the pressure gauge.

#### ▪ **Observation:**

During the process, the operator can observe the interior of the vessel through the sight glass. This is especially important for processes that require visual monitoring, such as reactions or sterilization.

#### ▪ **Controlled Release:**

After the process is complete, or at any point, when necessary, the pressure inside the vessel can be released using the outlet needle valve. This controlled release allows for safe depressurization.

▪ **Safety and Maintenance:**

- The extra outlet needle valve can be used to pass out gases and liquids from the vessel as needed. This can be important for safety or for transferring processed materials.
- The inlet needle valve is also used to control the introduction of gases and liquids into the vessel, ensuring precise control over the process parameters.

In summary, the autoclave is a pressure vessel equipped with components such as a pressure gauge, inlet and outlet needle valves, a sight glass, and an extra outlet needle valve. It is used for conducting various processes while controlling pressure, monitoring visually through the sight glass, and ensuring safety through controlled pressure release and material transfer. The specific application of the autoclave may vary depending on the user's needs, but it is commonly used in laboratory and industrial settings for processes requiring controlled pressure environments.

**3.1.2 Variac:**

A variac, short for "variable autotransformer," adjusts AC voltage in electrical circuits. It's a type of autotransformer with a single winding and a movable tap for voltage control. Variacs find common use in electrical testing, research, and industry for precise voltage control.

Their main purpose is to provide adjustable AC voltage smoothly, eliminating the need for multiple transformers. Variac's features a user-friendly knob or dial for gradual voltage changes, ideal for applications like lamp brightness control, electronic equipment testing, and motor speed regulation.

Variac's come in various sizes and voltage ratings, including single-phase and three-phase models. They are invaluable tools for AC voltage control in electrical engineering and electronics applications.

<b>Working fluid</b>	<b>Test and Heating</b>	<b>Insulating apparatus</b>
CO <sub>2</sub> gas	<ul style="list-style-type: none"><li>• Variac.</li><li>• Power Supply Unit.</li><li>• Nichrome Wire</li><li>• Thermometer</li></ul>	Glass fiver

### 3.2 Common Chemicals:

#### 3.2.1 TMOS as precursor:

The choice of precursor materials depends on the type of aerogel being produced. For silica aerogels, Tetramethyl Orthosilicate (98% concentration) is used for this process which chemical compound with the molecular formula  $\text{Si}(\text{OMe})_4$ , where Me represents a methyl group ( $\text{CH}_3$ ). TMOS is an organosilicon compound and a type of silicate ester. It is commonly used as a precursor or source material in the production of silica-based materials, such as silica sol-gels, silica aerogels, and various forms of silicon dioxide ( $\text{SiO}_2$ ) for applications.

Tetramethyl Orthosilicate properties:

Table 3: Tetramethyl Orthosilicate properties

SL. No.	Parameters	Symbol	Quantity	Unit
1.	Freezing temperature	$T_{\text{freeze}}$	-88°C	°C
2.	Boiling temperature	$T_{\text{boil}}$	160°	°C
3.	Density	P	0.936	kg/m <sup>3</sup>
4.	Specific heat (at 20°C)	$C_p$	0.59	Kj/kg-k
5.	Vapor pressure	$P_v$	1.33	kPa
6.	Molar mass	$M_s$	152.26	g/mol



Figure 10: Tetramethyl orthosilicate



### 3.2.2 Methanol as Solvents and Cleaning Agents:

Solvents like methanol are used to dissolve and create a gel from the precursor material.

Methanol is used as a cleaning agent which is used during gel preparation and processing to remove impurities and ensure a clean starting material. Lab grade Methanol (30% concentration) is used for the process.

Methanol, also known as methyl alcohol, wood alcohol, wood naphtha, or wood spirits (sometimes abbreviated as MeOH), possesses the chemical formula CH<sub>3</sub>OH. Initially, methanol earned the moniker "wood alcohol" due to its primary production through the destructive distillation of wood. However, contemporary methanol production involves an industrial catalytic process that directly synthesizes it from hydrogen, carbon dioxide, and monoxide.

Methanol, the simplest form of alcohol, presents itself as a transparent, lightweight, flammable liquid with a distinctive odor reminiscent of ethanol (common drinking alcohol). Diverging from ethanol, methanol is toxic and unsuitable for human consumption. It serves as a denaturant for ethanol and finds utility as an antifreeze, solvent, fuel, and polar liquid at room temperature. Additionally, methanol plays a role in the transesterification process essential for biodiesel production.

In nature, methanol naturally arises through the anaerobic metabolism of numerous bacterial species, often existing in trace amounts within the environment. Consequently, methanol vapor is scarcely present in the natural surroundings.

#### Methanol properties:

Table 4 Methanol properties

SL. No.	Parameters	Symbol	Quantity	Unit
1.	Freezing temperature	T <sub>freeze</sub>	-97.6	°C
2.	Boiling temperature	T <sub>boil</sub>	64.7	°C
3.	Density	P	792	kg/m <sup>3</sup>
4.	Specific heat (at 20°C)	C <sub>p</sub>	2.5	Kj/kg-k

SL. No.	Parameters	Symbol	Quantity	Unit
5.	Vapor pressure	Pv	13.02	kPa
6.	Molar mass	Ms	32.04	g/mol

### 3.2.3 Ammonium hydroxide as Catalysts:

In some cases, catalysts may be added to the precursor solution to facilitate gelation. For example, ammonium fluoride is often used as a catalyst in silica aerogel production. Lab grade (28% concentration) Ammonium hydroxide is used for the process.

Ammonium hydroxide, often referred to as ammonia solution, is a colorless liquid with a pungent, distinctive ammonia odor. It is an aqueous solution of ammonia gas (NH<sub>3</sub>) dissolved in water (H<sub>2</sub>O). Ammonium hydroxide is commonly used as a cleaning agent, in household cleaning products, and in industrial applications for its alkaline properties. It can be corrosive in concentrated forms and should be handled with care.

### Ammonium Hydroxide (NH<sub>4</sub>OH) Properties:

Table 5: ammonium hydroxide properties

SL. No.	Parameters	Symbol	Quantity	Unit
1.	Freezing temperature	T <sub>freeze</sub>	-57.5	oC
2.	Boiling temperature	T <sub>boil</sub>	37.7	oC
3.	Density (at 20°C)	P	0.91	g/cm <sup>3</sup>
4.	Specific heat (at 25°C)	C <sub>p</sub>	4.18	Kj/kg-k
5.	Vapor pressure (at 20°C)	Pv	3.7	kPa
6.	Molar mass	Ms	35.05	g/mol

### 3.2.4 CO<sub>2</sub> as Supercritical Fluids:

Supercritical carbon dioxide (CO<sub>2</sub>) is commonly used as a supercritical fluid in the supercritical drying process. It replaces the solvent within the gel structure and aids in solvent removal without

collapsing the aerogel's structure. CO<sub>2</sub> gas cylinder was used for the following experiment. Carbon dioxide (CO<sub>2</sub>), often referred to simply as CO<sub>2</sub>, is a fundamental chemical compound denoted by the molecular formula CO<sub>2</sub>. This CO<sub>2</sub> plays pivotal roles in everyday existence and the natural world. Historically, CO<sub>2</sub> was primarily a byproduct of various industrial processes, but its contemporary significance extends far beyond these origins. In today's world, carbon dioxide is generated through diverse means, including the combustion of fossil fuels, fermentation processes in brewing and winemaking, and industrial methods for capturing and concentrating it from the atmosphere. At standard room temperature and atmospheric pressure, carbon dioxide manifests as a colorless, odorless gas. It is approximately 1.5 times denser than air, causing it to accumulate in lower regions when released. Under specific temperature and pressure conditions, CO<sub>2</sub> can transition into a solid state, known as dry ice, or exist in a supercritical state, displaying the properties of both a gas and a liquid. Carbon dioxide finds itself involved in a multitude of practical applications across various industries. It is used to carbonate soft drinks, beers, and sparkling water, and it serves as a fire-extinguishing agent in fire extinguishers. Additionally, CO<sub>2</sub> is instrumental in extending the shelf life of perishable foods, and it has applications in medical procedures, such as cryotherapy and medical imaging. Industries benefit from its use in processes like welding and cryogenic cooling. However, carbon dioxide's significance extends even further as a greenhouse gas that contributes to climate change when its concentration in the atmosphere rises. Human activities, particularly the combustion of fossil fuels, have led to a substantial increase in atmospheric CO<sub>2</sub>, resulting in global temperature increases and associated environmental shifts.

**CO<sub>2</sub> properties:**

Table 6: CO<sub>2</sub> properties

SL. No.	Parameters	Symbol	Quantity	Unit
1.	Freezing temperature	T <sub>freeze</sub>	-78.5°	°C
2.	Boiling temperature	T <sub>boil</sub>	-56.6°C	°C

SL. No.	Parameters	Symbol	Quantity	Unit
3.	Density	P	1.977	kg/m <sup>3</sup>
4.	Specific heat (at 25°C)	C <sub>p</sub>	0.84	Kj/kg-k
6.	Molar mass	Ms	44.01	g/mol



Figure 11: Carbon dioxide gas cylinder

### 3.2.5 Distilled Water

Distilled water is water that has been heated into a vapor and then condensed back into liquid in a separate container. Any contaminants in the original water that do not boil at or below the boiling point of water are still present in the original container. So, distilled water is one kind of purified water.

## Distilled Water properties:

Table 7: Distilled Water properties

SL. No.	Parameters	Symbol	Quantity	Unit
1.	Freezing temperature	$T_{freeze}$	0	°C
2.	Boiling temperature	$T_{boil}$	100	°C
3.	Density	P	997	kg/m <sup>3</sup>
4.	Specific heat (at 25°C)	$C_p$	4.187	Kj/kg-k
5.	Vapor pressure	Pv	3.157/25 °C	kPa
6.	Molar mass	Ms	18.01528	g/mol

### 3.3 Making sol-gel

The following chemicals are needed for making the sol-gel:

- Methanol (CH<sub>3</sub>OH): 9.2 grams
- Tetramethyl orthosilicate (TMOS or Si(OMe)<sub>4</sub>): 4.256 grams
- Deionized water (H<sub>2</sub>O): 3.2 grams
- Ammonium hydroxide (NH<sub>4</sub>OH): 0.2 grams

#### 3.3.1 Preparation of Solution-1:

Mix 1.6 grams of Deionized water (H<sub>2</sub>O) and 0.2 grams of Ammonium hydroxide (NH<sub>4</sub>OH) in a clean container. This mixture is referred to as "Solution-1." Ensure thorough mixing to achieve a homogeneous solution.

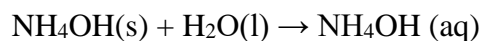




Figure 12:Preparation of Solution-1

### 3.3.2 Preparation of Solution-2:

In a separate container, measure out the following chemicals:

Methanol (CH<sub>3</sub>OH): 9.2 grams

Tetramethyl orthosilicate (TMOS or Si(OMe)<sub>4</sub>): 4.256 grams)

Combine Methanol and Tetramethyl orthosilicate (TMOS) to create "Solution-2." Stir the mixture until the chemicals are well-dispersed and form a homogeneous solution.

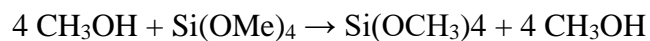


Figure 13:Preparation of Solution-2

### 3.3.3 Combining Solution-1 and Solution-2:

Gradually add Solution-2 into Solution-1 while stirring continuously. Additionally, include 1.6 grams of deionized water in this step. This combined solution is essential for the formation of the sol-gel. Continue stirring until you achieve a uniform mixture.

By including the 1.6 grams of deionized water along with Solution-2 into Solution-1, you ensure that the water content is appropriate for the sol-gel formation process, resulting in a more homogeneous mixture.

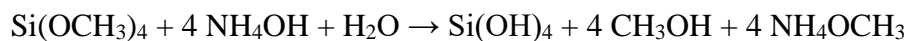


Figure 14: Combining Solution-1 and Solution-2

### 3.3.4 Gel Formation:

Allow the combined solution to stand undisturbed for a specified period to initiate the gel formation process. The gel will form as the precursor chemicals react.

The time required for gel formation can vary depending on the specific sol-gel system and conditions. It takes about 11 to 13 minutes in this following process.



Figure 15: Gel formation

### 3.3.5 Aging:

After gelation, the wet gel is left undisturbed to age for a certain period, typically several days to a few weeks. During this time, the gel structure undergoes chemical and physical changes. The aging process helps in the controlled shrinkage and strengthening of the gel network.

Aging is a vital step in fortifying the gel network before the drying phase, facilitating additional condensation. Key points regarding this process include:

- Typical aging periods vary from 24 hours to 4 days.
- For TMOS-based gels, the recommended aging duration ranges from 24 to 48 hours,
- It is advisable to replace the aging solvent, often methanol 1 to 2 times during the aging process to eliminate water content and impurities and promote condensation.
- Aging should be conducted within a sealed container to prevent evaporation and minimize the risk of cracking.
- Subsequent to aging, a solvent exchange procedure should be carried out 2 to 3 times over
- 1 to 2 days before proceeding with the drying phase.

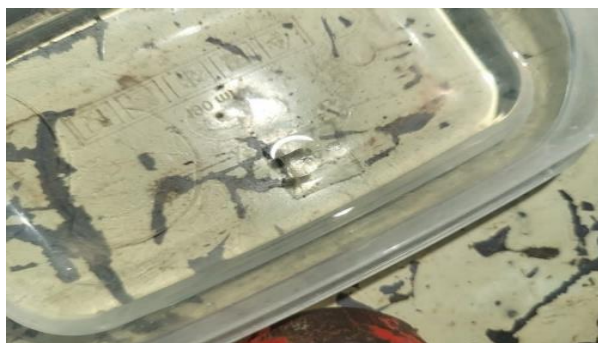


Figure 16: Aging



### 3.4 Aerogel Making

#### 3.4.1 Autoclave purging

Producing silica aerogels, a lightweight and highly porous material with exceptional insulating properties, requires the use of supercritical drying. Autoclave purging is an important step in this process to safely and effectively remove solvents from the silica gel structure while maintaining its unique aerogel properties. Before being placed in the autoclave, the gels must be purged (impurities removed) with methanol and liquid CO<sub>2</sub> to eliminate any contaminants. The cleansing stages are outlined below.

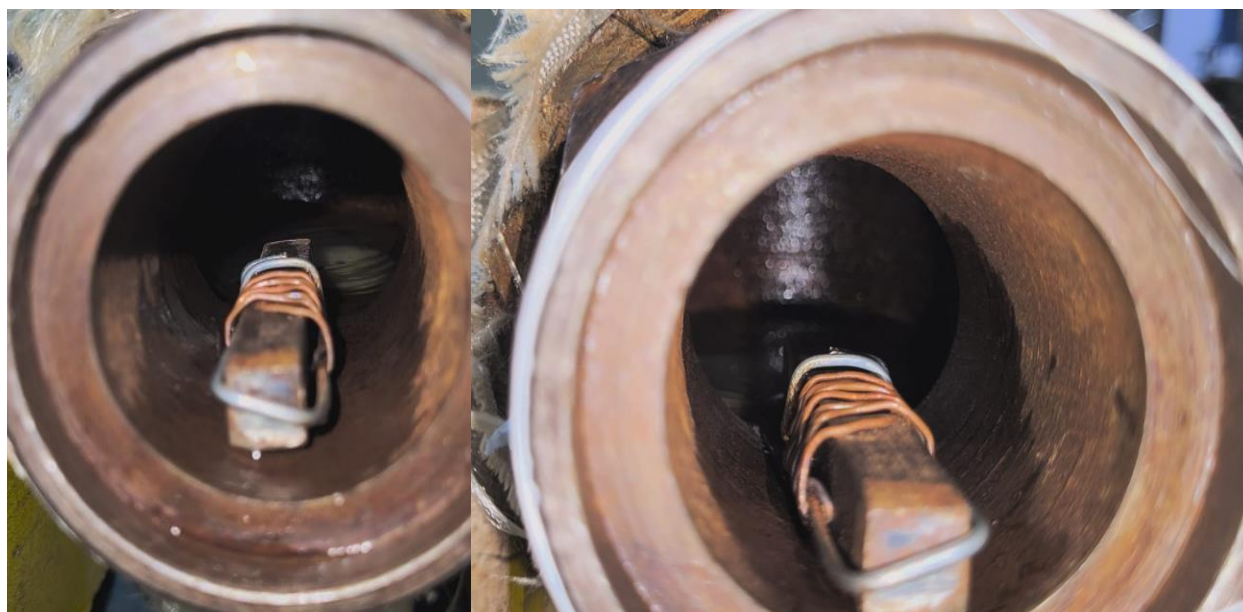


Figure 17: Submerged solgel into methanol

#### 3.4.2 Insertion of the Autoclave Vessel:

Open the pressure release valve and close the drain valve. Pour 1/3 or 600 ml of methanol into the autoclave through the opening at the top. Wrap Teflon tape over the threading of the nipple plug withdrawn from the top of the autoclave - make sure the wrapping direction is in the direction of friction force on the threads when screwing it back into the hole. Seal the sight glass (Borosilicate glass) of the Autoclaves. Screw the screw back into the aperture tightly.

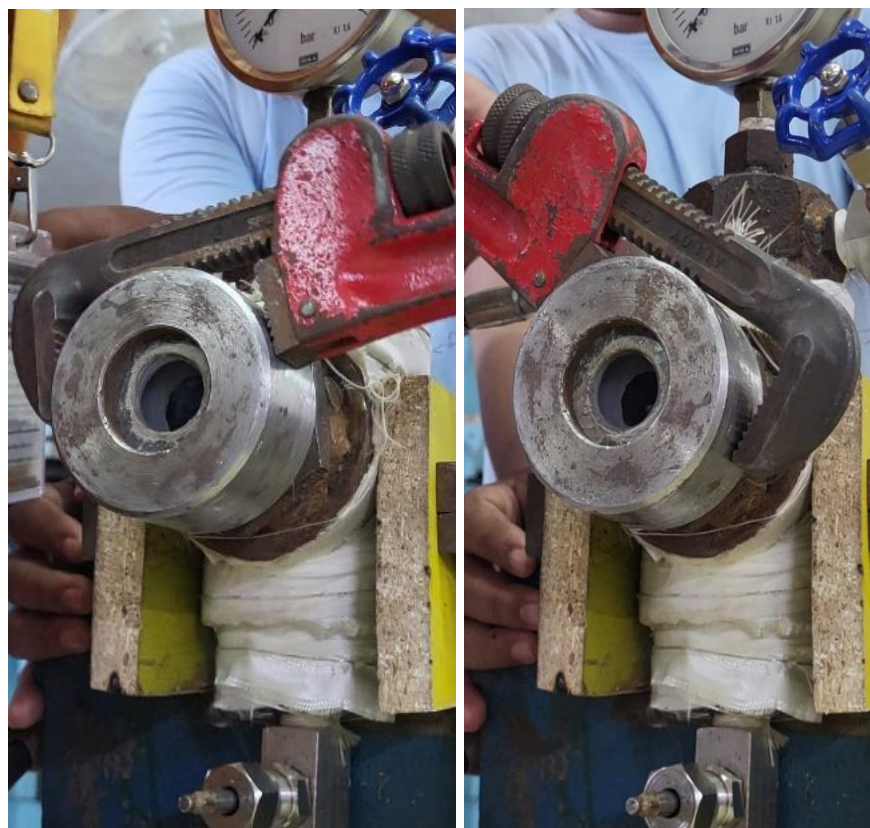


Figure 18: Threading process of autoclave

### **3.4.3 Autoclave Sealing:**

Emphasis is placed on ensuring a secure seal for the autoclave, thereby establishing a closed system that maintains the supercritical conditions necessary during the drying process.

### **3.4.4 Filling with Supercritical Fluid:**

To generate supercritical conditions in an autoclave, carbon dioxide ( $\text{CO}_2$ ) is injected as a supercritical fluid at a pressure increase rate of 1 bar (14.50 psi) per minute. This is preferred due to  $\text{CO}_2$ 's lower critical point. Temperature and pressure adjustments are made within the autoclave to achieve and maintain the supercritical state of  $\text{CO}_2$ , which can exceed  $31^\circ\text{C}$  and 73 bar. Once the liquid is filled, the inlet valve should be closed to maintain the desired pressure.



Figure 19: Filling with Supercritical Fluid

### 3.4.5 Solvent Extraction:

Supercritical CO<sub>2</sub> penetrates the pores of the silica gel and dissolves the liquid contents inside. This gradually replaces the solvent, expediting the transition from a liquid to a supercritical fluid state. This process needs to be repeated once a day until there is no more methanol extract draining. The goal is to ensure that the pores of the silica gel are filled with supercritical CO<sub>2</sub>.

### 3.4.6 Slow Methanol Draining

The following steps outline how the buffer methanol to complete a slow draining cycle, the autoclave must be emptied. Once the gels have been fed into the autoclave (or whenever it is decided to be drained methanol slowly:

Wait for 30-60 min after CO<sub>2</sub> gas inserting is done. Open the drain valve slightly until methanol starts dripping drop by drop. Adjust the drain valve every 5 – 10 min as the draining slows down. Keep watching the liquid level within the autoclave, and anytime it rises a little beyond the top of the cage containing the gels, immediately open the intake valve to prevent the sudden flow of CO<sub>2</sub>

into the autoclave so that it generates turbulence in the liquid in the autoclave and mixes the CO<sub>2</sub> and methanol. The draining might take up to 2 – 2.30 hrs.



Figure 20 : Methanol draining slowly

### 3.4.7 Supercritical drying

The autoclave was snugly cocooned with layers of glass fiber insulation, ensuring the retention of heat and provision of electrical insulation. The uniformly coiled nichrome wire, gleaming with anticipation, had been positioned around the autoclave chamber.

With all components in place, the connection between the chromium wire and the Variac was made, designating the Variac as the gatekeeper for temperature control in the experiment. Additionally, a thermometer was affixed to the autoclave's body for temperature monitoring and data collection.

Upon completing the setup, the experiment was initiated, and voltage, controlled by the Variac, was gradually increased. The chromium wire began to heat up, transferring heat to the autoclave body. Monitoring was facilitated through the use of a pressure gauge and thermometer, with experimental data being meticulously recorded with a stopwatch.

The occurrence of supercritical conditions was duly observed and recorded, and the experimental data table was constructed, detailing time, pressure, temperature, and voltage. Notably, supercritical conditions for CO<sub>2</sub> manifest at temperatures of up to 31.1 °C and pressures of 73.8 bar.

These recorded data points enabled the calculation of the duration during which supercritical conditions were sustained. In addition to numerical data, the transition into the supercritical state was visually ascertained through the sight glass by observing physical changes in the CO<sub>2</sub>.

the autoclave setup, comprising glass fiber insulation, nichrome wire, Variac, and monitoring instruments, allowed for the precise control and observation of the supercritical drying experiment. The data collected and the subsequent calculations were instrumental in characterizing the duration and conditions of the supercritical state, as well as verifying the transformation of CO<sub>2</sub> through visual observation.

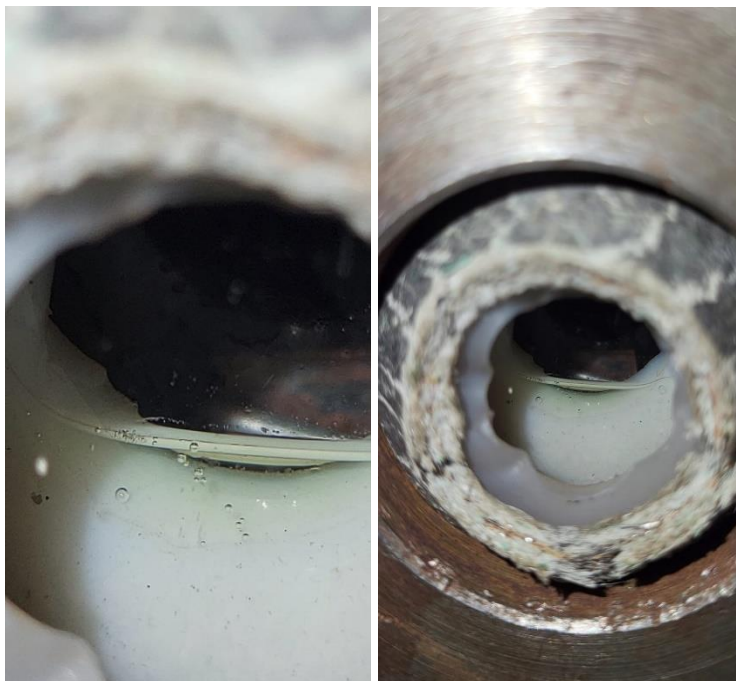


Figure 21: CO<sub>2</sub> getting its supercritical condition.

### 3.4.8 Gradual Reduction of Pressure:

Pressure reduction within the autoclave is carried out incrementally while maintaining a constant temperature. This gradual pressure reduction facilitates is the transformation of CO<sub>2</sub> back into its gaseous state, enabling it to exit the autoclave.

### 3.4.9 Aerogel Collection:

Once pressure and temperature have stabilized, the autoclave is opened, and the resulting silica aerogel is removed. The aerogel is characterized by its remarkable insulating properties, low density, and high porosity.

### 3.4.10 Post-Processing Considerations:

Depending on the intended application. Additional processing steps such as cutting, shaping, or specialized treatments may be necessary.

### 3.4.11 Safety Precautions:

Given the utilization of high pressures and supercritical fluids in autoclave purging for supercritical drying, stringent safety measures are implemented, including incorporating pressure release systems and providing appropriate training to mitigate potential hazards.

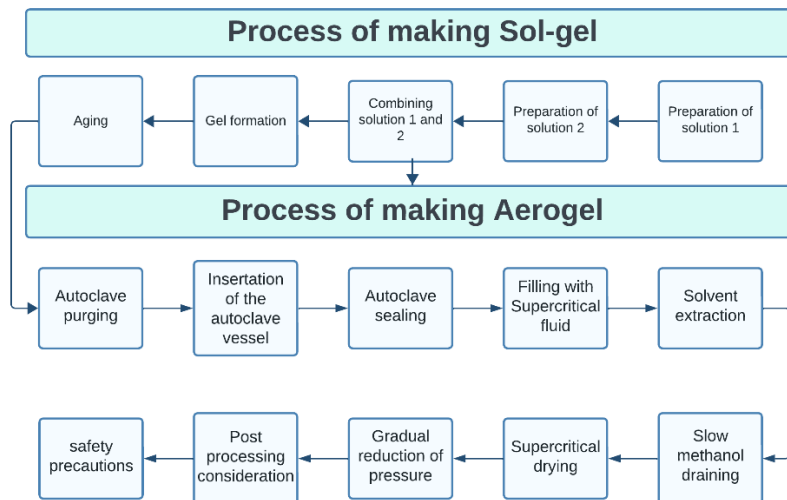


Figure 22:Block diagram of process of making solgel into aerogel.

# Chapter 4

## 4 Results & Discussions

This chapter will illustrate the findings visually and briefly explain the effect of Aerogel.

Excel & Word generated fascinating statistical visual graphs.

### 4.1 Condition of All Data: Result

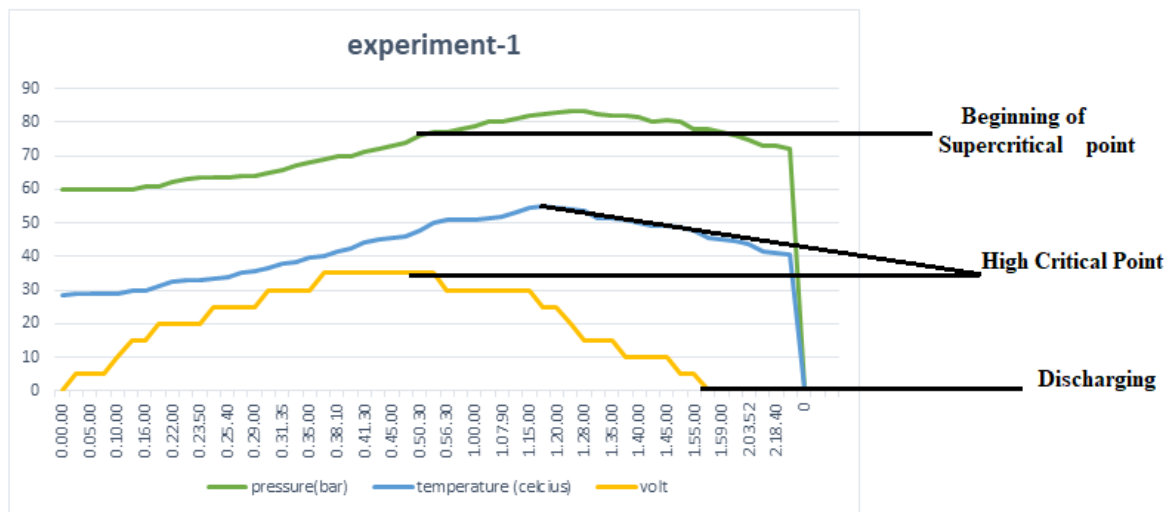


Figure 23.graph of experiment 1

The line graph illustrates the experiment of the supercritical state of CO<sub>2</sub> for creating an aerogel. Where blue, green, and yellow lines demonstrate temperature, pressure, and voltage variable in the y-axis in respect of time. The x-axis indicates the time variation. In the graph, the pressure and temperature begin at 61 bar and 28.6 degrees Celsius, and it takes 48 minutes and 50 seconds to reach the supercritical condition where pressure is 74 bar and temperature is 47 degrees Celsius. This condition develops corresponding to time and gets the highest-pressure position of 83.5 bar after 1 hour and 25 min owing to the temperature increase at 53.7 degrees Celsius. The supercritical state was endured for 1 hour, 16 min, and 52 seconds to stabilize the phase of CO<sub>2</sub> as a supercritical

fluid, and the voltage was concurrently raised at 0 to 35 volts every 10 to 20 minutes. This method was done in reverse of the experimental approach to maintain constant temperature during the experiment to achieve a better outcome. After the trial, the process was shut off. Therefore, additional data has not been gathered.

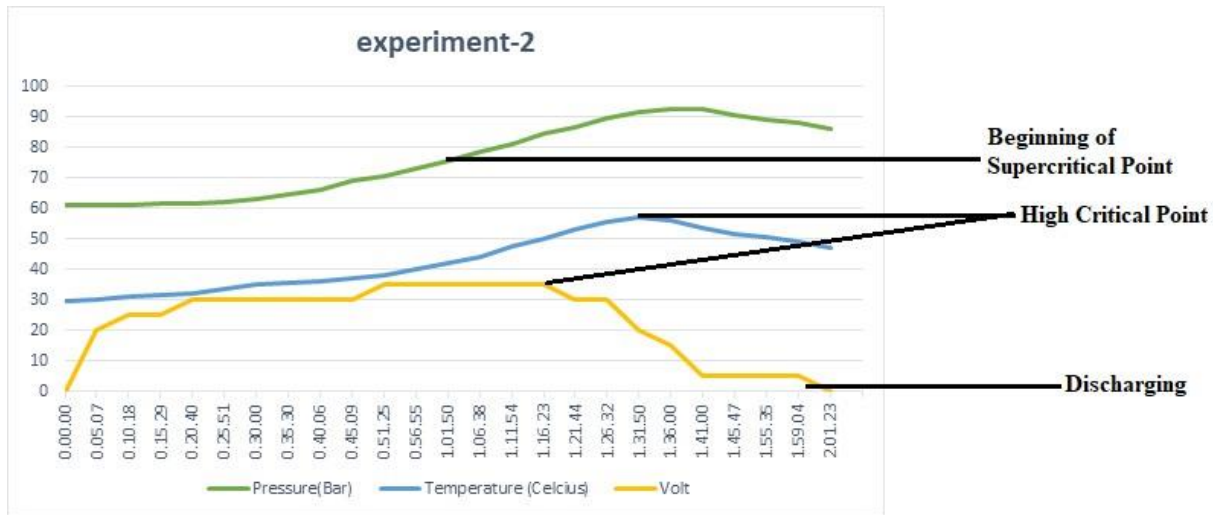


Figure 24. graph of experiment 2

The line graph illustrates the experiment of the supercritical state of CO<sub>2</sub> for creating an aerogel. The resulting graph showcases three key variables over time: temperature in blue, pressure in green, and voltage in yellow. Initially, the experiment starts at 29.8°C and 61 bar pressure with zero voltage. It took 60 minutes to obtain the supercritical state due to deficient volume of CO<sub>2</sub>, characterized by a temperature of 41°C and a pressure of 74 bar. Over time, the experiment displays a gradual increase in both temperature and pressure, reaching a peak pressure of 92.5 bar after 1 hour and 36 minutes, driven by a temperature rise to 56°C. The supercritical state remains stable for an impressive 1 hour, 1 minute, and 23 seconds, during which voltage control becomes pivotal, incrementally rising from 0 to 35 volts in intervals every 10 to 20 minutes. As a precision-enhancing measure, a reverse approach is adopted to maintain a constant temperature in the latter stages of the experiment. End of the experiment, it concludes with the shutdown of the process, with no further data collection in the synchronized data lines on the graph.



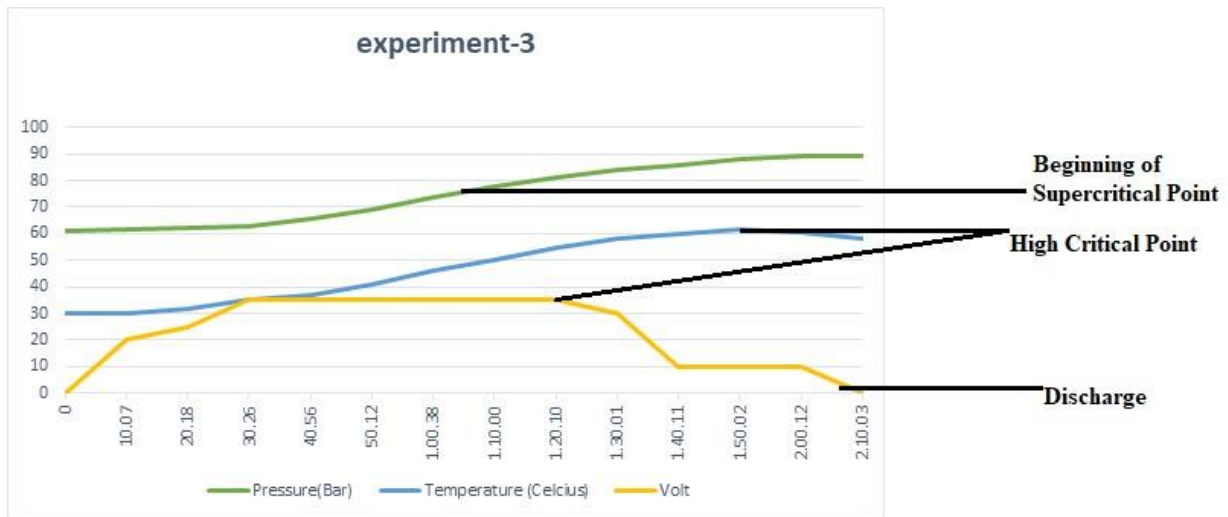


Figure 25.graph of experiment 3

The line graph illustrates the experiment of the supercritical state of CO<sub>2</sub> for creating an aerogel. It illustrates three key parameters over time: temperature (blue line), pressure (green line), and voltage (yellow line). Initially, the system starts at 29.8°C and 61 bar pressure with zero voltage. It takes approximately 1 hour and 2 min to reach the supercritical state pressure of 73.9 bar pressure (deficient volume of co<sub>2</sub>). Over time, temperature and pressure gradually rise, peaking at 89.5 bar due to a temperature increase to 53.7°C at the end of the experiment. This supercritical state remains stable for 1 hour, 8 minutes. During the process voltage incrementally increases from 0 to 35 volts and make constant volt of 35 for 1 hour later the voltage decrease in the later stages for safety precautions. After the experiment, the process is shut down, concluding without further data collection.

The reason of having different value in the following experiments is the volume of co<sub>2</sub> in the autoclave.

## 4.2 Discussion

Ordinary CO<sub>2</sub> has been used for the entire project, but Siphon type (liquid CO<sub>2</sub>) is needed for the making of Silica-Aerogel. Ordinary CO<sub>2</sub> gas cylinder is cheaper than Siphon type CO<sub>2</sub> cylinder, but it is likely not possible to produce aerogels and supercritical condition using Siphon type CO<sub>2</sub> cylinder. CO<sub>2</sub> gas will not diffuse into the solvent-filled pores to replace the solvent. Liquid CO<sub>2</sub> under pressure can penetrate the pores and replace the solvent. Supercritical CO<sub>2</sub> has no surface tension, allowing it to diffuse from the pores without collapsing the gel structure. Liquid CO<sub>2</sub> can transition to a supercritical state for drying. Gas cannot. After drying, CO<sub>2</sub> returns to gas for easy removal. So liquid CO<sub>2</sub> is used to penetrate pores, replace solvent, transition to a supercritical state for drying, and then be removed as gas. CO<sub>2</sub> gas alone cannot do solvent exchange, supercritical drying, or easy removal. Liquid and supercritical CO<sub>2</sub> enable damage-free solvent exchange and drying - which is critical for aerogel production. In short, liquid and supercritical CO<sub>2</sub> enables solvent exchange and surface-tension-free drying that retains the nanostructure of aerogels - features that CO<sub>2</sub> gas alone cannot do. This makes liquid CO<sub>2</sub> the favored option for supercritical drying in aerogel manufacturing.



Figure 26: Experiment 1



Figure 27: Experiment 2



Figure 28: Experiment 3

## Cost Analysis

Table 8: Cost Analysis of chemicals, autoclave and additional

Name of the chemicals	Quantity	Rate (TK)	Amount (TK)
Tetramethyl orthosilicate (TMOS)	1*100 ml	8,000	8,000
Methanol	3*1000 ml	600	1800
Ammonium hydroxide (NH <sub>4</sub> OH)	1*1000 ml	100	100
Deionized water	1*1000 ml	70	70
		<b>Total price (TK)</b>	9,970

Description	Quantity	Rate (TK)	Amount (TK)
2" *2" thread T (autoclave)	1	2600	2,600
4" plug (threaded)	1	1500	1500
2" plug (threaded)	1	1000	1000
2" nipple	1	800	800
2" threaded cap	1	1500	1500
Borosilicate temper glass	1	350	350
4" diameter Pressure Gauge	1	3000	3000
½" needle valve	3	1000	3000
½" * ½" bush	1	100	100
½" nipple	3	80	240
Teflon tape	10	16	160
CO <sub>2</sub> gas cylinder +refill	1	2200+400	2600
Hydraulic connecting pipe	1	620	620
Gasket		500	500

<b>Description</b>	<b>Quantity</b>	<b>Rate (TK)</b>	<b>Amount (TK)</b>
Nichrome wire		280	260
Glass fiber		500	500
Syring (mold)			150
			<b>Total: 18,880</b>
<b>Additional cost (TK)</b>		5000	

The aerogel manufacturing project is notably cost-effective, with the total cost amounting to just 34000 Bangladeshi Taka. When evaluated in the context of the global aerogel market, where manufacturing can often entail significant capital expenditure, this sum underscores the economical nature of our endeavor.

When converted to U.S. dollars using the current exchange rate of 109.59 BDT to 1 USD, as provided by the Bangladesh Bank, the total cost comes to approximately \$310.25 USD. This low financial outlay significantly affects our project's scalability and overall profitability.

The cost-effectiveness of our manufacturing process not only makes the project more accessible for investors but enables competitive pricing for the product. This creates an advantage in the market, as we can target a broader range of consumers whom more expensive aerogel options may have priced out. Moreover, it opens the possibility of more significant margins, thereby increasing the project's financial viability in both the short and long term.

This expanded explanation explains why your project's low manufacturing cost is a noteworthy advantage.

## Chapter 5

### Conclusions

The world of silica aerogels, often referred to as the "frozen smoke" of materials, has been explored extensively in this research. Remarkable characteristics, including exceptionally high porosity, low density, and outstanding thermal insulation properties, define these aerogels, primarily composed of silica. The central challenge addressed in this research has been the investigation of ordinary CO<sub>2</sub> gas cylinders as an alternative source for supercritical drying in the synthesis of silica aerogels. Traditionally, siphon CO<sub>2</sub> cylinders have been preferred due to their regulated release of CO<sub>2</sub> and consistent results. However, concerns about availability, cost, and environmental impact have led to examining conventional CO<sub>2</sub> gas cylinders as a more accessible and cost-effective option. The objectives of this research encompassed the manufacturing of silica aerogels using ordinary CO<sub>2</sub> cylinders, the evaluation of cost-effectiveness, and the assessment of product quality compared to siphon-type CO<sub>2</sub> cylinders. It must be emphasized that while ordinary CO<sub>2</sub> cylinders are more affordable, they may need to possess the necessary properties for producing aerogels under supercritical conditions. Liquid CO<sub>2</sub>, especially in its supercritical state, plays a pivotal role in solvent exchange, surface-tension-free drying, and the preservation of the nanostructure of aerogels. The hypothesis proposed that using gaseous CO<sub>2</sub> instead of liquid CO<sub>2</sub> for aerogel production could produce superior quality, safety, and environmental friendliness. However, this idea remains subject to validation through rigorous experimental tests and comprehensive outcome examinations. This research aims to bridge the knowledge gap concerning using ordinary CO<sub>2</sub> gas cylinders for supercritical drying in silica aerogel synthesis. While cost-effectiveness is a significant consideration, equal importance lies in ensuring the maintenance of desired aerogel characteristics. The findings of this research will not only inform material science and engineering. However, they will also guide the selection of CO<sub>2</sub> sources for various applications, potentially unlocking new possibilities in the realm of aerogel production.

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## Appendix:

Table 9: Experiment 1

<b>Time (Hour.Min.Sec)</b>	<b>Pressure(bar)</b>	<b>Temperature (Celsius)</b>	<b>voltage</b>
0.00.00	60	28.6	0
0.01.30	60	28.8	5
0.05.00	60	28.9	5
0.07.30	60	29	5
0.10.00	60	29.1	10
0.15.00	60	29.7	15
0.16.00	61	30	15
0.20.00	61	31.2	20
0.22.00	62	32.4	20
0.23.00	63	32.8	20
0.23.50	63.5	33.1	20
0.25.00	63.5	33.5	25
0.25.40	63.5	33.9	25
0.27.00	64	35	25
0.29.00	64	35.8	25
0.30.00	65	36.5	30
0.31.35	66	37.7	30
0.33.00	67	38.5	30
0.35.00	68	39.9	30
0.36.40	69	40.2	35
0.38.10	70	41.4	35

<b>Time (Hour.Min.Sec)</b>	<b>Pressure(bar)</b>	<b>Temperature (Celsius)</b>	<b>voltage</b>
0.40.10	70	42.3	35
0.41.30	71	44	35
0.43.00	72	45	35
0.45.00	73	45.5	35
0.48.50	74	46	35
0.50.30	76	48	35
0.54.00	77	50.08	35
0.56.30	77	50.8	30
0.57.00	78	50.8	30
1.00.00	79	50.9	30
1.02.00	80	51.6	30
1.07.90	80	52	30
1.13.10	81	53.2	30
1.15.00	82	54.6	30
1.17.06	82.5	54.8	25
1.20.00	83	54.4	25
1.25.00	83.5	54.3	20
1.28.00	83.5	53.7	15
1.33.40	82.5	51.3	15
1.35.00	82	51.3	15
1.38.30	82	50.9	10
1.40.00	81.5	50	10
1.43.30	80	49.2	10
1.45.00	80.5	49	10
1.47.00	80	48.6	5

<b>Time (Hour.Min.Sec)</b>	<b>Pressure(bar)</b>	<b>Temperature (Celsius)</b>	<b>voltage</b>
1.55.00	78	47.6	5
1.58.00	78	45.7	0
1.59.00	77	45.1	0
2.00.00	76	44.8	0
2.03.52	75	43.7	0
2.17.02	73	41.3	0
2.18.40	73	41.1	0
2.20.00	72	40.8	0
0	0	0	0

Table 10: Experiment 2

<b>TIME (Hour.Min.Sec)</b>	<b>Pressure (Bar)</b>	<b>Temperature (Celsius)</b>	<b>Voltage</b>
0.00.00	61	29.8	0
0.05.07	61	30.1	20
0.10.18	61	30.9	25
0.15.29	61.5	31.5	25
0.20.40	61.5	32.3	30
0.25.51	62	33.6	30
0.30.00	63	34.9	30
0.35.30	64.5	35.7	30
0.40.06	66	36.2	30
0.45.09	69	37	30
0.51.25	70.5	38.2	35
0.56.55	73	39.9	35
1.01.50	75.5	41.9	35
1.06.38	78.5	44	35
1.11.54	81	47.6	35
1.16.23	84.5	50	35
1.21.44	86.5	53.2	30
1.26.32	89.5	55.7	30
1.31.50	91.5	57	20
1.36.00	92.5	56	15
1.41.00	92.5	53.5	5

<b>TIME (Hour.Min. Sec)</b>	<b>Pressure (Bar)</b>	<b>Temperature (Celsius)</b>	<b>Voltage</b>
1.45.47	90.5	51.5	5
1.55.35	89	50.5	5
1.59.04	88	49	5
2.01.23	86	47.3	0

Table 11: Experiment 3

<b>TIME (Hour.Min. Sec)</b>	<b>Pressure (Bar)</b>	<b>Temperature (Celsius)</b>	<b>Voltage</b>
0	61	29.8	0
10.07	61.5	30.2	20
20.18	62	31.9	25
30.26	63	35.4	35
40.56	65.5	37	35
50.12	69	40.7	35
1.00.38	73.5	46.3	35
1.10.00	77.5	50.4	35
1.20.10	81	55	35
1.30.01	84	58.3	30
1.40.11	86	60.1	10
1.50.02	88	61.9	10
2.00.12	89	60.5	10
2.10.03	89.5	58.2	0