Development and Commercialization of a Novel Low-Cost Carbon Fiber

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Project ID #
LM048

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Overview

(Status Dec 2013)

Timeline

- Start Date – Oct 2011
- End Date – Jun 2015
- ~ 60% Complete

Budget

- Total project funding
  - DOE - $3,748,865
  - Contractor - $5,221,798
- ~ 45% Spent
- Substantial scale up tasks will catch up spending 2nd half 2014

Barriers

- Barriers addressed
  - Low cost carbon fiber
  - Inadequate supply base
  - High performance materials
- Targets
  - Cost = $5.00 / pound
  - Commercial product validation using existing manufacturing assets
  - Defined structural properties

Partner

- Weyerhaeuser Company
  - Provides low cost, high quality lignin polymer technology & supply source
  - Provides lab scale spinning technology and analytical testing

Objectives - Relevance

The objectives of this project are to develop and commercially validate a low cost carbon fiber based on renewable precursor raw materials and meeting DOE defined performance & cost targets.

Project defined targets:

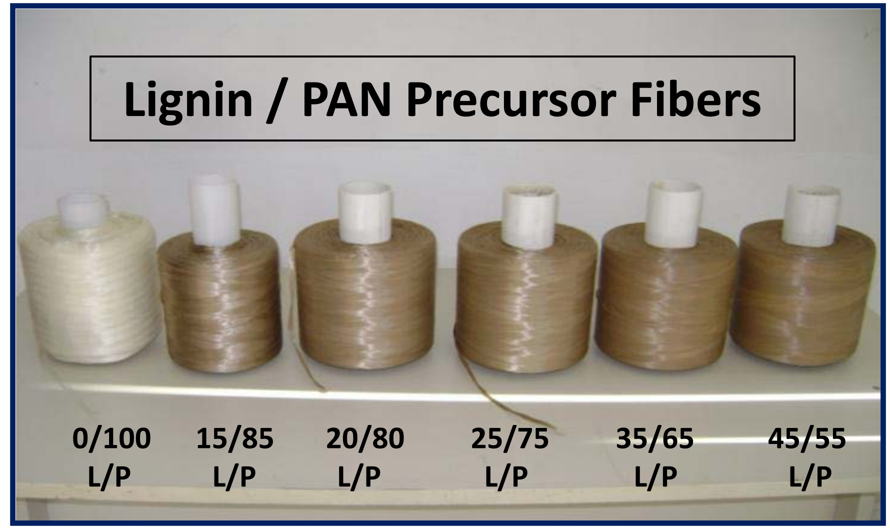
- carbon fiber cost = \$5.00 / lb (\$11.00 / kg)
- strength > 250,000 psi (1724 MPa)
- modulus of elasticity >25,000,000 psi (172 GPa)
- strain-to-failure > 1%

The specific cost and performance targets clearly address the Cost and Performance Barriers identified in the Vehicle Technologies Multi-Year Plan.

The commercial validation objective of this project will address the Inadequate Supply Base Barrier by demonstrating commercial scale production using existing manufacturing assets and approaches that have proven rapid capacity expansion capabilities.

Approach

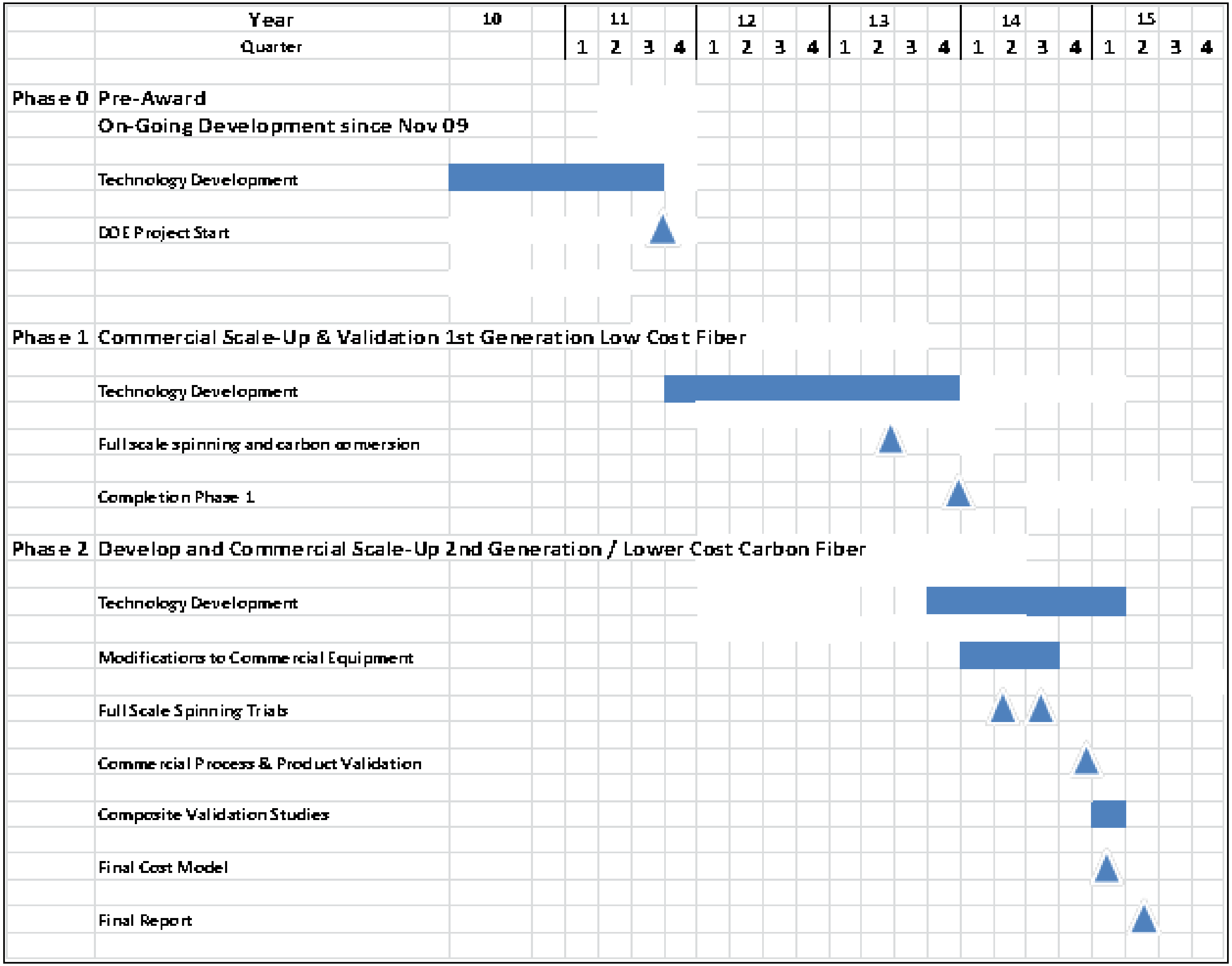
Primary Technical Approach:
Development of Lignin / PAN polymer blend precursor for carbon fiber using solution spinning process



Strategy:

- Different from prior developments with lignin; solution vs. hot melt spinning
- Allows use of existing production equipment → immediate commercialization
- Reduced reliance on PAN (petroleum based)
- Limitation 45% Lignin but still provides substantial cost reduction
- Additional cost reductions possible due to:
  - higher rate stabilization
  - higher carbon yield
- Further cost reductions achieved through operational efficiencies & energy efficiencies in carbon fiber manufacturing

Schedule / Milestones



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Schedule / Milestones

Month / Year	Milestone or Go / No-Go Decision	Status
Jun 2013	Milestone – first commercial scale demonstration	**Completed**
Dec 2013	Schedule – Completion of Phase 1	**Completed**
Oct 2013	Milestone – Initiate of Phase 2 Revised Technical Plan based on Phase 1 results	**Completed**
Jan - Sep 2014	Schedule – commercial equipment modifications for lignin based fiber production	**On-going**
Mar 2014	Milestone – 10,000 lignin delivered to Zoltek	**Plan**
May – Jul 2014	Schedule – Validate commercial scale HMW PAN go/no go decision	**Plan**
Jun – Aug 2014	Schedule – Validate commercial scale spinning go/no go decision	**Plan**
Dec 2014	Milestone – Commercial Process & Product Demo	**Plan**
Jan – Jun 2015	Schedule – Complete composite validation; Cost Model; Final Report	**Plan**

Accomplishments & Progress

March 2013
Commercial Scale Spinning Validated

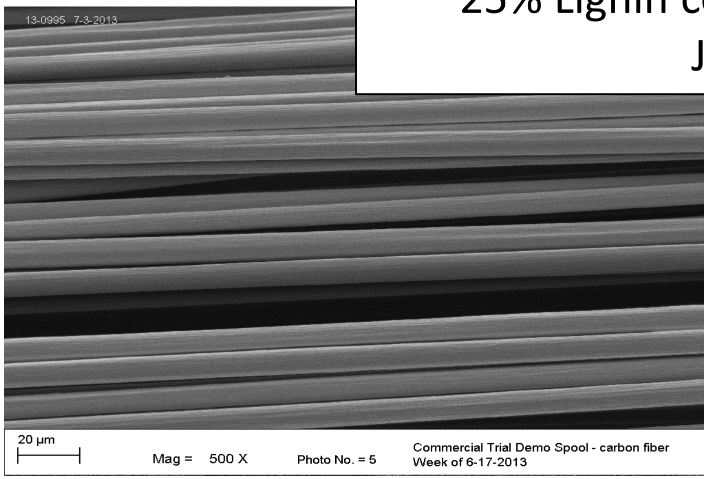
Commercial Precursor Spools containing 25% & 35% Lignin ready for shipment

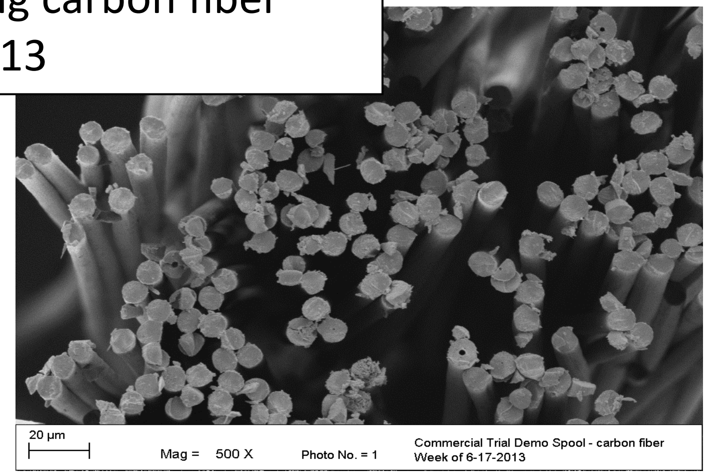
June 2013
Commercial Scale Carbon Fiber Validated

Commercial Carbon Fiber Spools containing 25% Lignin ready for shipment

Accomplishments & Progress

25% Lignin containing carbon fiber
June, 2013





Carbon fibers produced had good quality and properties exceeding targets

Parameter	Tensile Strength (Ksi)	Tensile Modulus (Msi)	Strain (%)	Density (g/cc)	Tow Mass (g/m)	Moisture (%)	Resistivity (10^{-3} Ω-cm)	Sizing (%)
AVERAGE	**325**	**31.5**	**0.98**	1.780	3.99	0.045	1.61	1.30
SD	49	2.2	0.09	0.013	0.10	0.027	0.09	0.10
CV	15.0	7.1	9.6	0.7	2.6	59.6	5.6	8.0
n	20	20	20	20	20	20	3	20

Accomplishments & Progress

Composite properties achieved from 25% Lignin carbon fiber are good and as expected based on fiber properties.

Composite Data from Prepreg & Pultrusion

Composite Data from	Prepreg	Pultrusion
Tensile Strength (Ksi)	137	137
Tensile Modulus (Msi)	16.7	21.0
Compressive Strength (Ksi)	187	
Compressive Modulus (Msi)	17.2	
Interlaminar Shear Strength (Ksi)	11.2	
Fiber Volume Fraction (%)	58	61

Composite Data from Sheet Molding Compound

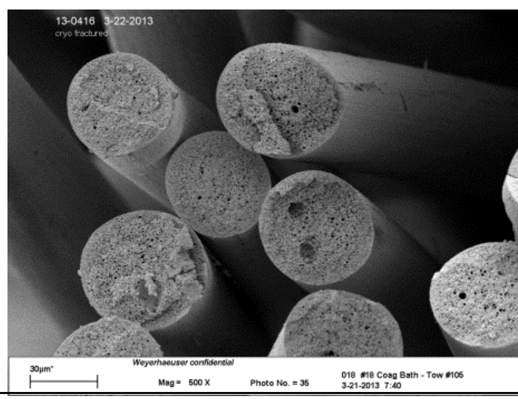
	Lignin Based SMC Composite	PAN Based SMC Composite
Flexural Strength (KSI)	47.9	61.2
Flexural Modulus (MSI)	3.74	4.06

Accomplishments & Progress

Phase 1 – Problems / Issues

- Commercial polymerization scale-up of high molecular weight PAN was not successful, forcing precursor spinning scale-up to use standard PAN
- Resulting commercial scale precursor had higher level of macro-voids than desired
- Only capable to successfully produce 25% lignin carbon fiber; void content too high in 35% lignin precursor
- Resulting carbon fiber yield was very low; again due to morphology problems
- Some lignin leaching occurred (2.5 – 3.0 %) during precursor spinning, creating production problems
- Fiber sticking occurred during carbon conversion, requiring increase of precursor sizing

These problems must be resolved in Phase 2



Too high level of macro-voids in precursor

Some Lignin leaching occurred in spinning creating production problems

ZOLTEK

Weyerhaeuser

Previous Year Reviewers' Comments

- Several comments addressed lack of understanding of chemistry of oxidation of lignin / Pan precursor fibers

  Understanding the chemistry and reaction kinetics of the oxidation of the lignin / PAN precursor fiber is quite challenging. We try to use thermal analysis (DSC) to try to predict reaction behavior but the boundary conditions and kinetics are different. We do not have a good procedure for analyzing the molecular construct of the oxidized fiber, so we are limited to measuring physical properties of the oxidized fiber, such as density and tenacity, and comparing these to the PAN baseline. Also using DSC to calculate the Aromatic Index of the oxidized fiber is a good indicator of carbonization performance

- Several comments addressed concern about achieving the $5.00 / lb. target

  Regarding $5/lb. target, there has been some confusion whether this means cost or selling price. DOE has indicated this is cost and that a reasonable margin would be added. With Zoltek's current low cost process, combined with lower cost of lignin polymer at 45% material substitution, and also additional cost reduction efforts of this project, the $5/lb. cost target appears achievable.

Collaborations

- Within the framework of this project, Zoltek is the prime contractor and Weyerhaeuser is the only subcontractor. In this regard, Zoltek has the administrative lead responsibilities for this project management, but technical and business decisions related to this development are shared jointly.
- Technical and business responsibilities are divided as follows:

Zoltek	Weyerhaeuser
PAN Polymer	Lignin Polymer
Pilot & Commercial Wet Spinning	Lab Scale Wet Spinning
Carbon Conversion	Lignin Polymer Commercialization
Carbon Fiber Commercialization	

- During the commercial scale validation portions of this project, several OEM and Tier 1 manufacturers have expressed interest in participating in product evaluations. In Phase 1, Magna International (largest Tier 1 automotive supplier) produced and tested Carbon Fiber Sheet Molding Compound (C-SMC) with the lignin based fiber. These results are included in the project reports. More comprehensive evaluations and applications demonstrations with OEMs and Tier 1 manufacturers are planned for Phase 2.

Future Work
2014 Development Plan

- Complete pilot scale optimization of spinning parameters
- Complete pilot scale optimization of HMW PAN polymerization & precursor spinning
- Validate at pilot scale best combination of all parameters
- Validate commercial scale polymerization of HMW PAN (may take several iterations)
- Complete equipment modifications to Spinning Line 1
- Validate commercial scale precursor spinning with optimized parameters & HMW PAN (may take several iterations); first 10,000 pounds of lignin to be delivered end Q1 2014
- Accelerate development of Dry-Jet Wet Spinning
- Complete equipment modifications to carbon line oxidation oven 1; validate throughput enhancement; if successful, make additional equipment modifications to oxidation ovens 2 & 3
- Validate carbonization of optimized commercial scale precursor
- Prepare for final process & product validation in Q4 2014

Summary

Objectives of this project are focused on very specific product and commercialization targets directly addressing Barriers defined in the Vehicle Technologies Multi-Year Plan:

Low Cost Carbon Fiber = $5.00 / pound
Tensile Strength > 250,000 psi
Tensile Modulus > 25,000,000 psi
Elongation > 1%
Commercialization and Demonstration of Capacity Growth Potential

In 2013, Phase 1 commercial scale validation was successfully accomplished, producing 1 metric ton of 25% lignin containing carbon fiber with properties exceeding projects targets.

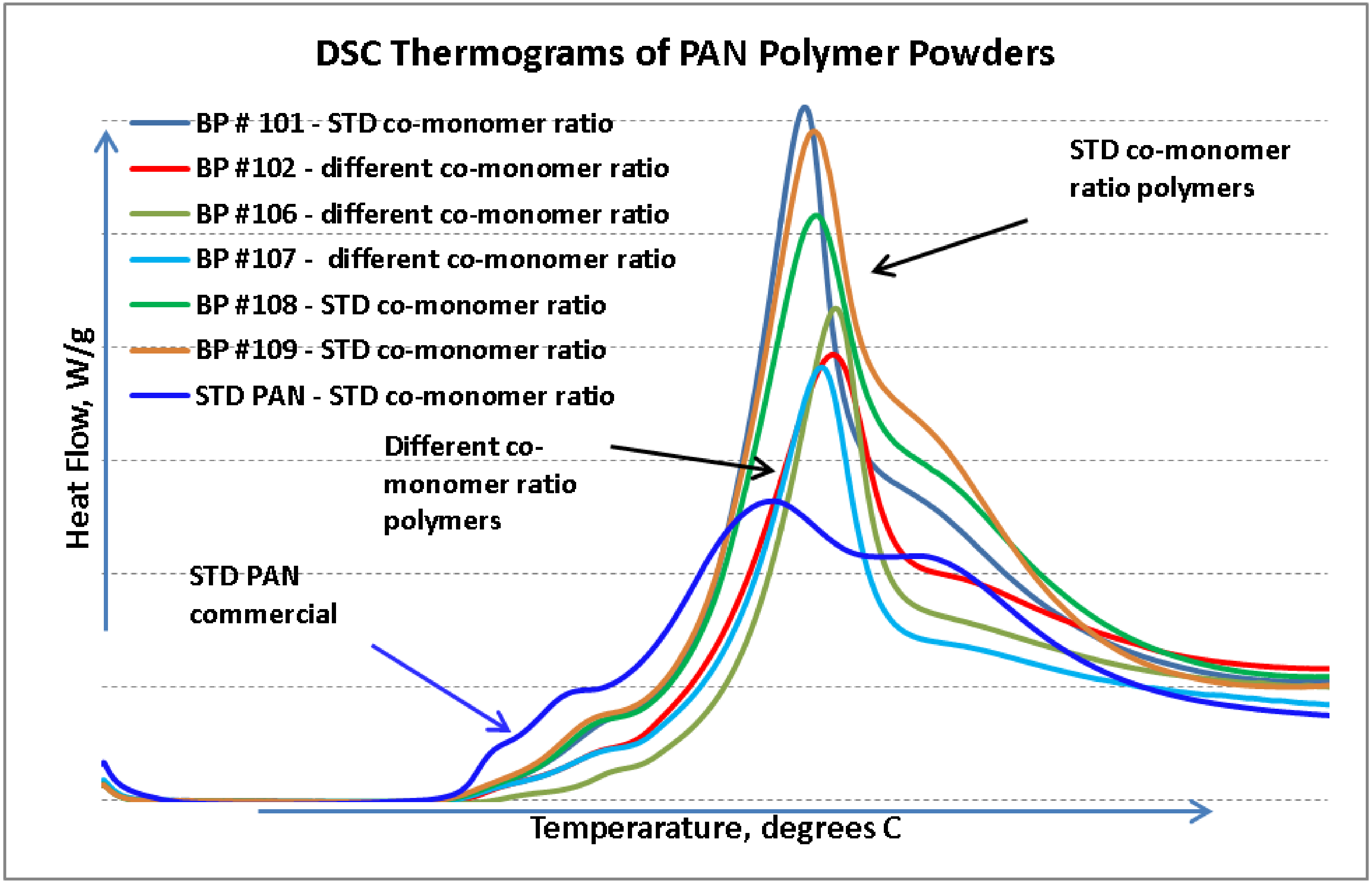
Tensile Strength = 325,000 psi
Tensile Modulus = 31,500,000 psi
Elongation = 1%

These fibers were subsequently processed in to composite laminates using prepreg compression molding, pultrusion, and sheet molding compound molding to successfully validate composite properties.

Issues identified during the Phase 1 development have been addressed in revised Phase 2 technical plan and schedule that was initiated in 4th quarter 2013

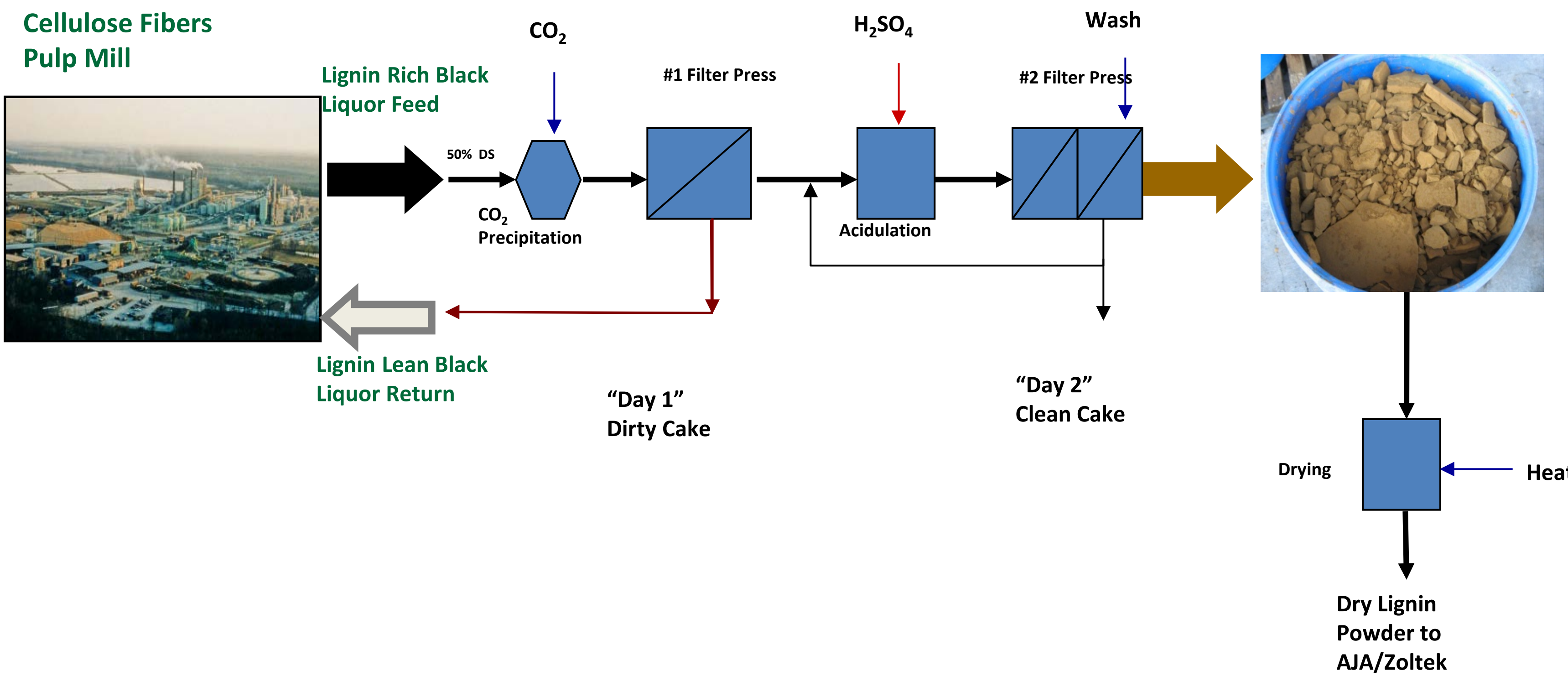
Technical Back-Up Slides

LP PROJECT

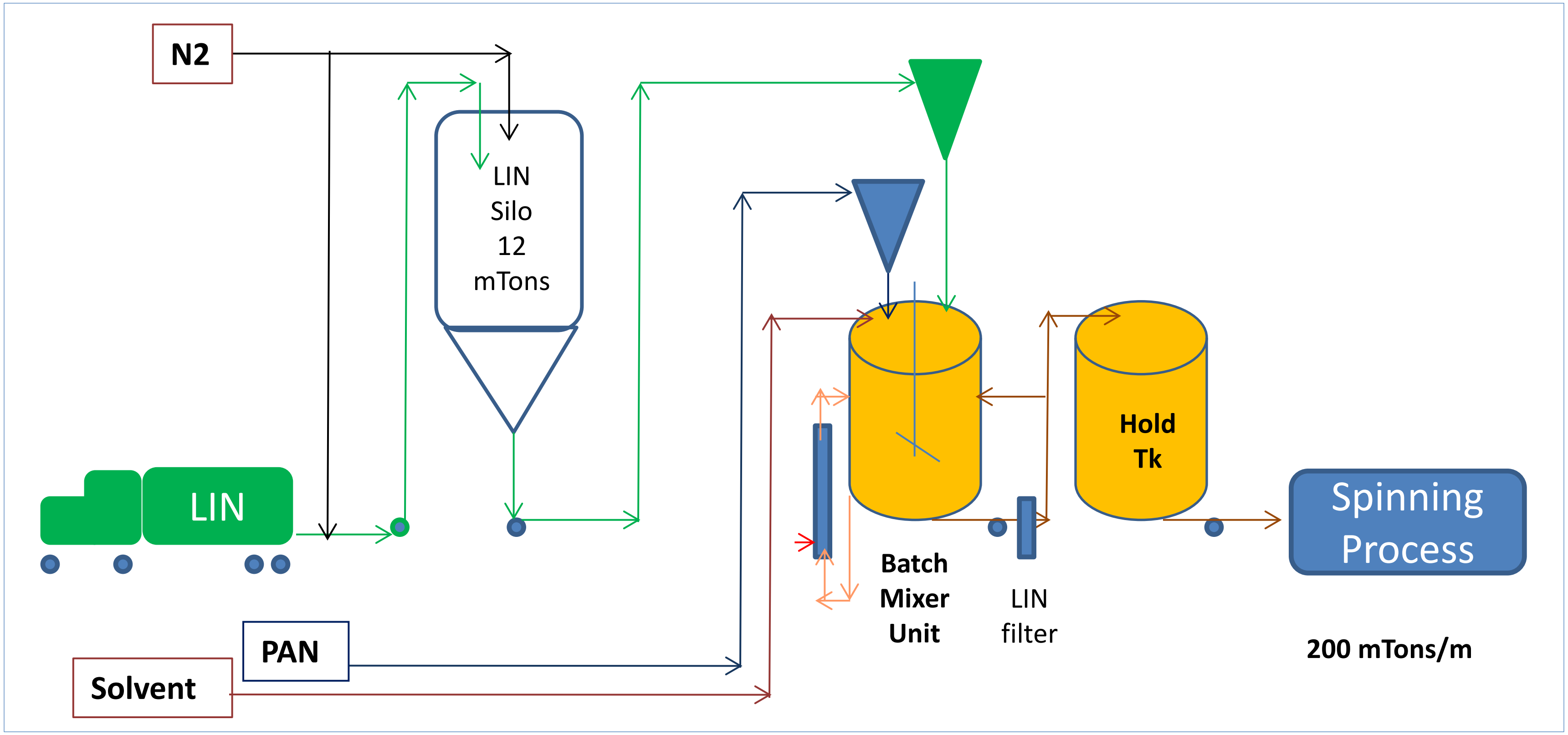


DSC Thermograms of PAN Polymer Powders
BP # 101 - STD co-monomer ratio
BP #102 - different co-monomer ratio
BP #106 - different co-monomer ratio
BP #107 - different co-monomer ratio
BP #108 - STD co-monomer ratio
BP #109 - STD co-monomer ratio
STD PAN - STD co-monomer ratio
STD co-monomer ratio polymers
Different co-monomer ratio polymers
STD PAN commercial
Heat Flow, W/g
Temperarature, degrees C

Weyerhaeuser Lignin Pilot Plant Process



Large Scale Mixing Facility



Zoltek Pilot Spinning Line

Zoltek Pilot Oxidation Line