



# Environment and Natural Resources Trust Fund

2022 Request for Proposal

## General Information

**Proposal ID:** 2022-189

**Proposal Title:** Processing biodegradable plastics from Cloquet pulp mill lignin

## Project Manager Information

**Name:** Simo Sarkanen

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

**Office Telephone:** (612) 624-6227

**Email:** sarka001@umn.edu

## Project Basic Information

**Project Summary:** Pollution from conventional plastics will be reduced by creating biodegradable replacements from pulp-mill byproduct lignins. Successful compounding and injection-molding conditions for these biodegradable plastics will attract industrial interest.

**Funds Requested:** \$198,000

**Proposed Project Completion:** June 30 2024

**LCCMR Funding Category:** Small Projects (H)

**Secondary Category:** Air Quality, Climate Change, and Renewable Energy (E)

## 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?**

Region(s): Central, NE, NW,

**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.**

In 2005, it was estimated that 1.3 billion tons of wood and plant biomass could be harvested from U.S. forest and agricultural lands annually, without encroaching on food production. Around 65–88% of these forest residuals and agricultural residues consist of cellulose fibrils (like cotton fibers) and various hemicelluloses in differing proportions. Both are composed of sugars (like glucose and xylose) that can be converted into organic chemicals, renewable fuels and plastics. Unfortunately, profitability has not yet been achieved. However, about 12–35% of structural plant materials and wood consist of lignins, namely, aromatic biopolymers that contain no sugars. So far, lignins have earned little value because of their customary use as recovery-boiler fuels in pulp mills. We will retrieve these losses by converting byproduct lignin from the Cloquet Sappi mill into biodegradable plastics. In Cloquet, aspen wood chips are converted by the kraft process into cellulosic pulp and byproduct kraft lignin. At 90% levels, surplus aspen kraft lignin, formed during increased pulp production, will be transformed into totally biodegradable plastics with strengths similar to polystyrene. We will show how these aspen kraft-lignin formulations are compounded and injection-molded into plastic items by processes that reflect current industrial practice.

### **What is your proposed solution to the problem or opportunity discussed above? i.e. What are you seeking funding to do? You will be asked to expand on this in Activities and Milestones.**

- [1] From the lignin that makes up 25% of the trunks and limbs of northern Minnesota aspen, we will create blends for completely biodegradable plastics that can be compounded and injection-molded to make consumer items that surpass polystyrene in properties. Starting materials will be the byproduct kraft lignins in the “black liquor” formed when aspen wood chips are pulped in the Sappi mill (Cloquet) to produce cellulosic fibers for making paper.
- [2] The aspen kraft lignin will be purified and a portion chemically methylated. Both preparations will be characterized according to their molecular weight distributions, thermal behavior, chemical structure, and molecular organization.
- [3] We will develop functional aspen kraft lignin blends by adapting our previously successful solution-cast compositions for Southern pine kraft lignin.
- [4] Viable conditions will first be identified for compounding and injection-molding methylated aspen kraft lignin blends. Then increasing proportions of unmethylated aspen kraft lignin will be progressively introduced into the formulations, and the compounding/injection-molding conditions will be responsively adjusted. The resulting relationships between composition and machine parameters will reveal the most efficient route to optimum formulations for injection-molded 90% aspen kraft lignin-based biodegradable plastics. Total biodegradability will also include the non-lignin blend components.

### **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?**

Biodegradable plastics with high (~90%) lignin contents will facilitate profitable biomass conversion into platform chemicals and renewable biofuels. The production costs of biodegradable aspen kraft lignin plastics are estimated to remain well below half of the polystyrene selling price. They will contribute to carbon capture and storage in the biosphere, thereby helping to reduce greenhouse gases. Unlike polystyrene, which causes pollution in almost all bodies of water, lignin plastics will not accumulate in the environment after disposal. The impending vista will promote conservation and enhancement of Minnesota’s renewable natural resources. Growth in green jobs and production facilities is expected.

## Activities and Milestones

### Activity 1: Formulations for biodegradable aspen kraft lignin plastics

**Activity Budget:** \$98,000

**Activity Description:**

Aspen kraft lignin will be isolated by acidifying kraft black liquor from the Sappi mill in Cloquet. It will be thoroughly washed with water and air-dried. We will develop fully biodegradable aspen kraft lignin plastics formulations with minimal modification. However, methylation will be carried out to create formulations suitable for determining initial injection-molding conditions (in Activity 2). Before and after methylation, the purified aspen kraft lignin will be characterized in regard to its molecular weight distributions (size-exclusion chromatography), glass-transition temperature (differential scanning calorimetry), chemical structure (nuclear magnetic resonance, NMR), and molecular organization (X-ray powder diffraction). Methylated and unmethylated aspen kraft lignin will be solution-cast into plastic test pieces on their own and with commercially available biodegradable blend components at levels below 10%. These plastics will be characterized with respect to tensile strength (Instron), thermal behavior (differential scanning calorimetry) and molecular organization (X-ray powder diffraction). The most promising formulations in terms of their mechanical properties will be candidates for compounding and injection-molding studies, as outlined in Activity 2.

**Activity Milestones:**

Description	Completion Date
Methylated and unmethylated aspen kraft lignin purified for lignin plastics	September 30 2022
Strengths of solution-cast unmethylated aspen kraft lignin plastics	March 31 2023
Characterization of aspen kraft lignins	June 30 2023
Strengths of solution-cast methylated aspen kraft lignin plastics	September 30 2023

### Activity 2: Compounding and injection-molding biodegradable aspen kraft lignin plastics

**Activity Budget:** \$100,000

**Activity Description:**

Injection molding is the most versatile plastics manufacturing process. A wide variety of parts, ranging in size and shape, can be produced on an industrial scale. The goal of Activity 1 is to identify the most promising biodegradable aspen kraft lignin-based plastics formulations by solution casting. Previous studies with softwood kraft lignin suggest that methylation of aspen kraft lignin may be necessary to achieve sufficient flow behavior initially for compounding and injection molding trials. Thus, our attention will first focus on methylated aspen kraft lignin formulations. These will be mixed to create homogeneous thermoplastic blends using an Xplore Microcompounder (5 mL size) at the Polymer Characterization Facility, University of Minnesota. The resulting materials will be injection-molded with a Haake Micro Jet II unit to produce pieces for mechanical testing (Instron). Then studies will be extended to the unmethylated kraft lignin. Varying proportions of unmethylated and methylated aspen kraft lignin will be used for compounding and injection-molding studies. The responsiveness of compounding and injection-molding parameters to variable blend formulations represents a high-throughput engineering problem in mechanical-property optimization of the resulting plastics. In this regard, processing conditions will be empirically optimized for selected blend compositions according to the Taguchi protocol.

**Activity Milestones:**

Description	Completion Date
Compounding parameters for methylated aspen kraft lignin plastics formulations	December 31 2023
Injection-molding aspen kraft lignin plastics	March 31 2024
Mechanical properties of injection-molded aspen kraft lignin plastics	June 30 2024



## Project Partners and Collaborators

Name	Organization	Role	Receiving Funds
Tom Radovich	Sappi North America (in Cloquet)	Mr. Radovich will supply aspen black liquor from which aspen kraft lignin will be isolated. Aspen kraft lignin is the starting material for producing biodegradable plastics.	No

## 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 be funded?**

As soon as promising results are secured from this LCCMR project, we will contact Sappi mill management in Cloquet to explore possible collaboration in bringing these biodegradable aspen kraft lignin plastics into the marketplace. Other companies and/or entrepreneurs will also be approached. At the same time, funds will be sought (from DOE and/or USDA) to extend studies to other kraft lignin sources such as those from “LignoBoost” purification. The optimization of formulations for compounding and injection-molding biodegradable plastics containing very high kraft lignin levels will be complemented with the development of reinforced lignin-based nanocellulose composites, for which funding will be sought elsewhere.

## Project Manager and Organization Qualifications

**Project Manager Name:** Simo Sarkanen

**Job Title:** Professor

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

While a faculty member at the University of Minnesota, Simo Sarkanen has overturned a 60-year-old idea about lignin structure that has blocked progress in some important areas of lignin research. Hence, his group is the only one so far that has created useful plastics containing over 90% levels of softwood kraft lignin with mechanical properties surpassing polystyrene. His group has also identified the first functional lignin-degrading enzyme. He and his coworkers are uniquely qualified to formulate fully biodegradable plastic test pieces by injection-molding blends of aspen kraft lignin produced by the Sappi mill in Cloquet.

### Recent Patents

Compositions Including Lignin and Methods for Making the Same: Chen, Y.-r.; Sarkanen, S. 2020. U.S. National Stage Patent Application Number 16/982,818, filed on September 21.

Compositions Including Lignin: Chen, Y.-r.; Sarkanen, S.; Wang, Y.-Y. U.S. Patent 2018, No. 10,119,027.

Lignin Degrading Methods and Compositions: Chen, Y.-r.; Sarkanen, S.; Wang, Y.-Y. U.S. Patent 2017, No. 9,796,993.

### Some Recent Publications

Chen, Y.-r., Sarkanen, S. Biodegradable lignin-based plastics. In (M. Dusselier, J.-P. Lange, eds.) Biodegradable Polymers in the Circular Plastics Economy; Wiley-VCH Verlag, Weinheim, Germany, 2021 (36 pages).

Chen, Y.-r., Sarkanen, S., Wang, Y.-Y. Lignin-only polymeric materials based on unmethylated unfractionated kraft and ball-milled lignins surpass polyethylene and polystyrene in tensile strength. *Molecules* 2019, 24, 4611 (15 pages).

Wang, Y.-Y., Chen, Y.-r., Sarkanen, S., Blend configuration in functional polymeric materials with a high lignin content. *Faraday Discuss.* 2017, 202, 43-59.

Sarkanen, S.; Chen, Y.-r.; Wang, Y.-Y., Journey to polymeric materials composed exclusively of simple lignin derivatives. *ACS Sustainable Chem. Eng.* 2016, 4, 5223-5229.

Wang, Y.-Y., Chen, Y.-r., Sarkanen, S., Path to plastics composed of ligninsulphonates (lignosulfonates). Green Chemistry 2015, 17, 5069-5078.

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

**Organization Description:**

The resources in the Department of Bioproducts & Biosystems Engineering along with those in the University of Minnesota Characterization Facility are sufficient to carry out the proposed research. The University of Minnesota Sponsored Projects Administration is the entity authorized by the Board of Regents to manage project agreements with LCCMR.

## Budget Summary

Category / Name	Subcategory or Type	Description	Purpose	Gen. Ineligible	% Benefits	# FTE	Classified Staff?	\$ Amount
<b>Personnel</b>								
postdoctoral researcher		characterization, compounding and injection-molding aspen kraft lignin-based plastics			26.7%	1.5		\$162,000
							<b>Sub Total</b>	<b>\$162,000</b>
<b>Contracts and Services</b>								
Arrow Laboratory Specialists	Professional or Technical Service Contract	maintenance and repair of centrifuges that will be routinely used for the project				0		\$4,000
Characterization Facility and NMR Center at the University of Minnesota	Internal services or fees (uncommon)	equipment usage fees for compounding (mixing) aspen kraft lignin plastics; facility usage fees for characterizing structure and molecular organization; these studies are essential for aspen kraft lignin-based plastics blend composition optimization purposes				0		\$12,000
							<b>Sub Total</b>	<b>\$16,000</b>
<b>Equipment, Tools, and Supplies</b>								
	Tools and Supplies	laboratory supplies: chemical reagents, nitrogen, solvents & laboratory consumables, etc.	producing and subsequent mechanical testing aspen kraft lignin-based plastics					\$20,000
							<b>Sub Total</b>	<b>\$20,000</b>
<b>Capital Expenditures</b>								
							<b>Sub Total</b>	-
<b>Acquisitions and Stewardship</b>								
							<b>Sub Total</b>	-

<b>Travel In Minnesota</b>								
							<b>Sub Total</b>	-
<b>Travel Outside Minnesota</b>								
							<b>Sub Total</b>	-
<b>Printing and Publication</b>								
							<b>Sub Total</b>	-
<b>Other Expenses</b>								
							<b>Sub Total</b>	-
							<b>Grand Total</b>	\$198,000



Classified Staff or Generally Ineligible Expenses

Category/Name	Subcategory or Type	Description	Justification Ineligible Expense or Classified Staff Request
---------------	---------------------	-------------	--

Non ENRTF Funds

Category	Specific Source	Use	Status	Amount
State				
			<b>State Sub Total</b>	-
Non-State				
			<b>Non State Sub Total</b>	-
			<b>Funds Total</b>	-

## Attachments

### Required Attachments

#### *Visual Component*

File: [8e6b8f00-b0f.pdf](#)

#### *Alternate Text for Visual Component*

Pollution from conventional plastics will be reduced by creating biodegradable replacements from pulp-mill byproduct lignins. Successful compounding and injection-molding conditions for these biodegradable plastics will attract industrial interest....

### Optional Attachments

#### *Support Letter or Other*

Title	File
Institutional Approval to Submit	<a href="#">b617b038-c7b.pdf</a>

## Administrative Use

**Does your project include restoration or acquisition of land rights?**

No

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

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? If so, describe here:**

Yes, Request funds to be transferred into a University of Minnesota account that supports continuing research in Project Manager's group.

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

Yes

**Does the organization have a fiscal agent for this project?**

Yes, Sponsored Projects Administration

# Transformation of Minnesota pulp mill byproduct lignins into biodegradable plastics reduces environmental pollution



Aspen often grows in nearly pure stands.  
Photo © University of Minnesota Extension



www.sappi.com

lignin  
kraft pulp  
for paper  
from aspen  
wood chips



www.xplore-together.com

Demonstration with lab  
compounder (mixer)

compounding and  
injection molding  
produce items from  
biodegradable  
lignin plastics for  
office, home & garden



www.creativemechanisms.com

Industrial goal: injection molding  
biodegradable lignin plastics

## Broad Range of Lignin Plastic Products



computer console; mainlinecomputer.com



automobile dashboard; kinlane.com



speaker enclosure; audioxpress.com



plastic chair, True Value

Less Pollution from Plastics made from Petrochemicals  
Waste Reduction and Better Environmental Stewardship  
Green Jobs and Production Facilities for Minnesota