# Natural Fiber Composites: Retting, Preform Manufacture & Molding

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# Overview

## Timeline

- Start - July 2007
- Finish - September 2010
- 50% complete

## Barriers

- Addressing vehicle weight reduction
- Material form capable of meeting high volume production

## Budget

- Total project funding
  - DOE $1200K
- Funding received in FY08
  - $400K
- Funding for FY09
  - $355K
- CR constraints for FY09

## Partners

- Ford Motor Company
- GM Corporation
- Ashland Chemical
- AOC, LLC
- Project lead

# Objective

- To develop, build, and demonstrate an economical, lab-scale, automated fiber retting process and apparatus suitable to bast based fibers including hemp, kenaf, and flax.

- To develop and demonstrate a thermoset polymer preform compression molding process, produce panels to develop a mechanical and thermal natural fiber polymer composite database.

# Milestones

| Month-Year | Milestone or Go/No-Go Decision |
|---|---|
| Mar-08<br>**(COMPLETED)** | Milestone: Preform Manufacture Process Development<br>Baseline Preform Process Defined |
| Apr-08<br>**(COMPLETED)** | Milestone: Deliver Retting Apparatus<br>Demonstrate Retting Apparatus |
| Apr-08<br>**(COMPLETED)** | **PROGRAM GO-NO GO DECISION**<br>External Review Meeting |
| Jun-08<br>**(COMPLETED)** | Critical Design Review - Preform Manufacture Apparatus<br>CDR Package and Presentation on Preform Manufacture Apparatus |
| Jan-09<br>**(COMPLETED)** | Milestone: Deliver Preform Apparatus<br>Demonstrate Preform Manufacture Apparatus |
| Mar-09<br>Revised Jul-09 | Deliverable: Submit Composite Molding and Characterization test Plan to External Review Committee |
| Apr-09<br>Revised Aug-09 | Milestone: Produce Fiber Preforms<br>Produce Natural Fiber and Hybrid Preforms of desired architecture |
| Apr-09<br>Revised Aug-09 | **PROGRAM GO-NO GO DECISION**<br>External Review Meeting |
| Jul-09<br>Revised Sept-09 | SMC Preforms<br>Deliver SMC Thermoset Natural Fiber Preforms |
| Sept-09<br>Revised Oct-09 | Deliverable: Molding Delivery<br>Complete All Quantities of Composite Molding (SMC) |

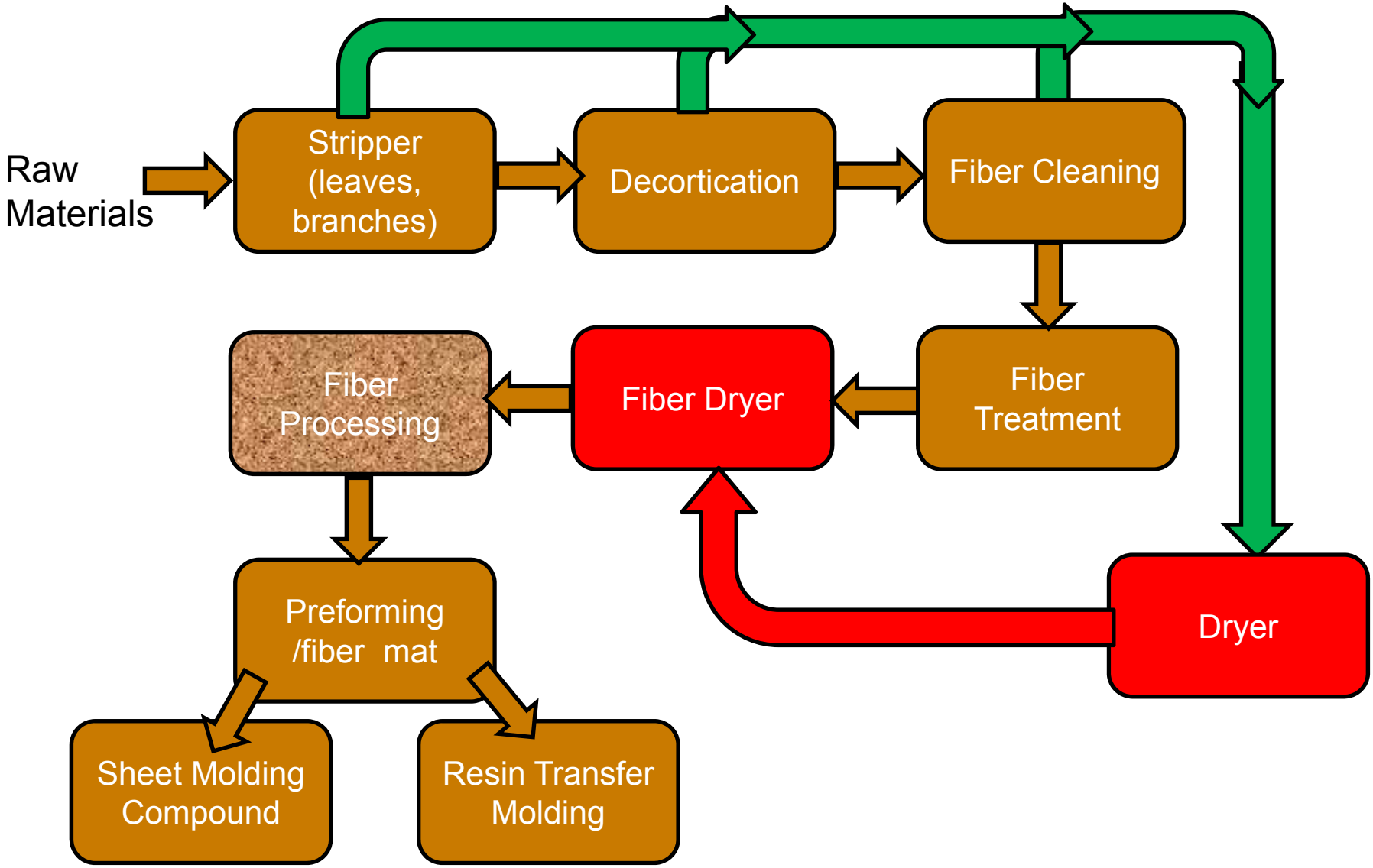
# Barriers

1. **Develop an alternative mechanical-physical-chemical system to the 3-4 month field retting process.**
   - How do we break the low-MW organics that anchor fibers within the plant?
   - What process technologies can be brought to bear on this problem?
   - What portion of the ~20% lignin in bast fibers should be removed?
   - Can this be captured as a process fuel source?

2. **Develop a *fluid-free* natural fiber preform manufacture process and apparatus.**
   - Is it possible to produce a preform composite from dry fiber?
   - Is this process capable of hybridizing preforms for RTM and compression molding?

3. **Develop natural fiber SMC thermoset composites in conjunction with industrial resin suppliers.**
   - What scalable process can be developed that is amenable to natural fibers?
   - Is it possible to produce both ester and urethane SMC from such a process?
   - Are we capable of producing SMC materials based on bio-polyols?

# Program Approach -Process Flow Diagram for RPM²



# Technical Accomplishments
## (FY08 Milestones)

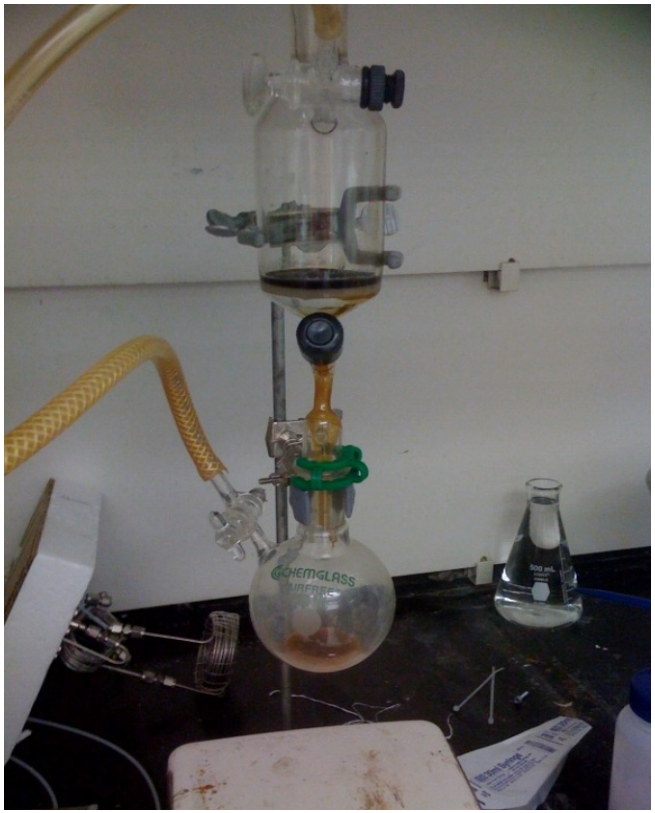
1. Natural fiber preparation process
    1. Lab scale process front end designed, built, and in operational modification. (completed)
    2. Fiber chopper procured and modified. (completed)
    3. Fiber decordication process explored, lab-scale process unit built. (75% completed)
    4. Advanced fiber treatment processes being explored.
        - Super-critical fluid treatment. (25% completed)
        - Thermal fiber treatment (15% completed)
        - Advanced extrusion methods. (25% completed)
        - Ionic liquid methodology. (75% completed)
    5. Fiber separation procedure identified; small scale unit running.
        - Based on carding and cotton gin technology. (50% completed)

# RPM – Technical Accomplishments

- Received 1 ton of kenaf from each – Kengro, Inc., Charleston MS & USDA Prosser, WA Research Center
  - Experiments complete on Kengro kenaf fiber
  - Initial experiments completed on Prosser kenaf fiber
  - Baseline fiber compared to fiber from SE Asia, Texas, and Canada.
- Fiber process line nearly complete

# RPM - Accomplishments

- Preliminary design of ionic liquid extraction process completed.
- Initial results of ionic liquids used to chemically remove the lignin present in lignocellulosic material has been tested and shown to be both possible, and effective in extracting lignin without attacking the cellulose fiber
- Completed spectroscopic analysis of all candidate fibers and correlated results with cellulose, hemi-cellulose, lignin, and LMW organic content from literature values
- All test are based on TAPPI and ASTM Standards




Lignin reaction with ionic liquid

# Chemical characterization of natural fibers

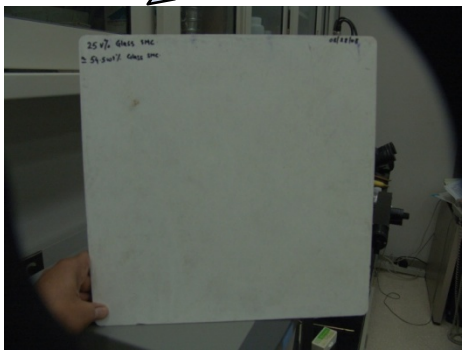
| Fiber | Ash | Extractives | Klason Lignin | Acid Soluble Lignin | Cellulose | Hemi-cellulose |
|---|---|---|---|---|---|---|
| Hemp | 2.42 | 0.59 | 8.12 | N/A | 71.43 | 8.43 |
| Bangladesh Retted Kenaf | 1.24 | 0 | 12.39 | N/A | 63.11 | 19.96 |
| Mechanically Separated Dew Retted Kengro Kenaf | 1.39 | 0.15 | 7.75 | 5.19 | 63.65 | 21.01 |
| Hand Separated Non-Retted Kengro Kenaf | 4.11 | 1.39 | 12.10 | 5.19 | 45.98 | 18.63 |
| Texas Field Retted Kenaf | 4.35 | 0.27 | 8.36 | N/A | 57.28 | 25.35 |
| Prosser Non-Retted Kenaf | 6.32 | 1.11 | 8.99 | 3.56 | 48.03 | 33.1 |
| PAPRICAN (standard) | 0.5 | 0.26 | 4.16 | 0 | 83.04 | 9.99 |

** All numbers reported as percentages of total mass on an oven dry basis.*

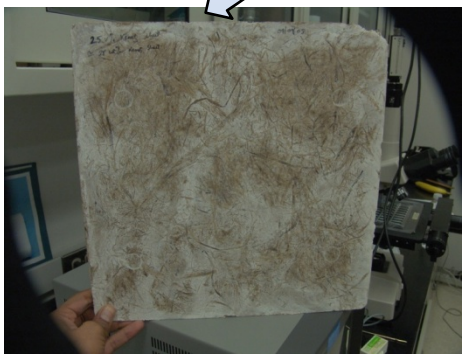
# RPM - Accomplishments

- Tool complete for molding (ACC approved)
- Molding of panels initiated

RTM Process



25 vol % Glass fiber SMC



25 vol % Kenaf fiber SMC (25% lighter than the glass SMC panel)

# Future Work – Fiscal Year 2009

1. Complete fiber preparation sample experiments
   - Determine most effective method (s) to prepare fiber for surface treatment.
   - Quantify the fiber process to make final process decision.
2. Design and produce lab-scale unit to process fiber
3. Conduct characterization study of processed fiber
   - Fiber/core cleanness, separation effectiveness, defibrillation
4. Begin natural fiber SMC development
   - (3rd Quarter, year 2)

# Future Work – Next Fiscal Year

1. Complete development of fiber preform process
   - Capable of integrating surface modification strategies/chemistries.
2. Complete design lab-scale preform manufacture apparatus
   - Complete design review with ACC.
   - Produce unit and quantify performance metrics.
   - Complete prototype manufacture of natural fiber and hybrid units.
3. In parallel, begin composite mechanical, thermal, and environmental characterization

# Summary

1. **Natural fiber composites show great promise in support of a bio-based manufacturing infrastructure within the United States**
   - There is potential for significant petroleum displacement through fiber reinforcement and bio-polyol development.
2. **PNNL efforts address critical needs in support of natural fiber composite development for transportation**
   - Fiber preparation process including delivery time and cost
   - Preform development expanding fiber architectures
   - SMC development enabling rapid processing of natural fiber composites
3. **Develop natural fiber SMC and hybrid-fiber architectures; produce panels and characterize in mechanical, thermal, and environmental**
4. **PNNL continues to establish and work with those commercial relationships to help rapidly insert developments into industry**