Inexpensive Superinsulation for Cryogenic and Highly Insulating Applications

Cooperative Research and Development Final Report

CRADA Number: CRD-21-18076

NREL Technical Contacts: Chaiwat Engtrakul and Lin Simpson

NREL is a national laboratory of the U.S. Department of Energy
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Technical Report
NREL/TP-5900-97751
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15013 Denver West Parkway
Golden, CO 80401
303-275-3000 • www.nrel.gov

NOTICE

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Cooperative Research and Development Final Report

Report Date: October 1, 2025

In accordance with requirements set forth in the terms of the CRADA agreement, this document is the CRADA final report, including a list of subject inventions, to be forwarded to the DOE Office of Scientific and Technical Information as part of the commitment to the public to demonstrate results of federally funded research.

Parties to the Agreement: Insula Inc.

CRADA Number: CRD-21-18076

CRADA Title: Inexpensive Superinsulation for Cryogenic and Highly Insulating Applications

Responsible Technical Contact at NREL:

Chaiwat Engtrakul | Chaiwat.Engtrakul@nrel.gov

Lin Simpson | Lin.Simpson@nrel.gov

Name and Email Address of POC at Company:

Doug Proctor | doug@insula.co

Sponsoring DOE Program Office(s): Office of Energy Efficiency and Renewable Energy (EERE), Advanced Manufacturing and Materials Technologies Office

Joint Work Statement Funding Table showing DOE commitment:

Estimated Costs	NREL Shared Resources a/k/a Government In-Kind
Year 1	$353,513.00
Year 2, Modification #1	$82,817.00
Year 3, Modification #2	$93,670.00
Year 4, Modification #3	$000.00
TOTALS	$500,000.00

Executive Summary of CRADA Work:

This effort will use our novel insulation formed into effective “solid” (as compared to powder) insulating panels and/or wraps to demonstrate important technical achievements including scaling appropriate crosslinking to directly and cost effectively address vacuum insulated panel (VIP) applications and cryogenic component wrapping to help reduce energy use. This project will improve and scale-up processes (from 3” diameter disks) to make larger samples (e.g., 12” x 12” x 1”) Wow, cfor external evaluation and testing.

CRADA benefit to DOE, Participant, and US Taxpayer:

- Assists laboratory in achieving programmatic scope,
- Uses the laboratory's core competencies, and/or
- Enhances U.S. competitiveness by utilizing DOE developed intellectual property and/or capabilities.

Summary of Research Results:

Purpose

To solve world energy demand and resolve global environmental problems, we must go well beyond inexpensive renewable energy generation and include transformational and fundamental changes in our approach and conviction to energy efficiency/savings. The ultimate goal of this collaboration is to develop novel thin, lightweight, and inexpensive super insulating materials that will be superior alternatives for standard insulations (e.g., Batt-fiberglass, extruded polystyrene or polyiso rigid foams) in terms of R-value per inch and cost per R-value, for a broad range of applications to substantially improve energy efficiency. Our unique approach (Figure 1) creates small pockets where 70% to 85% of the insulation volume is vacuum (no conduction or convection) along with novel materials in between the vacuum pockets that substantially reduce the residual air conduction and virtually eliminates radiative transport. Our unique materials integration approach addresses the higher thermal conduction pathways of similar materials that have absolute/effective thermal conductivities (ATCs) greater than 0.03 W/m-K. This project will improve and scale-up processes to make large enough samples (e.g., 12" x 12" x 1") for external evaluation and testing to demonstrate that the superinsulation has a viable technical and economic path for the VIP, cryogenic and other similar markets.

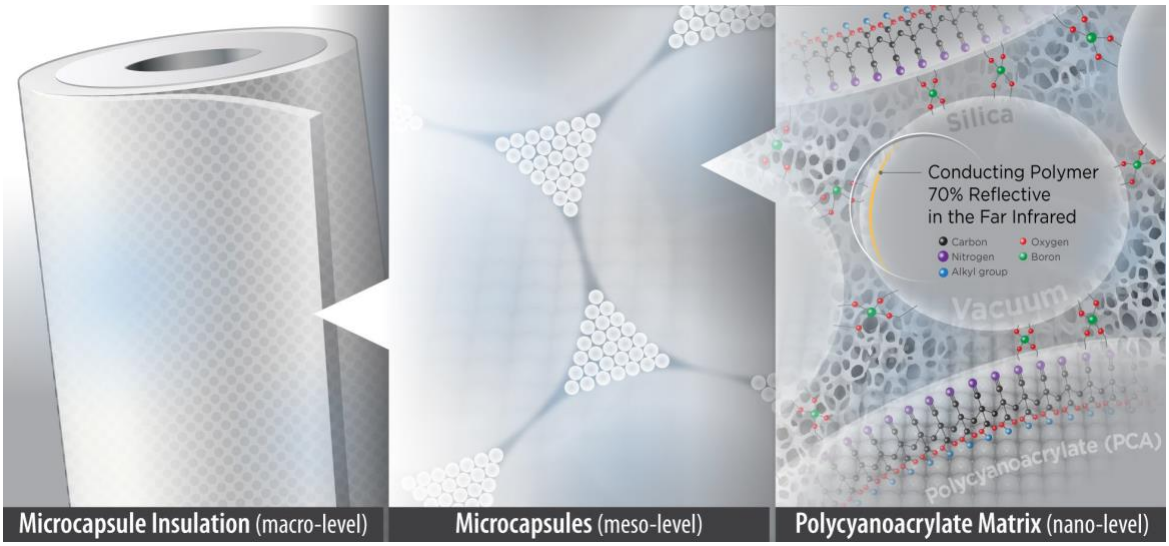

Figure 1. Pictorial of our polymer-crosslinked nanoporous lightweight (<0.1g/cc) solid superinsulation with >70% of the volume being vacuum-held in rigid evacuated microcapsules (MCs). An open-pore polymer matrix may be used to decrease the thermal conductivity of the air outside the MCs, and a conducting polymer coating should result in MC insulation with <0.002 W/m-K apparent/effective thermal conductivity.

Overall, NREL has demonstrated the ability to use solution and vapor processes to crosslink glass microcapsules (MCs), to coat them with conducting polymers that should reduce radiative transport, and to form strengthened porous but solid hydrophobic insulating materials with the desired mechanical properties. For this TCF project, NREL and Insula will need to go well beyond our initial demonstration efforts to integrate multiple approaches to reducing thermal conductivity while balancing the impact of these approaches to form solid advanced superinsulation with the desired mechanical, water interaction, flammability, and environmental impact properties needed for specific applications. Thus, this TCF effort will begin with a set of material selection and optimization activities that:

1. Reduce the thermal conductivity of the base materials (i.e., evacuated microcapsules) by decreasing the true density and removing air conduction inside the capsules.

2. Reduce the effective thermal conductivity of the air-filled pores on the outside of the MCs by increase the packing density of the microcapsules (decreasing the volume with air) and creating "nanocavities" in these spaces where the Knudsen effect can reduce air conduction by 50% to 75%.

3. Balance crosslinking and other strengthening processes with thermal conductivity.
    a. Designs to reduce TC while maintaining overall strength.
4. Select and optimize materials (e.g., conducting polymers, opacifiers, etc) to eliminate radiative transport (ensure processes are compatible with crosslinking).
5. Investigate and select other novel approaches to reduce thermal conductivity between MCs.
    a. Icephobic geometries (critical for cryogenic applications) and approaches to reduce condensed water thermal shorting and reduce contact area via surface roughening. Optimize surface properties based on impacts of hydrophilic and hydrophobic surface structures and chemistry.

One of the major goals of this effort is to provide samples for external testing and validation. At the outset, this suggests that our goal must include developing materials synthesis and processing commensurate with constructing samples that are approximately 12" x 12" x 1". This is a substantial leap from our present

~3" diameter by ¼" thick samples made for the initial demonstration effort. This will require that we scale- up several materials synthesis and processes including:

1. MC selection and/or sorting. At the very least, we will need to increase the packing density of MCs in order to optimize desired thermal conductivity properties through methods which will include sieve-based sorting to obtain properly sized MCs, amongst other approaches. These efforts will include a variety of efforts to determine the optimal balance between thermal conductivity, material cost minimization and manufacturing process simplicity. Initially, we will aim to establish high confidence baselines for readily achievable pack density of MCs without significant sieving or pre-selection activities as well as to determine appropriate theoretical targets for the relationship between pack

density and thermal conductivity, which in turn will guide experimental efforts to explore bimodal and/or multimodal mixing methods for different sized MCs. Insofar as it is appropriate, we will also continue discussions with MC manufacturers who have markets for different sizes and appear willing to sell any unused portions to other customers.

2. Increasing the packing density for MCs will require combining properly sized MCs in appropriate amounts and controlled mixing. This will require substantial scale-up of the mixing process.
3. Environmentally controlled atmospheric reactors. The vapor phase processing will require the design and development of a reactor chamber with the built-in control needed for precise materials delivery and reaction control to form 12" x 12" x 1" samples. The vapor phase processing already identified is amenable to atmospheric pressures and virtually any size. So the goal will be to develop processing expertise that is applicable to inexpensive scaleup of larger sizes and more importantly continuous rapid throughput processing that will occur in pilot plant development. We anticipate that some scaleup evaluations will be done by Insula during this project and NREL will support these activities.
4. MC preparation will be needed to form mixtures of MCs in the correct template(s) with other materials, while maintaining the desired properties, so that vapor phase processing can be performed.
5. Procure and install scale-up and/or testing equipment (suitable to process 2-3 L of MC materials and 12" x 12" areas).
    a. Sieving equipment and/or third-party industrial-scale sieving services in order to sort MCs according to required sizes (for mixing and packing density optimization). Part of this evaluation will be identifying which sieve products can be combined to achieve the desired packing densities, and to the extent appropriate, which will be left over for sale by MC vendors to other customers.
    b. Upgrade evacuation system to accommodate 1-5 L of MCs
    c. 3 to 5 L dual rod double movement mixer or equivalent, with vacuum and perhaps isotropic pressure capabilities
    d. Additional environmental control chamber for sample processing

Table: Project Milestones:

Task	Milestone/Deliverable Description	Type	Responsible Party	Due Date
1.2	Design, construct, and test system to make 8” to 12” diameter samples. Make initial crosslinked MC sample at least 8” diameter and measure thermal conductivity.	Milestone 1.2.1	NREL	Q1
1.1	Demonstrate higher packing density via multimodal mixing (e.g. 60-70%+)	Milestone 2.1.1	NREL	Q2
1.2	Down select a crosslinking method and MCI formulation optimum vis-à-vis cost (e.g., <$0.08/R/ft2) and required physical properties (e.g., compressive strength >5 psi, flexural strength >10 psi) for providing samples for testing.	Milestone 2.2.2	NREL	Q2
1.3	Identify potential processes (e.g., the use of fumed silica or other highly porous materials) that can form nanopores between the evacuated MCs and adjust the surface energy (e.g., integration of materials/processes that change the hydrophobicity of the MCs to between 50° and 110° water contact angle).	Milestone 3.3.1	NREL	Q3
1.3	Demonstrate conducting polymer and/or opacifier integration with MCI and greater than 0.001 W/m-K ATC reduction	Milestone 4.3.1	NREL	Q4
2.5	Formal TEA model that demonstrates MCI manufacturing costs less than $1/sf with vapor phase processing	Deliverable 5.5.1	Insula	Q5
1.3	Produce 12” x 12” x 1” sample with R-factor >8/in	Milestone 6.3.1	NREL	Q6
2.5	Detail energy modeling results that identify potential technical improvements and markets where MCI has the highest impact	Milestone 7.5.1	NREL	Q7
2.6	Document industrial collaborations that identify MCI cryogenic and/or VIP superinsulation first adopter markets,	Deliverable 8.6.1	Insula	Q8

TASK DESCRIPTIONS:

Task 1. MCI Technical Development

Subtask 1.1. MC Preparation and High-Density Packing

Lead: NREL and Insula

Via R&D and collaborators, NREL will identify appropriate and best practices to increase vacuum capsule and other MCI component packing density to increase from present capabilities of an estimated ~50% to between 70% and 95%. To achieve this goal, we will develop capabilities to sort hollow capsules by specific sizes, evacuate the capsules, identify and integrate ultra-low thermal conductivity materials between and/or around the vacuum capsules, mix the

components to form a composite with high packing density and ultimately integrate the resultant mixture into desired forms or structures.

1.1.1. Capsule size selection.

Presently, capsules are typically commercially procured and have a relatively broad size distribution due to the manufacturing process used. These materials can be further sorted to provide more discrete size ranges that are typically needed for higher packing densities. The initial approach for this project will be to use commercial sieving equipment to provide the desired size ranges. Different sizes will be combined to achieved desired packing densities with the goal of identifying different sets of sizes so that more of the sieved material can be used. In addition, if specific sizes cannot be used, we will work with MC vendors to identify which ones can be used in other applications. Throughout these efforts, the NREL and Insula teams will be critically evaluating the trade-offs between thermal conductivity performance, material cost and manufacturing process complexity -- the NREL team will look to Insula in particular to shape research and development priorities based on signal from potential customers.

1.1.2. Evacuate capsules.

Presently, ~0.1 L of capsules can be evacuated in a batch process. NREL's degas system used to do this will be scaled up with increased vacuum capacity and a larger heater and capsule holder to evacuate 1 to 3 L of capsules in a single batch. The integrated mass spectrometer of the system will be reconfigured to provide useful information at higher degas volumes. Based on measured degas times and desired vacuum, we will be able to design a continuous feed and degas system that uses residual heat from cooling evacuated capsules to preheat unevacuated capsules to reduce energy requirements and identify optimum throughput to evacuate the microcapsules. We will also perform a set of experiments to confirm initial results that indicate that the thin glass does not allow atmospheric gas diffusion through at a rate sufficient to significantly impact thermal conductivity and insulation performance. We plan to conduct durability studies in which we evacuate the capsules, leave them for extended periods of time (1 month – 2 years), and then attempt to degas them again after the specified time period. For example, we may place MCs into our evacuation chamber that has an integrated mass spectrometer such that it will detect if gases are evacuated during the 2nd evacuation step after the specified time period. These experiments will start in Q1 of the project (or as soon as is feasible) such that the longer time periods can be assessed during the timeframe and scope of this TCF project.

1.1.3. Strengthening MCI and decoupling intercapsule point contacts.

Preliminary results indicated that addition of cellulose fibers (or equivalent) and boron chemical treatments can be used to increase the MCI strength. In addition, fumed silica or equivalent can be used between the capsules to reduce thermal conductivity, primarily by inducing the Knudsen effect. NREL will investigate and develop improved and/or alternative materials to increase strength while reducing thermal conductivity.

1.1.4. Component particle mixing and packing density.

Based on discussions with consultants, if available, we will identify and develop processes to increase capsule packing density while decreasing the effective thermal conductivity. This will involve developing and procurement of appropriate mixing and other packing equipment.

1.1.5. Integration of MCs into forms.

Based on the composite material properties after mixing and compaction, additional procedures will be developed to integrate the particulate composition into forms or other structures for crosslinking and integration with other components (e.g., formed insulation structures for fitting around cryogenic piping and tanks, OSB or paper for integration with structures).

Milestone 2.1.1: Demonstrate higher packing density via multimodal mixing (e.g. 60-70%, or as appropriate given thermal conductivity value / complexity trade-offs). This may require selection and procurement of a larger sieve system, mixer and other components needed to achieve 70% to 95% packing density, or alternatively working with an outsourced industrial-scale sieving third-party.

Task 1 Results:

A stop work order was issued by Insula, Inc (project partner) mid-October 2021 just after officially starting the project in late September 2021. Thus, only a small amount of funds were spent during the ~1-month period the project was active. During this period, project activities included kick-off and planning meetings, development and optimization of several key laboratory processing steps to construct superinsulation samples. Insula's request for work to be stopped was due to apprehensions about whether vacuum could be obtained and retained in the superinsulation material. Thus, the project was officially terminated October 2022

Subtask 1.2. MC Crosslinking

Lead: NREL and Insula

NREL will demonstrate effective crosslinking of all component particulates to obtain the appropriate required insulation properties (e.g., strength, packing density, thermal conductivity, and vapor transport) for samples up to 12” x 12” x 1” in size. We will consult frequently with Insula, technoeconomic modelers, IP counsel, and commercial consultants, if available, to ensure that the development is guided by cost, end-market, commercial manufacturing and IP considerations.

1.2.1. Vapor phase process development and optimization for larger samples.

Three dimensional environmental control (e.g., temperature, air composition, and air flow parameters) will be evaluated and specific parameters quantified that significantly impact to scale up of uniform processing of larger samples. Design of experiments will be performed to map out the different parameter space to provide insights into the design and implementation of temperature and flow control of the sample and sources, as well as other important environmental parameters. The goal is to demonstrate effective crosslinking of large MCI samples.

1.2.2 Optimization of cross-linking process in the presence of MCs and physical strengthening components.

Intrinsic to MCI performance is limiting the amount of higher thermally conducting strengthening materials while achieving the required strength needed for specific applications. Issues involving fiber type, physical characteristics of the fibers, and chemical augmentation processes will be evaluated based on overall impact to performance and costs. Additional work will be done to improve the flexibility, while still achieving substantive thermal properties at very cold temperatures (i.e., down to 77 K).

Milestone 1.2.1: Design, construct, and test system to make 8" to 12" diameter samples. Make initial crosslinked MC sample at least 8" diameter and measure thermal conductivity.

Milestone 2.2.2: Down select a crosslinking method and MCI formulation optimum vis-à-vis cost (e.g., <$0.08/R/ft^2) and required physical properties (e.g., compressive strength >5 psi, flexural strength >10 psi) for providing samples for testing.

Results:

CRADA was terminated before this task could be started (or completed).

Subtask 1.3. MCI Thermal Transport Optimization

Lead: NREL

Explore coatings and additives to improve the thermal and other properties of MCI. These may include IR reflective coatings (low-e) to reduce radiative heat transfer and/or hydrophobic coatings to render MCI water repellant while maintaining water vapor breathability. This will involve evaluating insulating properties as a function of amount of crosslinking, packing density via multimodal mixing, integration of low-e coatings, particle incorporation outside capsules (e.g. opacifiers), and integration of strengthening agents.

Milestone 3.3.1: Identify potential processes (e.g., the use of fumed silica or other highly porous materials) that can form nanopores between the evacuated MCs and adjust the surface energy (e.g., integration of materials/processes that change the hydrophobicity of the MCs to between 50° and 110° water contact angle).

Milestone 4.3.1: Demonstrate conducting polymer and/or opacifier integration with MCI and greater than 0.001 W/m-K ATC reduction.

Milestone 6.3.1: Demonstrate 12” x 12” x 1” MCI w/ ATC <0.017 W/m-K (R8/in) measured w/ heat flow meter or equivalent.

Results: The CRADA was terminated before this task could be started (or completed).

Subtask 1.4. Thermal Conductivity and Material Property Performance Measurements

Lead: NREL and Insula

1.4.1. Instron strength testing.

Identify and implement procedures for measuring compression, tensile, and flexural strength.

1.4.2. *Thermal conductivity measurements and models.*

Utilize a Cryostat 500 to evaluate thermal conductivity for optimized MCI samples. Implement identified procedures to enable the hot disk thermal conductivity measurement system to provide thermal conductivity measurements of MCI and component materials. Quantify accuracy and ability to provide rapid absolute and/or relative results to provide rapid feedback. Improve the MCI and component material thermal transport models to help ensure accuracy of measurement results and provide insights to improving R-value.

1.4.3. *Packing density.*

Identify and implement techniques (e.g., pycnometer, Zeta potential) to accurately determine packing density, pore size distribution, and/or the volume between the vacuum capsules.

1.4.4. *Air permeance and water vapor transport rate.*

Identify and implement techniques to quantify air/water permeance.

Milestone 9.4.1: Produce a 12” long MCI and complete initial strength (e.g., flexural between 10 psi and 75 psi), durability (e.g., vacuum, crosslinking, and other components projected to last 10 to 25 years), and breathability/wettability (e.g., water vapor permeance 0.03 to 100 Perms/in, and <<1% water absorption) tests demonstrating different physical properties for different applications.

Milestone 12.3.1: Produce 12” x 12” x 1” sample for external evaluation with R-factor >20/in.

Results:

The CRADA was terminated before this task could be started (or completed).

Task 2. MCI Business Development

The CRADA was terminated before this task could be started (or completed).

The Following Subtasks were omitted from the Executed Agreement: 2.1, 2.2, 2.3a, 2.4.

Subtask 2.5. Energy and Thermal Modeling with Technoeconomic Analysis including Manufacturing Cash Flow and Payback

Lead: NREL and Insula

2.5.1 Energy and thermal modeling.

NREL's team has substantial expertise with energy and thermal modeling in general, including for residential and commercial buildings, windows, and even for transportation via the national vehicle climate control energy and fuel use analysis process, developed under prior DOE VTO programs. NRELalso have specific expertise for insulating materials modeling and will investigate contact point thermal resistance and shorting issues that are so critical to our MCI success. *NREL will use this expertise to analyze opportunities and potential impacts for cryogenic and superinsulation markets. NREL will augment this extensive tool set in thermal management with expertise in net-zero energy analysis to identify promising applications and quantify the advanced insulation's effect for different use cases.*

2.5.2. Technoeconomic analysis (TEA).

Technoeconomic work to estimate manufacturing costs at scale. Much of the technical research will involve estimated cost/benefit and optimization of insulation and structural properties vs. cost of manufacture. For example, cost/benefit analysis will be needed to optimize radiative transport technology and to identify hydrophobicity goals. Accordingly, knowing cost of manufacture will be essential to guide research direction. Finally, NREL TEA experts will provide input and feedback to Insula for commercial manufacturing analyses including manufacturing Insula, Inc. cash flow. Technical researchers will interact frequently with technoeconomic modelers.

Deliverable 5.5.1: Formal TEA model that demonstrates MCI manufacturing costs less than $1/sf with vapor phase processing.

Milestone 7.5.1: Detail thermal modeling results that identify potential technical improvements and markets where MCI has the highest impact.

Deliverable 11.5.1: Validated TEA and final technology-to-market plan.

Results:

The CRADA was terminated before this task could be started (or completed).

Subtask 2.6. Industrial Collaboration and Outreach: Business Development, Market Assessments, and Consultants

Lead: Insula Duration: M3-M36

2.6.1. Sample product demonstrations.

NREL will work with Insula and consultants, if available, to identify and provide MCI samples that can be used for business development, manufacturing planning, and market assessments, especially for early market adopters. The initial focus will be on samples for cryogenic and VIP applications. This may include samples formed for integration with cold piping and/or more rigid sheet insulation that can take the place in VIP applications.

2.6.2. MCI process and performance expertise.

NREL will work with Insula to answer product and manufacturing questions to Insula management team, and manufacturing and buildings consultants, if available. NREL will also assist Insula in designing and planning potential pilot plant manufacturing and market identification activities.

Deliverable 8.6.1: Document industrial collaborations that identify MCI cryogenic and/or VIP superinsulation first adopter markets, value proposition, and technology-to-market plan based on initial TEA.

Milestone 10.6.1: Identify initial licensing/manufacturing partners.

Results:

The CRADA was terminated before this task could be started (or completed).

Task 3. Project Management

Subtask 3.7: Project Management

Lead: NREL and Insula

This subtask will ensure that all project milestones and deliverables are completed on time. At the end, a technical report (i.e., CRADA Final Report) summarizing all results and learnings will be prepared by NREL and Insula.

Milestone: Execute every 2nd week team meetings to discuss progress.

Deliverable 14.7.1: Complete and submit CRADA Final Report in accordance with Article X.

This report serves to meet the requirement for the CRADA Final Report with preparation and submission in accordance with the agreement's Article X.

Task 4. Other Work

Lead: NREL

Other work at the direction of the Participant, consistent with the scope and subject to the availability of funding.

The CRADA was terminated before this task could be started (or completed).

References:

None

Subject Inventions Listing:

None

ROI #:

None