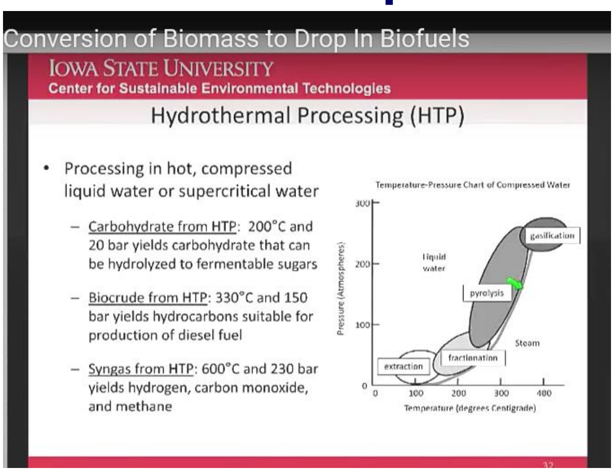
Liquefied Wood: What is it, and what can be done with it?

Cornelis F de Hoop, Associate Professor
Jiulong Xie, Research Assistant
Xingyan Huang, Research Assistant
Chun-yun Hse, Research Scientist, USDA

Conventional Liquefaction



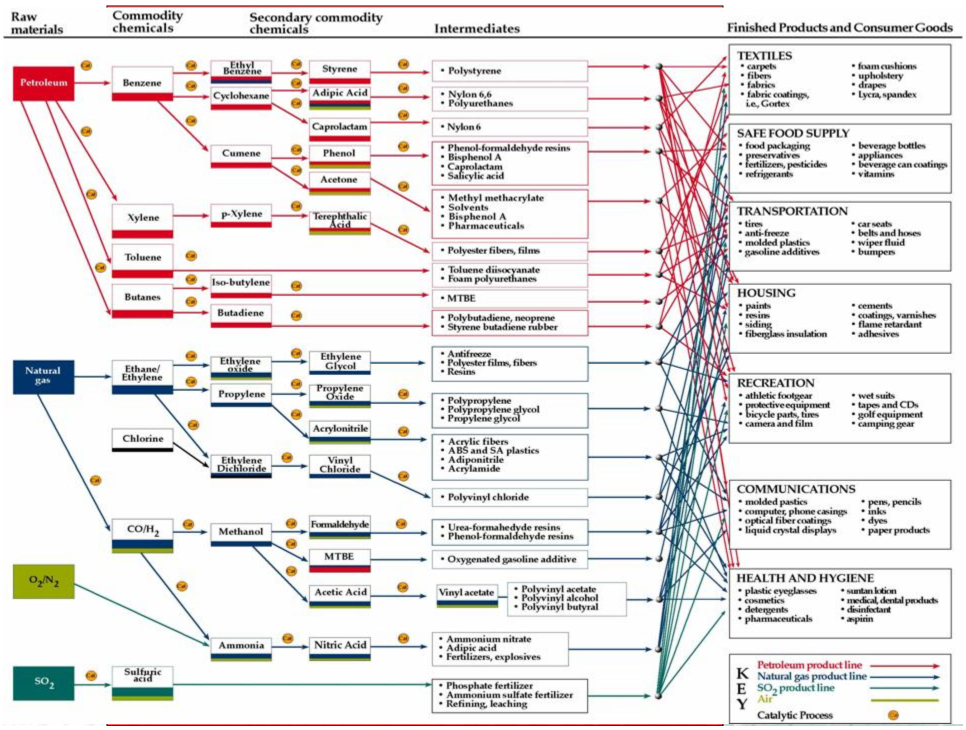
Credit: Robert Brown, CenUSA BioEnergy & Iowa State University

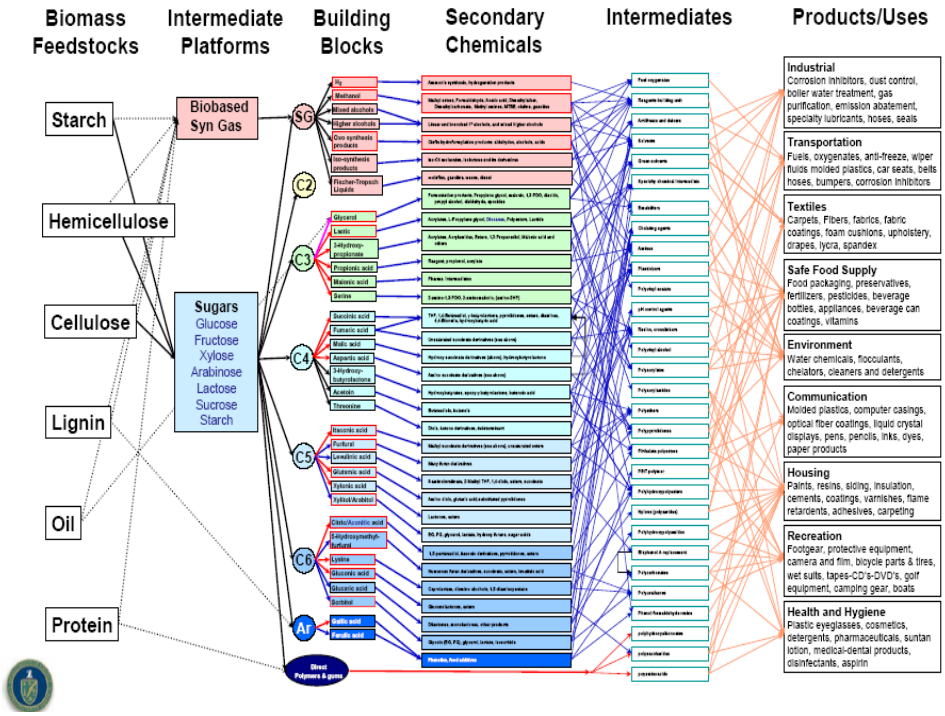
https://www.youtube.com/watch?v=Ua8She55qTc 2/14/2013

innovate . educate . improve lives
for the latest research-based information on just about anything, visit our Web site: www.lsuagcenter.com
LSU AgCenter Research & Extension

Major Problems

- **Energy input / Efficiency**
- **Do we really want to burn a valuable product?**





Heat Required

- **Energy efficiency = $$$$$**

innovate . educate . improve lives

for the latest research-based information on just about anything, visit our Web site: www.lsuagcenter.com

LSU AgCenter Research & Extension

Microwave

- Energy efficient
- More even heating
- More controlled heating
- Can still use solvents
  - Acids
  - NaOH, H_2O_2
  - Glycerin

Microwave

- Energy efficient
- Easier to develop a valuable product
  - Carbon nanofibers
  - Polyurethane Foam
- End of Service treated wood
  - Extract metals from CCA treated wood

3-step Converting CCA Treated Wood into High-value Added Products

Xingyan Huang

School of Renewable Natural Resources
Louisiana State University

Chromate Copper Arsenate (CCA)

- **C: Chromium (Cr) : a fixing agent**
- **C: Copper (Cu) : an effective fungicide**
- **A: Arsenic (As) : insecticide and fungicide**

(Kazi and Cooper, 2002)

A Mixture of HEAVY METALS

Fence

Deck

Pole

innovate . educate . improve lives
for the latest research-based information on just about anything, visit our Web site: www.lsuagcenter.com

BACKGROUND

- **Why should we recycle CCA treated wood?**

Diffusing heavy metals into environment and threatening human health (Kakitani et al., 2006)

Estimated spent CCA-treated wood in the U.S. would increase to 16×10^6 m^3 by 2020 (Copper 1993)

- **What the traditional ways to recycle?**

Particleboard

OSB

WPC

Fiberboard

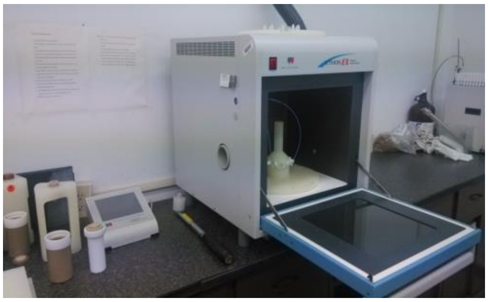
- **STEP 1 DETOXIFICATION**

Traditional Means of Detoxification

Biological, chemical, steam explosion, electro-dialytic

- **Previous recycling methods are too costly and/or too slow to be commercially viable**
- **Not really "green"**

Our Approach: Microwave-enhanced Detoxification



Microwaves can generate high temperatures and relatively high pressure rapidly, resulting in a higher liquefaction efficiency with a faster reaction rate and a shorter reaction time, compared to conventional heating.

Economical and Eco-friendly.

- **STEP 2 DELIGNIFICATION**

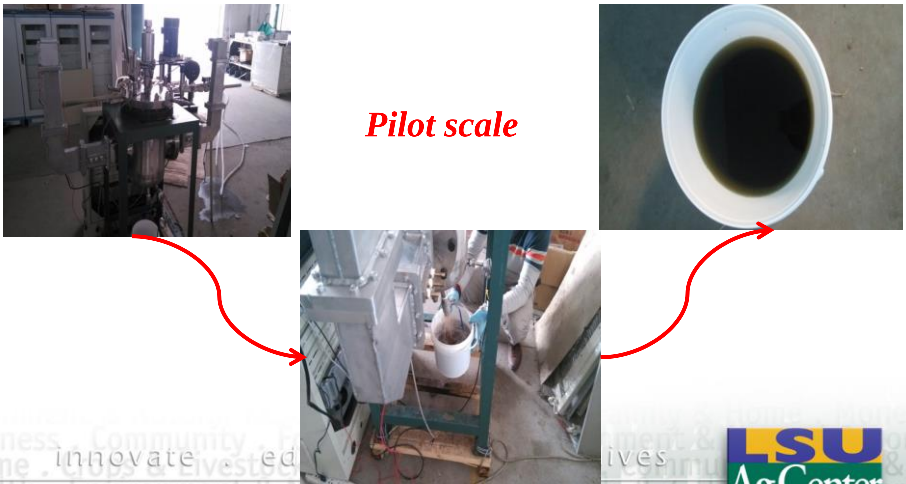
Traditional Means of Delignification

Biological, chemical, steam explosion, electro-dialytic

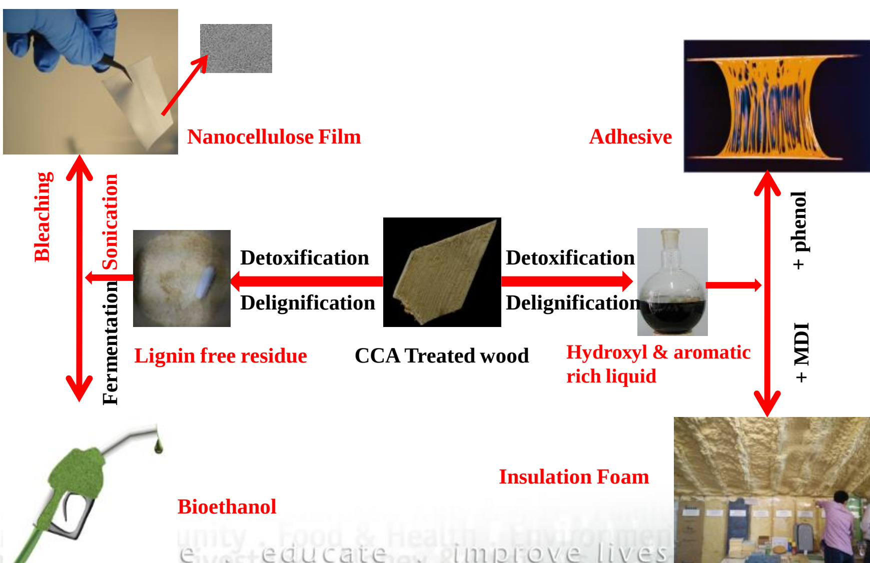
- **Acidic/alkaline pretreatment (Saha et al. 2007)**
- **Wet oxidative (Lissens et al. 2004)**
- **Liquid hot water (Perez et al. 2007)**
- **Team explosion (Sun et al. 2008)**
- **Ultrasound delignification**

- **Disadvantages:**
- **Cannot be in industrial scale**
- **Low efficiency**
- **Expensive**
- **environmentally-friendly**

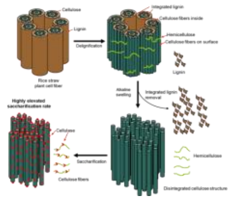
Our Approach: Microwave-assisted Delignification



- **STEP 3 CONVERTING**



- **REVIEW**



DETOXIFICATION **DELIGNIFICATION** **CONVERSION**

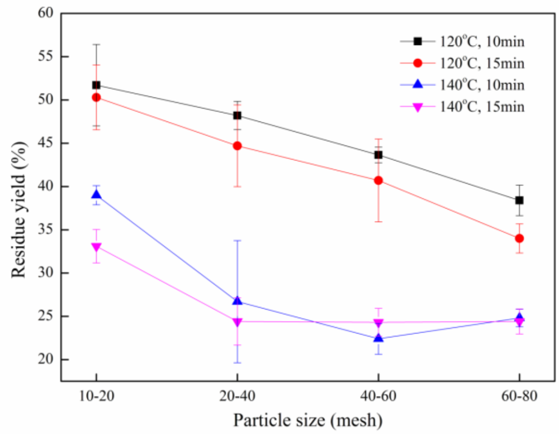
Thank you, Xingyan Huang

Liquefied Bamboo

- **Jiulong Xie, Xingyan Huang**
  - **Research Assistant**
  - **LSU AgCenter**

innovate . educate . improve lives
for the latest research-based information on just about anything, visit our Web site: www.lsuagcenter.com

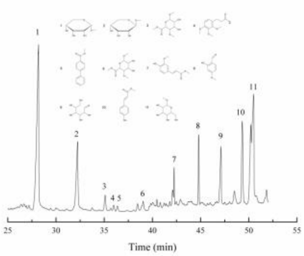
Residue Content (cellulose)



Note: Error bars represent the standard deviations

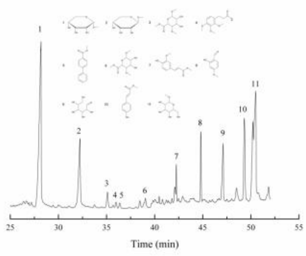
Chemical composition of biopolyols

- **Presence of C5 & C6 sugars from hemicellulose & cellulose indicate degradation of carbohydrates.**
- **Aromatics indicate decomposition of lignin.**



Chemical composition of biopolyols

- **Since the sugar derivatives processed 2-5 hydroxyl groups, the biopolyols have potential in polyurethane foams because of their large amount of hydrogen bonds.**



Characteristics of liquefied residues

The liquefied residues mainly exhibited a fiber structure with remaining cellulose and recondensed lignin fragments indicated by damaged cell wall and small granules.

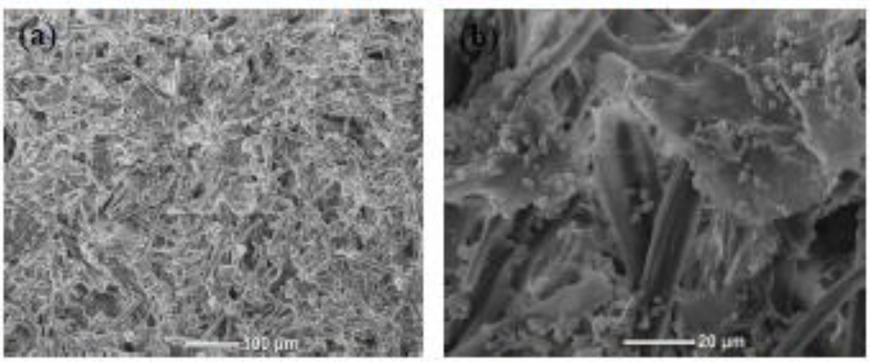


Figure 3. SEM images of traced cellulose structure of liquefied residues obtained from 140°C, 60-80mesh, 10min. (a) 150×; (b) 800×.

Production of nanofibers from residues

Table 1. Yield of chemically purified cellulose fibers and nanofibrillated cellulosic fiber from microwave liquefied bamboo residues.

Sample	Yield of chemically purified cellulose (%)	Yield of nanofibrillated cellulosic fiber (%)
120°C/2.0%	85.60	10.75
120°C/25%	77.78	31.96
120°C/45%	69.59	37.18
140°C/2.0%	78.07	37.17
140°C/25%	67.09	50.20
140°C/45%	65.61	52.02

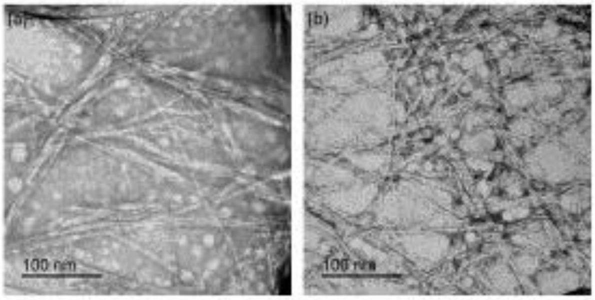
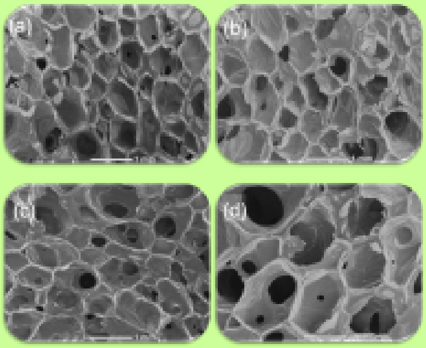


Figure 5. Transmission electron microscope (TEM) images of the nanofibrillated cellulosic fiber extracted from microwave liquefied bamboo residues; (a) 120°C/2%, ultrasonic time, 25 min; (b) 140 °C /25%, ultrasonic time, 5min

Polyurethane Foam from Lignin from Switchgrass

- **Thank you Jiulong Xie and Xingyan Huang**

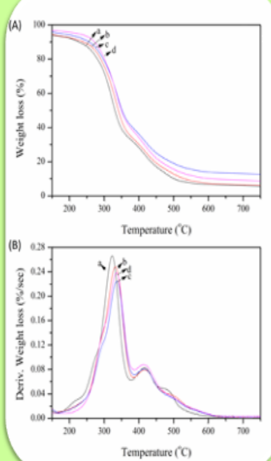
Morphological Structures – Polyurethane Foam



SEM images of (a) pure polyol, (b) 5% lignin, (c) 10 lignin, and (d) 15 lignin content polyurethane foams

- ❖With the introduction of lignin into the PU matrix, the PU foam color became brown and the cell diameter tended to be larger.
- ❖The addition of 15% lignin into the PU matrix had a significant effect on the foam cellular structures.
- ❖High lignin content affected the cell nucleation process in the preparation of PU foams.

Thermal stability



(a) pure polyol, (b) 5% lignin, (c) 10% lignin (d) 15% lignin

Temperature and residue yield of the PU foams with different lignin content

Lignin content (%)	T_{onset}(°C)	T_{max}(°C)	Char yield (%)
0	132.60	323.17	5.37
5	139.93	331.74	5.80
10	177.55	334.97	12.42
15	223.52	333.95	8.42

❖Lignin could improve the thermal stability of the foams, and the foam with 10% lignin had the best thermal stability.

for the latest research-based information on just about anything, visit our Web site: www.lsuagcenter.com
Research & Extension

Conclusions

- Using microwave energy simplifies liquefaction.
- Promising uses:
  - Recover CCA
  - Polyurethane foam (insulation)

Thanks to

- Jiulong Xie, LSU AgCenter
- Xingyan Huang, LSU AgCenter
- Chung-yun Hse, USDA Forest Service, Southern Experiment Station
  - This work is funded by the USDA Forest Service 2015 Wood Innovations Funding Opportunity program, Agreement 15-DG-11083150-054.

Louisiana Forest Products Development Center

cdehoop@Lsu.edu