Aerogel: A Nanostructured Material with Fascinating Properties and Unlimited Applications

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AEROGEL RESEARCH LAB

Established in 1996 with the mission to investigate both the fundamental properties and the cutting-edge applications of aerogels.

Basic Science
Diffusion on a Fractal Length Scale

Applied Research
Biological Warfare Detection

Courtesy of Prof. Pamela Norris, UVA
What is a Sol-gel Material?

A Gel That is Derived From a Sol

Three-Dimensional, Highly Porous Solid Network

Colloidal Suspension of Particles

Solid Network Can Be:

- Made from: Silica, Alumina, Titania, etc.
- Filled with: Water (Hydrogel), Alcohol (Alcogel), Air (Aerogel), Vacuum (Vacuugel?) etc.
- Surface Modified with: Silane Coupling Agents (Alkyl, Amine, Sulfhydryl, Carboxyl, Formyl) -> Ligands, Enzymes, Immunoglobins, Chelating Agents, etc.

Courtesy of Prof. Pamela Norris, UVA
Aerogel Production Process

Si(OCH₂CH₃)₄
H₂O
Catalyst
Ethanol

Add dopants to modify material properties

Supercritical or surface modification drying

Adjust viscosity to prepare thin films and fibers

Atmospheric drying

Xerogel

Aerogel

Sol

Adjust reaction conditions to control surface area, density, porosity, and pore size

Alcogel

Courtesy of Prof. Pamela Norris, UVA
Aerogel Microstructure

Aerogel

Macroporosity $d > 200$ nm

Mesoporosity $5$ nm $< d < 200$ nm

Microporosity $d < 5$ nm

Surface Silanol Groups

Silanol Groups

Courtesy of Prof. Pamela Norris, UVA
Microstructure

SEM micrographs of various aerogels formed with increasing concentrations of polyethylene glycol (PEG).

Courtesy of Prof. Pamela Norris, UVA
# Aerogel Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Aerogel&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Silica&lt;sup&gt;b&lt;/sup&gt;</th>
<th>Air</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dielectric Constant</td>
<td>1.007-2</td>
<td>---</td>
<td>1.0</td>
</tr>
<tr>
<td>Thermal Conductivity (W/mK)</td>
<td>0.01-0.3</td>
<td>1.3</td>
<td>0.03</td>
</tr>
<tr>
<td>Density (kg/m&lt;sup&gt;3&lt;/sup&gt;)</td>
<td>3-400</td>
<td>2200</td>
<td>1</td>
</tr>
<tr>
<td>Surface Area (m&lt;sup&gt;2&lt;/g&gt;)</td>
<td>600-1500</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Speed of Sound (m/s)</td>
<td>100-300</td>
<td>5000</td>
<td>330</td>
</tr>
<tr>
<td>Acoustic Impedance (kg/m&lt;sup&gt;2&lt;/sup&gt;s)</td>
<td>10&lt;sup&gt;3&lt;/sup&gt;-10&lt;sup&gt;5&lt;/sup&gt;</td>
<td>10&lt;sup&gt;7&lt;/sup&gt;</td>
<td>400</td>
</tr>
<tr>
<td>Refractive Index</td>
<td>1.0-1.4</td>
<td>1.4</td>
<td>1.0</td>
</tr>
</tbody>
</table>

<sup>a</sup> Silica aerogel  
<sup>b</sup> Sintered silica (polycrystalline)  

Courtesy of Prof. Pamela Norris, UVA
Historical Development

- Early 1930’s: Discovery by Kistler, Stanford
- Early 1960’s: Teichner, France--Rocket propellant storage, quicker production method
- Early 1980’s: Henning, Sweden--Cherenkov radiation detectors, first manufacturing facility
- Late 1980’s: Hunt, LBL--Safer production method
- 1990’s: Shuttle missions, Mars Rover
- 1996: Founding of the Aerogel Research Laboratory at UVA

Courtesy of Prof. Pamela Norris, UVA
Potential Applications

- Thermal, acoustic, and electrical insulators
- Chemical catalysis
- Thin film dielectrics
- Acoustic delay lines
- Desalination of seawater
- Subatomic particle detectors
- Micrometeroid collectors
- Supercapacitors
- Thermal insulation for Mars Rover
- Optical coatings and anti-reflective films
- Filter and absorption media

Courtesy of LBL

Courtesy of Prof. Pamela Norris, UVA
Aerogels in the News
“Stardust’s Butterfly Net”

NASA's Stardust spacecraft, shown in this artist's conception, collected particles from Comet Wild 2's coma in 2004. Samples from the comet — as well as samples of interstellar dust — were captured in an aerogel-filled collector during separate periods of exposure.

Courtesy of Prof. Pamela Norris, UVA
Aerogels as Thermal Insulation

A classic picture of a flower insulated from a Bunsen burner by a 1-cm thick tile of aerogel

Note that the flame side of the tile is heated to incandescence

Courtesy of LBL

Courtesy of Prof. Pamela Norris, UVA
Comparison of Insulating Quality

For same insulation (R11), you could use

- 90 mm Fiberglass Bat
- 60 mm Urethane Foam
- 40 mm Plain Air filled Silica Aerogel
- 18 mm Plain Evacuated Silica Aerogel
- 5 mm Optimized Evacuated Aerogel

Courtesy of Prof. Pamela Norris, UVA
Carbon-Loaded Battery Case

Silica aerogel loaded with carbon and cast into a 80 mm x 80 mm cylinder with approximately 8 mm wall thickness

Carbon introduced in suspension prior to gelation

Quarter

Courtesy of Prof. Pamela Norris, UVA
Aerogel Research Lab

Capabilities:
- Small to medium scale production of aerogels
- Thin films, beads, fibers, powders, bulk monoliths, composites
- Machining and special shape production of aerogels
- Full structural characterization
  - Porosimetry: surface area, porosity, pore size
  - Spectroscopic: IR, Mass Spec, NMR, SEM
- Acoustic measurements
- Flow characterization
- Aerosol collection research
- Pore-size engineering

Courtesy of Prof. Pamela Norris, UVA
**Smart Aerogel**

Nontargeted Molecules

Target Molecule

SIGNAL

Specific Receptors

Aerogel
Bulk View

Expanded Aerogel
Pore View

Collection

Identification

Assay

"Smart Aerogel", US Patent 6,447,991
Inventors: J.S. Brenizer, C.E. Daitch, B. Hosticka, L.R. Mason, and P.M. Norris

Courtesy of Prof. Pamela Norris, UVA
Images of circuits fabricated on aerogel thin film. a) A photograph of half of a silicon wafer with circuits deposited. b) A magnified view of the circuits. c) SEM showing a side view of the 0.72 μm thick aerogel film with device made from 0.258 mm thick platinum.

Courtesy of Prof. Pamela Norris, UVA
Illustration of Smart Aerogel

- SEM micrograph of *E. coli* adhering to aerogel with large pore structure.

- Ability to immobilize bacteria within aerogel allows for utilization of porosity and high surface area in biosensor development.

- Aerosolized virus particles induced the expression of Green Fluorescent Protein in aerogel immobilized *E. coli* cells.

- Immobilized organisms can be used as sensors to detect chemicals or organisms in the environment.

US Patent 6,447,991

Courtesy of Prof. Pamela Norris, UVA
Gels in Micro-analytical Systems

- Micro Capillary Electrophoresis
- Micro Capillary Gel Electrophoresis
- Micro Capillary Immunoassays
- Micro Size Exclusion Chromatography
- Micro Electrochemical Cells
- Micro Perm-Selective Membranes
- Micro Capillary Electrochromatography
- Micro Supported Liquid Membranes

Courtesy of Prof. Pamela Norris, UVA
Gel Micropads

with A. Mirzabekhov, et al. Argonne National Labs

- 3-Dimensional Immobilization Surfaces
- Increased SNR for Fluorescent Detection
- Patterned Polyacrylamide Pads
- Current Research is Focused on Forming Sol-Gel Pads

Courtesy of Prof. Pamela Norris, UVA
Sol-gel Based Pads

Benefits Include:

• Liquid Phase Chemistry Preparation
• Fewer Preparation Steps
• Inorganic Matrix (Chemically Inert)
• Rigid Scaffold, Reduced Swelling
• Controllable Pore Size
• Controllable Surface Chemistry
• Reduced Background
• Printable

Courtesy of Prof. Pamela Norris, UVA
Lab on a Chip Applications

- On-chip DNA quantitation
- Integration of Solid Phase Extraction (SPE) and on-chip DNA quantitation
- Characterization of SPE matrices made from different materials for different flow rates, buffer properties etc.

Courtesy of Prof. Pamela Norris, UVA
Solid Phase Extraction

**CONDITIONING LOADING**

**WASHING**

**ELUTION**

○ - analyte of interest
□ △ - contaminants

Courtesy of Prof. Pamela Norris, UVA
Solid Phase Extraction

CONDITIONING  LOADING  WASHING  ELUTION

○ - analyte of interest
□ △ - contaminants

Courtesy of Prof. Pamela Norris, UVA
Solid Phase Extraction

CONDITIONING  LOADING  WASHING  ELUTION

- analyte of interest
- contaminants

Courtesy of Prof. Pamela Norris, UVA
**Versatility of the Sol-gel Process**

Si(OR)₄ + H₂O → Sol

Catalyst + Ethanol

Gel → Drying → Aerogel

SiO₂, TiO₂, ZrO₂, Fe₂O₅, V₂O₅, PtO₂

Cellulose

Agarose

Carbon

Surface Area

Pore Size (10nm - 0.1mm)

Density

Surface Chemistry

Shape, Size (Machine)

Adhere to:

Wood

Metal

Plastic

Ceramic

Receptors:

Pharmaceuticals

Antibodies, Enzymes

DNA/RNA

Cells

Conducting Polymers

Metal Fibers

Carbon Powder

Fluorophores, Dyes

Spatially Separate

Dopants;

“Compartmentalize”

Courtesy of Prof. Pamela Norris, UVA
Classic Mechanisms of Heat Transfer

• Conduction
  – through the solid phase
  – through the gas phase

• Convection
  – within gas filled pores
  – (from surface to environment)

• Radiation
  – through the composite material
Scaling of Solid Phase Heat Conduction

Heat conduction through different forms of a given solid should scale as the density and sound velocity.

- High Density Silica, High Heat Transfer
- Lower Density Silica, Lower Heat Transfer
- Aerogels with Tortuous, Longer Paths, Lower Sound Velocity and Heat Transfer

Courtesy of Prof. Pamela Norris, UVA
Solid Phase Heat Conduction (300 K)

- $K_{\text{solid}}$ Aerogel = 0.002 W/mK at 300 K (best value measured)
- $K_{\text{solid}}$ Silica = 1.4 W/mK at 300 K
  - Low density
    - Silica = 2.2 g/ml, Aerogel = 0.1 g/ml,
    - $K_{\text{silica}}$ = 1.4 W/mK, Predicted $K_{\text{aerogel}}$ = 0.064 W/mK
  - Low velocity of sound
    - Silica = 5000 m/s, Aerogel = 100 m/s
    - $K_{\text{silica}}$ = 1.4 W/mK, Predicted $K_{\text{aerogel}}$ = 0.028 W/mK
- Combined low density and low sound velocity
  Predicted $K_{\text{solid}}$ Aerogel = 0.0013 W/mK
- Loose particles may have lower solid phase heat conduction than monoliths due to the low contact area between particles

Courtesy of Prof. Pamela Norris, UVA
Cryogenic Solid Phase
Heat Conduction

• High temperatures (above 50 K)
  – the porous structure results in long path lengths for thermal conduction
  – the long path lengths also cause low sound velocity

• At low temperature (below 50 K)
  – the phonon wave length becomes long in relation to the structure and interactions with the material become important
  – when phonon wavelength is longer than the pore size, heat capacity and heat conduction decrease dramatically

Courtesy of Prof. Pamela Norris, UVA
Solid Phase Heat Conduction Determined by Phonon Interactions

Phonon wave length $\lambda$, inversely proportional to temperature

I macro pore region
   all pores smaller than $\lambda$

II fractal pore region
   pore size equals $\lambda$

III only micropores are
   smaller than $\lambda$

IV no pores are smaller than $\lambda$

Courtesy of Prof. Pamela Norris, UVA
Gas Phase Heat Conduction

- Requires gas-particle to gas-particle interactions
- Monolithic aerogels do not need high vacuum
  - At STP, air has a mean free path of about 70 nm
  - Mean pore size is about 50 nm, therefore, full gas conduction is NOT developed at STP
  - At pressure less than 10 kPa essentially NO gas conduction and only solid phase heat conduction need be considered
- Microspheres or aerogel powder may need lower pressures to eliminate gas phase conduction in the intergranular pores

Courtesy of Prof. Pamela Norris, UVA
Gas Phase Conduction

Thermal Conductivity (W/mK) vs. Log Gas Pressure (kPa)

- Red line: Monolith (50 nm pores)
- Blue line: 200 µm spheres

Courtesy of Prof. Pamela Norris, UVA
Convective Heat Transfer

• NONE within pores of aerogel
  – Pores are too small to support convection cells
  – At low pressure (10 kPa) there may or may not be a gas molecule within a given pore

• No special effect on convection at wall of insulation made from aerogel
Radiative Heat Transfer

Silica aerogel is transparent or translucent to visible light

- blocks most of IR allowing use as a Greenhouse cover
- allows IR around 4 $\mu$m to pass reducing effectiveness of high temperature insulation
- loading carbon into gel will block the 4 $\mu$m IR
  - Mix carbon into suspension before gelation
  - Add organic into solution before gelation and carbonize after final supercritical drying
  - Adsorb organic vapor into dried aerogel and subsequently carbonize

Courtesy of Prof. Pamela Norris, UVA
Infrared Radiative Window

IR “window” allowing radiative heat transfer

\[ Q_R \approx T^{2.6} \quad 250 \, K < T < 450 \, K \]

(which is a curve-fit to the convolution of the blackbody spectrum and the transmission window)

Infrared window in plain aerogels can be “closed” by dispersing carbon in the silica aerogel

Courtesy of Prof. Pamela Norris, UVA