Cooperative Research and Development Agreement (CRADA) Final Report

Report Date: 12/18/2024

Parties to the Agreement: VERDE Nanomaterials Inc. & Berkeley Lab

CRADA number: FP00015574

CRADA Title: Simple and Scalable Process for Nanocellulose Production from Residues and Waste

Responsible Technical Contact at Berkeley Lab: Melanie Sonsteng

Name and Email Address of POC at Partner Company(ies):
Danielle Uchimura Pascoli <dpascoli@verdenano.com>

Sponsoring DOE Program Office(s): AMMTO

LBNL Report Number: LBNL-2001615

OSTI Number:
[SPO to complete]

Joint Work Statement Funding Table showing DOE funding commitment:

DOE Funding to LBNL	$100,000
Participant Funding to LBNL	$0
Participant In-Kind Contribution Value	$100,000
Total of all Contributions	$200,000

Provide a list of publications, conference papers, or other public releases of results, developed under this CRADA:
(Publications must include journal name, volume, issue, Digital Object Identifier)

Not applicable.

Provide a detailed list of all subject inventions, to include patent applications, copyrights, and trademarks:
(Patents and patent applications are to include the title and inventor(s) names. When copyright is asserted, the Government license should be included on the cover page of the Final Report)

Not applicable.

Executive Summary of CRADA Work:
This project successfully developed an innovative, cost-effective, and scalable method for converting low-cost agricultural waste feedstocks into nanocellulose—a sustainable material with wide-ranging industrial applications. It addressed the critical challenges of high production costs and limited global supply, which have hindered the widespread adoption of nanocellulose in industry. Through this work, significant advancements were made in optimizing resource use, improving process efficiency, and reducing costs. Notable achievements included a 50% reduction in water usage, 30% lower chemical consumption, and 20% energy savings, all while maintaining high-quality product standards. The technical feasibility of the process was further validated at a 10L scale in collaboration with the Advanced Biofuels and Bioproducts Development Unit (ABPDU), demonstrating scalability and replicability.

Key challenges in reducing nanocellulose production costs were addressed by utilizing low-cost biomass feedstocks and implementing low-temperature conversion reactions, leading to lower capital and operating expenses. The project also mitigated financial risk by generating critical data on the feasibility, adaptability, and scalability of the conversion technology, paving the way for its commercial implementation. Public benefits include advancing the circular bioeconomy, reducing environmental impact, fostering job creation, and enabling a shift toward biobased materials as sustainable alternatives to fossil-based products. These outcomes align closely with national goals to reduce greenhouse gas emissions and promote sustainable technological innovation.

Summary of Research Results:
This project focused on converting undervalued agricultural residue feedstocks into nanocellulose via a simple process that employs mild reaction conditions and environmentally-friendly chemicals. The specific objectives of this project included:

1. Produce nanocellulose from low-cost agricultural waste feedstocks
2. Optimize the conversion process, with focus on reducing production costs
3. Characterize the nanocellulose produced
4. Perform scale-up experiments using large 10L reactors

The methods used in this project were based on those previously established by Dr. Pascoli during her doctoral studies. The conversion process to produce nanocellulose can be summarized into three main steps: pulping reaction, oxidation pretreatment, and mechanical fibrillation. For objectives 1 and 2, these steps were performed in lab bench scale using beakers for reactions and a waring blender for mechanical fibrillation (Figure 1). Objective 3 consisted of characterizing nanocellulose using SEM, XRD, FTIR, and TGA, which was possible through a user proposal at The Molecular Foundry at Lawrence Berkeley National Laboratory (LBNL). Finally, objective 4 was performed in collaboration with the Advanced Biofuels and Bioproducts Development Unit (ABPDU) at LBNL, where scientists helped test the first reaction of the process (i.e., pulping) in a large 10L reactor.

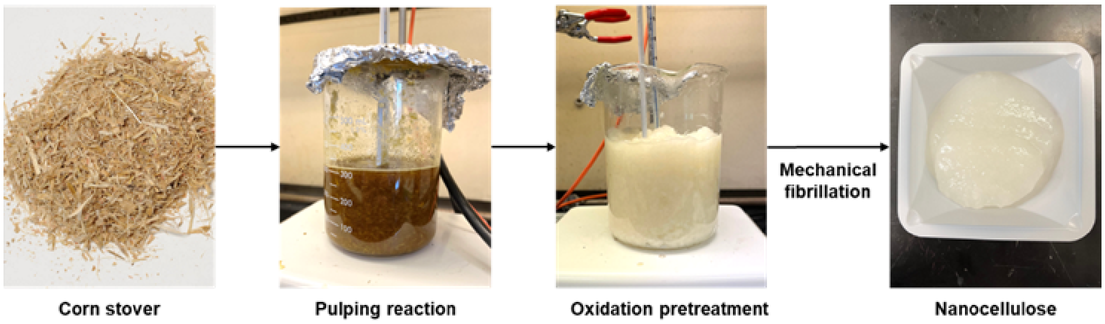

Figure 1. Summary of the process carried out to produce nanocellulose from corn stover feedstock.

During this project, we effectively optimized our manufacturing process to produce nanocellulose from corn stover biomass, enhancing cost savings in both resource and energy usage. We achieved this by 1) simplifying the mechanical fibrillation process by using a single equipment (waring blender) and eliminating the energy-intensive ultrasonicator that was previously used, and 2) reducing the amount of chemical used during pulping reaction and oxidation pretreatment. By doing these improvements, we were able to reduce water usage by 50% during sample preparation, reduce chemical usage by 30%, and achieve 20% energy saving compared to the original process, without compromising the quality of nanocellulose (Figure 2).

We also successfully scaled up the pulping reaction of corn stover using a 10L batch reactor in collaboration with ABPDU (Figure 3). This is a significant milestone of this project, as it proves that the lab-bench (300 mL) process can be easily scalable using common off-the-shelf equipment. The yield and chemical composition of the pulp obtained from the scale-up pulping reaction were also virtually equivalent to those from the lab scale, demonstrating our pulping process is robust and replicable at larger scale.

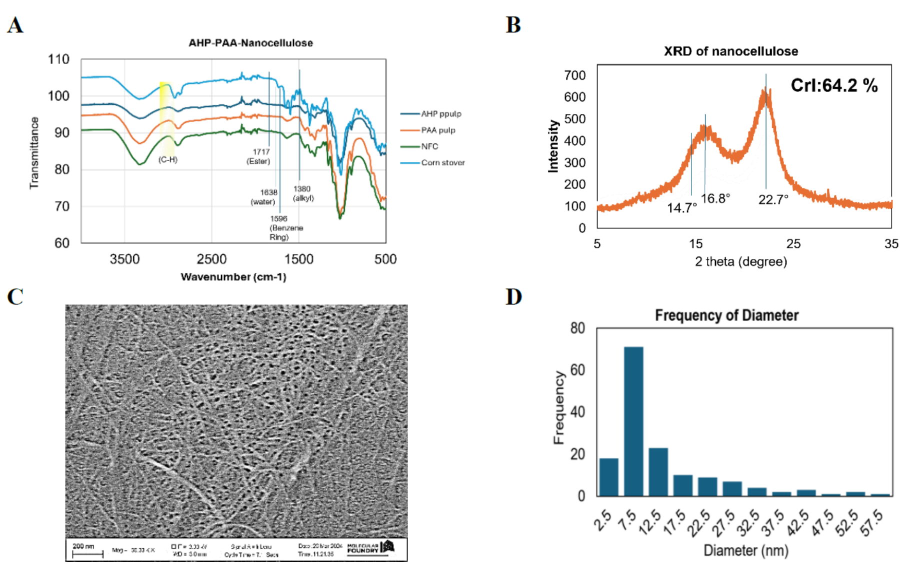

Figure 2. Characteristics of pulps and nanocellulose obtained from the optimized process: (A) surface chemistry of AHP pulp (produced after pulping), PAA pulp (produced after oxidation

pretreatment), and nanocellulose (produced after mechanical fibrillation), (B) crystallinity of nanocellulose by XRD, (C) morphology of nanocellulose by SEM, (D) frequency histogram of nanocellulose diameter.

Figure 3. Scale-up process at ABPDU: (A) ½ inch corn stover, (B) whole view of 10L reactor, (C) pulp washing after reaction, (D) resulting pulp.