Fire Retardant Aerogels Made from a Polybenzimidazole Backbone

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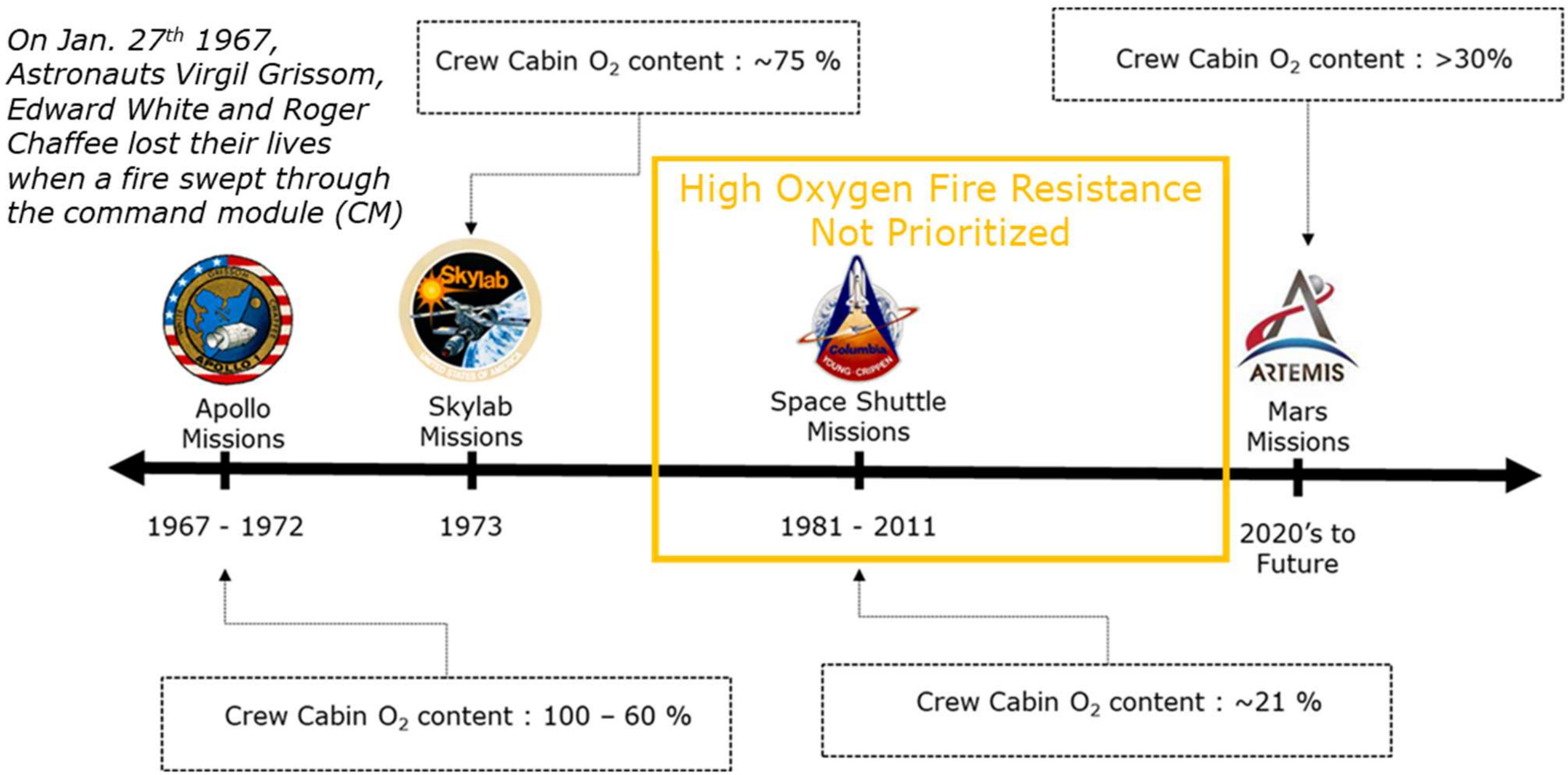
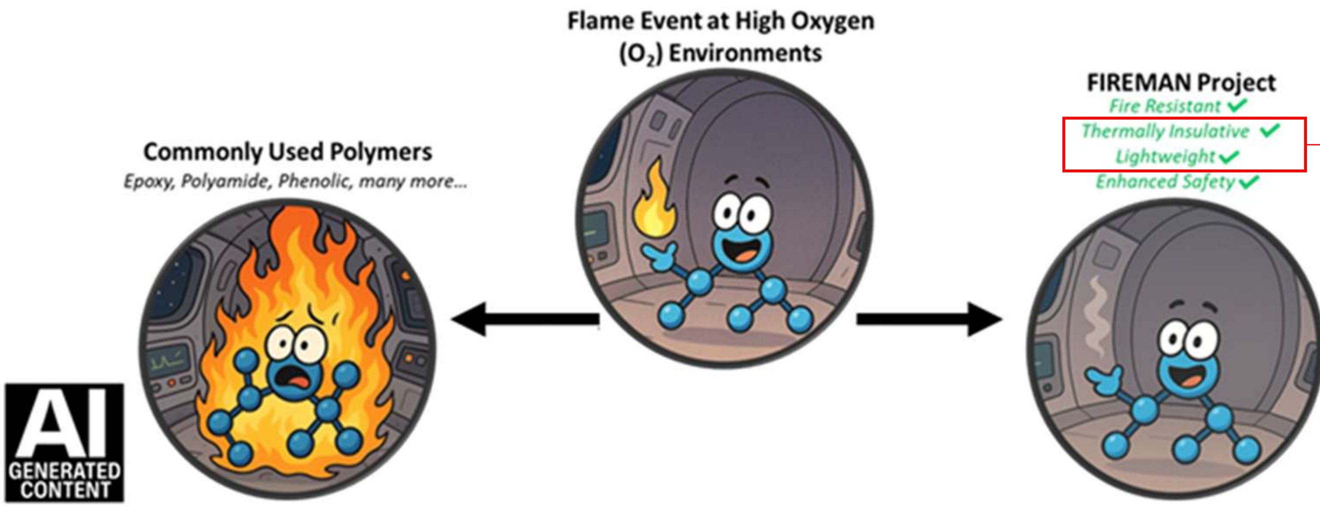
Timeline of Oxygen Environments for Crew Cabins



- **Due to the return of oxygen content levels similar to Earth's atmosphere, the research and development of fire-resistant materials was abandoned**
- **Some of the previously developed materials stopped being commercially produced**

Fireproof Insulation for Resistive Environments in Materials for Aerospace and Navigation (FIREMAN)

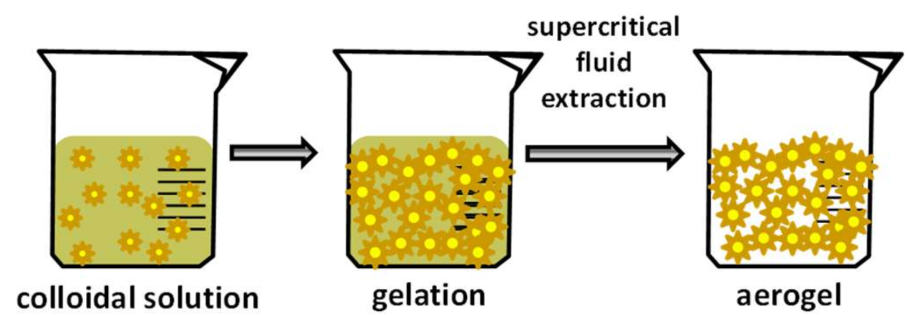


Typical properties

- High porosity (>85%)
- Pore size 10-40 nm
- High specific surface area
- Low thermal conductivity
- Low density

What is an Aerogel?

An open-celled, light weight, porous material derived from a gel in which the liquid is replaced by gas while maintaining the self-assembled three-dimensional structure



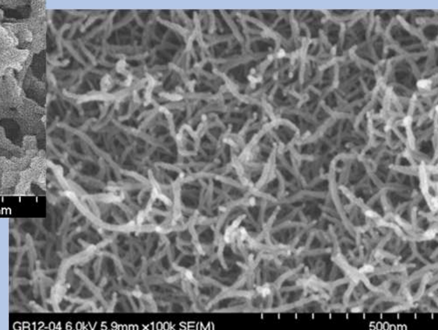
Aerogels made using sol-gel synthesis and supercritical fluid extraction

Aerogel Properties Dependent on Structure and Chemistry

Structure

Present in all aerogels

carbon



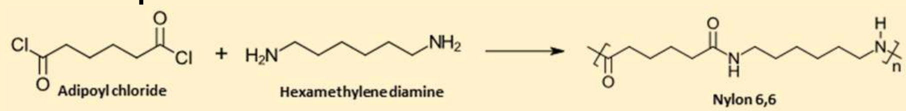
polymer

- High porosity
- Nano-scale pore size
- Low density
- High internal surface area
- Low Tc and dielectric

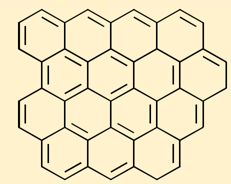
Chemistry

Varies in aerogels

- Aliphatic in backbone increases flexibility



- Aromatics in backbone increases strength and temperature resistance

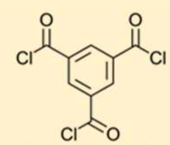


Graphene

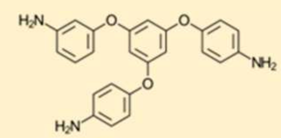
- Functional groups increase capabilities

4,4′-(Hexafluoroisopropylidene)diphthalic Anhydride (6FDA)

- Crosslinkers increase chemical stability and mechanical strength



1,3,5-benzenetricarbonyl trichloride (BTC)



Tris-1,3,5-aminophenoxy benzene (TAB)

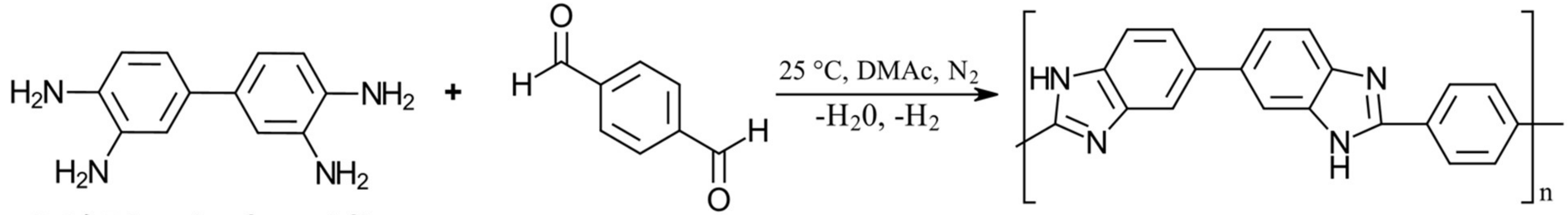
Polymer Backbone impacts Limiting Oxygen Index (LOI)

- LOI is the minimum concentration of oxygen *in the environment* needed to support combustion of a polymer
- The higher LOI is, the more flame resistant

	Polymer	LOI (%)
	Polymethylmethacrylate (PMMA) [1]	17
	Polypropylene (PP) [1]	17
ISS / Earth Atmosphere (O_2 Concentration: ~21%)	Polyethylene (PE) [1]	17
	Polystyrene [1]	18
	Vinyl Ester [1]	22
	Epoxy [1]	25
	Polyamide (Nylon 6,6) [1]	26
	Polycarbonate [1]	26
Exploration Atmosphere (O_2 Concentration: >30%)	Polybenzoxazine (PBz)[3]	26
	Polyamide (Nomex®)[2]	29
	Phenolic[4]	30 - 38
	Polyetheretherketone (PEEK) [2]	35
	Polyimide (Kapton®) [2]	37
	Polyaramide (Kevlar®) [2]	38
	Polyetherketoneketone (PEKK) [2]	40
Halogenated Polymers	Polybenzimidazole (PBI) [2]	42
	Polyvinylchloride (PVC) [1]	45
	Polytetrafluoroethylene (PTFE) [1]	>90

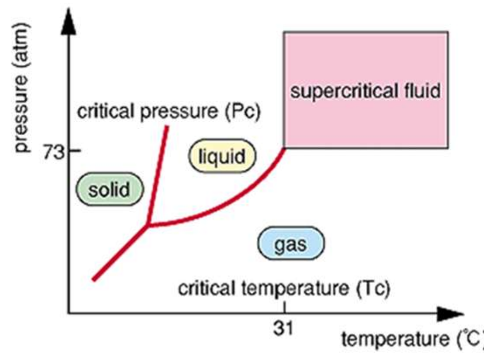
[1] D. Watson, D. Schiraldi, Biomolecules as Flame Retardant Additives for Polymers: A Review, Polymers 2020, 12(4), 849; https://doi.org/10.3390/polym12040849
[2] J. Quintiere, Appendix 15 in *Fundamentals of Fire Phenomena* (2006) Ed. 1 pp. 432
[3] C. Jupslip, C. Panyawanitchakun, S. Rimdusit, Flammability and thermomechanical properties of dianhydride-modified polybenzoxazine composites reinforced with carbon fiber, Polym. Comp. 34 (12) (2013) pp. 2067 – 2075. https://doi.org/10.1002/pc.22615
[4] C. Mougel, T. Garnier, P. Cassagnau, N. Sintes-Zydowicz, Phenolic foams: A review of mechanical properties, fire resistance, and new trends in phenol substitution, Polym. 164 (15) (2019) pp. 86 – 117. https://doi.org/10.1016/j.polymer.2018.12.050

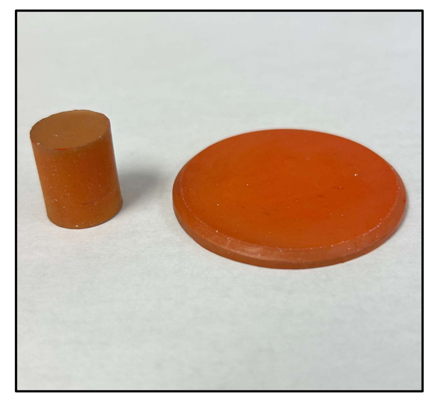
Fabrication Process of PBI Aerogels



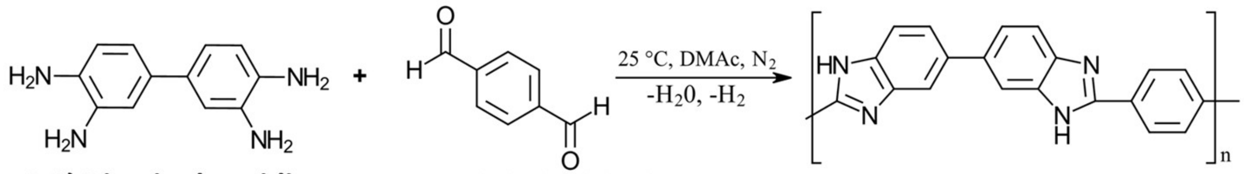
3,3′-Diaminobenzidine (DAB) | Terephthalaldehyde (TPA) | Polybenzimidazole (PBI)

Polymer Synthesis/ Gelation → ***Solvent Exchange*** → ***Supercritical Fluid Extraction***





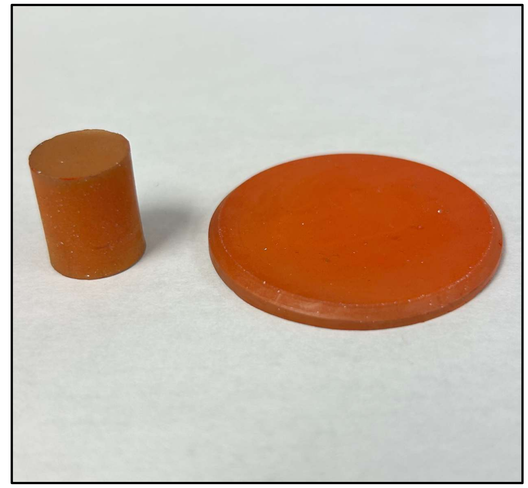
Physical Properties of PBI Aerogels



3,3′-Diaminobenzidine (DAB)

Terephthalaldehyde (TPA)

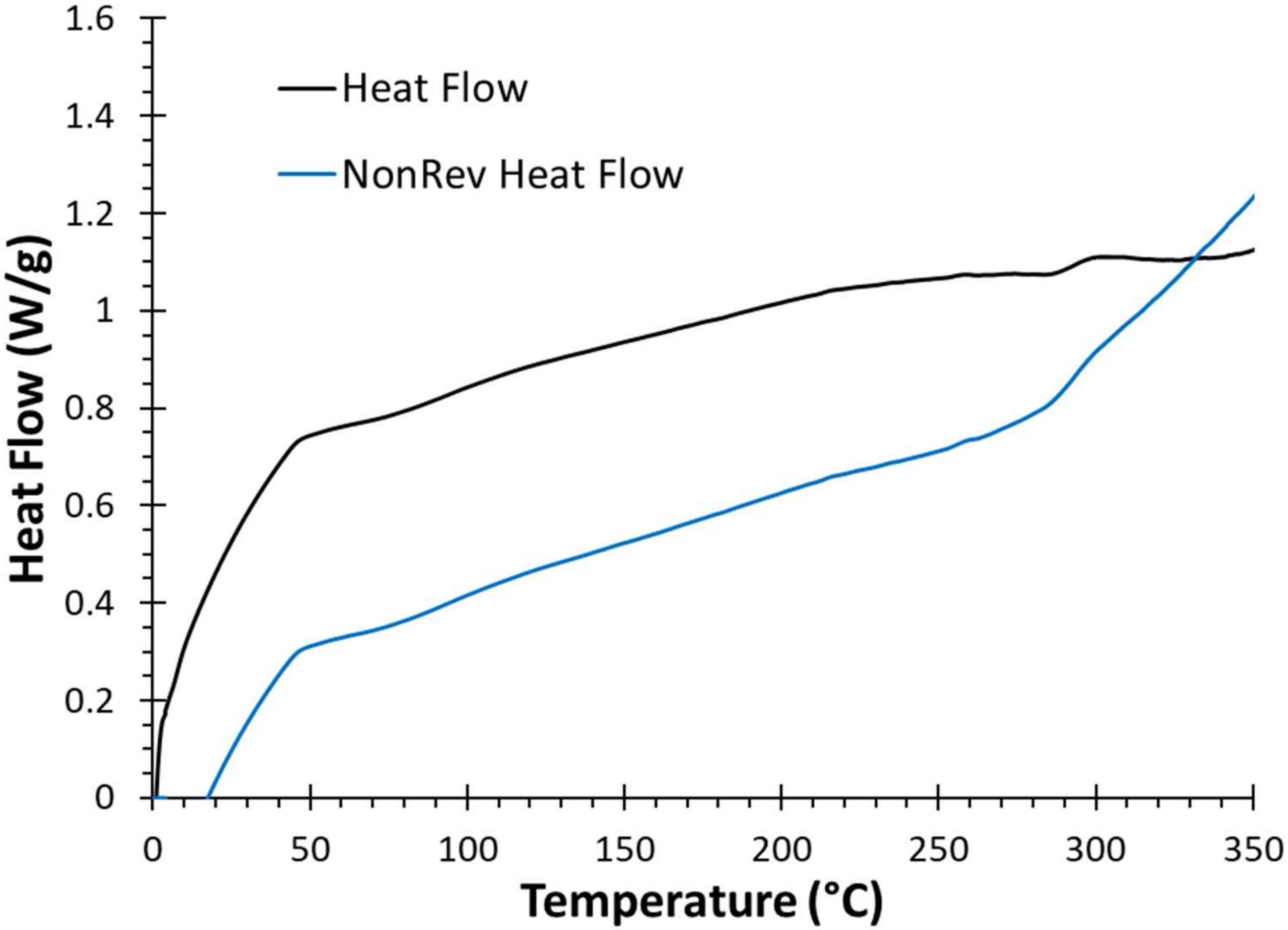
Polybenzimidazole (PBI)



PBI wt %	Gel Time (min)	Bulk Density (g cm^{-3})	*Porosity (%)	Shrinkage (%)	Surface Area (m^2 g^{-1})
20	~3	0.39 ± 0.01	70.8 ± 0.5	25.2 ± 0.3	173
15	~5	0.31 ± 0.01	76.2 ± 0.1	26.7 ± 0.2	233
10	~10	0.22 ± 0.01	83.3 ± 0.5	28.5 ± 0.5	237
7.5	~20	0.19 ± 0.01	85.6 ± 0.3	32.1 ± 0.2	183
5	~40	0.11 ± 0.01	91.9 ± 0.4	29.8 ± 0.3	216

*Skeletal density: ~1.30 g cm^{-3}

Differential Scanning Calorimetry (DSC) Data of PBI aerogels



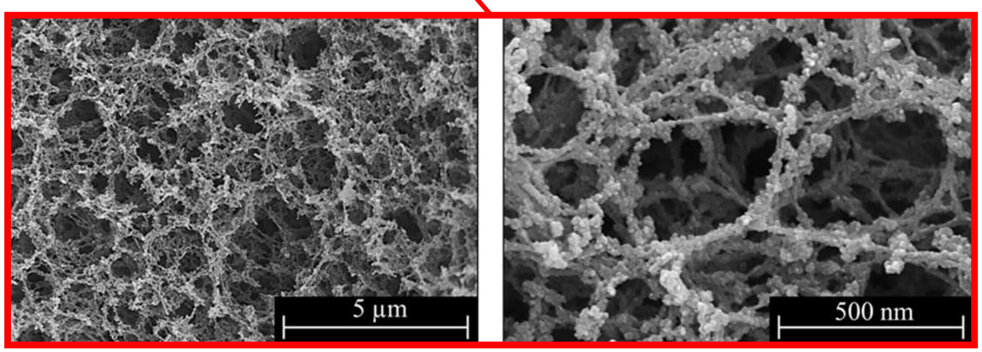
- Non-reversible heat flow present at >200 °C indicating the presence of further reaction

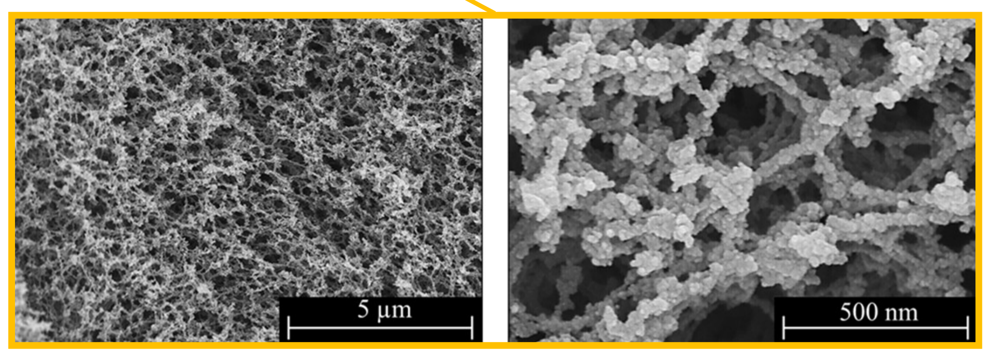
Impact of Higher Temperature Post Cure

200°C
24 hrs.

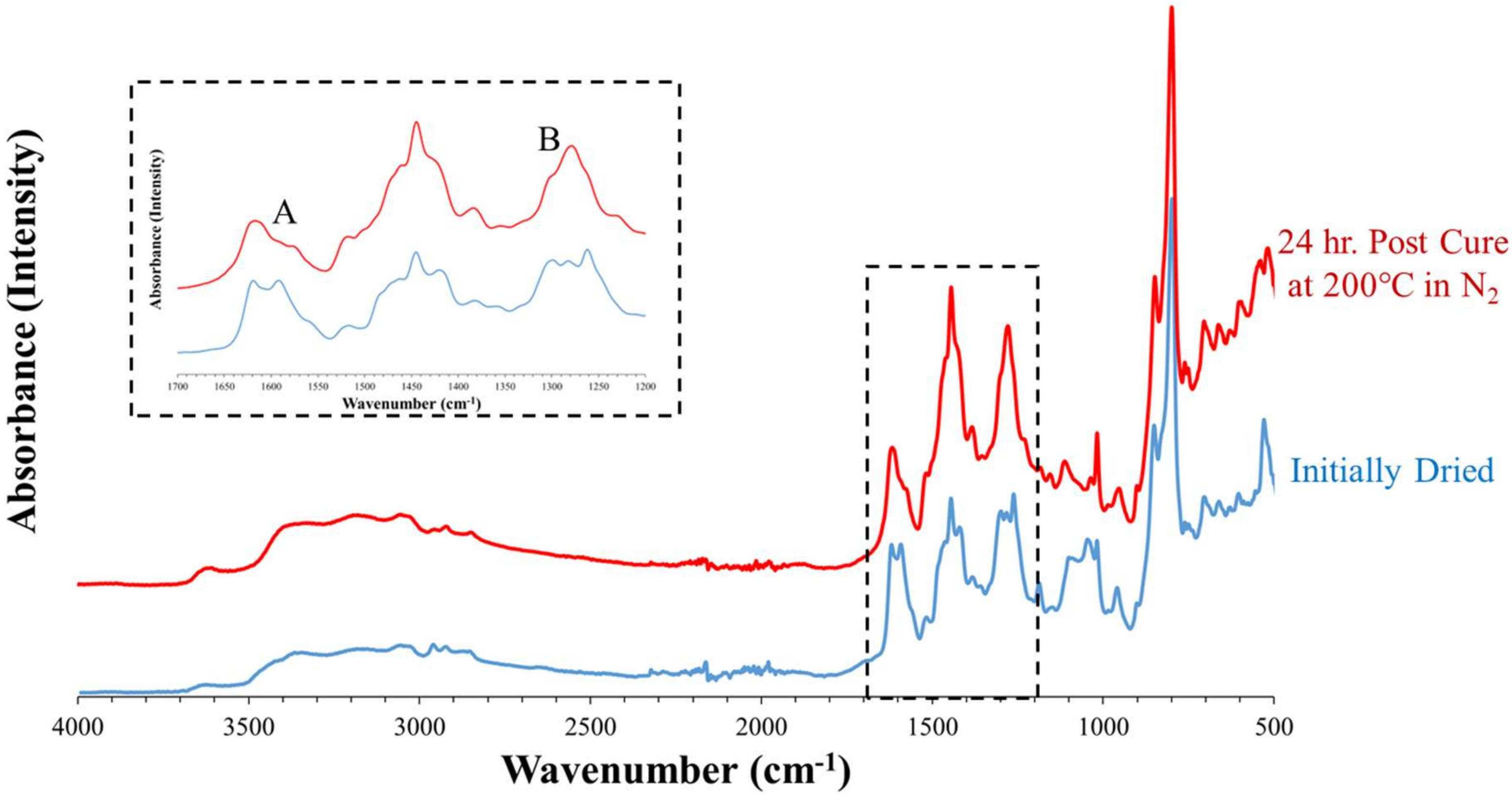
N_2

- Surface area reduction: ~ 30%
- Additional Shrinkage: ~1%
- Porosity Reduction: ~2%
- Skeletal Density change negligible
- Bulk Density Increase: ~2%



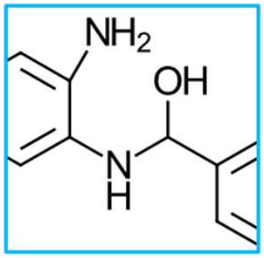


Fourier Transform Infrared Spectroscopy (FTIR) of PBI Aerogels

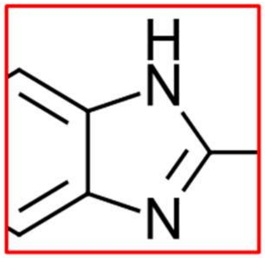


<u>A:</u> 1620 cm^{-1} band N-H stretching (two bands because both primary and secondary)

<u>B:</u> 1330 - 1250 cm^{-1} C-N stretch aromatic amines (two bands because both primary and secondary)

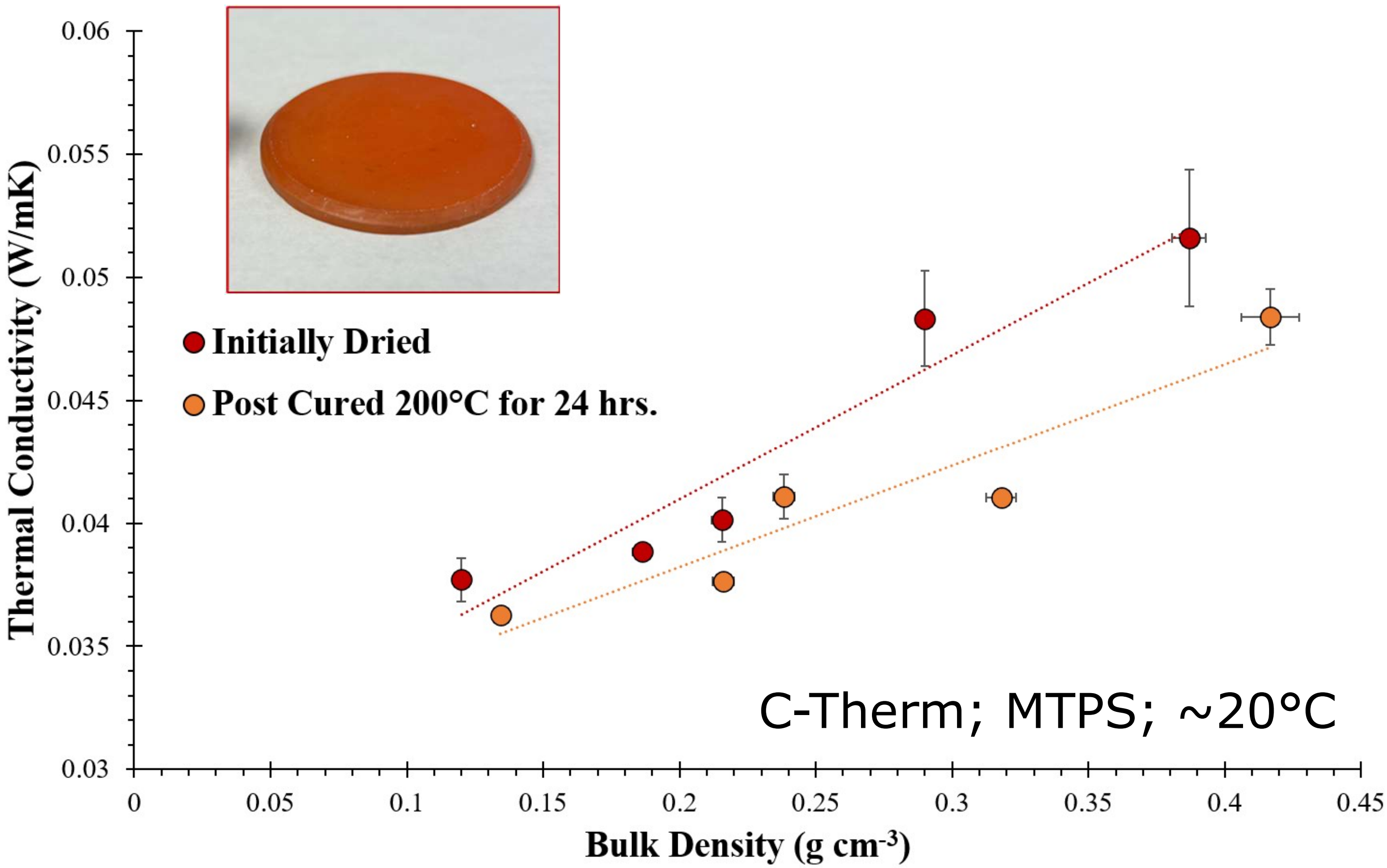


Primary / Secondary



Secondary

Thermal Conductivity of PBI Aerogels

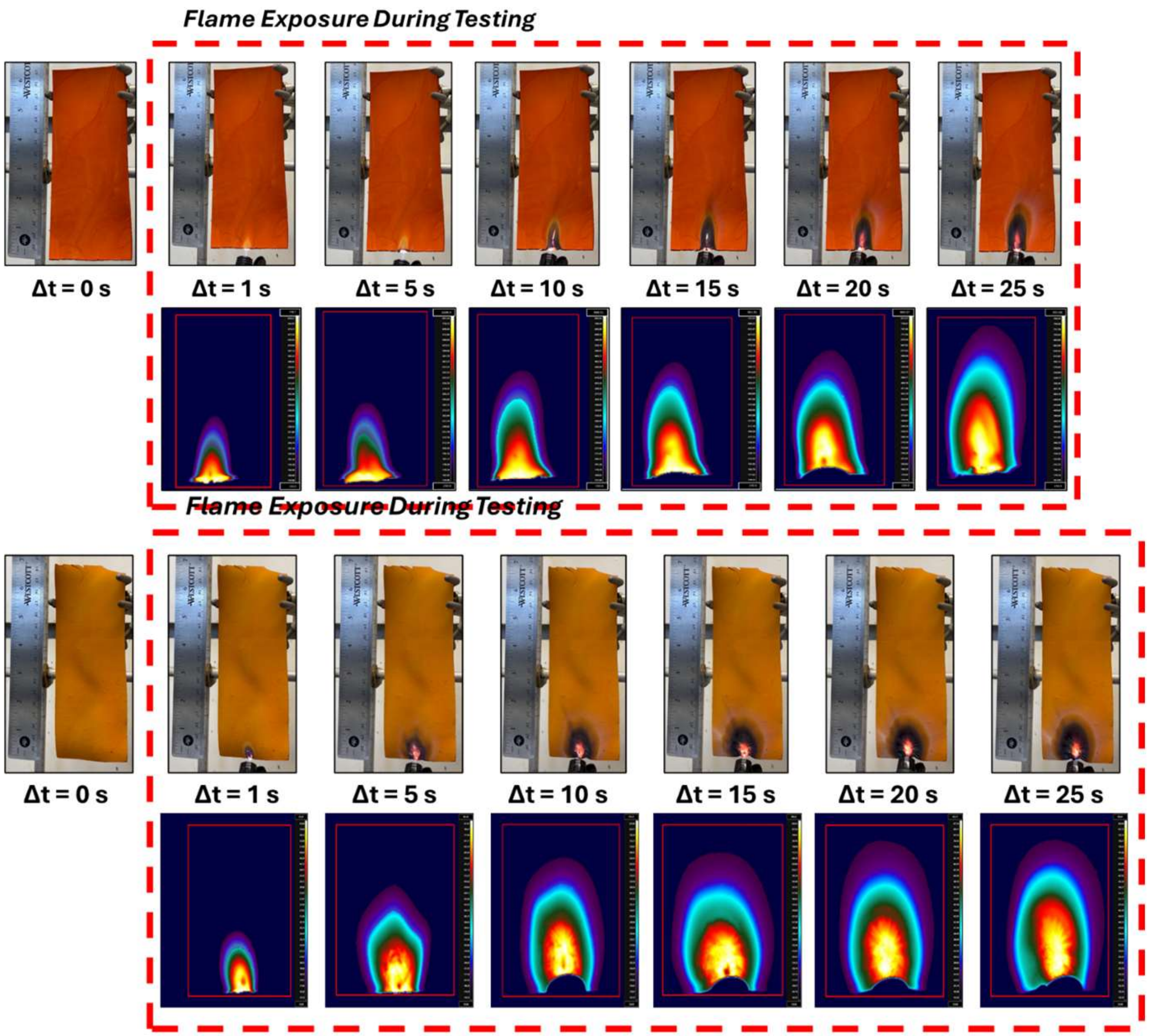


- Reduced thermal conductivity in post cured PBI aerogels

PBI Aerogel Scale-Up Fabrication

- Fabricated 20” x 20” x 0.2” PBI panels equating to ~0.75 kg material

Flame Propagation Screening in Earth Atmosphere



- Applied ~1000°C flame to bottom of 3”x6” sample for 25 seconds
- Measured flame propagation and temperature using FLIR camera

Flame Propagation Screening in Earth Atmosphere

Run 1

Run 2

Run 3

- Flame Influence: 2.3 ± 0.5 cm
- Consumed Length: 3.8 ± 0.4 cm
- Heat Damage: 5 ± 0.5 cm

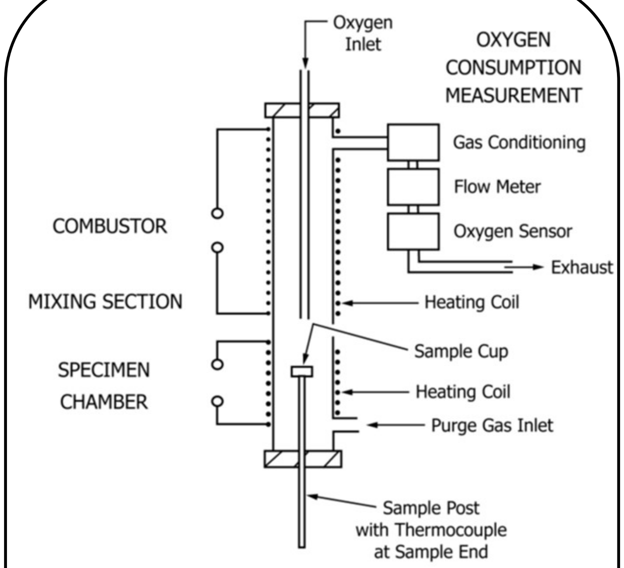
Run 1

Run 2

Run 3

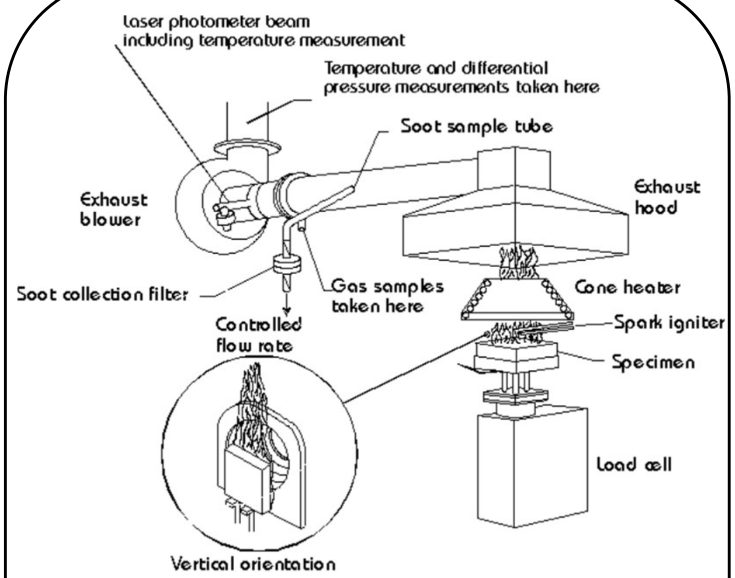
- Flame Influence: 1.5 ± 0.3 cm
- Consumed Length: 3.3 ± 0.3 cm
- Heat Damage: 4.3 ± 0.4 cm

Advanced Fire Resistance Testing - University of Dayton Research Institute



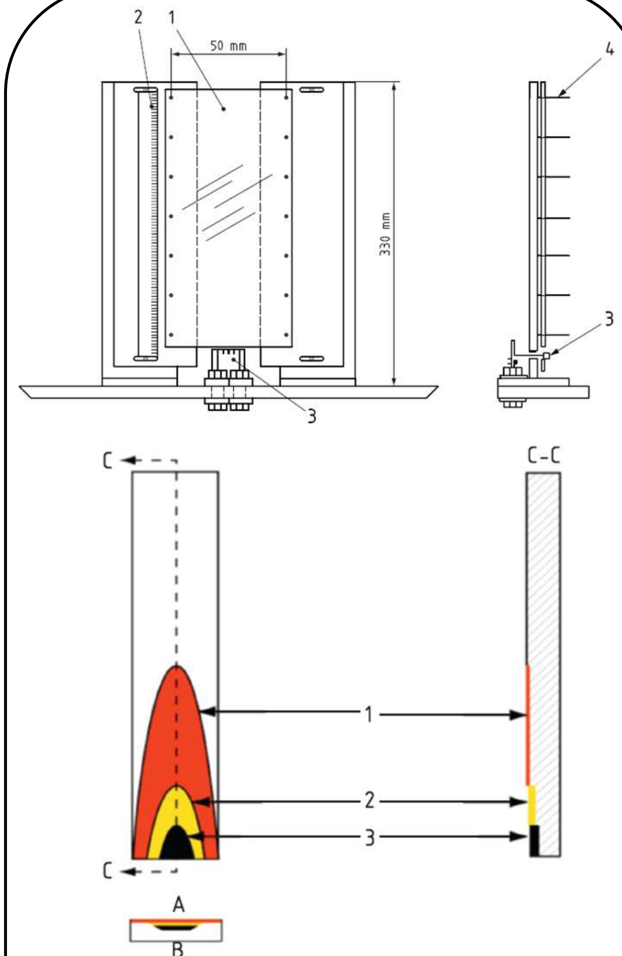
Micro Combustion Calorimeter

Ref: ASTM D7309



Cone Calorimeter

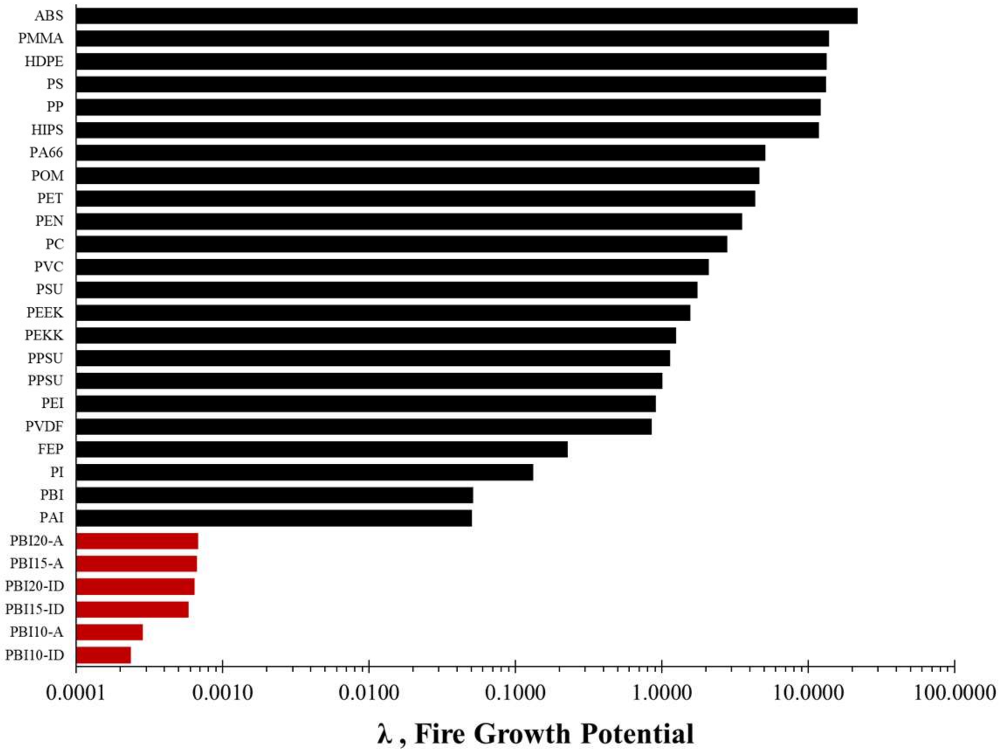
Ref: ASTM E1354



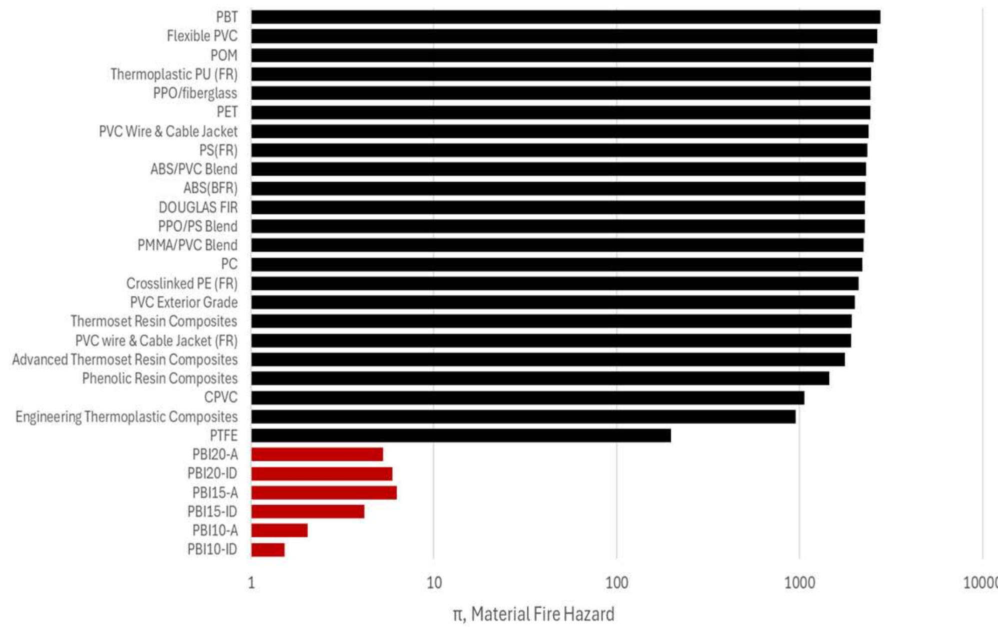
Vertical Burn Test

Ref: ISO 14624-1

Cone Calorimetry Testing Summary



Ref: R. Lyon, Journ. Fire Sci. 2024, Vol. 42(6) 487-513.

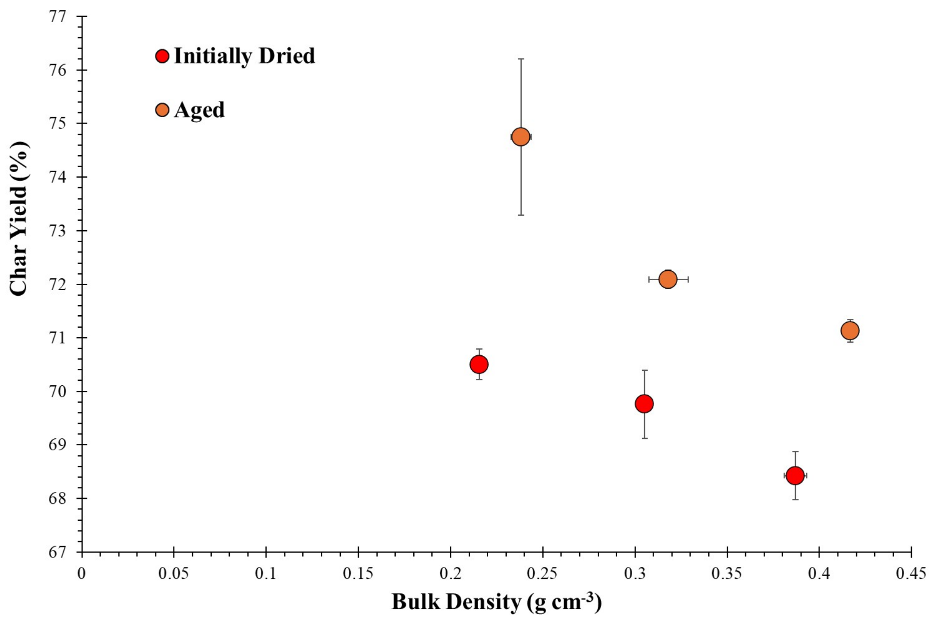


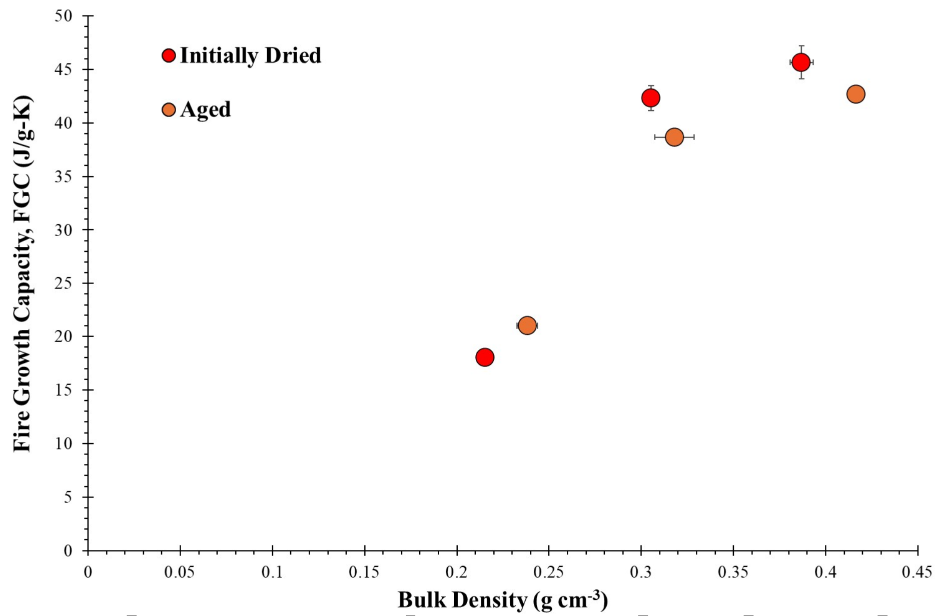
Ref: R. Lyon, Journ. Fire Sci. 2024, Vol. 42(6) 487-513.

GRC-derived insulation is 3-5 orders of magnitude (1000x) more fire resistant than common polymeric materials!

Credit: Dr. Alexander Morgan, University of Dayton Research Institute

Microcombustion Cone Calorimetry Testing Summary



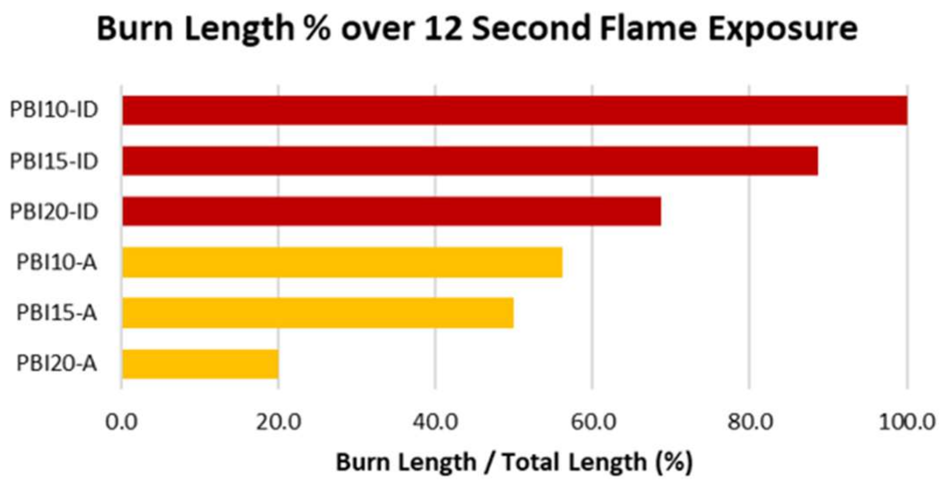


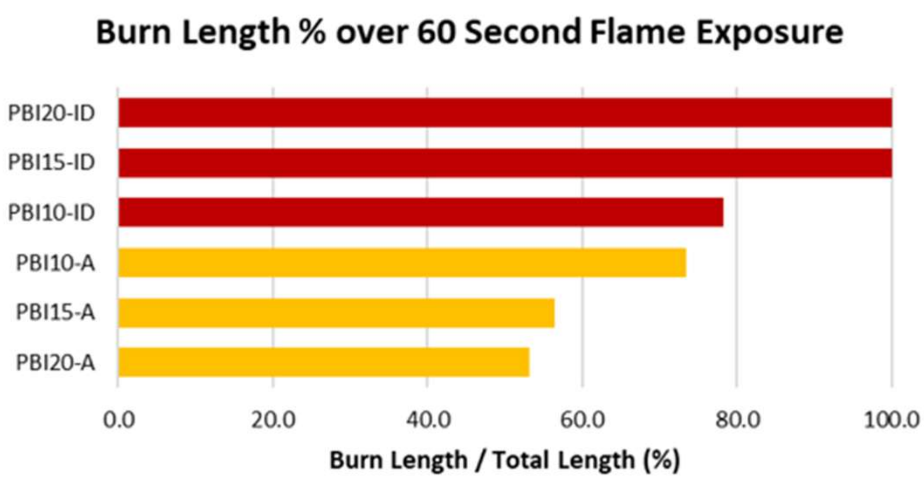
- **<u>Char yield:</u>** Measure of residual mass remaining after pyrolysis

- **<u>Fire Growth Capacity (FGC):</u>** Calculated within ASTM D7309 to give insights into the potential for a material, as a function of its chemical structure, to ignite and grow in fire intensity once ignited

Credit: Dr. Alexander Morgan, University of Dayton Research Institute

Vertical Burn Testing Summary

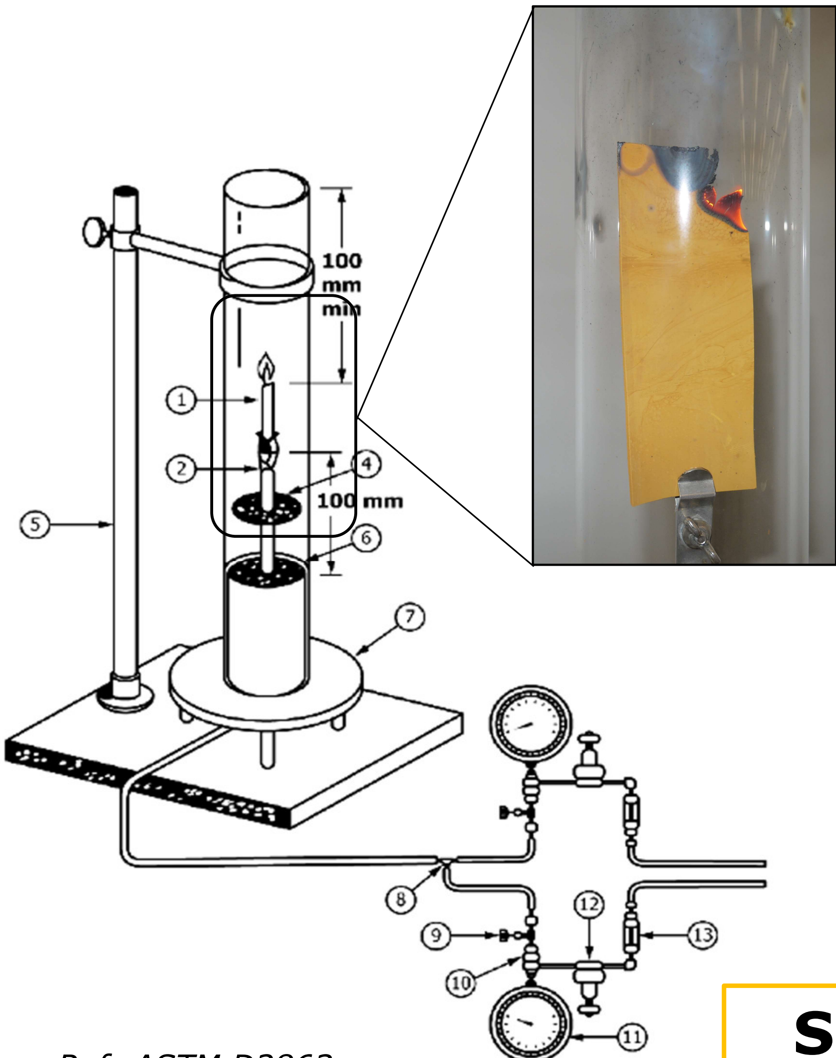




- Sample passes test if flame does not consume past 6” (sample length), is self extinguished, and there is no melting.

Credit: Dr. Alexander Morgan, University of Dayton Research Institute

Limiting Oxygen Index (LOI) Testing Summary



Ref: ASTM D2863

- LOI of initially dried: 33.9%
- LOI of post cured: 35.6%

Self-extinguishing behavior with limiting oxygen index (LOI) >35 %

Acknowledgements

- Funding with the Center Innovation Fund (CIF) through Space Technology Mission Directorate (STMD)
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Polymer Aerogel Innovation and Research (AIR) Laboratories

The Polymer Aerogel Innovation and Research Laboratories are dedicated to advancing the science and engineering of lightweight, high-performance aerogel materials to address the most demanding challenges in aerospace, aeronautics, and advanced technology sectors. Our mission is to pioneer innovative solutions in thermal management, structural insulation, energy absorption, and multifunctional material systems through the design, fabrication, and characterization of next-generation polymer aerogels. By fostering collaboration across government, industry, and academia, we strive to accelerate the transition of laboratory breakthroughs into real-world applications that enable safer, more efficient, and more technically advanced exploration and innovation.

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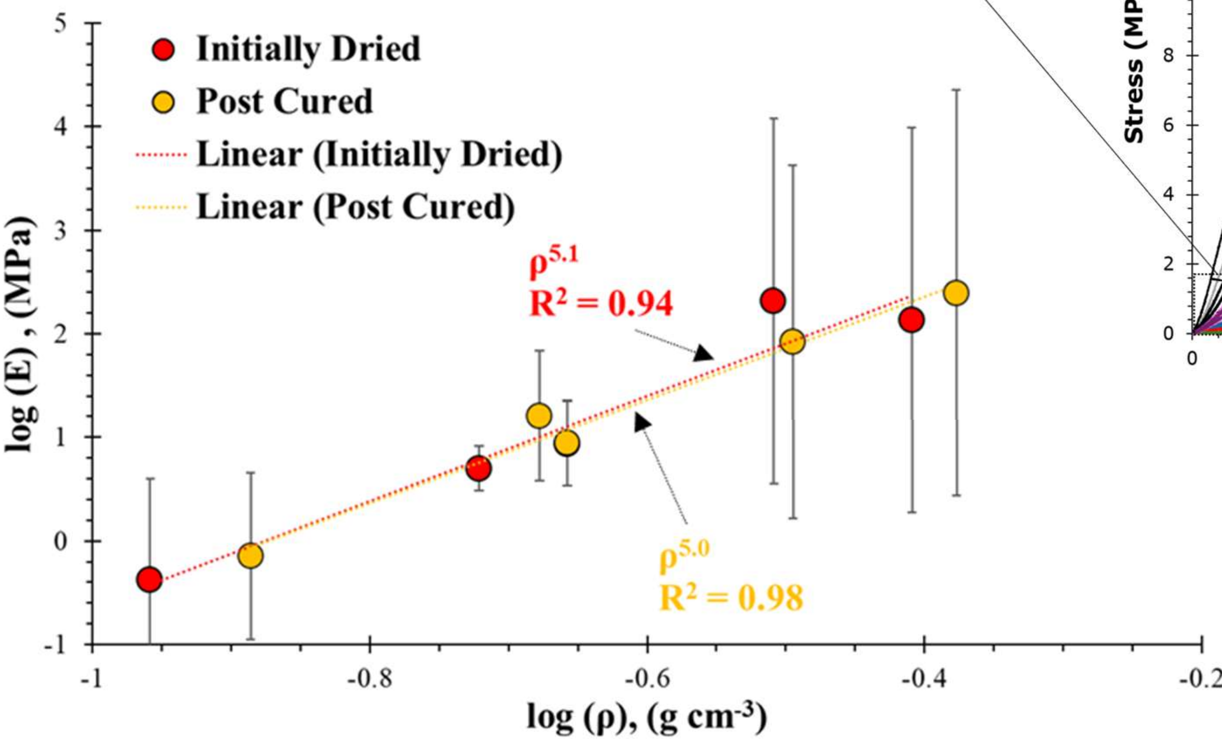
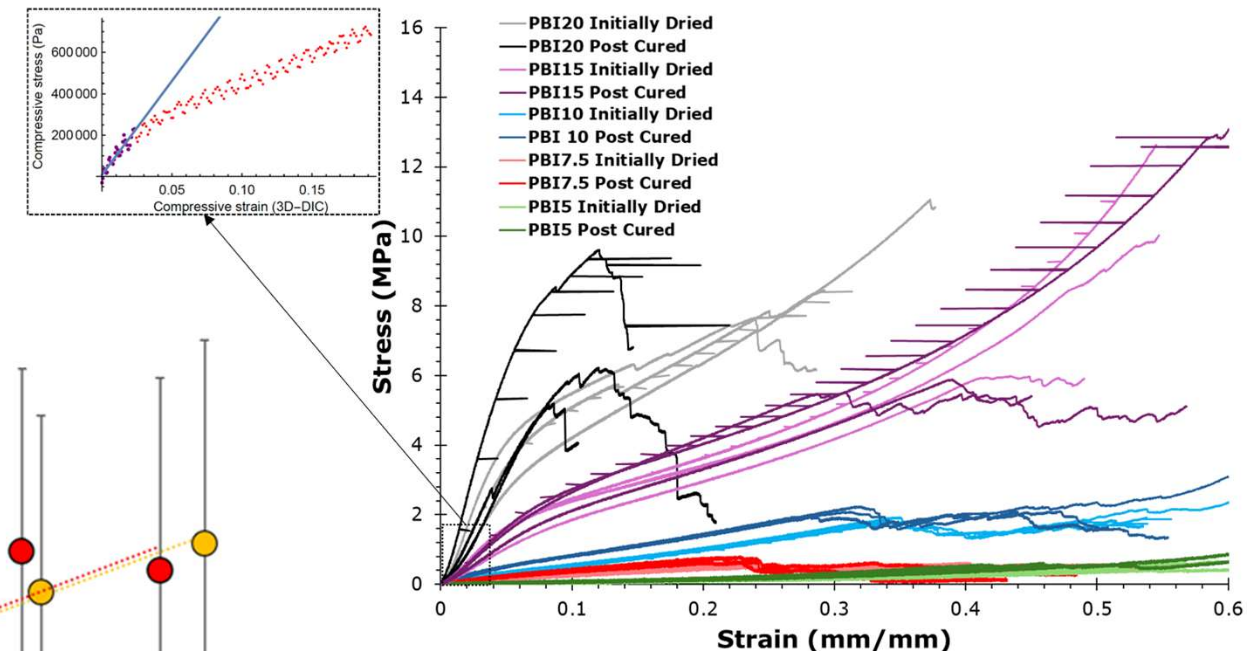
The University of Akron

Dr. Michael Chauby
PhD Chemical Engineering
Rowan University

Compression Testing of PBI Aerogels





- Modulus range 0.3 – 250 MPa
- Yield Strength: 0.2 – 5.4 MPa

- Average increase of 24 % in yield strength between initially dried and post cured PBI aerogels
- Average increase of 82 % in modulus