NEXTGEN
BATTERY TECHNOLOGIES, LLC.
Driving Innovation through Chemistry

High-density Hydrogen Storage in Space-filling Polyhedral Sorbents

Project ID: st150

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NextGen Battery Technologies, LLC

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Overview

Timelines

Start: April 9, 2018

End: October 8, 2018

Barriers

A. System weight and volume

J. Thermal management

E. Charging/discharging rates

Budget

Phase I SBIR

Total project budget: $143,783

Federal share: $143,783

Partners

NEI Corporation

Relevance

Objective

Increase the packing density of adsorbent materials *using a scalable process* without compromising their hydrogen storage properties or adversely affecting storage temperatures and pressures, gravimetric capacity, or reversibility

Parameter	DOE 2020 Technical Target	Project Impact
System volumetric capacity	0.030 kg H_2/liter system	Increase >50% (materials dependent)
System gravimetric capacity	0.045 kg H_2/kg system	No change
Min/max delivery temperature	-40 °C / +85 °C	No change
Min delivery pressure	5 bar	No change
Minimum full flow rate	0.02 (g H_2/s)/kW	No change

Approach

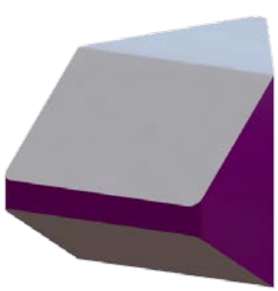
- Demonstrate the formation of space-filling polyhedral microliths from an activated carbon, a metal organic framework material, and a porous polymer
- Demonstrate the packing of space-filling polyhedral sorbents in a 1-liter container

	Milestone	Date	Performance
1	Formation of space-filling polyhedral shapes from at least two distinct classes of base materials with <5% change in key properties	08/09	Demonstrated for activated carbon
2	High-pressure hydrogen storage in space-filling polyhedral microliths from at least two distinct classes of base materials with <5% change in properties	09/09	Not started
3	Provide samples of space-filling polyhedral sorbents to NREL to validate adsorption measurements	10/09	Activated carbon available today
4	Demonstrate the packing of space-filling polyhedral sorbents in a 1-liter container with >95% of the theoretical packing fraction	10/09	Demonstrated at 250 ml

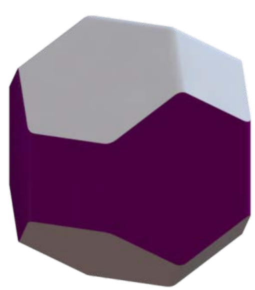
Accomplishments and Progress: *Shapes*

- Hardware for fabrication of space-filling microliths developed
- Pressing/casting conditions established
  - Carbon: 70 – 140 MPa (10 – 20 kpsi)
  - MOF: 50 MPa (7 kpsi)
  - Polymer: cast
- 6 kg of activated carbon rhombic dodecahedra fabricated

Space-filling polyhedral dies for a high-speed rotary tablet press



Gyrobifastigium



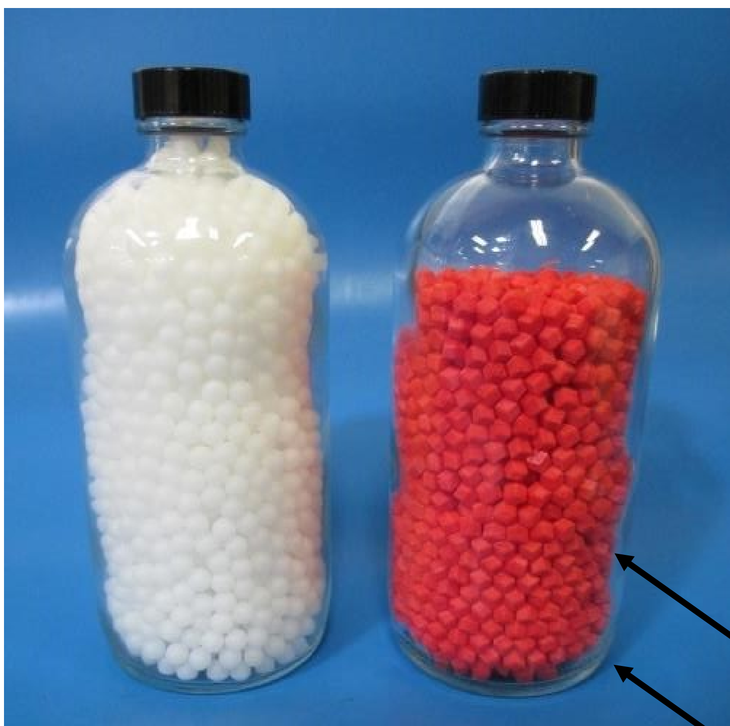
Truncated Octahedron

Gyrobifastigium   Truncated octahedron

Accomplishments and Progress: *Packing*

- Demonstrated packing of space-filling microliths in a 250 ml container with >96% of the theoretical packing fraction

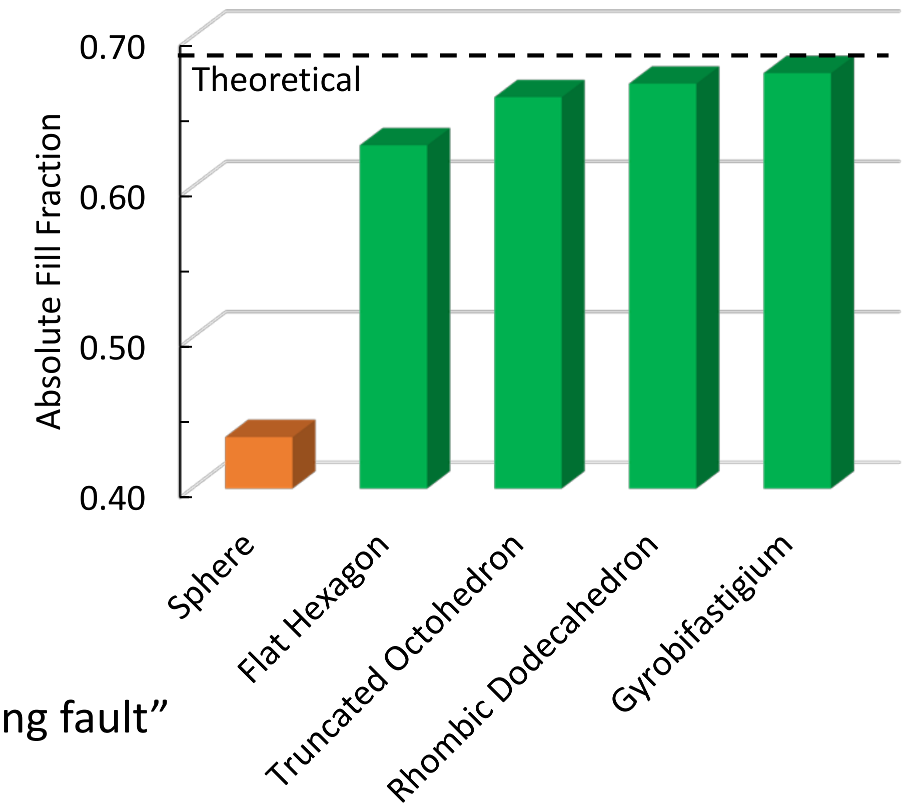
Comparable volume of spheres (white, 43% packing) and rhombic dodecahedra (red, 66.9% packing) in 250 ml jars



“Stacking fault”

Seed layer

Packing of space-filling polyhedral microliths in a 250 ml jar



Accomplishments and Progress:
Responses to Previous Year Reviewers' Comments

This project was not reviewed last year

Collaboration and Coordination

- Sub-awardee: NEI Corporation
- Small business outside the DOE Hydrogen and Fuel Cell Program
- Assist with materials synthesis, shaping, characterization (surface area, density, low-pressure hydrogen uptake), tank filling
- *Critical to the success of the project*

NEI Corporation

Remaining Challenges and Barriers

- Within the project
  - Procurement and/or synthesis of MOF-5
  - Synthesis of hyper-crosslinked polymer
  - Additional materials to test?

- To reach DOE hydrogen storage goals
  - Higher hydrogen density storage materials

Proposed Future Work

- Complete formation of space-filling microliths from MOF-5 (or other MOF), hyper-crosslinked polymer, other? – Milestone due 08/09
- Measure hydrogen uptake at low (1 atm) and high (100 atm) pressures for all samples – Milestone due 09/09
- Optimize filling of 1 liter vessel with space-filling microliths – Milestone due 10/09
- Outreach to industry for feedback – ongoing
- Write successful SBIR Phase II proposal (FY19) – TBD

Any proposed future work is subject to change based on funding levels

Technology Transfer Activities

- US patent 9,370,765: *Space-filling Polyhedral Sorbents* (issued June 21, 2016)
- Collaboration with InnerProduct Partners (venture capital)
- Industrial outreach begun

Summary

Goal

- Demonstrate the formation of space-filling polyhedral microliths from an activated carbon, a metal organic framework material, and a porous polymer and show that their hydrogen storage capacity has not been diminished from that of the base material by more than 5%

Accomplishments

- Hardware for fabrication of space-filling microliths developed
- Pressing/casting conditions established
- 6 kg of activated carbon rhombic dodecahedra fabricated
- Demonstrated packing of space-filling microliths in a 250 ml container with >96% of the theoretical packing fraction

Technical Back-up Slides

Shape Packing

	Advantages	Disadvantages
Spheres	• Added through the neck of a standard gas cylinder	• Low packing density • Poor thermal conductivity
Monoliths	• Near 100% packing fraction • Improved thermal conductivity	• Expensive and difficult to form • Low gas flow rate • Custom cylinder
Space-filling polyhedra	• Near 100% packing fraction • Added through the neck of a cylinder • Improved thermal conductivity • High gas flow rate	

	Monodispersed Spheres	Bimodal (10X) Size Distribution	Monolith	Space-filling Polyhedra
Random packing	0.534	0.726	N/A	0.97
Ideal	0.740	0.933	1.0	1.0

Spheres: S. Yamada, et al., IPSJ Trans. Math. Model. and Appl. **4** (2011) 23
Monoliths: ATMI/Entegris Space-filling polyhedra: US patent 9,370,765

Sorbent Agnostic: Literature Observations

	Measurement	Powder	Monolith
Pvdc-based activated carbon (BrightBlack) (1)	Size	200 μm	10+ cm
	Surface Area	1030 m^2/g	1030 m^2/g
	Density	0.68 g/cm^3	1.10 g/cm^3
	Pore Volume	0.4 cm^3/g	0.4 cm^3/g
	CO_2, CH_4 Uptake	Identical	
MOF 5 (2)	Size	0.25 μm	0.5 cm
	Surface Area	2762 m^2/g	2265 m^2/g
	Density	0.13 g/cm^3	0.5 g/cm^3
	Pore Volume	1.37 cm^3/g	1.12 cm^3/g
	H_2 Uptake	7.4 g/l	26 g/l
Hyper-crosslinked polymer (3)	Surface Area	>2000 m^2/g	>2000 m^2/g
	Pore Volume	0.70 – 0.78 cm^3/g	0.70 – 0.78 cm^3/g
	CH_4 Uptake	0.156 g/g and 82.7 cm^3/cm^3	0.156 g/g and 164 cm^3/cm^3

(1) ATMI/Entegris

(2) J. J. Purewal, et al., Int. J. H. Energ. **37** (2012) 2723

(3) W. T. Yongqin and F. Svec, Appl. Energ. **183** (2016) 1520