

# **Environment and Natural Resources Trust Fund**

# 2026 Request for Proposal

# **General Information**

Proposal ID: 2026-346

Proposal Title: Recyclable/Reversible Thermosets for Reducing Microplastics in Minnesota

# **Project Manager Information**

Name: William Tai Yin Tze Organization: U of MN - College of Food, Agricultural and Natural Resource Sciences Office Telephone: (612) 624-2383

# Project Basic Information

Email: wtze@umn.edu

**Project Summary:** We will reduce microplastics in Minnesota by creating a heat-hardened (thermoset) polymer which is not easy to wear down, yet reversible in structure upon stimuli to allow reuse and recycling.

ENRTF Funds Requested: \$499,000

Proposed Project Completion: June 30, 2029

LCCMR Funding Category: Land (F)

# **Project Location**

- What is the best scale for describing where your work will take place? Region(s): Metro
- What is the best scale to describe the area impacted by your work? Statewide

#### When will the work impact occur?

In the Future

# Narrative

#### Describe the opportunity or problem your proposal seeks to address. Include any relevant background information.

Microplastics are accumulating worldwide including the water and land ecosystems of Minnesota. One significant source of microplastics is their breakdown from larger plastics during use or when left degraded in the environment. Even plastics that are designed to be compostable releases microplastics if they are left to weather over time in an incompatible ecosystem. Obviously, regulated disposal practices and consumer awareness (reduce, reuse, recycle) are key factors in reducing microplastics pollution. From the product design perspective, materials that are not easily worn down to microplastics could be an upstream strategy to prevent such pollution. Thermoset, or polymers that are heat-cured into a crosslinked rigid network, are one such durable material. However, the favored durability feature is also the limitation for their recycling. Unlike thermoplastics such as PET bottles, thermoset polymers like epoxy cannot be melted or reshaped when heated, making them difficult to recycle. Yet, thermoset polymers have their place in certain end-use applications owing to their strength and long-lasting benefits. Their recycling does matter to keep them out of landfills and to reclaim high value fibers in the case of fiber-reinforced thermosets. It is thus worthwhile investing efforts on studying reversible thermosets to allow re-processing and recycling.

# What is your proposed solution to the problem or opportunity discussed above? Introduce us to the work you are seeking funding to do. You will be asked to expand on this proposed solution in Activities & Milestones.

Using a commonly used thermoset such as epoxy as a benchmark, we will incorporate into the formulation chemical species that will form reversible, covalent crosslinks in the rigid network of the cured thermoset. When triggered by heat, these dynamic linkages will break up into their constituent reactive species. Consequently, the network integrity will be disrupted, macroscopic flow facilitated, and re-processing enabled during which the reactive species will pair with adjacent segments to re-form covalent network. This adaptive reversibility endows the (modified) thermoset recyclability.

We will harness the reversible Diels-Alder reaction between furan and maleimide to realize crosslinks and un-crosslinks in the thermoset network. As more dynamic crosslinks are enabled, the ease of recycling increases but the cured thermoset would become brittle. We will use an oligomeric bismaleimide and nanocellulose to tune mechanical performance for mitigating the trade-off. Chemical recycling will also be examined to recover the formulated Diels-Alder resin and pre-blended fiber. Additionally, we will examine the thermoset in lab settings for UV, thermal, fresh and salt water durability. Overall, the optimized formulation will serve as a resin for thermo-reversible thermoset products. It could also be used as a recyclability enhancer by blending to cured thermoset of conventional (irreversible) formulations.

# What are the specific project outcomes as they relate to the public purpose of protection, conservation, preservation, and enhancement of the state's natural resources?

This project will bring the outcome of exemplifying how thermoset polymer, which is designed to last, can be made recyclable. The project uses as an example epoxy thermoset which is widely used so that the finding's impacts can be maximized. Yet, the obtained result will serve as a model for reducing waste of other thermoset in Minnesota's environment. A consequent benefit is the reduction of microplastics pollution. From sustainability perspective, the recyclable thermoset affords material circularity. This reversible formulation, if successful and further developed, will help recycle existing epoxy structures such as wind turbine blades in the long run.

# Activities and Milestones

# Activity 1: Formulations of reversible Diels-Alder (DS) thermoset and their characterization

Activity Budget: \$170,452

#### **Activity Description:**

The objective here is to formulate and evaluate a reversible epoxy thermoset. Existing protocols for epoxy network crosslinked with reversible Diels-Alder bonds will be refined and adopted for this study. One modification will be the use of oligomeric bismaleimide (alternative or supplementary to monomer bismaleimide). The synthesized resin will be analyzed using nuclear magnetic resonance spectroscopy or/and mid-infrared spectroscopy to verify its chemical structure. A portion of the resin will be added with nanocellulose for mechanical reinforcement. The nanocellulose will be freeze-dried prior to mixing with the liquid resin. The resin will be cured via thermal molding. Bar specimens will be tested for thermo-reversibility as the specimen stiffness is monitored as a function of temperature in a thermomechanical analyzer (TMA). Some of the cured thermoset will be milled in liquid nitrogen to prepare powder for differential scanning calorimetry (DSC) study which measures the mobility of the cured thermoset in response to (bond dissociation and association) temperature. Test specimens will also molded for mechanical, UV, thermal, and liquid degradation tests.

The specific outcome is to acquire know-how in formulating/synthesizing recyclable thermoset resin (Activity 2), which can be used in lieu of conventional thermoset or promoting its recyclability (Activity 3).

#### **Activity Milestones:**

Description	Approximate Completion Date
Synthesis and curing conditions optimized for Diels-Alder thermoset	March 31, 2027
Formulations characterized for reversible behavior	June 30, 2027
Diels-Alder thermoset characterized for mechanical performance and durability	June 30, 2027

# Activity 2: Recycling of cured Diels-Alder (DS) thermoset

#### Activity Budget: \$162,006

#### **Activity Description:**

The objective of this activity is to examine and demonstrate the mechanical and chemical recycling of the cured thermoset. The cured thermoset will be pulverized into powder and thermally consolidated to simulate mechanical recycling. The hot pressing parameters (pressure, time, and temperature) first need to be optimized to afford dissociative Diels-Alder bonds for re-crosslinking. Once the protocol is devised, we can repeat the milling-hot pressing cycles for the desired number of times. The recycled samples can then be analyzed using DSC and TMA (refer to Activity 1) to relate to bond reversibility, and tested in tension or/and bending mode for their strength and ductility. Particle consolidation will be examined using electron microscopy. For chemical recycling, selected organic solvents will be tested at various temperature. The dissolved resin ingredients and non-dissolved fiber (if applicable) will be separated using a rotary evaporator. The recovered quantity and the time taken will be used to quantify recovery efficiency.

The specific outcome here is a devised protocol for mechanical recycling of the formulated Diels-Alder thermoset. This knowledge is useful for effectively harnessing the thermo-reversibility of the thermoset. Through strength retention data, we will know when to replenish the recycled with fresh Diels-Alder resin.

#### **Activity Milestones:**

Description	Approximate Completion Date
Optimizing hot pressing parameters for consolidating powder made from cured thermoset	December 31, 2027
Mechanical recycling of DS thermoset and characterization of the recycled product	March 31, 2028
Chemical recycling of DS thermoset and re-polymerization of the recovered resin components	June 30, 2028

# Activity 3: Application of Diel-Alder thermoset to impart recyclability

#### Activity Budget: \$166,542

#### **Activity Description:**

This activity is aimed at addressing the mechanical recycling of the irreversibly cured thermoset. Two approaches will be tested -- adding solid cured Diels-Alder thermoset or fresh liquid resin. The ratio between the reversible (Diels-Alder) resin and irreversible thermoset will be varied. The resulting recycled products will be thermally and mechanically tested as described in Activity 2. The particle consolidation after recycling will also be examined using optical or/and electron microscopy.

The specific outcome for this activity is the information about what can be done, in the long run, pertaining to recycling of the existing, irreversibly cured structures when they are no longer in use.

#### **Activity Milestones:**

Description	Approximate Completion Date
Blending fresh DS liquid resin to cured conventional (irreversible) thermoset	December 31, 2028
Blending cured DS solid resin to cured conventional thermoset	June 30, 2029

# Long-Term Implementation and Funding

# Describe how the results will be implemented and how any ongoing effort will be funded. If not already addressed as part of the project, how will findings, results, and products developed be implemented after project completion? If additional work is needed, how will this work be funded?

After project completion, funds will be sought to examine the life cycle performance of the formulated product, and investigate their degradation in natural ecosystems. Potential funding agencies are EPA, DOE, and USDA. Results obtained will be used to further improve our formulations or protocol. When our work is significantly advanced, we will approach companies for incorporating the refined formulation to eventually bring the product to the market place.

# Other ENRTF Appropriations Awarded in the Last Six Years

Name	Appropriation	Amount Awarded
Eco-Friendly Plastics From Cloquet Pulp-Mill Lignin	M.L. 2021, First Special Session, Chp. 6, Art. 5, Sec. 2, Subd. 07b	\$193,000

# Project Manager and Organization Qualifications

#### Project Manager Name: William Tai Yin Tze

Job Title: Associate Professor

Provide description of the project manager's qualifications to manage the proposed project.

Experience with managing previous LCCMR project: ID Number 2020-018.

Organization: U of MN - College of Food, Agricultural and Natural Resource Sciences

#### **Organization Description:**

In the College of Food, Agricultural and Natural Resources Sciences (CFANS) at the University of Minnesota, we look at the bigger picture. When we envision a better tomorrow, it includes disease-resistant crops, products that protect our health, lakes free from invasive species, and so much more. We use science to find answers to Minnesota and the world's grand challenges and solve tomorrow's problems. Almost 93 percent of students who earn CFANS undergraduate degrees find jobs in their career field or enter graduate school within six months of graduation. The Department of Bioproducts and Biosystems Engineering, in CFANS, discovers and teaches solutions for the sustainable use of renewable resources and the enhancement of the environment. We discover innovative solutions to address challenges in the sustainable production and consumption of food, feed, fiber, materials, and chemicals by integrating engineering, science, technology, and management into all degree programs.

We have a public impact through community engagement and extension efforts. We develop and deliver high quality, regionally and nationally-recognized research-based programs to meet current and emerging needs of industry and communities. We also have a long-standing tradition of close partnerships with alumni, industry professionals, organizations, government agencies, donors, and community members.

# Budget Summary

Category / Name	Subcategory or Type	Description	Purpose	Gen. Ineli gible	% Bene fits	# FTE	Class ified Staff?	\$ Amount
Personnel								
Lead PI - 2 weeks summer salary annually		Direct all research and personnel			36.6%	0.12		\$24,729
Post doc researcher		Assist lead PI, conduct research on synthesis, chemical analysis, applications of liquid resin, and chemical recycling.			25.9%	3		\$244,530
Graduate Student		Conduct experiments and analysis on curing, reinforcement, durability, and mechanical recycling of thermoset; work with undergrad students, education			83.6%	3		\$174,275
Undergraduate students		set up and take down experiments			0%	0.3		\$10,218
							Sub Total	\$453,752
Contracts and Services								
lab services	Internal services or fees (uncommon)	chemical and other analysis, including spectroscopy, calorimetry, microscopy, mechanical testing, etc.				0		\$6,000
							Sub Total	\$6,000
Equipment, Tools, and Supplies								
	Equipment	non-capital equipment: Freeze dryer (\$5,000), grinder mill compatible with liquid nitrogen (\$3,500), and photochemical reactor (\$4,800)	To conduct the research proposed: 1) The freeze dryer is to dry nanocellulose without sticking for mixing with liquid resin; 2) the grinder mill is to pulverize cured samples without thermally modifying them to prepare for subsequent recycling; 3) the photochemical reactor is to induce ultra-violet energy to the cured samples to					\$13,300

Took and Supplies       lab supplies       lab supplies       chemicals, regents, lab both based reactor (2148), other glassware, vials, pipettes, filter paper, Parafinin, gloves, masks, protective eyewear, etc.       lab       lab       Sub Total       \$5,148         Capital Expenditures       Image: Capital Expenditures <th></th> <th></th> <th></th> <th>examine their UV durability or</th> <th></th> <th></th> <th></th>				examine their UV durability or			
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Total							\$499,000

# Classified Staff or Generally Ineligible Expenses

Category/Name	Subcategory or Type	Description	Justification Ineligible Expense or Classified Staff Request
Travel Outside	Conference	8 trips @ \$2600: 600 air, 900	this new research has no other funders right now - all student personnel to at least one
Minnesota	Registration	registration/membership, 250 per	conference
	Miles/Meals/Lodging	diem, 100 ground, 750 3 nights	
		lodging	

# Non ENRTF Funds

Category	Specific Source	Use	Status	Amount
State				
			State Sub	-
			Total	
Non-State				
			Non State	-
			Sub Total	
			Funds	-
			Total	

Total Project Cost: \$499,000

This amount accurately reflects total project cost?

Yes

# Attachments

#### **Required Attachments**

*Visual Component* File: da0811b7-cb8.pdf

#### Alternate Text for Visual Component

The reversibly crosslinked thermoset allows both mechanical and chemical recycling. It would also make conventional thermoset to be able to recycle. The impact is thus reduced thermoset waste and reduced risks of microplastics pollution in Minnesota....

#### Supplemental Attachments

#### Capital Project Questionnaire, Budget Supplements, Support Letter, Photos, Media, Other

Title	File
UMN board resolution/authorization	e774aa5a-8fc.pdf
Background Check Certification Form	<u>3f6ce651-cbc.pdf</u>
Audit	ff41f097-f87.pdf

# Administrative Use

Does your project include restoration or acquisition of land rights?

No

Do you understand that travel expenses are only approved if they follow the "Commissioner's Plan" promulgated by the Commissioner of Management of Budget or, for University of Minnesota projects, the University of Minnesota plan?

Yes, I understand the UMN Policy on travel applies.

Does your project have potential for royalties, copyrights, patents, sale of products and assets, or revenue generation?

Yes

Do you understand and acknowledge IP and revenue-return and sharing requirements in 116P.10?

Yes

Do you wish to request reinvestment of any revenues into your project instead of returning revenue to the ENRTF? No

Does your project include original, hypothesis-driven research? Yes

Does the organization have a fiscal agent for this project?

No

Does your project include the pre-design, design, construction, or renovation of a building, trail, campground, or other fixed capital asset costing \$10,000 or more or large-scale stream or wetland restoration?

Do you propose using an appropriation from the Environment and Natural Resources Trust Fund to conduct a project that provides children's services (as defined in Minnesota Statutes section 299C.61 Subd.7 as "the provision of care,

#### treatment, education, training, instruction, or recreation to children")?

No

Provide the name(s) and organization(s) of additional individuals assisting in the completion of this proposal:

Wendy Moylan. University of Minnesota

Do you understand that a named service contract does not constitute a funder-designated subrecipient or approval of a sole-source contract? In other words, a service contract entity is only approved if it has been selected according to the contracting rules identified in state law and policy for organizations that receive ENRTF funds through direct appropriations, or in the DNR's reimbursement manual for non-state organizations. These rules may include competitive bidding and prevailing wage requirements

N/A