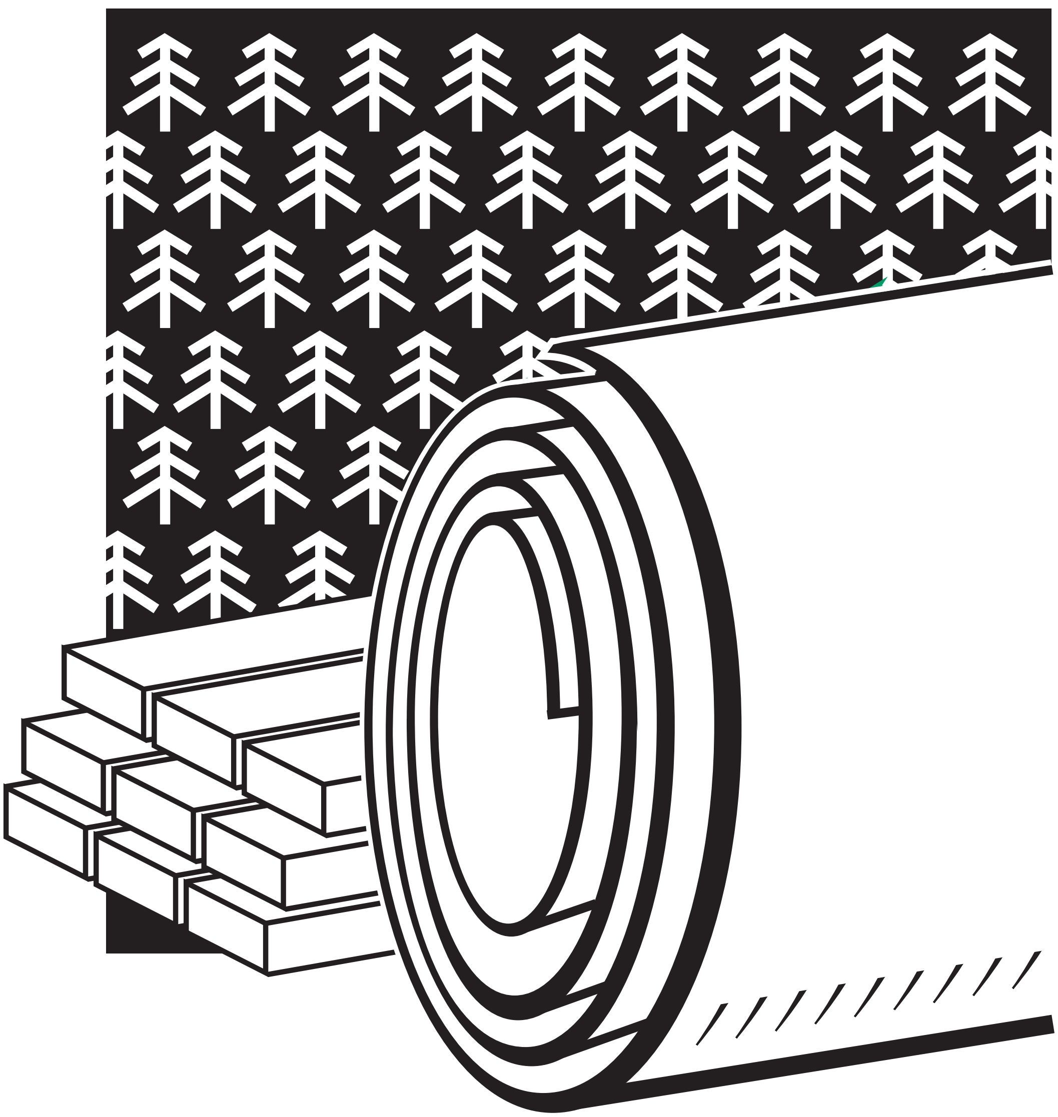
Forest Products Industry of the Future

Quarterly Status Reports

As of March 31, 2007



U.S. DEPARTMENT OF ENERGY

02-GA50113-09

Forest Products

Industry of the Future

Quarterly Status Reports

As of March 31, 2007

Contacts

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Enhanced Raw Materials

Mill Processes: Pulping

Mill Processes: Papermaking

Mill Processes: Process Control

Mill Processes: Recovery / Environmental

Improved Fiber Recycling

Wood Processing

Engineering of Syringyl Lignin in Softwood Species Through Xylem-Specific Expression of Hardwood Syringyl Monolignol Pathway Genes

Joshi: Michigan Technological University, NREL

ID14440

QUARTERLY PROGRESS REPORT

Project Title: Improved Wood Properties Through Genetic Manipulation: Engineering of Syringyl Lignin in Softwood Species Through Xylem-Specific Expression of Hardwood Syringyl Monolignol Pathway Genes

Covering Period: January 1, 2007 to March 31, 2007

Date of Report: April 30, 2007

Recipient: Michigan Technological University
1400 Townsend Drive, Houghton, MI 49931-1295
Congressional District: MI Ist

Award Number: DE-FC36-03ID14440

Subcontractors: North Carolina State University, Room 1 Leazer Hall, Campus Box 7514, Raleigh, NC 27695-7514
Matt Ronning, Associate Vice Chancellor
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Maud A. W. Hinchee, Chief Technology Officer
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(2) MeadWestvaco, P.O. Box 1950, 180 Westvaco Road, Summerville, SC 29484
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(3) Weyerhaeuser Company, WTC, 2D39, P.O. Box 9777, Tacoma, WA 98063-9777
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(4) International Paper Company, P.O. Box 7910, Loveland, OH 45140-7910
Richard B. Phillips, Senior Vice President-Technology
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Contact(s):

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Project Objective: Our long-term goal is to genetically engineer higher value raw materials with desirable wood properties to promote energy efficiency, international competitiveness, and environmental responsiveness of the U.S. forest products industry. The immediate goal of this project is to produce the first higher value softwood raw materials engineered with a wide range of syringyl lignin quantities.

Background: The most important wood property affecting directly the levels of energy, chemical and bleaching requirements for kraft pulp production is lignin. Softwoods contain almost exclusively chemically resistant guaiacyl (G) lignin, whereas hardwoods have more reactive or easily degradable lignins of the guaiacyl (G)-syringyl (S) type. It is also well established that the reactive S lignin component is the key factor that permits much lower effective alkali and temperature, shorter pulping time and less bleaching stages for processing hardwoods than for softwoods. Furthermore, our pulping kinetic study explicitly demonstrated that every increase in one unit of the lignin S/G ratio would roughly double the rate of lignin removal. These are clear evidence that softwoods genetically engineered with S lignin are keys to revolutionizing the energy efficiency and enhancing the environmental performance of this industry.

Softwoods and hardwoods share the same genetic mechanisms for the biosynthesis of G lignin. However, in hardwoods, three additional genes branch out from the G-lignin pathway and become specifically engaged in regulating S lignin biosynthesis. In this proposed research we will simultaneously transfer aspen S-specific genes into a model softwood, black spruce, to engineer S lignin.

Status

In the past quarter, we continued to work on selection of the transformed calli, culture of somatic embryo maturation and germination, regeneration of transgenic seedlings, and greenhouse planting and seedlings maintenances. Particularly, we have intensively conducted the transformation with the newly constructed multiple-genes constructs. The work is related to the planned Project Milestone, NCSU Task 3 (Transformation of black spruce with aspen *CAld5H*, *AldOMT* and *SAD* gene expression constructs via *Agrobacterium*-mediated multigene transfer and regeneration and propagation of transgenics). The work that have been done in this period included: (1) subculture for continuous selection of the transformed calli using the constructs of aspen S-lignin genes under the control of double 35S constitutive promoter (pBI-K35S-PtCAld5H, pBI-PtAldOMT, and pBI-PtSAD, designated as the D35S construct set) and the constructs of the S-lignin genes under the control of spruce xylem-specific promoter (pBI-PmP$_{4Cl}$-PtCAld5H, pBI-PmP$_{4Cl}$-PtAldOMT, and pBI-PmP$_{4Cl}$-PtSAD, designated as the SXS construct set), (2) somatic embryo maturation, germination, and regeneration of the transgenic seedling, (3) growing putative transgenic seedlings in greenhouse, (4) transformation with new multiple gene constructs.

Note: The late start of the project and prorated project budget have caused considerable interruptions to an effective, continual progress of the project. As a result, the timing and therefore progress on regeneration of transgenic plants, in particular, has been most seriously affected, requiring an extension of the project period. A no-cost extension of the project to October 31, 2007 has recently been approved.

1. Subculture for continuous selection

We continued to subculture and select the calli putatively transformed with the D35S construct set and the SXS construct set. The selection of the transformed calli was through continuous subcultures with an interval of 2 weeks on the medium containing kanamycin antibiotics. After 2-3 month selection, approximate 10% of callus masses were selected as transformed callus masses for the subsequent embryo maturation. Up to now, we have selected a total of more than 200 masses transformed with each set of constructs.

2. Somatic embryo maturation, germination, and regeneration of the transgenic seedling

After 2-3 month selection culture, the positive callus mass was cultured onto embryo regeneration medium for embryo formation. The embryos maturation took about 8 -10 weeks to form somatic embryos that can be harvested. When somatic embryos were formed and grew to ~ 2 mm in length, they were harvested from the callus mass and cultured on the germination medium. Now the embryos are continuously harvested from various batches of the transformations. After embryo harvested, it took about 3 months for germination and elongation. The putative transgenic seedlings have been continuously regenerated.

3. Growing putative transgenic seedlings in greenhouse

To plant regenerated seedlings, we have tested and optimized various conditions for planting the transgenic seedlings in greenhouse. We have successfully planted 14 batches totaling more than 650 transgenic seedlings in greenhouse. The seedling survive rate is about 50%.

4. Multiple gene construct preparation and transformation

Considering low efficiency of the simultaneous transformation with spruce embryonic callus, we have prepared the construct combined with 2 and 3 target genes together in order to increase the efficiency of multiple gene integration. We have completed the construction of a binary plasmid containing both *CAld5H* and *AldOMT* genes individually controlled by a xylem-specific promoter and another binary plasmid containing *CAld5H* and *AldOMT* and *SAD* genes that are individually controlled by a xylem-specific promoter in the construct. These new multiple-genes constructs have been used to transform spruce callus tissue. A total of 15 batches of transformation have been completed with these constructs. In order to increase the transformation efficiency, we developed suspension-cultured cells (reported last time) and used for the new transformation. Currently most of the new transformations are cultured in the selection stage. From these new transformations, the transgenic seedlings with a higher rate of multiple-genes co-integration can be expected.

We will continue subculture and regenerate the already-transformed callus tissues, which now are at various developing stages. We will put more efforts on selection and generation of the transgenic plantlets co-transformed with multiple genes. At the same time, we will try to characterize the transgenic plants. However, the transgenic plants grow slowly in the greenhouse. A longer time than expected is needed to grow these plants to a size that can be harvested for detail characterization.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Comments
NCSU Task 1	Cloning of spruce xylem-specific promoter	6/1/04	Complete
NCSU Task 2	Preparation of aspen *CAld5H*, *AldOMT* and *SAD* gene expression constructs and *Agrobacterium* strains	8/1/04	Complete
NCSU Task 3	Transformation of black spruce with aspen *CAld5H*, *AldOMT* and *SAD* gene expression constructs via *Agrobacterium*-mediated multigene transfer and regeneration and propagation of transgenics	6/1/06	Complete
NCSU Task 4	Molecular genetic and biochemical characterization of transgenic black spruce plants	10/1/07	In progress & on schedule
NCSU Task 5	Lignin content and S/G protocol establishment	6/1/05	Complete
NCSU Task 6	NIR-based characterization of cellulose and xylan contents	6/1/06	Complete
NREL Task	Py-MBMS quantification of the S/G ratios and other lignin structural details for the finalist transgenic spruce	6/1/06	In progress
	Final Report	12/1/07	See “Note” in the “Status” section

			of this report

Patents: None

Publications/Presentations: None

Budget Data (as of date): The actual spending should reflect the money actually spent on the project in the corresponding periods.

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	9/30/04		**600,406.09**		**128,491.96**	**728,898.05**
4Q04	10/1/04	12/31/04		**012,000.08**		**119,186.03**	**131,186.11**
1Q05	1/1/05	3/31/05		**000000.00**		**000000.00**	**0.00**
2Q05	4/1/05	6/30/05		**119,446.26**		**000000.00**	**119,446.26**
3Q05	7/1/05	9/30/05		**9,950.03**		**0.0000.00**	**9,950.03**
4Q05	10/1/05	12/31/05		**59,038.19**		**42,200.82**	**101,239.01**
1Q06	1/1/06	3/31/06		**71,182.66**		**0.00**	**71,182.66**
2Q06	4/1/06	6/30/06		**49,476.56**		**0.00**	**49,476.56**
3Q06	7/1/06	9/30/06		**49,030.76**		**79,993.34**	**129,024.10**
4Q06	10/1/06	12/31/06		**17,005.37**		**22,116.05**	**39,121.42**
1Q07	1/1/07	3/31/07		**98,683.69**		**-9,284.04**	**89,399.65**
2Q07	4/1/07	6/30/07	**167,309.76**		**33,741.90**		
3Q07	7/1/07	9/30/07	**167,309.76**		**33,741.90**		
4Q07	10/1/07	12/31/07					
Totals				**1,086,219.69**		**382,704.16**	**1,468,923.85**

* Update quarterly

General Note: DOE Laboratory partner spending should not be included in the above table. If a DOE Laboratory is a partner, report their spending and spend plan information in the table below (use separate tables if multiple DOE Laboratories are involved):
Note 1: Leave blank. Only the actual DOE/Cost Share amounts spent through 6/30/04 are needed.
Note 2: Amount for this quarter and subsequent quarters should be updated as necessary on a quarterly basis. Estimates need to be provided for the entire project. If spending for a given quarter is different than estimated, then the remaining quarter's estimates should be updated to account for the difference. Total DOE and Cost Share amounts should be the same as the Award amount.
Note 3: This should match the amount on the SF269A section 10.c. Column III (10.j. Column III on the SF269).
Note 4: This should match the amount on the SF269A section 10.c. Column II (10.j. Column II on the SF269).
Note 5: This should match the amount on the SF269A section 10.b. Column III (10.i. Column III on the SF269).
Note 6: This should match the amount on the SF269A section 10.b. Column II (10.i. Column II on the SF269).
Note 7: This should match the amount on the SF269A section 10.a. Column III (10.d. Column III on the SF269).
Note 8: This should match the amount on the SF269A section 10.a. Column II (10.d. Column II on the SF269).

DOE Laboratory Spending Table (if applicable):

DOE Laboratory Partner Spending and Estimate of Future Spending					
Quarter	**From**	**To**	**Estimated DOE Lab Amount***	**Actual DOE Lab Amount**	**Total**
	Start	6/30/04			
3Q04	7/1/04	9/30/04	Note 2		
4Q04	10/1/04	12/31/04			
1Q05	1/1/05	3/31/05			
2Q05	4/1/05	6/30/05			
3Q05	7/31/05	9/30/05			
4Q05	10/1/05	12/31/05			
1Q06	1/1/06	3/31/06			
Etc.					
Totals					

Increasing Yield and Quality of Low-Temperature, Low-Alkali Kraft Cooks with Microwave Pretreatment

Compere: Oak Ridge National Laboratory

Agr id:10295

QUARTERLY PROGRESS REPORT

Project Title: Increasing Yield and Quality of Low-Temperature, Low-Alkali Kraft Cooks with Microwave Pretreatment

Covering Period: January 31 2007 through March 31, 2007

Date of Report: May 7, 2007

Recipient: Oak Ridge National Laboratory (ORNL), operated by UT-Battelle
P. O. Box 2008
Oak Ridge TN 37831-6150

Award Number: DE-AC05-00OR22725

Subcontractors: H. Jameel and M. J. Kocurek, North Carolina State University (NCSU)
A. L. Fricke, Emeritus Professor, University of Florida

Other Partners: D. Parent, Communications and Power Industries

T. S. Bigelow and W. L. Griffith, ORNL

Contact(s): A. L. Compere (865-574-4970) compereal@ornl.gov

Project Team: Drew Ronnenberg, DOE program manager; AF&PA program managers and mentors

Project Objective: The project goal is development of a predictive understanding of the effect of different microwave pretreatment parameters, including frequency, application pattern, and wood shape (logs or chips) and water content, on yield and quality of Kraft pulp produced using low-temperature or low-cooking-chemical digestion regimes. In addition to yield evaluation of Kraft pulps, pulp bleachability and handsheet quality (brightness, strength, freeness) will be evaluated for selected processing conditions. The ultimate project goal is development of proof-of-concept demonstration of a microwave/RF pretreatment process which could: 1) increase the yield of Kraft pulps, 2) decrease the cooking chemicals and cooking temperature required for production of a given quality pulp, 3) increase Tomlinson boiler throughput, or 4) decrease lime kiln energy usage.

Background: This project started ten years ago as a two-year study supported by ORNL in-house funding to determine whether it was possible to develop “green chemistry” pulping technology. The project approach was to change the structure of wood to facilitate penetration of cooking chemicals. Pulping studies to evaluate the effect of pretreatment on ORNL hardwood coppice were performed by Thomas Joyce of North Carolina State University through his small business. Beloit and Bechtel staff provided advice on pulping technologies.

Initial proof-of-concept experiments were performed on small sections of wood (~200 g) in a small microwave unit, and later experiments evaluated pulp quality and yield of sycamore sections up to 3.75 inches in diameter treated with a high power microwave. The data consistently showed increased yield and lower Kappa number at conventional Kraft pulping conditions. Tom Joyce also found that a substantially smaller amount of cooking chemicals were required to achieve equivalent pulp quality. Bleaching and handsheet tests indicated that samples from pretreated wood were slightly brighter and slightly stronger than control samples. However, these proof-of-concept tests did not systematically evaluate processing schemes such as low-temperature or low-chemical cooks, which could minimize process energy and material requirements. Additionally, these tests did not systematically evaluate the effect of pretreatment on softwoods or address the ability to process chips. Both of the factors are critical to adoption of this process technology by the pulping industry.

The first part of this project, as funded by the Industrial Technology Program, had relatively modest goals: 1) to evaluate methods and develop technology for decreasing pulping chemicals, process time and energy (as H-factor), and cost required to produce a given amount of pulp and 2) to develop the data required to transfer the technology to both the forest products industry and manufacturers of large industrial microwave/RF systems. It was thought that shorter process times and lower cooking chemical use would permit an existing mill to produce more pulp at lower cost and with less environmental impact. However, initial expectations were a limited reduction, perhaps 2% active alkali (AA), in pulping chemicals and a modest reduction, perhaps 5%, in H-factor (time at temperature), a measure of process energy. It was also thought that the technology would facilitate pulping of oversized (12 mm thick) chips.

Using a small (100 mm diameter) continuous 915 MHz microwave applicator, the project team was able to show that very large sections (up to 100 mm long X 100 mm wide) of bark-on microwave pretreated hardwood harvested on the ORNL reservation could be pulped at greatly reduced cooking chemical concentrations (12% AA vs. 16-17% for untreated conventional chips) or significant reductions in H-factor (50%).

The project was extended and, as requested by advisors, the research team worked with industrial partners to develop a prototype industrial applicator which for use on chips, logs, and lumber. This was accomplished, and our industrial partner, Communications and Power Industries, constructed the applicator and subsidized its lease to ORNL. The applicator was specifically designed to provide information which will facilitate their scale up of this technology.

Using both softwood and hardwood chips, we have been able to show reductions in both process energy (45% reduction in H-factor for pine, 50% for sycamore) and in pulping chemicals (45% reduction in H-factor for pine, 40% for sycamore). Most important, reductions in cooking chemicals and in H-factor could be achieved in the same pulping runs. Additionally, screened yield, handsheet properties, and kappa number equal or exceed those of paired, untreated, conventionally-pulped samples. Reductions in pulping chemicals are becoming particularly significant because natural gas is typically used to fire the kiln used to prepare lime used in white water preparation (chemical recycle). Since the lime kiln consumes the bulk of natural gas in a pulp mill, process chemical reductions translate directly into reductions in natural gas usage and into energy conservation.

To assess the potential impact on the industry, periodic energy and mass balance assessments have been prepared. Using sample cooks matched for kappa number and yield, but with varying pulping chemicals, the impact of lowered H-factor on mill throughput was also assessed. The results indicated that microwave pretreatment could, for the samples evaluated, increase batch mill throughput by 9% to 35% due to reduced sample pulping times.

Finding methods, such as hemicellulose separation, which could increase both the throughput and yield of existing pulp mills, also has the potential to facilitate rapid adoption of microwave pretreatment technology.

Status: The project team is evaluating sites to support an in-mill demonstration of microwave pretreatment using the current project applicator. An initial survey of the capacities of 68 domestic batch or tube digester pulp mills expected to be operating during FYs 2007 and 2008 showed that the average digester capacity of half of these mills makes them potential candidates as demonstration sites.

In the last quarter, the project team obtained lists and contact information for sixty-eight domestic pulp mills using batch or very small scale pulping technologies. These mills were located in 23 states.

Mill data obtained from Lockwood-Post and the Sloan Foundation Center for Paper Business and Industry Studies at Georgia Institute of Technology were used to evaluate the average output of digesters, pulping process used, and mill furnish with the goal of selecting mill demonstration sites which would have broad applicability.

A list of mill characteristics (scoring criteria) which would be likely to lead to a successful demonstration was developed. These factors included: 1) previous successful experience as a technology demonstration site; 2) scale, site location, processes, and products; 3) management enthusiasm for demonstration projects like this one; 4) sufficient space on the operating floor in which to place a microwave/RF generator and applicator, including conveyor belts; 5) several batch digesters; 6) available electric power on the operating floor; 7) manpower; 8) any operating requirements (such as EPA or state EPA restrictions on a mill, union labor requirements, or standard use of proprietary information); 9) company operating requirements (such as formalized procedures) which could slow down equipment installation; 10) accessibility to the project team members; and 11) willingness to share their demonstration results with other pulping companies and, with management agreement, permit DOE to host a site technology demonstration; 12) accessibility to project staff and DOE. Mills were then screened and downselected.

Mills which used uncommon pulping process, such as: 1) the five mills which use neutral-sulfite semichemical pulping, 2) the three mills which produced dissolving pulp, and 3) the two mills which pulp flax for specialty papers were eliminated from consideration as demonstration sites because their technology would be directly useful to a small section of the pulping industry.

Accessibility of potential demonstration sites to both research staff, DOE management staff, technical advisors and reviewers, and to industry is critical in encouraging adoption of a technology. Limited accessibility resulted in the removal of one mill from consideration.

Planned use of an existing applicator in the demonstration limited site consideration to mills in which individual digesters produce approximately 50 or fewer air-dried tons of pulp per day. This eliminated 25 mills from consideration as demonstration sites.

Technical and supervisory staff from the most promising mills – those, which pulped both hardwood and softwood, had digester sizes in a feasible range, and were accessible - were contacted by Mike Kocurek. Mills interested in participating were visited, evaluated in more detail, and asked to provide chips for pulping evaluation. The six mills likely to provide the most promising demonstration sites are discussed below. Particular attention was paid to the methods used for handling chips as these vary considerably across the industry.

Contacts and initial visits were arranged and performed by Professor Michael J. Kocurek of North Carolina State University. Professor Kocurek has extensive experience in arranging pilot and demonstration projects for new pulp and paper technology because he served as Executive Director of the South's oldest research organization, the State of Georgia's Herty Foundation from 1986-1997. The Herty Foundation maintains pilot facilities for pulp and paper. In 1996, a $27 million modernization program was completed that created the largest independent contractual R & D pilot laboratory in the U.S. This background has given Professor Kocurek extensive experience in setting up and managing pilot evaluations of new process technology for the pulp and paper industry.

In 1997, he came to North Carolina State University as Head of the Department of Wood and Paper Science. Mike is a TAPPI Fellow (1983) and his TAPPI activities and contacts (Distinguished Service Award, 1994; longest-standing short course, *Introduction to Pulp and Paper*, taught to over 5000 employees from 50 different forest products corporations across 25 years) give him a wide range of contacts and access to mills. Mills in the North Central, Southeast, and Northeast were contacted and visits scheduled. During the visit, a contact responsible for interactions with the project team was requested. Synopses of mill visits and initial evaluations, arranged alphabetically by mill operator, are given below.

<u>Blue Ridge Paper Company, Canton, North Carolina.</u> The visit was hosted by Michael Ferguson, Superintendent, Pulp Manufacturing, and Brian Farley, Maintenance Coordinator, Pulp Manufacturing. Professor Kocurek reviewed background and scope of the microwave pretreatment project, and discussed the next phase of conducting a demonstration of the technology at a mill site. He reviewed the Canton mill chip preparation process and requested digester and pulping information so that pulping cooks which duplicated mill conditions could be

conducted at NCSU. Professor Kocurek discussed demonstration unit requirements and answered mill staff questions. Interest hosting in a demonstration is high and this mill has considerable experience in fielding novel environmental technologies. Using developed selection criteria, the Canton site receives a preliminary high score.

The Blue Ridge Paper Company has 18 digesters each of 3300 cubic feet, so digester size at this mill fits well with the prototype applicator and the large number of digesters makes the mill operations flexible. The mill pulps both bleached hardwood and softwood Kraft for use in printing, writing, and specialty papers.

Hardwood and softwood chip accepts and rejects were provided. Both untreated and pretreated chips are being pulped at NCSU. If the data demonstrate success under mill conditions, a second visit and evaluation by an expanded team is recommended.

The Blue Ridge mill has some location advantages – it is a short drive (20 miles) from Asheville, which is both a good site for conferences and a manageable drive from both Oak Ridge and NCSU. The mill has experienced some environmental protests and, as a result, has adopted and successfully fielded advanced chemical recovery technologies. However, as a result of attention from the environmental protection staffs of North Carolina, Tennessee and the federal government, the project team will need to be sure that environmental requirements are met and that the environmental protection staffs are fully briefed before and throughout any demonstration. As it may be possible to use this as a method of obtaining environmental approval for microwave pretreatment, this is not necessarily a problem, but care has to be taken that mill and project schedules are not disrupted. *Canton should be tentatively considered a semifinal 'short list' candidate mill for further evaluation*.

Domtar Industries, Nekoosa, Wisconsin. The Nekoosa mill was visit was hosted by Paul Braun, Senior Process Engineer, Pulp & Power Department; Chris Bodet, Pulp Mill Manager; Rob Goggins, Superintendent, Chip Preparation and Sulfite Mill; and Susan Molinarolo, Manager, Technical Services.

Professor Kocurek reviewed both background and scope of the microwave project, and the next phase of conducting a demonstration of the technology at a mill site. Interest in the project by the mill staff is good. He also discussed the Nekoosa chip preparation methods and requested information on digester and pulping conditions. Digester size at this mill, which ranges between 2600 and 3600 cubic feet, fits well with the prototype applicator and the 9 digesters at this mill provide operational flexibility.

Chips were requested, but were delayed due to inclement weather, a factor that could pose problems during a mid-winter demonstration. (The mill is 80 miles north of Madison, Wisconsin.) Both untreated and pretreated chips are being pulped at NCSU. If the data demonstrate success, a second visit and evaluation by an expanded team is recommended.

The Nekoosa mill has some special advantages. Because forest products is a major industry, Wisconsin state energy staff actively support, and, in some cases provide funding for, demonstrations of new pulp mill technologies. This could assist DOE in technology deployment. Second, the reorganization to form the “new” Domtar, which incorporates significant U.S. ownership and a number of smaller U.S. mills, is now complete. Both the Canadian staff of the “old” Domtar and the Weyerhaeuser staff transferring into the “new” Domtar have actively and ably partnered Agenda 2020 projects at Oak Ridge. Initial contacts with Domtar central management have been positive.

The major disadvantages lie in the distance of this site from ORNL, NCSU, and Washington, DC and the inclement winter weather. *The Nekoosa mill should not be placed on the semifinal ‘short list’ at this time, but should be tentatively considered an alternate candidate site*.

Georgia-Pacific, Palatka, Florida. The visit was hosted by Dr. Adel Kassebi, Technical Services Supervisor. Professor Kocurek the reviewed background and scope of the microwave pretreatment project, and discussed the next phase of conducting a demonstration of the technology at a mill site. He reviewed the Palatka mill chip preparation process and requested digester and pulping information so that comparable condition pulping cooks could be conducted at NCSU. Professor Kocurek discussed demonstration unit requirements and answered mill staff questions. Interest hosting in a demonstration is high. Using developed selection criteria, the Palatka site receives a preliminary high score.

Using developed selection criteria, the Palatka site receives a preliminary high score. Digesters are a bit larger than desired, but other criteria are very good. Hardwood and softwood chip accepts and rejects were provided. Both untreated and pretreated chips are being pulped at NCSU. If the data demonstrate success, a second visit and evaluation by an expanded team is recommended.

The Palatka mill has several unique advantages. As a mill, Palatka is perhaps the most flexible and versatile in the Southeast in terms of the large number of digesters, varied furnish, and varied products. The site, 65 miles southwest of Jacksonville, Florida is readily reached from the Jacksonville airport and, barring a hurricane, would be accessible to DOE and project team staffs most of the year. The mill appears to have very high quality, fresh furnish and to control furnish quality well.

Palatka also has a significant research background on which to draw. First, Adel Kassebi completed his education at NCSU and is very familiar with the faculty, staff, and equipment in the Wood and Paper Sciences Department. Professor Arthur Fricke, who serves as a consultant to this project, performed detailed, well-controlled evaluations of Palatka’s major softwood furnishes under a very wide range of experimental pulping conditions and consulted with the mill staff on recovery boiler operations. Additionally, Koch, which acquired Georgia-Pacific, has a lengthy and successful history in the development of high-quality equipment for emerging chemical processes. *Palatka should be tentatively considered a semifinal ‘short list’ candidate mill for further evaluation*.

International Paper Company, Riegelwood, North Carolina. Professor Kocurek visited with Wes Petrea, Process Engineer, and Tim Corey, Process Engineer Supervisor, at Riegelwood. He also met briefly with the Riegelwood head of manufacturing. During the visit Professor Kocurek the reviewed background and scope of the microwave pretreatment project, and discussed the next phase of conducting a demonstration of the technology at a mill site. He reviewed the Riegelwood mill chip preparation process and requested digester and pulping information so that parallel pulping cooks may be conducted at NCSU. Professor Kocurek discussed demonstration unit requirements and answered mill staff questions. Interest in hosting a demonstration is good.

Location of the Riegewood mill, which is approximately 60 miles southwest of Raleigh, is an advantage because it would permit access to NCSU staff and facilities as needed. The mill produces bleached pulp for use in bleached foodboard and paperboard.

The Riegelwood mill has 16 batch digesters ranging between 3200 and 6300 cubic feet. Riegelwood also has a particularly flexible arrangement because it uses relatively similar conditions for pulping both hardwood and softwood chips. This permits the staff to change digester furnish from hardwood to softwood as needed without upsetting the mill chemical processing and recycle systems.

The Riegelwood mill provided both hardwood and softwood accepts and rejects. The reject chips from this mill are particularly large – many in the 6 to 9 inch length range. Additionally, the mill receives approximately one-third of its chips from sawmills, which might result in variations in chip size or moisture.

The Riegelwood staff with whom Mike Kocurek met indicated that volatile emissions generated during a demonstration might require an additional permit. The mill staff also indicated that metal fragments or bullets from hunting which might be present in chips could require assessment. *Riegelwood should be tentatively considered a semifinal 'short list' candidate mill for further evaluation*

Thilmany Papers, Kaukauna, Wisconsin. Robert Erickson; Fiber Line Manager; Scott Gigot, Pulp Process Engineer; Jeff Osterberg, Process Engineer; and Mark Szczpanik, Utilities Superintendent hosted Professor Kocurek's visit. During his visit, professor Kocurek reviewed both background and scope of the microwave project, and the next phase of conducting a demonstration of the technology at a mill site. Interest in the project by the mill staff is high. Professor Kocurek also discussed Kaukana chip preparation methods and requested information on digester and pulping conditions. Using developed selection criteria, the Kaukana site receives an intermediate score. The mill's digester size, 6000 cubic feet, is slightly larger than would be preferred, and the small number of digesters (four) limits process flexibility.

The mill pulps both hardwood and softwood chips to produce unbleached grades of Kraft pulp. Both untreated and pretreated chips are being pulped at NCSU. If the data demonstrate success, a second visit and evaluation by an expanded team is recommended.

The Kaukana mill has some advantages. Because forest products is a major industry, Wisconsin state energy staff actively support, and, in some cases provide funding for, demonstrations of new pulp mill technologies. This could assist DOE in technology deployment. Thilmany is a smaller pulping company with more centralized decision making. The major disadvantages lie in the distance of this site from ORNL, NCSU, and Washington, DC and the inclement winter weather. The Kaukana should not be placed on the semifinal 'short list' at this time, but should be tentatively considered an alternate candidate site.

<u>Wausau Papers, Mosinee, Wisconsin.</u> Tony Marzofka, Vice President of Operations; Scott McNutt, Director of Manufacturing; and Brian Maahs, Pulp Mill Superintendent, hosted Professor Kocurek.

Professor Kocurek reviewed both background and scope of the microwave project, and the next phase of conducting a demonstration of the technology at a mill site. Interest in the project by the mill staff is high. Professor also discussed the Mosinee chip preparation methods and requested information on digester and pulping conditions. The digester size, which is 2600 cubic feet, fits well with the prototype applicator, and process flexibility will likely be reasonable for a site demonstration although the mill has six digesters. The mill pulps hardwood and softwood for use in unbleached industrial Kraft papers.

However, the Mosinee site receives an intermediate score, primarily because the mill uses a specialized furnish which might limit the utility of a site demonstration. The nontraditional chips consist of very thin veneer slice accepts and very large, dry reject chunks. The material appears to have been derived from wood composite or lumber operations as the larger chunks have smooth surfaces (probably the result of planing or veneer production) and some are coated with paint on the ends.

Mosinee mill advantages include the active support of the state energy agency which support, and, in some cases provide funding for, demonstrations of new pulp mill technologies. This could assist DOE in technology deployment. In addition to the non-traditional furnish, the major disadvantages lie in the distance of this site from ORNL, NCSU, and Washington, DC and the inclement winter weather.

The Mosinee mill should not be placed on the semifinal 'short list' at this time, but should be tentatively considered an alternate candidate site. The atypical furnish may be a temporary problem caused by the limited availability of wood chips in the north central region. This will be discussed with the mill contact.

The six mills which were visited provided large samples of both accept and reject (oversized or overthick) hardwood and softwood chips for pulping evaluation. (The Domtar Nekoosa mill provided only reject hardwood and softwood chips.) They also provided mill pulping conditions and discussed their process constraints and questions related to microwave pretreatment of chips with Mike Kocurek.

Also, mills have been particularly interested in evaluation of pretreatment of their entire chip stream to save energy, increase production, and improve or maintain pulp quality. Although this represents a significantly larger stream than oversized / overthick, interest by the mills is valuable because treatment of the whole stream would provide a full technology demonstration which would support deployment. This was discussed with NCSU staff during pulping trials and Richard Phillips, who is presently at NCSU, suggested a method which might permit a mill trial demonstration of the process on digester loads of batch chips.

Pulping studies on mill chips at mill conditions, with matched control and pretreated chips, are being conducted using a small (4 kW) batch applicator taken to NCSU. These studies are expected to be complete around the end of April. Mill pulping conditions (H-factor, chemicals, maximum temperature) are being replicated in these cooks.

Also, during his visits, Mike Kocurek made a list of areas where pulping runs would be required to determine whether mill conditions could be met. A typical example is the limitation of pulping liquor in batch digesters, which leaves some chips out of the liquor. These conditions are being evaluated as well as possible.

Plans for Next Quarter: Initial small digester cooks of chips is at mill conditions are expected to be completed around the end of April. Staff from ORNL and CPI expect to work with Hasan Jameel and Mike Kocurek to evaluate potential sites for a mill demonstration trial of microwave pretreatment of oversized or mill run chips. Mills are very interested in evaluations of pulping their whole combined stream. Results from pulping untreated and pretreated chip streams from selected mills will be evaluated and a report prepared for release. A series of pulp cooks at progressively increasing sizes are planned later to improve understanding of process scale up and to ensure that we can meet mill quality goals.

Patent: Leitten, C. F., Jr.; Griffith, W. L.; and Compere, A. L. Method for improving separation of carbohydrates from wood pulping and wood or biomass hydrolyzate liquors. U.S. Patent Application Serial No. 11/563,286.

Publications/Presentations: None.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.1	Complete preliminary small digester experiments evaluating low-temperature and low-cooking-chemicals pulping	10/31/2001	11/02/2001	Presented as TAPPI Pulping Conference paper
1.2	Complete draft preconceptual design report	10/31/2001	09/30/2001	
1.3	Initiate softwood studies	09/30/2002	09/30/2002	
1.4	Larger cooks and papermaking evaluations	09/30/2002	09/30/2002	
2.1	Pretreatment tests of logs	03/31/2003	03/31/2003	
2.2	Complete initial handsheet and bleaching tests	09/30/2003	09/30/2003	
2.3	Initiate large cooks to prepare enough pulp for evaluation	12/31/2003	03/15/2004	Funding received late, equipment problems.
2.4	Handsheet and bleaching tests	09/30/2004	09/30/2004	
2.5	Select demonstration site	02/28/2007		
2.6	Develop plans for demonstration tests	09/30/2007		
2.7	Install microwave system in mill	12/31/2008		

Budget Data (as of 1 April 2007)

Phase / Budget Period			Approved Spending Plan			Actual Spent to Date		
			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	To						
Year 1	10/99	9/00	200000	108000	308000	68391	108000	176391
Year 2	10/00	9/01	350000	108000	458000	251715	108000	359715
Year 3[a]	10/01	9/02	350000	108000	458000	279120	108000	387120
Year 4[a]	10/02	9/03	372000	122200	494200	238722	110200	348922
Year 5	10/03	9/04	395000	134200	529200	308692	106200	414892
Year 6[b]	10/04	9/05	421000	104200	525200	212325	110200	322525
Year 7	10/05	9/06				253395	110200	363595
Year 8	10/06	9/07				125513	55100	180613
		Totals	2088000	684600	2772600	1737873	815900	2553773

[a]Only $250,000 in DOE funding was received during FY.
[b]Only $120,000 in DOE funding was received during FY.

Integrated Forest Products Refinery

Van Heiningen: University of Maine

GO14306

QUARTERLY PROGRESS REPORT

Project Title:	**Integrated Forest Products Refinery (IFPR)**
Covering Period:	January 1st, 2007 – March 31st, 2007
Date of Report:	April 30, 2007
Recipient:	**University of Maine, Corbett Hall, Orono, ME, 04469**
Award Number:	FC36-04GO14306
Other Partners:	International Paper

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Project Objectives:
Hemicelluloses will be extracted from residual wood chips prior to pulping. The extract is then recontacted with kraft fibers to increase pulp yield and quality. The remaining hemicelluloses in the extract constitute a feedstock for sugar based polymers and chemicals. The sugar-based polymers may replace fossil fuel based chemicals and resins in wood composites for the development of bio-composite materials.

The objectives of the project are organized in the following tasks:
1. Extraction of Hemicelluloses from Residual Wood Chips and its Integration in Pulp Production.
2. Development of Bio-Composite Materials using cellulosic fibers and polyesters.

The organizations which will work on the above listed 2 tasks are: The University of Maine and International Paper. Specifically, the University of Maine will address the laboratory experiments for tasks 1 and 2, while International Paper will take the lead on the industrial implementation of the findings of Task 1. The tasks will be executed over a period of 3 years.

In task 1 the benefits of hemicellulose extraction will be quantified in terms of: improved pulp yield and quality, decreased overall alkali consumption during kraft cooking, and a reduced organics load going to black liquor recovery. Extraction, pulping, oxygen delignification and pulp bleaching trials will be performed at the Pulp and Paper Process Development Center of the University of Maine to determine the optimum process conditions for the several operations involved in this task.

Task 2 concentrates on the production of Sheet Molding Compounds (SMCs) using natural fibers (such as micro-crystalline cellulose, wood pulps, and agricultural fibers rather than the commonly used glass fibers) and conventional polyester resin systems. The manufacturing operating parameters and properties of the SMCs will be determined, and the technical, process and marketing issues associated with the production of SMCs will be identified.

Background:
Managed forests in the United States have enormous untapped potential as sources of bioenergy and biomaterials. Realizing this potential requires an approach that integrates new separation and conversion technologies with existing forest products procurement, production and delivery infrastructure.

US chemical pulp mills receive more than 120 million dry tons of wood each year. The objective of this project is to evolve existing chemical pulp and wood product mills into Integrated Forest Products Refineries (IFPRs) that produce new biomaterials and export renewable energy while continuing to meet growing demand for pulp and paper products. The transition to IFPRs will be enabled by economic returns from improved ongoing pulp and paper production, while producing additional biomaterials and renewable energy.

In an IFPR it is most cost effective to use trees principally for solid wood production, while the residual wood fiber and biomass (bark) are used for pulp and power production respectively. The dominant alkaline pulping process is then used to release the highest quality fiber for pulp production from essentially all hardwood and softwood species with minimal environmental impact. The pulp yield is only about 50% because most of the hemicelluloses and almost all the lignin end up in the spent pulping stream, called black liquor. The black liquor is combusted for steam and electricity generation, while the dissolved inorganic cooking chemicals are recovered and recycled for pulping. Since the heating value of the carbohydrates is only half that of lignin, the combustion of dissolved hemicelluloses does not constitute optimal economical use of this resource. Therefore, in the present IFPR a significant amount of the hemicelluloses are extracted from the residual wood chips prior to pulping. The relatively pure extract of hemicellulose oligomers is then recontacted with the delignified fibers to increase pulp yield and quality. The remaining hemicelluloses in the extract may be used as a feedstock for the production of ethanol, sugar based polyester polymers or other chemicals. The waste streams of the conversion processes will be combined with the black liquor to recover the heat value, and recycle sodium and/or sulfur. The sugar-based polymers may serve to replace fossil fuel based chemicals and resins in wood composites for the development of bio-composite materials.

The economic, energy and environmental benefits of the project are estimated assuming a pulp yield increase of 2% compared to conventional alkaline pulping, and implementation of the hemicellulose extraction technology in 50% of the chemical pulp mills. The higher pulp yield leads to an extra pulp production of 1.2 million tons at a value of about \$600 million. The incremental pulp production is equivalent to an additional income of about \$30/ton pulp produced. For the same scenario it is envisioned that the mills will produce 2 million tons of sugar-based chemicals and resins, representing an additional revenue stream of \$2400 million/year or \$80/ton pulp produced. The production of hemicellulose-based polyesters could save up to 85×10^{12} BTU/yr in energy in the year 2020 and 218×10^{12} BTU/yr when fully implemented.

Summary of Previous Work Completed on Task 1:

Fundamentals of pure water extraction of Southern Mixed Hardwoods (SHM)

Hemicelluloses are predominantly removed during pre-extraction of SHM with water at 150 ºC. Xylan dissolves as oligosaccharides and then it depolymerizes slowly into monomeric xylose at longer extraction times (up to 500 minutes). No significant amount of furfural, was generated under the present extraction conditions. The xylan that remains in the wood is only slightly deacetylated. The percentage of 4-O-MGA in xylan retained in wood decreases significantly with increasing extraction time, and a linear relationship exists between xylan dissolution and 4-O-MGA removal during pre-extraction. A much smaller amount of glucomannan is initially removed as oligosaccharides. Further hydrolysis into monosugars is faster than found for dissolved xylan. Approximately 1% of cellulose (based on wood) dissolves mostly in polymeric form. This most likely represents low molecular weight cellulose in SHW. Arabinan and galactan are completely removed from wood as monomers at the end of the extraction process. During the first 30 minutes all acetyl groups are removed while still bound to oligosaccharides. Then acetic acid is released by deacetylation of the dissolved oligosaccharides. A significant amount of lignin (up to 5% based on wood) is extracted, part of which is still covalently bound to hemicelluloses.

Extraction/Kraft Cooking of Southern Mixed Hardwoods

Southern mixed hardwood chips obtained from International Paper were extracted with pure water and aqueous alkaline solutions. The extracted chips were then cooked at standard conditions (160 °C, 12-15% EA charge, L/W of 4.5 L/kg and variable H-factor). Variables were the temperature of extraction, the extraction time, the amount of alkali charged and the form of the alkaline species. It was found that about 10% of the organics may be extracted from the wood chips with pure water or 10% NaOH. Unfortunately the total yield of the final kraft pulp after water extraction is lower than that of the control (i.e. no extraction), and the yield loss increases with extraction temperature. For example at 150 °C the total pulp yield is more than 5% lower than the control, and at 160 °C the yield loss is almost 7 %. Extraction with 10 % NaOH also gives a lower pulp yield, similar to that obtained with pure water extraction. For pure water extraction the yield loss after cooking is mostly due to xylan loss, while the yield loss after alkaline extraction and cooking is due both to cellulose and xylan loss.

To avoid the large (5-7%) pulp yield loss experienced with water and caustic extraction, we have adopted a different approach. With this new approach we are now able to maintain the pulp yield (based on the original od weight of wood) to that of a kraft cook control. Other benefits from this approach are:

- A reduction in EA charge during cooking by 3%
- An increase in delignification rate of up to 30%
- A reduction in the amount of rejects at higher kappa numbers
- A decrease in organic load to the recovery boiler by up to 10% based on od wood

A US patent has been filed in December 2006 for modified extraction and cooking of hardwoods which maintains pulp yield (based on original unextracted wood) compared to the control.

Readsorption of Extract on Kraft Pulp

Adsorption experiments with the novel extract were conducted to study the hemicellulose adsorption on pulp. The extract was mixed in a plastic bag with the unbleached kraft pulp obtained after extraction and kraft cooking. After vacuum filtration, washing and drying, the adsorption yield and kappa number was measured. The results indicate that a yield increase of almost 2% can be achieved. The kappa number after adsorption was about 2 to 3 units higher than that of the brownstock pulp.

Bleaching of the Extracted and Non-Extracted Hardwood Pulps

Two hardwood pulps obtained after the novel extraction method (Kappa number of about 18 and 28% ISO brightness) performed on the mixed southern hardwood chips were bleached using a standard $D_0E_PD_1$ sequence. The ISO brightness obtained for two different chlorine dioxide (and NaOH) charges were 81.5 and 87%, exactly the same as for the control kraft pulp (also Kappa number of about 18 and 28% ISO brightness). This shows that the pre-extraction does not influence the bleaching response of the pulps.

Extraction/Kraft Cooking of Loblolly Pine

Loblolly Pine chips were extracted with pure water at temperatures varying from 160 to 190 °C for different times up to 90 minutes. It was found that the wood weight loss yield as well as the dissolved total sugar yield, total hemicellulosic sugar yield, polymeric sugar yield, cellulose yield and lignin yield at the different time and temperature conditions are all satisfactorily described by the H-factor variable. A maximum polymeric sugar yield of about 8 % (on od original wood) in the extract is obtained at an H-factor of about 500. The lignin content at this H-factor is about 0.5%. This level of extraction may be obtained by extraction for about 60 or 25 minutes at 160 and 170 ^{o}C respectively. Subsequent pulping of H-factor 500 hours water extracted chips to a kappa number of 30 leads to a 60 % increase in delignification rate compared to that of the control, but also to a pulp yield which is 6% lower than the control. At a pre-extracting H-factor of 200 hours the pulp yield loss is 3% lower than that of the control.

Physical Properties of Loblolly Pine Pulp after Extraction/Kraft Cooking

Physical properties were determined of Loblolly Pine pulp after water extraction at an H-factor of 400 hours and then kraft cooking at 170 °C maximum temperature. The liquor to wood ratio was 4.5 L/kg and the kraft cook sulfidity 30%. As before, the total yield of the pre-extracted kraft pulps was significantly lower than that of regular kraft control pulps of the same kappa number. The pre-extracted kraft pulps require much more energy to develop the same degree of freeness as the kraft control pulp. This is expected since the pre-extracted kraft pulps contain considerably less hemicelluloses. It was found that the specific volume (or bulk) is the same for the handsheets prepared from the pre-extracted kraft and kraft control pulps at the same degree of beating. However the tensile strength of the pre-extracted kraft pulp handsheets is 20% lower than the kraft control. This indicates that the lower hemicellulose content in the pre-extracted kraft pulp leads to poorer bonding between the fibers in the handsheets at the same degree of beating. Similarly the burst strength is reduced by about 30%. The tear-tensile curves of the water pre-extracted kraft pulps are essentially the same as that of the control kraft pulps cooks. This suggest that the fiber strength is essentially the same for the two pulps (as was also implied by the pulp viscosity results), but that the significant lower hemicellulose content of the pre-extracted fibers leads to a loss in bonding strength between the fibers. The results suggests that dry strength additives and a high refining energy are needed for the water pre-extracted kraft pulps to make paper and board of the same quality as those from made from kraft reference pulps.

Summary of Previous Work Completed on Task 2:

Production and Testing of Sheet Molding Compounds (SMCs)

A compression mold made of carbon steel was designed and fabricated to produce a Sheet Molding Compound (SMC) of approximately 18 cm by 18 cm. To make the molded SMC, an industrial B-stage SMC was acquired from AOC Resins. The B-stage SMC was successfully compression molded into C-stage SMC at the Advanced Engineered Wood Composites Center,(AEWC). Samples of 7mm x 3 mm x 45mm were cut from the SMC. These samples were dried to constant weight, then sanded to 1.8 mm thickness for DMTA analysis from -60ºC to 260°C at 1 Hz frequency and 1°C per minute ramp rate with a MKIV-Rheometrics DMTA to obtain physical property and environment aging data for the SMC. The Tan Delta for the hydro thermally soaked samples was determined with the DMTA and was used to make a hypothesis about the possible chemical reactions taking place in the SMC matrix. The hydrothermal tests simulate an accelerated exposure of the SMC to the environment.

Hydrothermal aging of the SMC has been completed with DMTA testing. The results agree with previous literature results in many aspects. The peaks indicating a thickening reaction for the polyester and metal oxide intensified with hydrothermal aging. The peak intensity for the glass transition of poly(vinyl acetate) decreased with aging as has been previously reported. However, HPLC results indicate that no acetic acid was formed during the hydrothermal aging. This disagrees with literature reports of the hydrothermal decomposition of poly(vinyl acetate) degrading to poly(vinyl alcohol) and acetate. The HPLC indicated the presence of large molecules in the aging solution. Considering the decrease in intensity for the poly(vinyl acetate), the large molecules may be some of the low profile additive. Since no glass transition peak was found for poly(vinyl alcohol) in previous work, and in the current investigation, the hypothesis that the large molecules in the aging solution may be poly(vinyl acetate) is plausible. Styrene was run using HPLC and compared with the aging solution and is shown to be leaching into water. A paper describing this work was submitted to the Journal of Material Science on February 14th 2007.

Inverse gas chromatography, (IGC), with bast Kenaf fibers has been done. Initial experimental work focused on getting meaningful results by proper packing of IGC columns and designing experiments. Experiments were conducted at three temperatures, 27°C, 35°C, 45°C. Inverse gas chromatography is done to characterize fiber and matrix surface energies to determine appropriate fiber sizing agents. The IGC has been completed with the Kenaf fibers. Acid base interactions for the bast Kenaf fibers have been determined. The acid base constants for the fibers will be compared to the acid base character of the matrix to determine appropriate sizing agents to maximize the interaction of the matrix with the fiber. The polyester prepreg material has been characterized. Because of the heterogeneous nature of the material new flow rates as well as operating parameters for the IGC experiments had to be determined.

Complete characterization of the polyester prepreg material from 30°C to 120°C, 15 fiber types, and chopped glass reinforcement have been completed and two manuscripts for submission to research journals are in rough draft stage at the time of this report.

Prepreg SMC was donated by AOC resins; Kenaf by Kenaf industries, glass from Owens Corning, and peroxide initiator from Akzo Nobel. This material will be used to compound SMC with glass and natural fibers.

Project Status Task 1

Adsorption of mixed southern hardwood extract on hardwood brown stock pulp is presently under investigation. Very promising results have been obtained, but because the work has not been completed, and because of possible intellectual property implications no further details can be provided at the present time.

The effect of temperature during pure water extraction of southern mixed hardwood on the properties of the extract and extracted wood chips has been completed. The development of the lignin-hemicellulose carbohydrate bonds during extraction has been determined. Because the chemical analysis is not fully completed at the end of the present quarter, this work will be reported during the next quarter

Project Status Task 2

Production and Testing of Sheet Molding Compounds (SMCs)

SMC material used for the experiments comprised of 28.10% dicyclopentadiene derived polyester resin molecular weight, MW, 12,000, 13.08% polystyrene, MW 250,000, 0.97% polyethylene powder, 6.27% styrene, 0.02% BHT, di-tertiarybutylhydroxytoluene, inhibitor, 0.48% compatibilizer, 0.15% of 5% pBQ, parabenzoquinone solution, 0.05% black pigment, 2.42% zinc stearate, 48.45% calcium carbonate. The SMC was crosslinked at 150˚C for 30 minutes. After crosslinking, the material was placed in double lined plastic bags and hammered to coarse particles. The coarse particles were ground to a powder with a mortar and pestle. The ground polymer was screened first by a 45 mesh screen then by a 60 mesh screen for a maximum particle size of 60 mesh. The power sample was heated for 3 days at150˚C to drive off volatiles. IGC columns were packed and the samples were conditioned at 150˚C in the IGC with 15 standard cubic centimeters, (sccm) per minute of helium until the flame ionizing detector recorded a background signal of <5 pA at 30˚C.

Chopped strand glass fibers, Owens Corning brand 973, were also run using IGC analysis. The glass fibers are sized for medium solubility resin compatibility with either polyester or vinyl ester resins. The strands were packed "as is" with lengths over 1 inch into the IGC column. The glass fibers were conditioned at 103˚C for 24 hours with 10 sccm/min of helium until the flame ionizing detector recorded a background signal of <5pA at 30˚C.

After conditioning the crosslinked composite material, experiments were conducted using a Hewlett-Packard HP 6850 Gas chromatograph equipped with an automatic injector and a fully automated Surface Measurements Systems SMS IGC with head space temperature control. Teflon column tubing with a 2.5 mm inner diameter was used with the HP 6850 at temperatures of 65˚C, 75˚C, 85˚C, 100˚C, 110˚C, and120˚C and custom silane treated glass tubes are used for the SMS IGC at the temperature of 30˚C, 35˚C, 40˚C, 100˚C, 110˚C, and 120˚C. The glass fibers were run using the SMS IGC at temperatures of 30˚C, 35˚C and 40˚C. Vapors of HPLC grade polar and non-polar probes were sampled by micro syringe and an infinite dilute concentration of probe was injected into the packed column and the retention time measure by a flame ionization detector.

Figure 1 shows how the dispersive energy is related to temperature compared to polystyrene and polyester values reported in the literature. Results for the dispersive component of the surface energy, γ_S^d, demonstrated slightly higher values for the SMC material, (46.46mJ/m^2 at 30°C), seen in Fig. 1, as compared to previous literature work for pure polyester, (43 mJ/m^2 at 30°C) and polystyrene, (39.15 mJ/m^2 at 30°C)

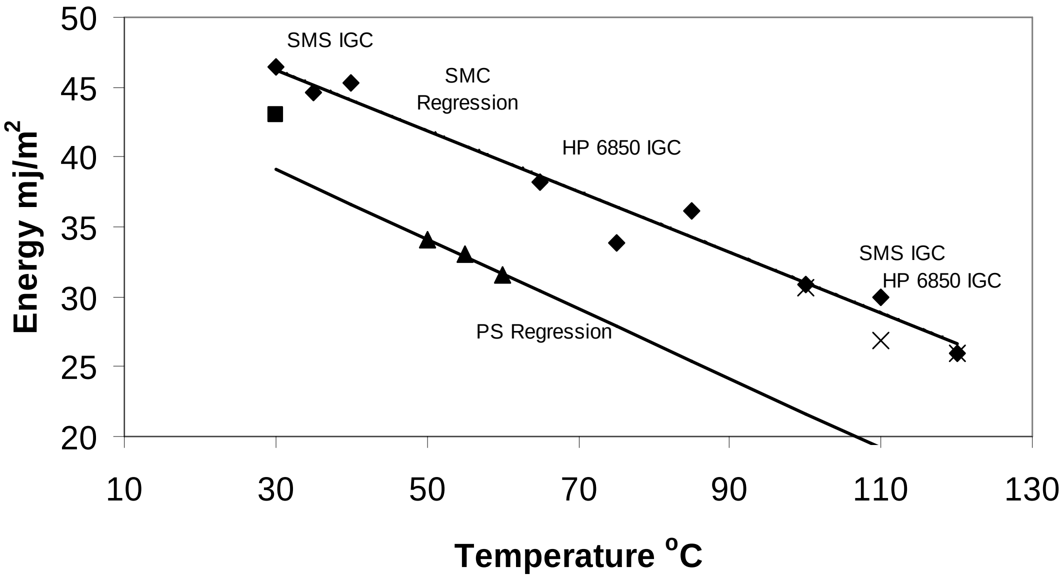

Figure 1. Dispersive energy for SMC material◆, polystyrene,(PS), ▲(12)*, polyester ■(3)*, and SMC material repeat at high temperature x, (* refers to reference). Three temperatures were run per experiment and the instrument used is listed by the data. SMC regression R^2=0.97, PS regression R^2=0.99.

In figure 2 Donnet's polarization method is used to determine the K_a and K_b for the polyester and this same method is used for the glass and various other fibers.

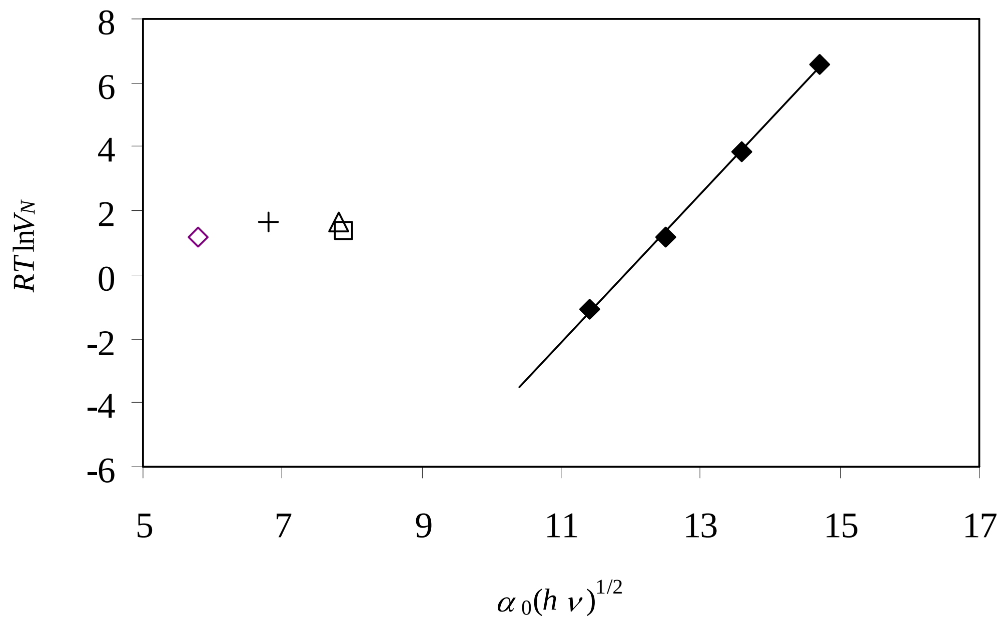

Figure 2. Polarization plot at 100˚C to determine acid base characteristics of the SMC material with n-alkanes◆, ethyl acetate☐, tetrahydrofuran +, chloroform Δ, acetone ◇

In Figure 2 the n-alkanes form a linear relationship representing the apolar characteristic of the polyester composite material. This relationship is used to determine γ_S^d. The polar interactions are represented by the probes that fall off the linear relationship and are used for determining K_a and K_b that is graphically demonstrated in Figure 3.

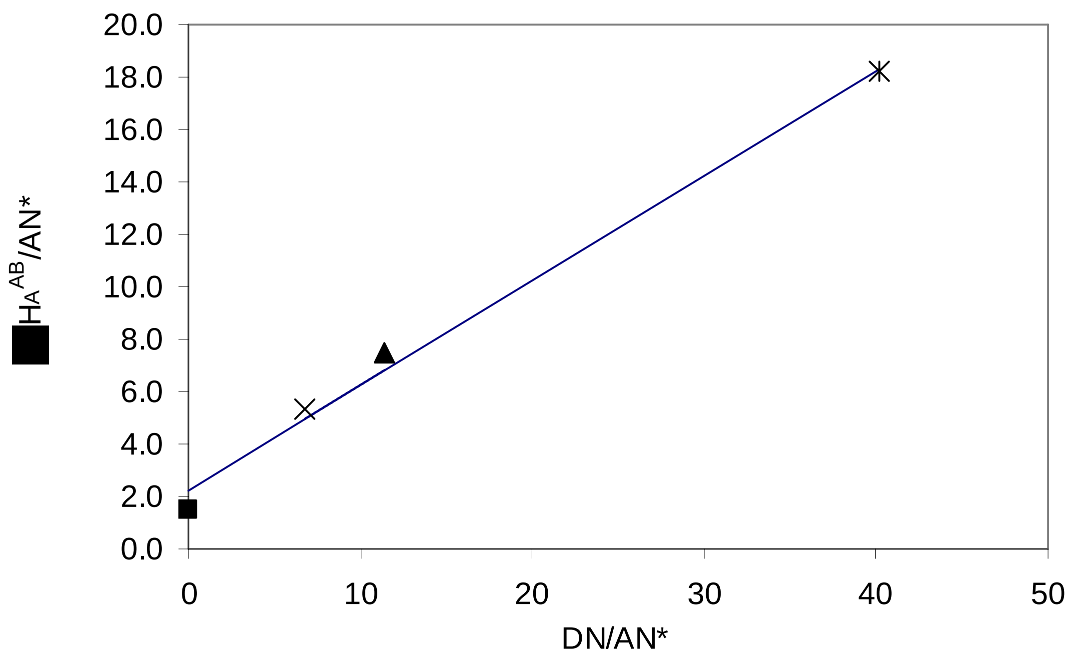

Figure 3. Relationship for determining Ka and Kb using data from the polarization method at 100˚C, 110˚C, and 120˚C for the SMC material with chloroform ■, ethyl acetate ▲, acetone ✕, and tetrahydrofuran ✳

From the slope and intercept of the data in Figure 3, K_a and K_b are determined. K_a was found to be 0.40 and K_b is 2.23. Previously, polystyrene has been reported to have a K_a of 0.06, and a K_b of 0.35[1], or 0.29 and 0.48[2]. The very high K_b for the composite material is most certainly because of the $CaCO_3$. The slightly higher K_a value may be due to the polyester.

With the K_a and K_b for the multi component polyester SMC material, the design of the reinforced composite can be concluded with knowledge of the K_a and K_b for the fiber reinforcement. Tze *et. al*.[2] reviewed how the acid-base interactions in a composite can be maximized to enhance the mechanical strength of a composite.

$$I_{a-b} = K_{a,f} K_{b,m} + K_{b,f} K_{a,m} \qquad (1)$$

Table 1. Sized and untreated cellulose fiber K_a and K_b values for calculating the interaction parameter with the SMC material

	Ka	Kb	Ia-b
SMC material	0.4	2.23	
Sized cellulose			
untreated cellulose, (lyocell)	0.36	0.39	0.96
Polyester sized glass	0.06	0.58	0.37
Amino-silanated	0.33	0.52	0.94
Phenylamino-silanated	0.32	0.42	0.88
Phenyl-silanated	0.34	0.43	0.93
Octadecyl-silanated	0.33	0.27	0.84
SMA-Grafted	0.42	0.57	1.16

From Table 1 it is apparent that the only sizing agent that gives a higher interaction parameter as compared to the untreated fiber is styrene-maleic anhydride, (SMA-Grafted). The reason is apparent considering that SMA-Grafted is the only sizing agent listed that increases the acidic character of the fiber. Due to the high K_b for the SMC material, any fiber reinforcement in the material will need to have a high acidic character to promote a good interaction. The polyester sized glass has a much lower interaction parameter as is compared to the biobased fibers indicating that the biobased fibers may have an advantage in overall cohesion within an SMC matrix.

Many other fiber types have been run using the IGC and dispersive energies calculated (see Table 2). The Ka and Kb's are still being determined.

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[1] Riedl B., Matuana L.M.; Encyclopedia of Surface and Colloid Science, (2002), 2842-2855

[2] Tze, W.T.Y, Wålinder M.E.P., Gardner D.J.; J. Adhesion Sci. Technol., vol 20, no. 8, (2006), 743-759

Table 2. Dispersive energy as a function of temperature and regressed value at 20˚C for various fiber types calculated by the Schultz and Lavielle method.

Fiber type	40(°C)	35(°C)	30(°C)	20(°C)	
	Dispersive energy mJ/m^2	Dispersive energy mJ/m^3	Dispersive energy mJ/m^4	Dispersive energy mJ/m^5	Regression Coefficient at 20˚C
flax	32.1	34.1	35.7	39.3	1.00
kenaf	34.0	35.3	36.9	39.7	1.00
kapok	34.7	35.0	36.4	37.8	0.85
hemp	33.1	34.8	36.4	39.7	1.00
cokos	33.6	34.8	36.1	38.5	1.00
cotton	35.7	35.3	36.4	36.9	0.46
rice	36.3	37.0	38.6	40.7	0.96
Poplar seed fibers	35.8	35.7	36.7	37.4	0.66
wheat straw	32.6	34.5	35.1	37.7	0.90
wheat pulp bleached	35.5	36.3	37.4	39.3	0.99
Reed	34.3	35.3	36.5	38.6	1.00
aspen	36.4	36.2	36.2	35.7	0.81
jute	40.6	39.2	40.0	39.1	0.17
abaca	38.2	37.2	37.0	35.7	0.90
sisal	38.0	36.6	35.4	32.7	1.00

One paper has been submitted for the earlier DMTA and HPLC work and two more papers will be derived from this work.

Plans for Next Quarter
Task 1:
Adsorption of Hemicellulose Extract on Southern Mixed Hardwood Pulp

1. An extraction and cooking procedure has been developed for hardwood which maintains pulp yield (based on original wood) compared to pulp of the same kappa number using conventional kraft cooking. Adsorption of this hardwood extract on the pre-extracted hardwood kraft pulp is presently under investigation. During the next quarter the adsorption procedures will be studied with the objective to obtain a maximum overall pulp yield.

2. Determine the bleaching response of the pre-extracted Southern Mixed Hardwood kraft pulps with adsorbed hemicellulose.

Fundamentals of pure water extraction of Southern Mixed Hardwoods

1. The effect of temperature during pure water extraction of southern mixed hardwood on the properties of the extract and extracted wood chips will be written up in the form of a paper.

2. The measurement of the development of the lignin-hemicellulose carbohydrate bonds during extraction will be completed.

Task 2:
Production and Testing of Sheet Molding Compounds (SMCs)

1. Fabricate SMC sheets at AEWC at University of Maine made with varying degrees of biobased reinforcement for testing and comparison with the standard "industrial" synthetic SMC. All material has been acquired and the B-Stage SMC is being fabricated.

2. From IGC analysis, sizing agents are being determined and will be purchased to make B-stage SMC fabricated with varying degrees of reinforcement, with and without sizing, and compression molded. After compression molding of the B-stage SMC, tan delta and storage modulus curves will be determined with DMTA, impact testing ASTM D 256-00, tensile testing ASTM D 638-01, and water uptake as has been done previously.

Presentations by Adriaan van Heiningen

A. van Heiningen, S. Shaler, J. Genco, H. Pendse, S-H. Yoon, S. Tunc, R. Jara, J. Paredes, S. Walton and J. Mitchell, "University of Maine Forest Bioproducts R & D", 93rd Annual Meeting PAPTAC, Montreal, QC, February 7th, 2007

A. van Heiningen, "A Concept and Analysis for Converting a Kraft Mill into a Forest Biorefinery", Finnish Paper Research Community Serving Europe, Helsinki, January 23, 2007

A. van Heiningen and S. Yoon, "Integration of Extraction of Hemicelluloses in Pulp Production", Final Review Meeting with International Paper, University of Maine, Orono, ME, January 10th, 2007

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.1a	Explore optimal extraction conditions for hardwood using ASE 100 of Dionex	3/31/05	10/31/05	
1.2a	Complete extraction and cooking of hardwood chips with profiling digester	8/31/05	7/31/05	
1.3a	Complete the deposition study of hardwood hemicelluloses on kraft pulp	8/31/05	6/30/07	Optimization study is ongoing
1.4a	Complete Bleaching of hard wood kraft pulp with adsorbed hemis	8/31/05	6/30/07	
2.1a	Produce SMCs from conventional polyesters	12/31/04	12/31/04	
2.1b	Physical properties and accelerated aging of conventional SMCs	8/31/05	3/31/06	
1.1b	Explore optimal extraction conditions for softwood chips	12/31/05	12/31/05	
2.2	Produce biobased SMCs	12/31/05	6/30/07	
1.2b	Complete softwood chips extraction and cooking with profiling digester	8/31/06	9/30/06	
1.3b	Complete the deposition study of softwood hemicelluloses on kraft pulp	8/31/06		Abandoned due to permanent pulp yield loss after extraction
1.4b	Complete oxygen delignification of soft wood kraft pulp with adsorbed hemis	8/31/06		
1.5	Complete bleaching of softwood and hardwood oxygen delignified pulp	8/31/06		
1.7	Go/No Go decision for feasibility study of hemi extraction technology at mill	8/31/06	5/01/07	IP will decide based on data produced in Tasks 1.3a and 1.4a
2.2	Complete SMC composites evaluation	6/30/07		
1.6	Completion of mass balance analysis of optimized and integrated process	6/30/07		
1	Experimental confirmation of integrated hemi extraction for soft and hardwood	6/30/07		
1+2	Complete final report - End of project	8/31/07		

Budget Data (12/31/06):

Quarter	From	To	Estimated Federal Share of Outlays*	Actual Federal Share of Outlays	Estimated Recipient Share of Outlays*	Actual Recipient Share of Outlays	Cumulative
4Q04	Start	12/31/04	$84,000	39,140.63	$25,000	18,522.38	$57,663.01
1Q05	1/1/05	3/31/05	$33,314	33,412.19	$40,524	12,263.43	$45,675.62
2Q05	4/1/05	6/30/05	$33,314	44,088.23	$40,524	8,645.04	$52,733.27
3Q05	7/1/05	9/30/05	$33,315	74,267.43	$40,524	2,290.42	$76,557.85
4Q05	10/1/05	12/31/05	$81,368	42,141.98	$39,702	32,870.90	$75,012.88
1Q06	1/1/06	3/31/06	$41,367	56,990.74	$39,702	49,640.83	$106,631.57
2Q06	4/1/06	6/30/06	$41,367	56,080.17	$39,702	23,866.58	$79,946.75
3Q06	7/1/06	9/30/06	$41,367	32,695.63	$39,702	13,191.42	$45,887.05
4Q06	10/1/06	12/31/06	$26,793	20,217.47	$29,703	58,858.00	$79,075.47
1Q07	1/1/07	3/31/07	$26,793	73,522.67	$29,703	-17,891.13	$55,631.54
2Q07	4/1/07	6/30/07	$26,793		$29,703		
3Q07	7/1/07	9/30/07	$26,793		$29,703		
Totals			**$496,584**	**$472,557**	**$424,192**	**$202, 258**	**$674,815**

Highly Energy Efficient Directed Green Liquor Utilization (D-GLU) Pulping

Lucia: NCSU

GO14308

QUARTERLY PROGRESS REPORT

Project Title:	HIGHLY ENERGY EFFICIENT D-GLU (DIRECTED GREEN LIQUOR UTILIZATION) PULPING
Covering Period:	January 1, 2007 through March 31, 2007
Date of Report:	30 April 2007
Recipient:	North Carolina State University (NCSU)
Award Number:	DE-FC36-04GO14308
Subcontractors:	Georgia Institute of Technology (GIT), Evergreen Pulp, Inc.
Other Partners:	N/A
Contact:	Lucian A. Lucia, 919.515.7707 (W), lucian.lucia@NCSU.edu
Project Team:	Dimitris Argyropoulos (NCSU), Sujit Banerjee (GIT), Rod Ledbetter (Evergreen Pulp, Inc.), Gibson Asuquo (DOE)
Upload Site:	https://www.eere-pmc.energy.gov/SubmitReports.aspx

Project Objective & Update: We are currently running mill support work to simulate the LoSolids® process that we intend to implement at Evergreen Pulp. We are currently seeking another project associate to support the implementation of the process at Evergreen and also to solicit increased industry support for our DOE project through D-GLUCOSE (Directed-Green Liquor Utilization Consortium Operating for Savings in Energy).

STATUS AS OF 30 APRIL 2007

I. MILESTONE 5: Complete engineering studies on implementing D-GLU at hardwood and softwood mills

II. MILESTONE 1: Answers to feasibility and planning of GL pulping process (pulp properties, secondary effects on mill) in target mill

Mill Trial Support

Additional laboratory pulping experiments were done at IPST to determine the total alkali requirements and split between the upper and lower cooks to reach the mill's target kappa no. of 35-36 when green liquor was used as part of the chemical make-up in the upper cook.

The procedures and equipment were the same as reported last quarter.

Chips from the Evergreen mill consisting of a mix of redwood and Douglas fir were used for the testing. Mill white, green, and black liquors were also used. The measured concentrations are shown in Table I.

Table I. Evergreen Liquor Composition for Lab Cooks

Liquor Composition	White	Green	Black
AA, g/L Na_2O	94.6	39.5	7.19
EA, g/L Na_2O	82.3	27.1	7.19
TTA, g/L Na_2O	122.1	111	15.6
% Sulfidity on AA	26.1	62.8	0
NaOH g/L actual chemical	90.3	19.0	9.3
Na_2S g/L actual chemical	30.9	31.2	0
Na_2CO_3 g/L actual chemical	47.0	122.3	14.3

Two cooking zones were simulated in the lab cooks, an upper zone and a lower zone. The basic conditions are shown in Table II. Preheated white or white + green liquor was added to the chips for the upper cook. After 30 minutes, 2 L of liquor was extracted, simulating the upper cook extraction flow. White liquor, diluted with a fixed volume of black liquor was added for the lower cook, which was conducted for 210 minutes at a final temperature of 165^{o}C. For control cooks without green liquor added, the total white liquor charge was split with 49% to the upper cook and 51% to the lower.

Previously reported results showed that at the maximum amount of green liquor added (0.6 L) with no corresponding reduction in white liquor AA the kappa no. was decreased by 30%. At the same mill kappa target of 35, the white liquor in the upper cook could be reduced by 10% with the maximum amount (0.6 L/kg) of green liquor added.

Additional cook results are shown in Table III. Adding the white liquor that was taken off the upper cook circulation (0.9% as AA) to the lower cook resulted in a kappa reduction of 17% compared to the control. With 0.6 L/kg green liquor in the upper cook, the mill can maintain their target kappa with a reduction in the upper cook AA from 9.1 to 8.2% at the same lower cook white liquor addition of 9.4%. With 0.4 l/kg of GL in the upper cook, a somewhat higher kappa number (36.4) can be achieved with the same upper cook (8.2%) and lower cook (9.4%) white liquor AA.

Table II. Conditions for Lab Cooks

Upper Cook		
Chip charge	o.d kg	1.0
Temp. start	^oC	100
Temp., final	^oC	135
Total L:W		4
Time cook	min.	30
UC extraction	L	2
Lower Cook		
Time ramp	min.	
Time, cook	min.	210
Temp.	^oC	165
White liquor	L	1.11
Black Liquor	L	0.8
L:W		3.91

Table III. Evergreen Simulation Lab Pulping Results

Sample	**Upper Cook**			**Lower Cook**		**Total Yield**	**Screened Yield**	**Rejects**	**Kappa no.**	**Residual Alkali**	
	WL AA	**Green Liquor**	**GL AA**	**WL AA**	**Final H factor**	**%**	**%**	**%**		**UC**	**LC**
		L	%							g/L Na_2O	
Control	9.1	0	-	9.4	2000	44.3	44.0	0.34	35.0	3.4	5.4
GL/Kraft	8.2	0.6	2.4	10.3	1945	42.7	42.4	0.29	29.0	6.4	9.1
GL/Kraft	8.2	0.6	2.4	9.4	1939	44.8	44.2	0.60	34.0	4.7	8.1
GL/Kraft	8.2	0.4	1.6	9.4	1928	44.1	43.9	0.22	36.4	7.4	10.0

Effect of Thiourea

Testing over the next quarter will address the effect of adding thiourea as a pulping additive for increased pulping efficiency and yield and potential synergy with the green liquor addition. The thiourea addition will be compared to the effect of anthraquinone (AQ).

? By synthesizing key lignin compounds we intend to investigate the mechanism of action of thiourea when used as an additive in Green Liquor pulping
? For this purpose we have recently hired a synthetic organic chemist with significant isolation and mechanistic skills to probe these important reactions.
? This aspect of the work intends to supply an in-depth understanding of the effect of thiourea on the pulping chemistry with the aim to eventually optimize our mill trial.

Hemicellulose Isolation

- ? During this quarter we also initiated work that focuses at understanding the amount of hemicelluloses that are dissolved in the spent green liquor.
- ? Our efforts will be focused at understanding and defining the optimum conditions for their maximum extraction in the stream
- ? An additional effort is focused at understanding and devising novel methods of coagulating the dissolved hemicelluloses from spent green liquor streams.

Understanding the Science Behind Green Liquor Pulping

Our efforts have continued in this front mainly by further elucidating the way our previous findings of sulfur carry-over with the lignin is occurring. Evidence is accumulating pointing in the direction of a new (yet undocumented) way that certain lignin structures strongly bind with elemental sulfur under Green Liquor Impregnation conditions. The consequences of this *"sulfur carry over"* phenomenon are very significant since they provide a means for augmenting the local sulphidity of the cook during subsequent kraft pulping. Obviously, this important finding is indented to be fully explored and charted for the benefit of our project.

Task 1

We are currently establishing a consortium of companies that includes North (USA, Canada) and South American (Brazil, Chile) and Finnish companies interested in implementing GL pulping.

A. Consortium Update

D-GLUCOSE (**D**irected-**G**reen Liquor **U**tilization **C**onsortium **O**perating for **S**avings in **E**nergy); Shown below are details of its development:

D-GLUCOSE **UPDATE:**

We are in the process of developing a WINGEMS model of a one and two bottleneck kraft pulp mill case using a model derived by Dr. Kirkman at NCSU:

1. Reduction of black liquor solids to the recovery boiler (1 & 2% yield increase)
2. Reduction of processed GL

We have drawn up the necessary paperwork and membership agreement for the consortium, but will hire a staff/student over the summer to develop the modifications to the base case model so that we can derive the energy and material balance. These numbers will then go into an economic model that Dr. Richard Phillips has developed here at NCSU for repurposing a "pulp mill to bioethanol producer" feasibility study. We anticipate that at the beginning of the summer, we will have all of the information necessary (including the beginning of the Evergreen Pulp Mill trial) to attract the following companies:

1. Georgia Pacific (US)

2. Aracruz (Brazil)
3. Susano (Brazil)
4. Botnia (Finland)
5. UPM (Finland)

B. Connections with Finland

- Under the auspice of the honorary Finnish Distinguished professorship that Dr. Argyropoulos recently received, we have initiated a series of contacts with Metso Botnia in Finland so as to transfer the technology and work with these experts who have done a very serious amount of work in this area in the past.
- It is anticipated that these contacts will allow us to further augment our technical know how and guide our future activities by avoiding otherwise unsuccessful scenarios.

These contacts are to be further leveraged to enhance our competitive position toward the creation of our industrial consortium

EVERGREEN PULP INC.

DATE: April 27, 2007

TO: Lucian Lucia

FROM: Andrew Kulchin

SUBJECT: D-GLU Trial at Evergreen Pulp Update 04272007

The mill installed a new lime kiln dust ESP during the annual maintenance shutdown in April. Several other unrelated environmental negotiations still trump the D-GLU project for environmental permitting; although, the D-GLU project is in the cue but it is not yet next in line.

We may be able to begin a trial involving the synergistic D-GLU digester additive, thiourea, in advance of the hardware and environmental modifications that the D-GLU system will require. The mill has run trials for other digester additives in the recent past and the stainless steel tubing delivery system is still in place. Based on the lab work, thiourea could have considerable value for lime kiln gas reduction in and of itself.

Sampling preparations have been made for safe handling and transportation of the large volume of samples that will be required for more detailed lab simulations.

Patents: We anticipate at this point allowing the consortium to handle all Intellectual Property issues.

Publications

1. Guerra, A.; Lucia, Lucian A.; Argyropoulos, Dimitris S. "Lignin from Various *Eucalyptus* Wood Species," *Holzforschung* **2007**, submitted.
2. Ban, W.; Song, J.; Lucia, L.A. "The Influence of Natural Biomaterials on the Absorbency and Transparency of Starch-Derived Films: An Optimization Study," *Ind. Eng. Chem. Res.* **2007**, accepted.
3. Guerra, A.; Gaspar, A.R.; Contreras, I.S.; Lucia, L.A.; Crestini, C.; Argyropoulos, D.S. "On the Propensity of Lignin to Associate: A Size Exclusion Chromatographic Study with Native Lignins Isolated from Different Wood Species," *Phytochem.* **2007**, in press.
4. Liu, Q.; Lucia, L. "New Manufacturing Study of Linerboard Materials." *TAPPI J.,* submitted, **2007.**
5. Liu, Q.; Ban, W.; Wang, S.; Lucia, L.A. "The Influence of Green Liquor and AQ-Modified Kraft Pulping on the Fiber Hexenuronic Acid and Carboxyl Group Content for High Lignin Pulps," *Appita* **2007**, accepted.
6. Filpponen, I.; Guerra, A.; Hai, A.; Lucia, L.A.; Argyropoulos, D.S. "Spectral Monitoring of the Formation and Degradation of Polysulfide Ions in Alkaline Conditions." *Ind. Eng. Chem. Res.* **2006**.
7. Ban, W.; Lucia, L.A. "Kinetic Profiling of Green Liquor Pretreatment." *Ind. Eng. Chem. Res.* **2006**.
8. Guerra, A., Filpponen, I., Lucia, L., Argyropoulos, D. S., "A Comparative Evaluation of Three Lignin Isolation Protocols, with Different Wood Species" *Journal of Agricultural & Food Chemistry* **2006**.
9. Filpponen, I., Guerra, A., Hai, A., Lucia, L., Argyropoulos, D. S., "Spectral Monitoring of the Formation and Degradation of Polysulfide Ions in Alkaline Conditions" *Industrial Enginering Chemistry Research* **2006**.
10. Guerra, A., Filpponen, I., Lucia, L., Saquing, C., Baumberger, S, Argyropoulos, D. S., "Toward a Better Understanding of the Lignin Isolation Process From Wood" *Journal of Agricultural & Food Chemistry* **2006**.
11. Liu, Q.; Singh, J.M.; Wang, S.; Ban, W.; Lucia, L.A. "The Influence of Green Liquor and AQ-Modified Kraft Pulping on Fiber Hexenuronic Acid and Carboxyl Group Content for Linerboard Grade Pulp." *Cell. Chem. Technol.* **2005**.

Presentations

12. New Insights into Eucalyptus Lignin & Pulping Chemistry. Third International Colloquium on Eucalyptus Pulp (Belo Horizonte, Brazil, March 4-7, 2007). Guerra, A.; Lucia, L.A.; Argyropoulos, D.S.
13. Kinetic and Molecular Features of Green Liquor-Modified Versus Conventional Kraft Pulping. 5th INWFPPC and 3rd ISETPP Conference (Guangzhou, China, November 8-10, 2006). Lucia, L.A.; Filpponen, I.; Guerra, A.; Ban, W.; Argyropoulos, D.SLucia, L.A. "A Quantitative Comparison of the Kinetic Features of Green Liquor-Modified Versus Conventional Kraft Pulping," Wood and Paper Science Departmental Graduate Seminar Series (Raleigh, NC, 19 January 2006).
14. Banerjee, S.; Lucia, L.A.; Jacobson, A. "Green Liquor Pretreatment Pulping: Importance of Diffusion." TAPPI High Yield Symposium: Growing Yield from the Ground Up. Atlanta, GA; May 16-18, 2006.
15. Lucia, L. A*., Filpponen, I., Guerra, A., Ban, W., Argyropoulos, D., S. *"Kinetic & Molecular*

Features of Green Liquor–Modified Versus Conventional Krafty Pulping" 3^{rd} International Symposium on Emerging Technologies of Pulping and Papermaking, Guangzhou, China, pp. 18-24, November 8-10, 2006

16. Filpponen, I., Guerra, A., Hai, A. A., Lucia, L. A*., Argyropoulos, D., S. "*Toward Understanding the Efficacy of Green Liquor Impregnation* " 9^{th} European Workshop for Lignocellulosics & Pulp, Vienna Austria , August 27^{th} -31^{st} , pp 254-257, 2006
17. Anderson Guerra, Ilari Filpponen, Lucian Lucia, Dimitris S. Argyropoulos* "Different Wood Scpecies Offer Different Yields, Lignin Structures and Molecular Weights when Isolated with the Same Method" 9^{th} ,European Workshop for Lignocellulosics & Pulp, Vienna Austria , August 27^{th} -31^{st} , pp. 274-277, 2006.
18. Filpponen *,I., Hai, AKM, Lucia, L.A., Argyropoulos, D.S. "Understanding the efficacy of green liquor pretreatment by using etherified lignin model compounds," 233^{rd}, American Chemical Society Meeting & Exposition, Atlanta, Georgia, March 26 - 30, 2006.
19. Argyropoulos,*D. S., Guerra, A., Xavier, A., Baumberger, S., Evtuguin, "Yield, Functional Groups & Molecular Weights, for Cost E41 Lignins", Cost E41 on "Analytical Tools with Applications to Wood and Pulping Chemistry" Grenoble France April 12^{th} -13^{th} 2006.
20. Guerra*, A., Xavier, A., Lucia, L., Argyropoulos, D. S. "The Effects of Milling Conditions on the Structure of Enzymatic Mild Acidolysis Lignin," 233^{rd}, American Chemical Society Meeting & Exposition, Atlanta, Georgia, March 26 - 30, 2006.
21. Hai*, A. K. M., Filpponen, I., Guerra, A., Xavier, A., Lucia, L.A., Argyropoulos, D.S. "A Kinetic Study of the Formation and Degradation of Inorganic Polysulfide Species: Quantitative Analysis by 1H-NMR Spectroscopy," 233^{rd}, American Chemical Society Meeting & Exposition, Atlanta, Georgia, March 26 - 30, 2006.
22. Fundamental Considerations for Green Liquor Pretreatment Technologies. 2005 AIChE Conference (Atlanta, GA, April 11-14, 2005). Lucia, L.A.; Ban, W.; Argyropoulos, D.S.
23. Understanding Lignin During Pulping and Bleaching. 2005 AIChE Conference (Atlanta, GA, April 11-14, 2005). Lucia, L.A.; Argyropoulos, D.S.
24. Lucia, L.A. "A Quantitative Comparison of the Kinetic Features of Green Liquor-Modified Versus Conventional Kraft Pulping," 2005 TAPPI Engineering, Pulping, and Environmental Conference (Philadelphia, PA, August 28031, 2005). Lucia, L.A.; Argyropoulos, D.S.; Ban, W.; Courchene, C.

MILESTONE STATUS TABLE:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments (1/31/2007)
1	Organization of pulping project mill consortium and agenda for implementation over four years	01/01/05	Currently pursuing: have interest from Aracruz, Stora-Enso, Botnia, Georgia Pacific, Susano	We are currently assembling the consortium of companies for mill implementation beyond Evergreen Pulp
2	Addressing the necessary research tasks to address mill implementation requirements at Evergreen Pulp, Inc.	08/15/05	04/01/2006	In progress with Andrew Kulchin at Evergreen Pulp, Inc.
3	Green liquor (GL) laboratory pulping studies associated with Task 2	12/01/05	04/15/2006	Baseline work has been completed to satisfaction of mill
4	Fundamental and applied determination of handling green liquor impregnation and liquor flows; work in coordination with Milestone 1 to eliminate any problems in mill; have supporting engineering design	06/30/06		In progress
5	Finalization for mill trial and implementation	07/01/06		Delayed by 10 months
6	Applied and fundamental studies for the pulping catalyst including role, fate, impact on pulp properties, impact on recovery, exploration for alternative organics	12/31/07		The reset of the tasks from this point on are part of phase 2 GL pulping
7	Selection of mill(s) that has conducted the appropriate mill studies for additive-enhanced GL pulping	04/01/08		Evergreen Pulp has expressed interest in this task as of 4/01/07
8	Exploration of mill specific issues related to odor, environmental issues, solid impacts, and corrosion for Milestone 4	06/30/08		
9	Mill trial	07/01/07		The PSD Review is nearly complete

10	Final Engineering report and lab work supporting mill trials for additive-enhanced GL impregnation	12/31/08		
11	Mill trial at selected sites based on engineering criteria developed with mill	09/30/09		
12	Final report for the industry and DOE	12/31/09		

MILESTONES:				
1	**MILESTONE 1: Answers to feasibility and planning of GL pulping process (pulp properties, secondary effects on mill) in target mill**	01/15/06	04/15/2006	We have addressed this as well as we will do at this point
2	**MILESTONE 2: Answers to the question of how the build up of scale in the pulp mill and downstream occurs**	12/31/06	01/31/2007	This milestone is not critical at this point – our work does not point to it being a "show stopper"
3	**MILESTONE 3: Answers to the possibility of significantly altering green liquor recausticization based on Task 5**	06/01/07		This is potentially a new avenue for used GL to be recycled – currenly not a high priority
4	**MILESTONE 4: Answers to the economic use and application of additive-enhance GL pulping**	02/01/08		Once the mill trial is established, this will be the second major phase of the project
5	**MILESTONE 5: Complete engineering studies on implementing D-GLU at hardwood and softwood mills**	03/31/07		We are currently working with a number of people to effect this engineering work at Evergreen Pulp; this will be part of the work done in the consortium for other companies
6	**MILESTONE 6: Demonstrate 15% energy savings at mill trials of D-GLU pulping technology**	07/01/07		This will be documented at Evergreen Pulp

BUDGET Quarterly Report Through 3/31/2007*

To	Estimated Federal Share of Outlays	Actual Federal Share of Outlays	*Estimated Recipient Share of Outlays	Actual Recipient Share of Outlays	Cumulative
9/30/2004	-	-		-	-
12/31/2004	-	-		-	-
3/31/2005	750	750.41	5,935.26	5,935.00	6,685.67
6/30/2005	37,713.92	37,713.92	5,757.81	5,920.00	43,633.92
9/30/2005	168,540.18	168,540.18	2,987.55	2,987.55	169,959.54
12/31/2005	86,152.73	86,152.73	12,391.61	1,419.36	102,938.15
3/31/2006	124,301.84	124,301.84	4,879.43	16,785.42	126,193.72
6/30/2006	26,988.22	26,988.22	41,579.51	1,891.88	68,567.73
9/30/2006	54,328.09	54,328.09	78,681.63	41,579.51	60,857.30
12/31/2006	89,397.43	89,397.43	98,388.59	21,085.29	110,482.72
3/31/2007	134,927.75	134,927.75	533,000.00	6,530.59	141,458.34
6/30/2007	324,178		533,000.00		-
9/30/2007	324,178		19,445.00		-
12/31/2007	324,178		19,445.00		-
3/31/2008	324,177		19,445.00		-
6/30/2008	324,177		19,445 .00		-
9/30/2008	324,177		19,445 .00		-
12/31/2008	150,000		12,963 .00		-
	2,818,166	723,100.57	1,260,283.00	107,676.26	830,776.83

* Updated quarterly

***This report corrects previous reports that lsted project cumulative in quarterly rows instead of quarterly cumulative.

Integration of the Mini-Sulfide Sulfite Anthraquinone (MSS-AQ) Pulping Process and Black Liquor Gasification in a Pulp Mill

Jameel: NC St U

GO16041

MSSAQ pulping March 2007:

In this quarter, we explore two different MSS-AQ procedures, MSS-AQ-700-22%TC with NaOH and other MSS-AQ-700-22% without NaOH. The most noteworthy difference between the procedures is the amount of NaOH used in the pulping liquor. Standard laboratory Kraft pulps were prepared and used as a baseline for comparisons made from the MSS-AQ. The breakdown of charged chemicals for the Kraft baseline cooks and the MSS-AQ cooks are shown in Table 1, and outline the differences between the procedures with respect to chemicals used and their relative amounts.

Table 1- Breakdown of chemicals charged in Kraft and alkaline sulfite methods

Pulping Method	**Total Chemical Charge (% as Na_2O on OD wood)**	**Component % of total chemical charge**				**AQ (% on OD wood)**
		Na_2SO_3	**Na_2S**	**NaOH**	**Na_2CO_3**	
Kraft	17.6% TTA	0	21	64	15	0
MSS-AQ (Different Split) With NaOH	22.0% TC	75	10	5	10	0.1
	22.0% TC	75	5	10	10	0.1
	22.0% TC	70	15	5	10	0.1
	22.0% TC	70	10	10	10	0.1
	22.0% TC	65	15	10	10	0.1
MSS-AQ (Different Split) Without NaOH						
	22.0% TC	85	5	0	10	0.1
	22.0% TC	80	10	0	10	0.1
	22.0% TC	75	15	0	10	0.1
	22.0% TC	70	20	0	10	0.1

Table 1- As seen in the table, the total chemical charge for the Kraft procedure is displayed as TTA, which represents the total titratable alkali, or the sum of NaOH, Na2S and Na2CO3 present in the liquor charged to the wood on oven dry basis. The nomenclature is different in sulfite pulping, where total chemical, TC, is used analogously to TTA and represents the sum of the components present pulping liquor.

The amounts of total titratable alkali, TTA, and total chemical, TC, used in these procedures fall in the typical range for each respective pulping method. Kraft pulping utilizes no Na_2SO_3, whereas alkaline sulfite pulping utilizes no Na_2S. The mini-sulfide sulfite pulping employs a mixture of these two chemicals, Na_2SO_3 being the major component, but only a small amount of NaOH. Na_2CO_3 represents a system dead load not having been converted to active cooking chemical. The amount of AQ charged in each procedure was held constant at 0.1 % on oven dry wood, ODw, basis.
The series of cooks was aimed at generating linerboard grade pulp with kappa number in the range 90-100. Under the selected operating parameters an h factor of 700 proved to produce pulps in this range. The operating parameters used in each laboratory cook procedure are shown in Table 2. The table displays the system active and total chemical charge, as well as the anthraquinone charge on ODw basis. Also shown was the pulping liquor to wood ratio, L/W, cooking temperature and h factor. The difference between active chemical and total chemical is the amount of Na_2CO_3 present in the pulping liquor.

Table 2- Operating parameters for Kraft and MSS-AQ methods

Item	**Units**	**Kraft**	**MSS-AQ**
Active Chemical	**% Na_2O**	**15.0**	**19.8**
Total Chemical	**% Na_2O**	**17.6**	**22.0**
AQ	**% on ODw**	**0.1**	**0.1**
L/W	**n.a.**	**4**	**4**
Temp.	**°C**	**160**	**160**
h factor	**n.a.**	**700**	**700**

MSS-AQ pulping (with and without NaOH) results:

The pulping data obtained through the outlined series of experimental cooks is outlined in Table 3. The table shows the black liquor pH, the total pulp yield, as well as the pulp kappa and brightness. The pH remains high in the Kraft. As expected, the black liquor pH is significantly lower in the MSS-AQ cooks which contain little or no NaOH. However, the black liquor pHs still remain above nine, where recrystallization of lignin becomes a problem.

MSSAQ Pulping with NaOH:

We explore MSS-AQ procedure with NaOH charges and without NaOH charge, keeping total chemical 22%, constant through out the experimental series. Following are the distribution of chemicals.

- Pulps generated from the MSS-AQ procedure is 10% greater than the kraft pulps at similar kappa number.
- Brightness point is higher by 12 points for MSS-AQ pulps.

Table 3- MSS-AQ Pulping with NaOH

Cook ID		(Na2SO3-Na2S-NaOH-Na2CO3	Kappa Number	Total Yield %	Brightness(unbleached) ISO	Black liquor PH
MSSAQ-1	22%TC	(75-5-10-10)	108.3	63.2	26.4	9.94
**MSSAQ-2	22%TC	(70-15-5-10)	104.3	63.1	23.2	10.02
MSSAQ-3	22%TC	(70-10-10-10)	111.0	61.5	22.8	10.10
**MSSAQ-4	22%TC	(75-10-5-10)	106.5	62.7	24.7	9.92
MSSAQ-5	22%TC	(65-15-10-10)	112.1	66.5	20.7	10.14
Kraft Baseline	15%AA/17.6%TC	Kraft Cook (25% sulfidity)	98.2	51.7	15.22	12.86

** Pulps taken for the strength properties measurement.

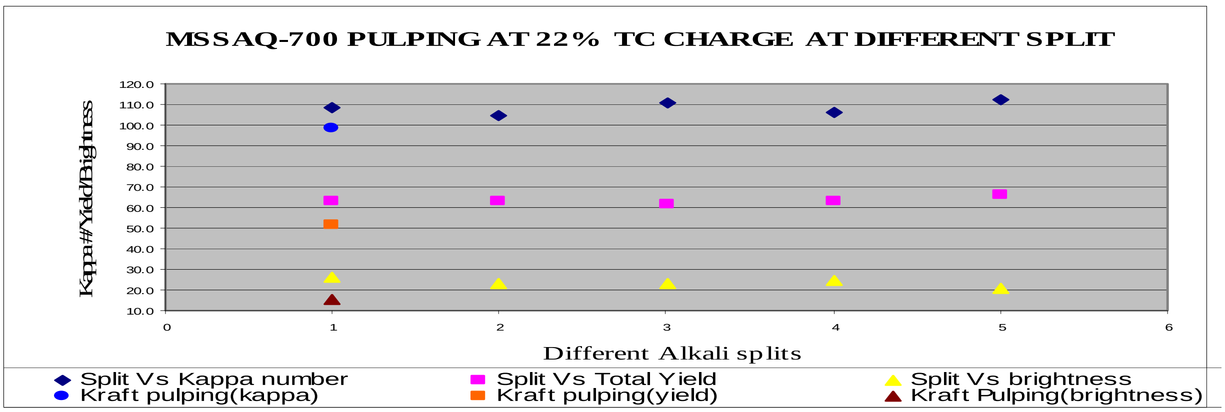

Figure 1 MSSAQ-700-22%TC- with NaOH

MSS-AQ Pulping without NaOH:

Strength properties measurement shows that MSSAQ-700-22% TC (with NaOH) pulps have comparable tensile and burst index but the tear index still remains lower than the base Kraft pulps.

Now we are trying to make MSSAQ pulps without NaOH, useful to eliminate lime kiln in the recovery cycle and also beneficial improving the brightness and strength properties. Following are the chemical split, total chemical was remain 22% and but there is no sodium hydroxide charge.

Table- 4 MSS-AQ Pulping without NaOH

Cook ID		(Na2SO3-Na2S-NaOH-Na2CO3)	Na2SO3:Na2S ratio	Kappa Number	Total Yield %	Brightness (unbleached) ISO	Black liquor PH
MSSAQ-1	22%TC**	(85-5-0-10)	17.0	102.2	66.3	27.44	9.61
MSSAQ-2	22%TC**	(80-10-0-10)	8.0	97.0	62.3	27.97	9.74
MSSAQ-3	22%TC**	(75-15-0-10)	5.0	105.5	61.0	23.1	9.98
MSSAQ-4	22%TC**	(70-20-0-10)	3.5	107.7	61.7	20.8	9.84
Kraft Baseline	15%AA/17.6%TC	Kraft Cook (25% sulfidity)	-	98.2	51.7	15.22	12.86

As shown in Figure 2, the MSS-AQ methods generate pulp yields significantly greater than the Kraft procedure at similar kappa. The MSS-AQ procedures resulted in yield increases ranging from about 10 to 14 % relative the Kraft baseline. The brightness of the MSSAQ-700-22%TC (without NaOH) was 6-12 points higher than the base kraft pulps.

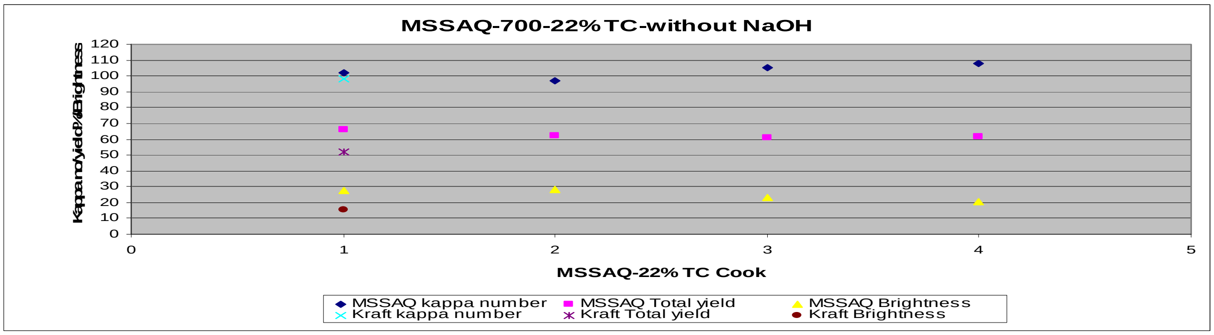

Figure 2- MSSAQ pulping without NaOH

Table 5- Strength properties of MSSAQ-700-22% TC Pulping

Pulp ID	Base kraft	Average Values				
PFI (revolutions)	Basis Wt. (g/m^2)	Apparent density (kg/m3)	Tear Index (mN*m2/g)	Tensile Index (N*m/g)	Burst Index ((kPa*m2/g)	CSF (mL)
3000	68.6	499.5	12.2	85.0	4.43	739
6000	67.1	546.9	9.8	99.3	5.36	655
9000	65.0	652.1	9.3	130.1	6.30	456
12000	64.7	657.1	8.2	132.7	6.42	255
Pulp ID	**MSSAQ-700-22%TC (75-10-5-10)**	**With NaOH**				
6000	66.4	576.1	10.1	101.1	5.97	688
8000	68.5	633.6	9.5	112.8	5.91	586
10000	67.8	634.1	8.7	112.5	5.87	405
12000	69.4	638.9	7.4	113.2	6.08	316
Pulp ID	**MSSAQ-700-22%TC (70-15-5-10)**	**With NaOH**				
3000	72.9	516.5	9.9	90.4	4.92	709
6000	70.9	629.6	9.4	96.7	6.05	638
9000	69.3	667.7	9.2	113.8	6.19	444
12000	70.5	681.5	8.2	114.0	6.25	268
Pulp ID	**MSSAQ-700-22%TC (85-5-0-10)**	**Without NaOH**				
3000	71.1	448.2	17.2	66.0	3.28	736
6000	69.5	582.1	15.9	95.6	4.68	684
9000	69.0	584.0	15.0	96.9	4.71	572
12000	68.0	658.6	13.4	111.6	5.66	356
Pulp ID	**MSSAQ-700-22%TC (80-10-0-10)**	**Without NaOH**				
3000	70.1	484.1	17.6	86.7	4.30	753
6000	68.5	574.0	16.3	101.0	5.33	693
9000	71.9	642.6	14.7	109.7	5.35	546
12000	68.1	659.5	13.5	118.0	6.32	288
****Pulp ID**	**MSSAQ-700-22%TC (75-15-0-10)**	**Without NaOH**				
3000	69.4	464.6	21.1	82.0	4.11	746
6000	70.6	542.5	17.2	98.8	5.09	685
9000	68.2	620.2	14.1	116.8	5.80	482
12000	68.3	662.2	13.4	122.1	6.25	284
Pulp ID	**MSSAQ-700-22%TC (70-20-0-10)**	**Without NaOH**				
3000	70.2	475.8	21.3	93.9	3.93	744
6000	69.9	547.1	19.1	98.1	5.14	682
9000	67.9	625.7	14.2	116.2	5.79	494
12000	68.2	664.6	14.0	116.3	6.26	285

** MSSAQ-700-22%TC (without NaOH) generate pulps of comparable strength with Kraft pulp

Comparison of kraft pulp and MSS-AQ pulp strength (Graphical presentation):

As the graph shows that the tear index was lower in the MSSAQ pulps obtained from NaOH charge but this problem is eliminated when we pulp without NaOH. In that case tear index was very fairly improved and 5-10 point higher than the Kraft pulps. Tensile and burst index were comparable to Kraft pulps.

As shown in Figure 3, the MSS-AQ pulp freeness response to refining is comparable to Kraft pulp. The sheet apparent density was calculated from the average hand sheet caliper and its' variation with level of refining is shown in Figure 4. The MSS-AQ pulps had an apparent density greater than the Kraft pulp, while the MSS-AQ pulps had values comparable to the Kraft pulp.

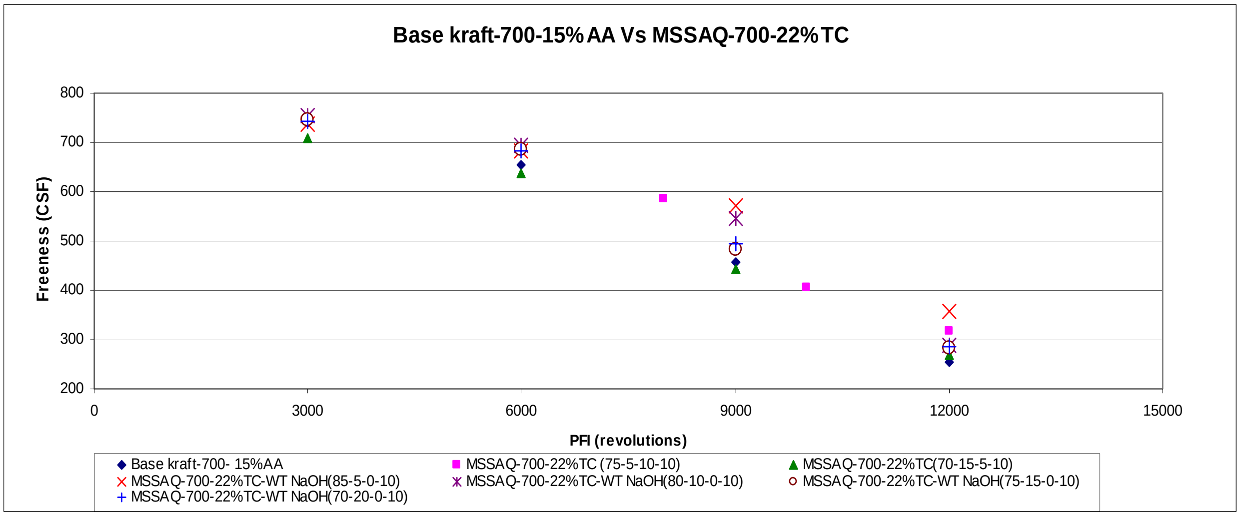

Figure 3- Freeness Vs PFI revolutions

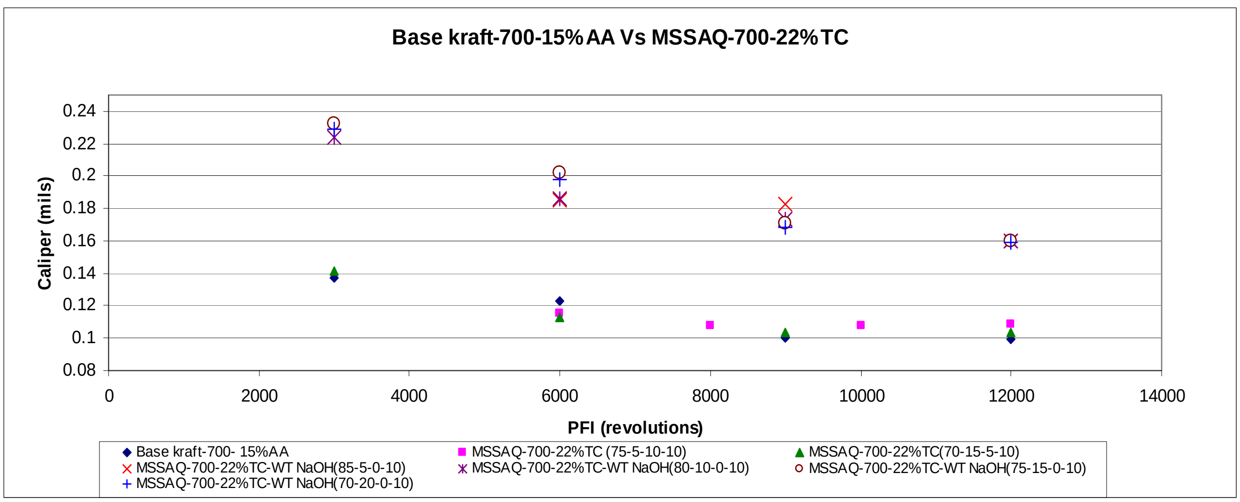

Figure 4- Caliper Vs PFI revolution

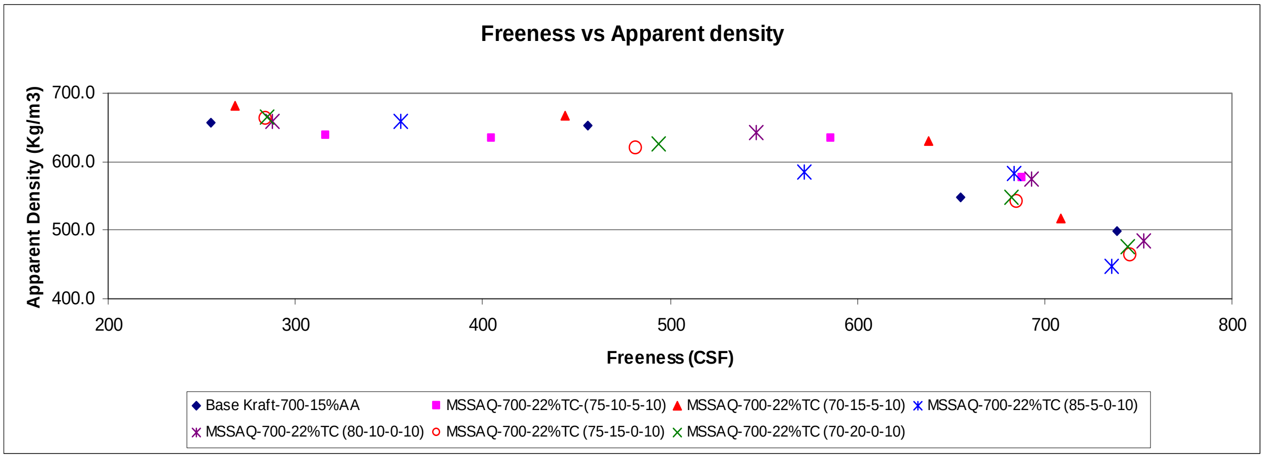

Figure 5- Freeness Vs Apparent density

The tensile index was plotted against the tear index for all samples in Figure 6. As shown, a tear index of MSSAQ pulps was lower for the comparable tensile index of kraft pulps. It was also seen that the response of the MSSAQ pulps without NaOH gives higher tear than the base kraft pulps at nearly comparable tensile values. As shown, at a comparable tensile index, the MSSAQ-700-22% TC (without NaOH) generated tear values 6 to 9 mN*m^2/g greater than those obtained for Kraft pulps. The promising results for tensile and tear are for MSSAQ-700-22%TC (80-10-0-10) and MSSAQ-700-22%TC (75-15-0-10).

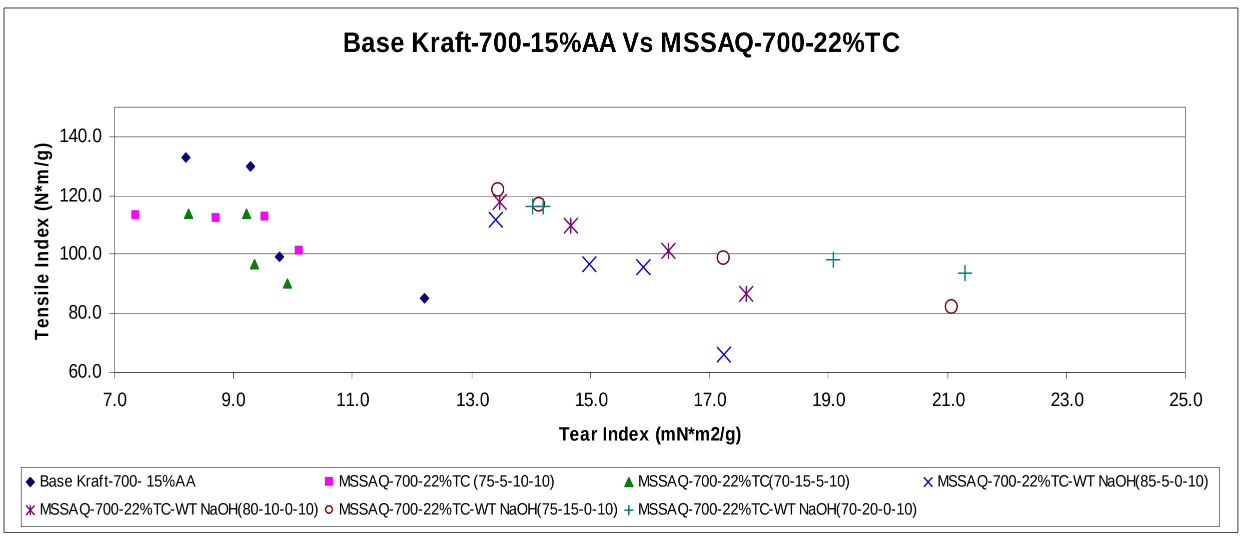

Figure 6- Tensile Index Vs Tear index

The pulp burst index was similarly plotted against the tear index for all pulps. The resulting graph is shown in Figure 7. As shown, The MSSAQ-700-22%TC (without NaOH) generated pulps comparable burst but higher tear values. Particularly the MSSAQ-700-22% TC (75-15-0-10) had best compared tensile and burst with higher tear index than kraft pulps. MSS-AQ (with NaOH), had tear index somewhat lower.

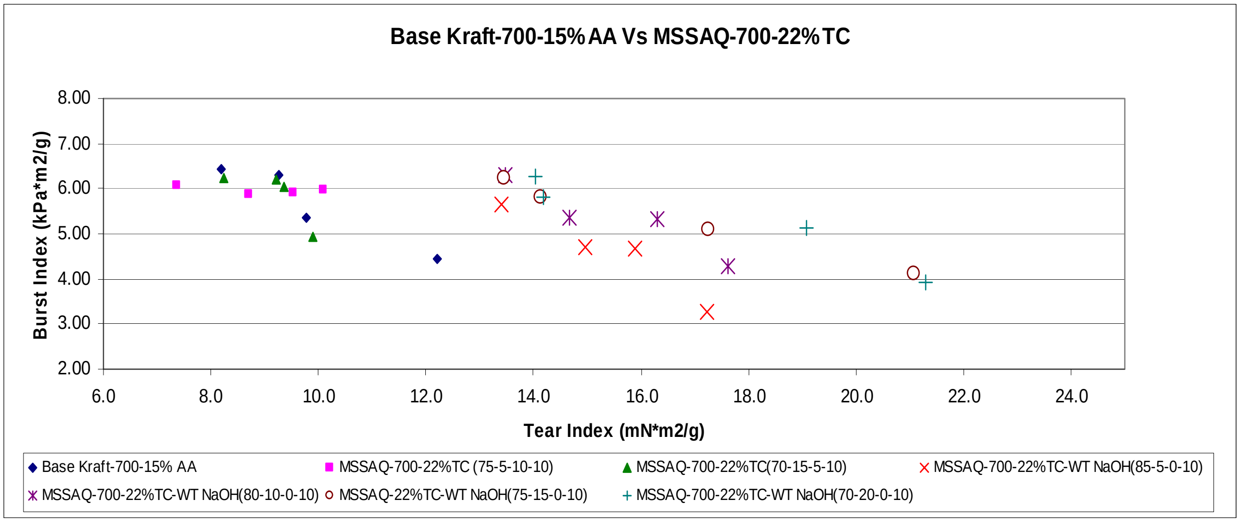

Figure 7- Burst Index Vs Tear Index

The results shown in Figures 6 and 7 indicate that the Kraft reference pulp generated sheets of comparable tensile strength, and burst strength relative the MSS AQ-700-22%TC (with NaOH) but lower tear index. MSS-AQ-700-22%TC (without NaOH) procedures produced pulps that were somewhat higher tear index and comparable tensile and burst than the kraft pulp.

Conclusions for MSS-AQ pulps

The purpose of the two different liquor compositions was to investigate the effect of NaOH and Na_2S concentrations on the pulping procedure and pulp properties. MSS-AQ (with NaOH) high kappa pulp had a total chemical 22%. From an operating cost perspective, it would be desirable to minimize the amount of NaOH used in the process, while optimizing the relative amounts of Na_2S and Na_2SO_3 as to their effect on process operation, output and pulp properties.

When comparing the process output results for the two procedures it is worth noting that there seems to be little difference in the cooking liquor initial pH and the black liquor final pH. Furthermore, when pulping to the same h factor, there is little difference in the resulting pulp yield, kappa and ISO brightness. This indicates that with regard to pulping operations and the listed pulp properties, there is little or no benefit of adding NaOH to the cooking liquor

When compared to the Kraft-AQ reference pulp, the MSS-AQ procedures produced pulps with a 10-15 % yield benefit and ISO brightness 3/2 times greater. The pulp refined little easy and had a slightly lower apparent sheet density. (In both the cases) At similar levels of tear index the MSS-AQ pulps also produced a comparable tensile and burst index. (MSS_AQ-without NaOH)

Future work:

In the next quarter, We will be generating bleached MSS-AQ pulps with suitable bleaching sequence and compare bleached strength properties with that of bleached kraft pulps.

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Direct Causticization for Black Liquor Gasification in a Circulating Fluidized Bed

Sinquefield: IPST (Ga Tech)

GO16042

Title: Direct Causticizing for Black Liquor Gasification in a Circulating Fluidized Bed
Project #: DE-FG36-06GO16042
Quarterly report #2
Period: Jan 1 – March 31, 2007
Recipient: Institute of paper Science and Technology at GA Tech via Georgia Tech Research Corporation

A series of gasification experiments has been completed in the IPST pressurized gasifer to compare the kinetics (carbon gasification) of Ti doped liquor versus un-doped liquor. The Weyerhaeuser New Bern liquor was used since we have extensive kinetic data from a recent PhD project using that liquor. Another series of PEFR experiments is under way to compare the influence of CO2 partial pressure on causticizing conversion at 850°C (this study) to conversion at 950°C (previous work). In all cases, char samples are analyzed for carbonate carbon and total carbon, as well as leached to compare the amount of recoverable hydroxide to the carbonate converted. We expect to have results of the chemical analysis for Quarter 2 experiments in Quarter 3.

In Quarter 3, we plan to continue with the experimental matrix outlined in the proposal.

Scott Sinquefield
April 29, 2007

Design and Demonstration of Multiport Cylinder Dryers

France: Argonne National Laboratory

Agr id:11430

QUARTERLY PROGRESS REPORT

Project Title: Development and Full-Scale Demonstration of Multiport Dryer Technology for the Forest Products Industry.

Covering Period: January 1, 2007 through March 31, 2007

Date of Report: April 30, 2007

Recipient: Argonne National Laboratory
Energy Systems Division, Bldg 212
9700 S. Cass Avenue
Argonne, IL 60439

Award Number: 49682

Subcontractors: Ability Engineering
Design Solutions, Inc.

Other Partners: The University of Illinois at Chicago
Kadant Johnson
International Paper

Contact(s): David France
Voice: 630-252-7361
Email: DFrance@uic.edu

Project Team: DOE-HQ contact: Drew Ronneberg

Project Objective: For retrofit applications, design, fabricate and test prototype Multiport Dryer inserts first for a full-scale test dryer and then for production paper machine dryers.

Background: Argonne National Laboratory (ANL) developed the Multiport Dryer design concept with the potential to increase drying effectiveness in paper-making machines from 20% to 50% over current technology. This Multiport Dryer technology represents only the second major innovation influencing steam dryer condensing heat transfer since the inception of the modern paper making process over 200 years ago. In a laboratory-scale proof-of-concept test, ANL demonstrated the feasibility of this technology. A series of steam condensing tests in ANL's Multiport Dryer Heat Transfer Test Facility showed that the condensing heat transfer coefficient for Multiport Dryers is approximately seven times greater than achieved in conventional dryers with spoiler-bar enhancement and 20 times greater than in conventional dryers without spoiler bars. These results translate into increased overall heat transfer rates in a steam drum dryer of up to 20% over spoiler bar technology and up to 50% over conventional technology without spoiler bars. Furthermore, the tests showed that dryer shell surface temperatures could be kept quite uniform in Multiport Dryers.

The Multiport Dryer technology has been advanced to the stage where a full-scale insert has been designed for retrofit into an existing full-size dryer for testing purposes. The insert has been partially manufactured and assembled. During that assembly, in the Kadant Johnson shortened version of their full-size test dryer, several items were identified for modification prior to completion of manufacturing of the full-length insert. These items are related to ease of field installation and to minimizing steam by-pass flow. Subsequently, modifications were designed addressing these items, and a subcontract was issued to complete the manufacturing of the full-size Multiport Dryer insert for testing in the Kadant Johnson full-scale test dryer. The materials necessary for this manufacturing process were ordered and received.

Status: During the reporting period, design drawings were modified for the Multiport Dryer insert for testing in the Kadant Johnson full-scale test dryer. The drawings now reflect the design modifications developed as a result of the earlier assembly of the Multiport Dryer insert in the shorted Kadant Johnson steam dryer. Fabrication of components commenced.

The Multiport Dryer insert will be tested simultaneously with spoiler bars in the Kadant Johnson full-scale steam drum. Installation requires a ten-foot attachment extension to properly place the Multiport Dryer insert in the steam drum. That attachment extension is part of the subcontract for completion of the manufacturing of the full-scale Multiport Dryer insert. During the reporting period, the attachment extension also received attention from the subcontractor.

Under a new DOE program, an experienced industrial contact was assigned to the Multiport Dryer project for the purpose of providing project guidance from an industrial prospective. During the reporting period, initial conversations were held under this DOE program, and large-scale testing considerations were discussed.

Plans for Next Quarter:

During the next fiscal quarter, Multiport Dryer insert manufacturing will continue, and there is a possibility that it will be completed. Upon completion, the insert will be delivered to Kadant Johnson in Three Rivers, Michigan for assembly and testing. ANL will interface with both the manufacturing and assembly processes. Assembly procedures will be prepared incorporating the as-built design modifications.

Future Plans: After the Multiport Dryer insert is assembled in the Kadant Johnson test dryer, heat transfer tests will be conducted. Heat transfer rates and surface temperatures will be measured in the Multiport Dryer section of the test dryer and in the spoiler bar section simultaneously. This procedure will give a direct comparison of the effectiveness of the two technologies. ANL and Kadant Johnson will work together in reducing, interpreting and presenting the test results.

Subsequent to the Kadant Johnson tests, the program will move into the Multiport Dryer demonstration phase in which an operating, production paper machine will be utilized. First, some insert redesign will be undertaken to provide flexibility in the Multiport Dryer insert to allow for retrofit into a large range of operating dryer drum sizes and designs. Then an operating paper machine suitable for Multiport Dryer testing will be sought. Subsequently, inserts will be prepared and installed in the paper machine. Drying rates and temperatures will be compared before and after the installation of the inserts.

Patents: U.S. Patent No. 6,397,489 B1 entitled, "Multiport Cylinder Dryer with Low Thermal Resistance and High Heat Transfer" issued on June 4, 2002 in the name of Stephen U. Choi (Disclosure No. ANL-IN-96-009 and DOE Case No. S-86200).

On-Line Fluidics Controlled Headbox

Aidun: IPST at Georgia Tech

GO10416

As of May 15, 2007, the PI has not submitted a status report for the quarter ending March 31, 2007.

The Lateral Corrugator

Schaepe: IPST at Georgia Tech

GO10616

QUARTERLY PROGRESS REPORT

Project Title: An Improved Method of Manufacturing Corrugated Boxes: Lateral Corrugator

Covering Period: January 1, 2007 – March 31, 2007

Date of Report: April 26, 2007

Recipient: Institute of Paper Science and Technology at Georgia Tech
500 10th St, NW
Atlanta, GA 30332-0620

Award Number: DE-FC36-01GO10616 M3

Subcontractors:

Other Partners: Temple-Inland Paperboard and Packaging, Smurfit-Stone Container Corp., MarquipWardUnited, Corrugated Gear, Albany International, Armstrong, Container Graphics Corporation, CUE, Harper-Love Adhesives, The Johnson Corporation, Corn Products International, Hardy Instrumentation, WIKA Instrument Corp., Chicago Electric, Pamarco Global Graphics, Bill Nikkel

Contact: Michael Schaepe, Senior Research Engineer, Principal Investigator
404-894-6640
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Perry Arrington, Robert Hall

Project Team: Gibson Asuquo, gibson.asuquo@go.doe.gov
Tim Ramsey, tim.ramsey@go.doe.gov
Beth Dwyer, beth.dwyer@go.doe.gov

Project Objective: The goal of this project is to develop a commercially viable lateral corrugating process. This includes designing and building a pilot lateral corrugator, testing and evaluating the pilot machine, and developing a strategy for commercialization.

Background:

Since paper is non-isotropic and fibers tend to orient in the machine direction, machine direction (MD) compressive strength of paper exceeds that of the cross-machine direction (CD). In a conventional corrugator, the paper machine direction is perpendicular to the flute direction. Therefore, a typical corrugated container cannot take advantage of the stronger compressive strength of the paper machine direction.

Experiments conducted at IPST demonstrated that combined-board with the linerboard MD orientation in the transverse direction of the combined-board generated box compression strength improvements of up to 30% over conventionally oriented board. Yet, with the medium MD orientated conventionally, flat crush, handling toughness, and board rigidity were maintained. It was found that a box utilizing 15% lighter materials with the linerboard transversely oriented generated comparable stacking strength to a conventional box.

A method to produce combined-board with the linerboard oriented in the transverse direction has been considered. This method of box manufacturing could reduce fiber consumption 15% and improve the compressive strength to weight ratio of corrugated shipping containers considerably, thereby **significantly reducing energy usage both in manufacturing and transportation**. The technology to produce such a combined-board would involve conventional fluting of the medium. The transverse orientation of the linerboard would be achieved through a sheeting operation. Single-facing and double-backing would utilize conventional but state-of-the-art corrugating technologies.

This project has been undertaken to construct a lateral corrugator and evaluate the resulting combined-board. The project will entail the development of a testing program, the design and construction of a pilot lateral corrugator, and the evaluation of conventional and lateral combined-board samples and boxes.

During the first year of this project two major program objectives were initiated. The lateral corrugator design was begun and experiments to explore the unique heat transfer opportunities of the lateral corrugator were undertaken. The design of the lateral corrugator proceeded as a retrofit to the newly completed double-backer at the IPST Industrial Engineering Center. The design incorporates a unique glue applicator system to allow the use of high solids adhesives and eliminates single-face festoons from the corrugating process. Both of these unique design features will **reduce the energy requirements** to produce combined-board. The heat transfer experiments aided in the selection of the post-heating elements to be used for the lateral corrugator.

The second year of the program focused on the fabrication and installation of the corrugating roll stack, the lateral corrugator drive system, the hydraulic and steam supply systems, and the glue machines. Also during the second year of the program the advantages of cut-to-width sheeting associated with lateral corrugator operation were investigated. Cut-to-width sheeting will reduce trim waste at the paper mill and box plant and simplify paper roll management. Since with lateral corrugating the paper is sheeted, matching paper-roll widths at the box plant would not be necessary and corrugator trim waste could be precisely controlled. For paper companies, the advantage of the lateral corrugator is very attractive since it would allow papermakers to reduce waste. With the cut-to-width capability of the lateral corrugator, paper companies could produce paper-roll widths to fully trim out paper machines. Also, papermakers could produce paper-rolls to optimize logistics, that is, maximize transportation efficiency. With these advantages **energy saving would be considerable**.

Future work will involve the integration of sheet feeders and splicing equipment into the lateral corrugator. The final result will be a pilot facility to produce lateral corrugated combined-board blanks of sufficient size to manufacture small boxes.

The IPST web page (http://www.ipst.gatech.edu/) includes information and pictures of the lateral corrugator. The lateral corrugator is highlighted under Research. To view the lateral corrugator program on the IPST web page, go to Corrugating and Converting, and then Lateral Corrugating. The web page includes a picture of the lateral corrugator, a schematic of how lateral corrugating works, a graph showing the strength enhancement benefits of lateral corrugating compared to conventional corrugating, and a description of the project.

Status:

Both glue machines have now been built and installed on the lateral corrugator including connection to the drive system. Glue dams are being fitted and the adhesive delivery and return systems are being plumbed. These will be completed within the next month.

The lateral corrugator was relocated to its finial position adjacent to the existing double-backer section. The move of a mere 5 feet was made difficult due to the heavy weight of the equipment and high center of gravity. The corrugator roll stack was removed to decrease the overall weight of the machine and lower the center of gravity. Railroad jacks were used to lift the lateral corrugator and trucks were placed under the feet. The machine was inched forward to its final location. Pictures of the move are included as Figures 1-2.

With the lateral corrugator in its final location, the steam system can now be connected. The steam lines and regulators will be installed within the next couple weeks.

Southland Electric has been engaged to wire the drive system. Several planning sessions have been held and electricians will begin wiring the lateral corrugator next month. Several electrical components are required including starters, disconnects, controllers and drive. Lead time on acquiring these devices is about two weeks. Once the wire is run and the electrical components acquired, electrical connection should take only a couple days. Still, frames and control stations will need to be built to house the machine drive and controls. Planning will allow the inclusion of sheet feeding system controls later in the project. This phase of electrical work should be completed mid way thought the next quarter.

Tri-Star Packaging, which is planned as the first location for commercial application of the lateral corrugator, is making progress constructing a corrugating facility in Tennessee. This plant will produce bulk boxes and plans are to incorporate a lateral corrugator with this corrugating facility. The lateral corrugator would be employed in the production of a triple wall corrugated structure to generate greater strength per unit weight. The integration of the lateral corrugator into a bulk box line is perhaps the best first application of the technology since corrugating speeds for bulk containers falls at the lower end of the production scale, and heavier weight material generates a disproportionately greater strength improvement over lighter weight materials. Specimens of triple wall construction have been built at IPST for testing. Controls and several permutations of possible construction configurations will be tested. The goal of this testing is to determine the impact of lateral corrugating on bulk box performance. All testing and comparisons to date have involved single-wall structures

Figure 1) Removing roll stack in preparation to move lateral corrugator
Figure 2) Using four railroad jacks, the lateral corrugator is lifted, trucks are placed under the feet and the machine is inched forward into its final location

Plans for Next Quarter:

The major goal for the upcoming quarter is to complete the electrical connection of the lateral corrugator and to demonstrate the unique roll stack configuration. To achieve this goal the hydraulic, drive, steam supply, and adhesive handling systems must all be connected and tested. Although all of these systems are in place they must be final plumbed and tied into a central control system.

Achieving this goal is a significant project milestone and DOE project management will be invited to attend a demonstration. Also, other parties, especially contributing and commercialization partners will be invited to attend demonstrations.

Specifically, the following tasks will be achieved in the next quarter:

- Glue dams will be fitted and the adhesive delivery and return systems will be connected
- Steam lines and regulators will be installed to provide steam to the corrugating and cure rolls
- Electrical connection will be completed including wiring, integrating hydraulics, providing trim control for the glue machines and double-backer, and building frames to mount all electric control devices.

Publications:

Previous quarterly project progress reports to DOE were distributed to selected IPST member companies and project commercial partners.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	Heat Transfer Experiments	June 2004	June 2004	
2	Splicer and Seam Analysis	June 2004	June 2004	
3	Address Economic and Production Considerations	Sept 2004	Sept 2004	
4	Identify Commercialization Partner	Dec 2004		Ongoing
5	Design Lateral Corrugator	July 2005	July 2005	
6	Build Lateral Corrugator			
6a	Roll Stack	Dec 2005	Dec 2005	
6b	Drive System and hydraulics	Jan 2006	Jan 2006	Design, fabrication and installation complete; electrical connection to be completed
6c	Glue Machines	Oct 2006	Jan 2007	
7	Demonstrate Lateral Corrugator (i.e., test roll stack configuration)	Nov 2006		Tasks to be completed: -Electrical connection -Plumb steam
8	Design Sheeter and Splicer	Feb 2007		Focus is on roll stack configuration (ID No. 6c)
9	Build and Integrate Sheeter/Splicer	Sept 2007		
10	Conduct Testing and Demonstrate Overall Concept	Dec 2007		
11	Final Report	Dec 2007		

DOE HQ Selected Milestones:

Recipient	CID	Milestones
Georgia Tech Research Corporation	DE- FC36 - 01GO 10616	Attend annual project review with industry
		Attend annual project review with industry
		Complete design of lateral corrugator
		Complete testing of lateral corrugator and demonstrate overall concept.

1) Attend annual project review with industry (2004):

- Michael Schaepe, "Lateral Corrugator", Agenda 2020 Technology Summit II, Peachtree City, GA, March 28-31, 2004
- Michael Schaepe, "Lateral Corrugator: An Improved Method of Manufacturing Corrugated Boxes", Session 32-3, TAPPI Paper Summit, Atlanta, GA, May 3-5, 2004
- Michael Schaepe, "An Improved Method of Manufacturing Corrugated Boxes", Program Management Group Meeting, Institute of Paper Science and Technology, Atlanta, GA, May 17, 2004
- Michael Schaepe, "An Improved Method of Manufacturing Corrugated Boxes: The Lateral Corrugator", Elective Research Consortium Meeting, Institute of Paper Science and Technology, Atlanta, GA, May 19, 2004
- Michael Schaepe, "Annual Project Report: An Improved Method of Manufacturing Corrugated Boxes", Institute of Paper Science and Technology, Atlanta, GA, August 10, 2004
- Michael Schaepe, "An Improved Method of Manufacturing Corrugated Boxes: The Lateral Corrugator", Poster Session, Forest Products Techno-Business Forum, Institute of Paper Science and Technology and the Center for Paper Business and Institute Studies, Atlanta, GA, October 26-27, 2004
- Michael Schaepe, "An Improved Method of Manufacturing Corrugated Boxes: The Lateral Corrugator", Poster Session and Facilities Tour, TAPPI International Corrugated Packaging Conference, Atlanta, GA, November 11, 2004

2) Attend annual project review with industry (2006):

- Forest Products Subprogram biannual Peer Review, Atlanta, GA, April 4-6th, 2006; a PowerPoint presentation and poster were presented
- Lateral corrugator, one program highlighted in the DOE's Energy Efficiency and Renewable Energy Publication Technology Solutions: Public-Private Partnerships Transforming Industry, May 15, 2006.
- Lateral corrugator project added to the DOE's commercialization tracking list
- Lateral corrugator project included in the DOE Energy Efficiency and Renewable Energy Industrial Technologies Program publication

3) Complete design of lateral corrugator –

- Lateral corrugator design completed July 2005
- Lateral corrugator glue machine design completed March 2006

4) Complete testing of lateral corrugator and demonstrate overall concept –

- Scheduled for completion December 2007

Budget Data:

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	6/30/03		24,521		142,869	**167,390**
3Q03	7/1/03	9/30/03		21,907		10,072	**31,979**
4Q03	10/1/03	12/31/03		14,093		3,104	**17,197**
1Q04	1/1/04	3/31/04		50,183		9,099	**59,282**
2Q04	4/1/04	6/30/04		80,454		103,110	**183,564**
3Q04	7/1/04	9/30/04		64,136		0	**64,136**
4Q04	10/1/04	12/31/04		88,733		36,522	**125,255**
1Q05	1/1/05	3/31/05		87,553		18,623	**106,176**
2Q05	4/1/05	6/30/05		57,620		9,732	**67,352**
3Q05	7/1/05	9/30/05		6,400		8,421	**14,821**
4Q05	10/1/05	12/31/05		16,259		8,421	**24,680**
1Q06	1/1/06	3/31/06		0		8,590	**8,590**
2Q06	4/1/06	6/30/06		0		6,493	**6,493**
3Q06	7/1/06	9/30/06		0		6,266	**6,266**
4Q06	10/1/06	12/31/06		19,567		2,001	**21,568**
1Q07	1/1/07	3/31/07		27,484		0	**27,484**
2Q07	4/1/07	6/30/07	40,000		70,000		**110,000**
3Q07	7/1/07	9/30/07	24,000		70,000		**94,000**
4Q07	10/1/07	12/31/07	14,028		51,531		**65,559**
Totals			**78,028**	**558,910**	**191,531**	**373,324**	**1,201,792**

Laser Ultrasonics Web Stiffness Sensor

Patterson: IPST at Georgia Tech, LBNL

ID14344

QUARTERLY PROGRESS REPORT

Project Title: Laser Ultrasonics Web Stiffness Sensor

Covering Period: January 1, 2007 to March 30, 2007

Date of Report: May 4, 2007

Recipient: Institute of Paper Science and Technology at Georgia Tech, Atlanta, GA

Award Number: DE-FC07-02ID14344

Subcontractors: none

Other Partners: ABB Industrial systems (Industrial partner)

Contact(s): Timothy Patterson, 404-894-4797, tim.patterson@ipst.gatech.edu

Project Team: DOE Program Manager: Asuquo Gibson; Research Team: IPST@GT: Gary Baum, David Huggins, collaborating at LBNL: Paul Ridgway

Project Objective: The objective is to provide a sensor that uses non-contact, laser ultrasonics to inspect the mechanical state of paper during the manufacturing process. Tasks include optimization of ultrasound generation on moving paper, development of interferometric detection schemes for on-line operation, and construction of a prototype for single point application on a paper machine.

Background: Laser ultrasonic methods have the potential to greatly extend the utility of on-line ultrasonic telemetry. Existing on-line ultrasonic techniques using contact transducers function only on board grades. Laser ultrasonic methods could perform at higher speeds without causing damage to lightweight papers. Laser ultrasonic methods are able to determine the bending stiffness of the paper. Bending stiffness is a property that is currently measured off-line on paper that determines end-product rigidity and is of great importance in a wide variety of paper grades. Laser ultrasonics could also provide single-sensor in-plane and out-of-plane characterization and give the first on-line gauge of stiffness orientation.

This project is the continuation of project DE-FC07-97ID13578. It is the combined efforts of two organizations with complementary experiences in paper physics and laser ultrasonics. Lawerence Berkeley National Laboratory (LNBL) is expert in the art of laser acoustic wave generation. Progress here is necessary to induce the largest possible ultrasonic disturbance without damaging the sheet. They developed a scanning-mirror, Mach-Zehnder interferometer that works well at high speeds on a web-simulator. IPST at GeorgiaTech contributes paper physics expertise and close relations with the paper industry as well as laser expertise. They have also demonstrated laser ultrasonic capabilities by constructing unique laboratory ultrasonic systems for use on paper. The group continues to work together to improve these technologies for web measurements. ABB is our industrial partner and is contributing web stablization technology, sensors enclosures, sensor design advices, and softwares.

Status:

In the previous quarterly report several items requiring actions were identified. These were

1. During a February 1 machine shutdown the sensor frame will be installed at the St. Helens mill. If time permits the actuator system for moving the sensor package on the frame will be tested. IPST personnel participating in the frame installation are T. Patterson and D. Huggins. P. Ridgeway from LBNL will also participate. This will be the first visit by the IPST personnel to the mill and will provide an orientation opportunity.
2. The new actuators will be installed in the sensor package. This will require a redesign of the mechanical structure and integration of the controller software into the existing control program.
3. The Graphical User interface for the control program needs to be redesigned to allow easy use by mill personnel.
4. A housing for the sensor package will be designed and built. This housing will protect the sensor package when it is off-machine and not in use.
5. Cabinets for the laser electronics and LUSS computer must be fabricated/purchased and the equipment installed.
6. Software for communication between the ABB system and the LUSS computer must be completed and tested.
7. The system is scheduled for installation at the mill during a machine shut down in late March 2007.

The actions taken were

1. The sensor frame was installed at the St. Helen's mill in February.
2. The new actuators for moving the generation laser have been installed in the sensor package. These actuators are more durable and capable of significantly higher speeds in comparison to the previously used actuators. This will increase both the utility and reliability of the system.
3. The graphical user interface s been partially redesigned, the remaining work will be completed prior to the end of May 2007.
4. The off sheet housing for the sensor package was designed, built and installed at the mill.
5. The St. Helens mill has provided an unused electrical cabinet for the project and has installed it adjacent to the paper machine.
6. The software for communication between the ABB and LUSS is not yet completed. The communication protocols have been established and programming is in progress.

7. The system was not installed on the machine in March. Delays in delivery of hardware (generation laser actuators, rotating mirror motor) made it impossible to have the system ready. The system will be installed during a May 31, 2007 shutdown. The system can only be installed during a shutdown.

In the upcoming quarter the following tasks will be completed

1. The system is scheduled for installation at the mill during a machine shut down on May 31, 2007.
2. The Graphical User interface for the control program needs to be redesigned to allow easy use by mill personnel will be completed in May.
3. Software for communication between the ABB system and the LUSS computer must be completed and tested.
4. The complete system will be tested at IPST during May 2007.
5. The system will begin operational testing at the mil following the installation.

Development of METHANE deNO$_X$ Reburning Process for Wastewood, Sludge, and Biomass Fired Stoker Boilers

Rabovitser: Gas Technology Institute

GO10418

QUARTERLY PROGRESS REPORT

Project Titles: Development of METHANE de-NOX® Reburning Process for Wood Waste and Biomass Fired Stoker Boilers

And

Utilization of Non-Condensable Gases as Reburn Fuel in FPI Wood Waste and Sludge-Fired Stoker Boilers

And

Advanced METHANE de-NOX for Woodwaste-Fired Stoker Boilers

Covering Period: January 1 to March 31, 2007

Date of Report: April 30, 2007

Recipient: Gas Technology Institute (GTI)
1700 Mount Prospect Road
Des Plaines, IL 60018-1804

Award Number: DE-FC36-99GO10418

Subcontractors: ESA Environmental Solutions, Sargent & Lundy LLC, Fluent Engineering, Reaction Engineering and University of Illinois at Chicago

Other Partners: Gas Research Institute, IGT Sustaining Membership Program, Detroit Stoker Company, Boise Cascade and Georgia-Pacific Corporation

Contact(s):

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Project Team: Gibson Asuquo, DOE project manager; Carrie Capps, DOE Project Monitor; David Highsmith, Georgia-Pacific Port Hudson Operations Project Manager; Tom Gilmore, Boise DeRidder mill project manager; Chad Stodola, Boise International Falls project manager; Larry Szymanski, ESA Environmental Solutions; Tom Giaier, Detroit Stoker Company project manager; Fluent Inc., Reaction Engineering Inc, University of Illinois at Chicago.

Project Objective: The primary project objective is to demonstrate the effectiveness of the METHANE de-NOX® (MdN) process for promoting more efficient use of wood waste and sludge for steam generation while keeping NO_x and CO emissions in compliance for biomass–fired stoker boilers in the Forest Products Industry (FPI). A second objective is to investigate the use of non-condensable gas (NCG) and stripper off gas (SOG) as reburn fuel in the MdN technology. This process extension enhances existing reburn technology benefits by further reducing natural gas consumption while providing an effective waste gas disposal option.

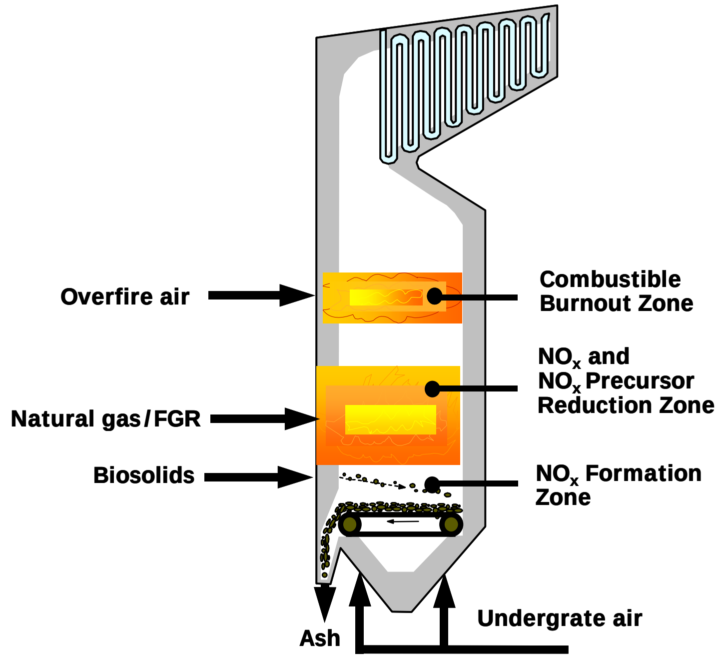


Figure 1. MdN reburn process uses fuel and air staging for combustion improvement and reduced NOx emissions

Background: The firing of biosolids can be limited by the low heating value and presence of bound nitrogen in these fuels. High moisture fuels result in inefficient combustion due to the latent heat of water vapor that is lost to the stack. High fuel moisture biosolids can also contribute to poor fuel distribution and piling, resulting in poor undergrate air distribution, uneven combustion at the grate, and increased emissions of CO and NO_x. Fuels with high nitrogen content such as secondary and tertiary treatment solids also contribute to increased NO_x emissions, limiting the ability to fire these fuels in boilers operating near their NO_x permit limit. Cofiring supplemental fuel such as natural gas through auxiliary burners helps to improve combustion effectiveness and to reduce NO_x emissions. However, these benefits are typically limited to the fractional input of the cofiring fuel.

The MdN reburn process uses both fuel- and air-staging to improve combustion and reduce boiler emissions. A small amount of natural gas and recirculated flue gas (FGR) is injected above the stoker grate to create a well-mixed, oxygen-deficient atmosphere immediately above the primary combustion zone (Figure 1). Air distribution between the undergrate and overfire air is also adjusted to reduce oxygen availability in the lower furnace and improve burnout in the upper furnace. Hotter and less-oxidizing conditions at the grate promote the decomposition of fuel-bound nitrogen compounds to reduce nitrogen oxide formation, while deeper air staging improves burnout in the upper furnace and allows operation with lower excess air. The added heat release and gas mixing at the stoker grate also improves combustion of difficult-to-burn woodwaste fuels. As a result, more high-moisture waste fuels can be burned, while reducing NO_x emissions, stabilizing combustion and improving boiler efficiency through reduced carbon losses and operation with lower excess air.

An MdN boiler retrofit consists of four primary components: 1) a natural gas supply and injection system, 2) a flue gas recirculation system, 3) air distribution adjustments that may include overfire air system modifications, and 4) control integration. Depending on many operational conditions and constraints, which vary widely for FPI woodwaste-fired boilers, the MdN process can reduce NO_x emissions by 30 –50% and improve boiler thermal efficiency up to 2%, while stabilizing grate combustion and increasing the ability to fire difficult-to-burn fuels.

The project has resulted in the successful demonstration of the MdN technology on a bark- and sludge-fired boiler at a Boise Cascade paper mill in International Falls, MN and baseline testing and evaluation of two additional bark-fired stoker boilers at other mills. As a result of this testing, a continuation project was awarded in 2001 under Cooperative Agreement DE-FC36-99GO10418 to extend the application and benefits of the technology to include a flexible combustion-based disposal option for NCG through their use as reburn fuel in bark and hog fuel boilers. In this project, an existing NCG collection and distribution system will be incorporated into a reburn system retrofit to be developed and demonstrated on a 200,000 lb/h MCR (maximum continuous rating) bark-fired boiler at a second Boise mill in DeRidder, Louisiana.

Another MdN system installed at a Georgia- Pacific paper mill at Port Hudson, LA in 2002 achieved NO_x reduction of over 30% on a 225,000-lb/h bark and gas-cofired boiler. A second continuation project awarded in May 2004 has resulted in the design and installation of a modified overfire air (OFA) and flue gas recirculation (FGR) system to further improve the boiler's energy and emissions performance.

Quarter Status for Base and Continuation Projects:
Currently, an MdN retrofit with NCG project is planned at Boise's DeRidder mill. An MdN with NCG design package by Energy Systems Associates (ESA), a licensee of the MdN technology, was already approved by DeRidder. GTI will provide technical assistance to ESA on an as-need basis during design, construction and system shakedown activities; a preliminary schedule estimated a mid-2007 completion. There is no work to report for this effort in the quarter.

Also, potential installation of an MdN overfire air system at Boise's I. Falls No. 2 Boiler remains on hold due to capital budget limitations.

Another project interest brought about by increased cost of natural gas is the possible utilization of highly reactive solid waste such as sander dust to replace a portion of MdN natural gas used for reburn fuel to control NOx emissions in hog-fuel stoker boilers. GTI has received preliminary interest from a plywood facility in Cleveland, Texas regarding this enhancement to the MdN process; however, this quarter there is no work to report.

The project final report is complete and submittal planned at the end of this time extension, 12/31/2007.

Base Project: Development of METHANE de-NOX® Reburning Process for Wood Waste and Biomass Fired Stoker Boilers

This development effort was divided into two phases. In Phase 1, a 300-MMBtu/h wood waste and sludge-fired stoker boiler at Boise Cascade's paper mill in International Falls, MN was retrofitted with MdN technology. Site testing was conducted in December 1999. Field results from 15 parametric tests proved the technology's effectiveness by meeting all projected performance goals as follows:

- Increased sludge firing by over 150% from the current 1.2-1.5 tph to 4 ton per hour
- Increased thermal efficiency for 40-100% load by 1 to 2%
- Reduced NO_x emissions by over 50% compared to previous cofiring mode
- Decreased natural gas input by 25% compared to the previous cofiring mode

Boise Cascade accepted the MdN retrofit and American Forest and Paper Association (AF&PA) recognized MdN's performance benefits with a 1999 Environmental and Energy Achievement Award in the Energy and Management and Innovation Category. The I. Falls system has logged over 4 years of continuous operation without a major problem reported since being placed into continuous full-scale operation on December 12, 1999.

Phase 2 activities in the base project consisted of a variety of tasks to promote the technology transfer and use of MdN within the Forest Products Industry. Please refer to the Milestone Status Table given later in the report for a task description.

Work Summary

Phase 1, Tasks 1 – 3 and Phase 2, Tasks 4 – 10; All work is complete.

Phase 2, Task 11 and 12; Tasks suspended: reallocated effort for development of an MdN Technology Manual per the Modified SOW.

Phase 2, Task 13 ; All work is complete. Commercialization activities have continued through collaboration with ESA, the technology licensee.

Continuation Project 1: Utilization of Non-Condensable Gases as Reburn Fuel in FPI Woodwaste and Sludge-Fired Stoker Boilers (MdN with NCGs)

The continuation project seeks to use waste gases produced during the pulp and paper process to enhance benefits already demonstrated by the base MdN process through a modification scheme that effectively processes these wastes to further improve energy and environmental performance from base MdN technology.

Background

Discussions with mill personnel at Boise's DeRidder Mill in Louisiana raised the possibility of using the mill's low volume high concentration (LVHC) and stripper off gases (SOG) as a portion of the reburn fuel for the system. This approach can reduce the use of expensive cofiring fuels such as oil and natural gas beyond the 25-30% reduction demonstrated with the conventional base reburn system at International Falls. By introducing LVHC and SOG in the primary combustion zone above the grate (Figure 2), the system should meet MACT control option requirements while reducing or eliminating the need for firing the auxiliary gas burners. NCG use has the potential to further increase gas savings from the reburn system due to substitution of NCG for a portion of the reburn gas (natural gas). In addition, if adequate and reliable destruction of NCG is demonstrated in the system, it will be possible to shut off the gas burners in one or both of the mill's bark-fired boilers when not needed for steam production.

MdN with NCGs performance objectives are:

- utilize to the maximum extent possible all low-volume high-concentration (LVHC) NCG and SOG waste gases consistent with safe and reliable operation
- decrease reburn natural gas consumption by more than 25% compared with original MdN operation
- reduce NO_x emission by over 40% compared to baseline (uncontrolled) conditions
- improve carbon burnout and increase boiler efficiency.

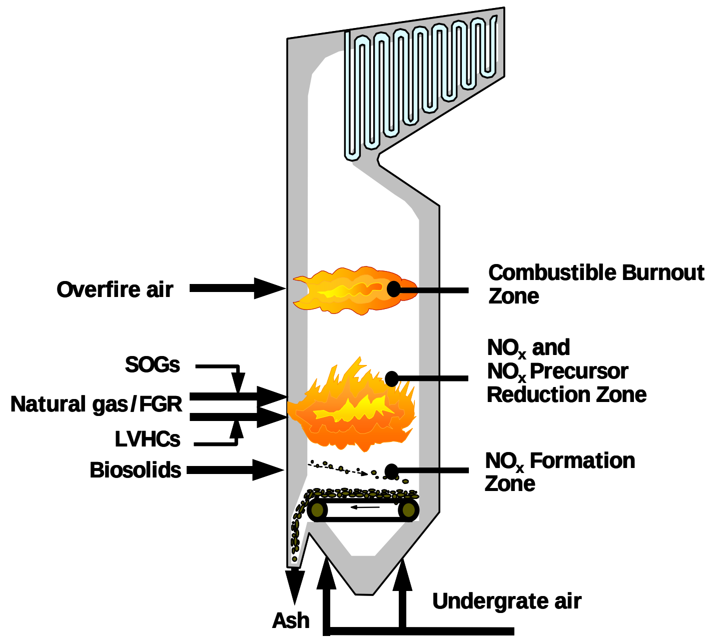


Figure 2. SOG and LVHC will be introduced separately into the natural gas/FGR injection headers

As noted in last quarter's report, the DeRidder mill is under new ownership and the planned MdN retrofit installation at the mill's No 2 Power Boiler is on hold. Subsequent discussions between GTI and DOE recognized the unlikelihood of proceeding with a field retrofit. Therefore a contractual modification to the statement of work and a Revised Statement of Objectives (SOO) was finalized with DOE in September 2005. The SOO reallocated planned project work and shifted funds to provide for preparation of an MdN Reburn Technology Manual (Manual) in lieu of the DeRidder MdN with NCG boiler retrofit.

Work Summary

Continuation Project 1: MdN with NCGs at DeRidder

Tasks 1 – 3; All work is complete.

Tasks 4 – 7; Task suspended, reallocated effort for development of an MdN Technology Manual.

Tasks 8 – 9; All work is complete.

Task 10; Project Management; Management of project activities and budget continued.

Continuation Project 2: Advanced METHANE de-NOX for Woodwaste-Fired Stoker Boilers

Tasks 1 – 4; All work is complete.

Task 5; Task suspended, reallocated effort for development of an MdN Technology Manual.

Revised Statement of Objectives (SOO)
A contractual modification was executed in September 2005, which changed the work scope; a Revised Statement of Objectives is summarized in the SOO Table on page 8. This change made for the development of an MdN Reburn Technology Manual. The manual includes MdN retrofit commercial designs for four (4) different boiler constructions as illustrated in Figure 3. Each commercial design includes boiler field test data; retrofit design protocols and retrofit engineering guidelines and specifications. The manual is complete and submittal is planned at project end, 12/31/07.

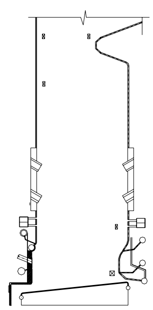
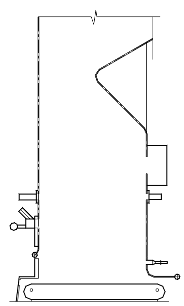
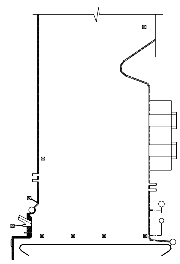
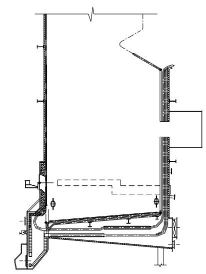
			
International Falls	DeRidder	Port Hudson	Wallula

Figure 3 Section views of Stoker Boilers having a Commercial Design Package prepared

Modified Statement of Work:

Task 1-17 International Falls; All work is complete.

Task 29 DeRidder Modeling, Design and Optimization: All work is complete.

Task 25 Port Hudson Modeling, Design and Optimization: All work is complete.

Task 31 Wallula Modeling, Design and Optimization: All work is complete.

Plans for Next Quarter:

1. Continue collaboration as needed with ESA and MdN with NCGs at DeRidder
2. Continue study of alternative reburn fuel options, especially use of highly reactive solids and solicit letters of interest from industrial clients for DOE funding consideration in 2007.

Patents and Publications/Presentations: None.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.0	**MdN at International Falls**			
1.1	Engineering Design for IF Unit #2	9/98	9/98	
1.2	Procurement/Installation at IF Unit #2	11/98	11/99[1]	
1.3	Field Parametric Testing of IF Unit #2	12/98	12/99[1]	
1.4	Long-term Performance Testing at IF Unit #2	7/00	8/00	
1.5	Pilot-Scale Testing at EPA	10/00	--	Task Suspended
1.6	Furnace Computer Modeling	01/01	02/01	
1.7	Boiler 2 Baseline Testing	08/00	04/01	
1.8	Boiler 3 Baseline Testing	08/00	11/01	
1.9	IF Unit #2 Simulation	12/00	10/01	
1.10	Data Processing and Analysis	02/01	12/01	
1.11	MdN Technology Database for Wood Firing	02/01		Task Suspended
1.12	MdN Engineering Design	02/01		Task Suspended
1.13	Commercialization and Technology Transfer	03/01	12/02	
1.14	Project Management and Reporting	04/01		See 3.6
2.0	**MdN with NCGs at DeRidder**			
2.1	Baseline Testing	04/02	06/02	
2.2	LVHC and HVLC Gas System Evaluation	05/02	03/03	
2.3	Modeling and Conceptual Design	08/02	06/03	
2.4	Detailed Engineering	12/02		Task Suspended
2.5	Procurement and Installation	04/03		Task Suspended
2.6	Parametric Testing	06/03		Task Suspended
2.7	Long-Term Testing	09/03		Task Suspended
2.8	Data Processing and Analysis	09/03	5/05	
2.9	Model Validation	11/03	4/05	
2.10	Project Management	09/03		See 3.6
3.0	**Advanced MdN at Port Hudson & DeRidder**			
3.1	Port Hudson Modeling and Conceptual Design	05/04	05/04	
3.2	Port Hudson Detailed Engineering and Installation	05/04	05/04	
3.3	Port Hudson Boiler Performance Testing	08/04	08/04	
3.4	Port Hudson Data Processing and Evaluation	08/04	08/04	
3.5	DeRidder Modeling and Conceptual Design Review	09/04	09/04	New Date 04/05[3]
3.6	Project Management	09/04		New Date 12/05[3]
	Final Report	03/31/05		New Date 12/31/07

Revised Statement of Objectives Status Table

Task Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1-17	**MdN at International Falls** Commercial MdN Design Package	12/06		New Date 12/31/06
29	**DeRidder Modeling, Conceptual Design and Optimization** CFD Optimization Study Conceptual Design Commercial Design Package	12/06		New Date 12/31/06
25	**Port Hudson Modeling, Conceptual Design and Optimization** CFD Optimization Study Conceptual Design Commercial MdN Design Package	12/06		New Date 12/31/06
31	**Wallula Modeling and Conceptual Design** CFD Modeling Study Conceptual Design Commercial MdN Design Package	12/06		New Date 12/31/06

Budget Data

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
	Start	9/30/2004		$ 1,410,810		$ 1,926,856	$ 3,337,666
4Q04	10/1/2004	12/31/2004	$ 26,831	$ 47,003	$ 5,380	$ 46,292	$ 93,295
1Q05	1/1/2005	3/31/2005	$ 50,000	$ 90,332	$ 5,382	$ 17,050	$ 107,382
2Q05	4/1/2005	6/30/2005	$ 100,000	$ 75,842	$ 5,382	$ 0	$ 75,842
3Q05	7/1/2005	9/30/2005	$ 120,000	$ 158,005	$ 5,382	$ 0	$ 158,005
4Q05	10/1/2005	12/31/2005	$ 120,000	$ 24,967	$ 5,382	$ 32,754	$ 57,720
1Q06	1/12006	3/31/2006		$ 11,910		$ 1,059	12,790
2Q06	4/1/2006	6/30/2006		$ 0		$ 0	$ 0
3Q06	7/1/200	9/30/2006		$ 0		$ 0	$ 0
Actual Totals				**$ 1,830,711**		**$ 2,024,011**	**$ 3,854,722**
Contract Totals				**$ 1,829,826**		**$2,024,011**	**$ 3,854,722**

*Update quarterly

Steam Cycle Washer for Unbleached Pulp

Salminen: Port Townsend Paper, INL

GO14304

QUARTERLY PROGRESS REPORT

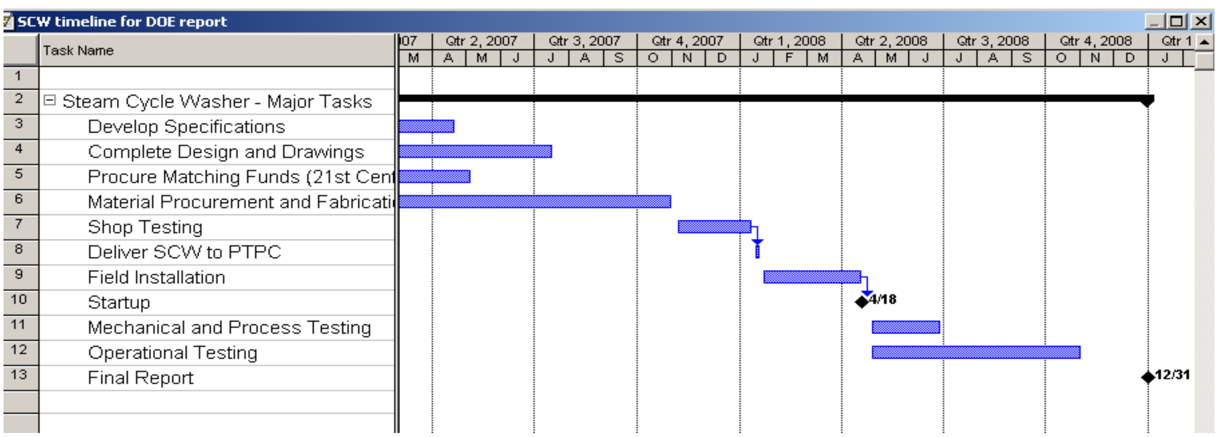
Project Title:	STEAM CYCLE WASHER
Covering Period:	1Q07 (January 1, 2007 to March 31, 2006)
Date of Report:	April 30, 2007
Recipient:	Port Townsend Paper Corporation
Award Number:	DE-FC36-04GO14304
Subcontractors:	21st Century Pulp & Paper, LLC 1329 State Street, Suite 202 Bellingham, WA 98225
Other Partners:	Idaho National Laboratory (INL) P.O. Box 1625 Idaho Falls, ID 83425-2210
Contacts:	Bruce McComas, Port Townsend Paper Corporation Vice President and Mill Manager 360-379-2158 brucem@ptpc.com Douglas Tolchin, 21st Century Pulp & Paper, LLC President 360-303-6122 doug@superpulp.com
Project Team:	Andy Karlsnes, Port Townsend Paper Corporation Recipient Business Officer 360-379-2074 andyk@ptpc.com Gibson Asuquo, Department of Energy DOE Project Officer 303-275-44910 gibson.asuquo@go.doe.gov Reijo Salminen, 21st Century Pulp & Paper, LLC Recipient Project Director 360-714-8416 reijo.salminen@yahoo.com Dean Harding, INL Advisory Engineer, Industrial and Material Technologies 208-526-6111 dean.harding@inl.gov

Project Objective: This project will provide a commercial scale demonstration of the Steam Cycle Washer (SCW) for Unbleached Pulp. It will show that the SCW will provide an innovative, energy efficient means to wash unbleached pulp using a pressure vessel charged with steam. The benefits of the SCW are that it will enhance mill profitability by significantly reducing energy costs, increasing product quality, and ensure environmental compliance exceeding current regulations.

Background: The feasibility of the SCW concept has been previously demonstrated through extensive pilot plant evaluations. Proprietary, unpublished test results confirm that the SCW will allow pulp mills to substantially reduce energy consumption, exceed environmental compliance requirements, increase fiber quality, and improve washer discharge consistency.

The Port Townsend Paper Corporation's pulp mill in Port Townsend, WA has been selected as the host site for conducting the demonstration of the SCW. This technology can be installed with minimal downtime and production impact to the mill by installing it in parallel to the existing brown stock washers. Startup and initial testing of the SCW is now not expected to take place until early 2008.

Steam Cycle Washer Major Tasks: (revised 1/21/07)



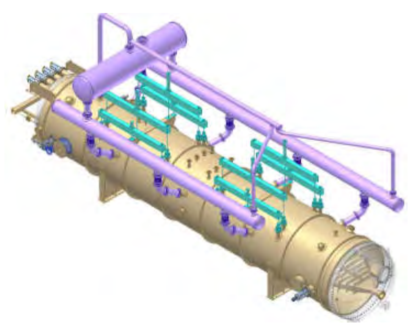
Status: This is the eleventh quarterly report (1Q07) of the SCW project. Activity in 1Q07 was minimal as 21st Century Pulp & Paper continued through the process of resolving their organizational and funding issues. It is anticipated that their issues will be resolved and a clear direction developed on how to proceed with the completion of the Steam Cycle Washer Project by the end of 2Q07. Checkmate Control Systems continued work on updating P&ID drawings, as well as designing a standard motor control interface between components to be supplied by 21st Century Pulp & Paper, the hydraulics vendor, and Port Townsend Paper Corporation. Fabrication on the washer internal components at Jesse Engineering was stopped. They have approximately 15% of their fabrication completed, and 90% of the materials cut and ready for assembly.

As the scope and design of the Steam Cycle Washer project was refined the expected cost to complete the demonstration project has significantly increased. 21st Century is presently completing the steps necessary to be able to attract investors into their company. When 21st Century's arbitration process is complete, and work with the securities lawyer is completed, funding for completion of the SCW fabrication can be obtained. This means that final design, drawing completion, and major fabrication activities on the project will be delayed another 2-3 months. I have modified the preceding schedule to show what I currently believe is a best-case scenario with respect to project timing.

On January 29th, 2007 Port Townsend Paper Corporation voluntarily filed for protection under Chapter 11 of the U.S. bankruptcy code. Under the consensual chapter 11 plan of reorganization PTPC will seek to implement an agreement that it has reached in principle with a majority of its bondholders. The agreement will allow PTPC to restructure its debt, while continuing to operate uninterrupted. I expect the entire process to take until mid 2007 as PTPC finalizes arrangements under the supervision of the U.S. courts. The Company is continuing to try to operate in a "business as usual" mode. It is our intent, subject to bankruptcy proceedings approvals, to continue to work with 21st Century Pulp & Paper to move ahead with the installation of the Steam Cycle Washer Project. It is not expected that this process will cause further delays in the SCW project installation.

Major Task 1: Design and Specifications for the SCW:

Subtask 1: Design and Specifications: 21st Century's work on the design completion slowed considerably during the quarter. The suspension of work on fabrication drawings by Alaskan Copper continued. Yet to be completed are the drawings for the ancillary tanks, piping, and equipment associated with the washer. At the end of 21st Century's arbitration process Alaskan Copper will complete these drawings and they will be delivered to Jesse Engineering for fabrication. Currently the expectations are that the final fabrication drawings will be completed in 3Q07. Bids for pressure vessel fabrication have been received. It is hoped that a fabrication contract can be released in 3Q07, with delivery of the pressure vessel to Jesse Engineering for final assembly expected in 4Q07 or 1Q07. Hypower has completed the design of the hydraulic system required for the SCW. They have not yet been contracted to convert their design sketches into final drawing for bid.



SCW External View

Subtask 2: Algorithms and P&Ids Development: Checkmate Control System's (CCSi) work on the development of the SCW process control software programming, and instrumentation design was slowed during the quarter. To continue P&ID development efforts Port Townsend Paper Corporation, with 21st Century's approval, diverted some of the DOE grant funds that are committed to 21st Century so that process and instrument design by CCSi could continue.

In general, this quarter has been spent mostly with the electrical design. To not cul-de-sac the PLC electrical design, the 115VAC digital inputs to were changed to 24VDC inputs as higher density modules can be used since module count was approaching the maximum of 30. We are still awaiting final confirmation from HyPower (the hydraulics package designer) regarding this change. Sketches, drawings, questions, and request for information on several topics were transmitted to several parties but, as yet, we have not received responses except from North Coast Electric (PLC vendor). Electrical design started, initially with resolving some questions regarding PTPC's standard MCC interface. Standard 11 x 17 drawing templates were generated for 21st Century Pulp & Paper. Research was done on UL508A and NEC409 interplay versus NEC670 wiring codes and their impact on industrial control panels. Also generated were integrated and detailed wiring diagram templates for the Rockwell 32-point, 24VDC digital input module (1769-IQ32 married to Interface Module 1492-IFM40D24A-2) and the 32-point digital output module (1769-OB32 married to Interface Modules 1492-XIM4024-16RF and 1492-XIM24-16RF). These will later be used as the basis to be fully populated with digital points from the P&ID drawings and Control System Component List database.

Subtask 3: Design Field Piping: Most of the tie-ins required for the SCW were completed during the annual plant shutdown in October 2005. Additional valve and piping relocations were completed during the mill's 4-day annual shutdown in November 2006. Design of additional field piping runs by Hipp Engineering and other field installation has been suspended until we start fabrication of the pressure vessel.

Major Task: 2: Fabrication and shop testing.

Subtask 1: Fabrication: All fabrication on the SCW has been suspended until 21st Century Pulp & Paper resolves their organizational issues and funding is obtained.

Alaskan Copper in Seattle, Washington was finalizing the engineering specifications and fabrication drawings for the pressure vessel, ancillary piping, and filtrate tanks when their work was suspended. Completion of the stress analysis of the SCW pressure vessel and finalization of the fabrication drawings by Alaskan Copper is not expected to resume until 3Q07.

Fabrication on the SCW interior components (e.g. wire table, perimeter frame, pressure plate, and wash header assemblies) was started at Jesse Engineering in 4Q05. During 4Q06 complex machining of the pressure plate was completed and delivered to Jesse Engineering. Their work was also suspended in 1Q07.

Budget pricing has been solicited and received for the fabrication of the pressure vessel. In 3Q07 I also expect a contract will be awarded for completion of the pressure vessel. When fabrication of the SCW pressure vessel is completed it will be delivered to Jesse Engineering for final assembly and shop testing.

Subtask 2: Shop Testing: Shop testing of the assembled washer will occur at Jesse engineering before delivery to the Port Townsend Paper mill. Current plans are that completion of SCW fabrication, assembly, and shop testing, will be completed during late 4Q07 or early 1Q08.

Major Task: 3: Delivery and Field Installation

Sub Task 1: Delivery: Port Townsend Paper has identified the pumps, motors, valves, instruments, and piping required for the new SCW installation at the mill. They have also identified changes required to the existing mill systems. Long delivery items such as pumps, motors, and electrical switchgear were delivered to Port Townsend during 2Q06. Bids have been solicited for installation of the foundations and support structure but a contract has not been awarded. Other short delivery items have not been ordered.

Sub Task 2: Field Installation: Installation of the structural steel piling and footings will start after 21st Century P&P completes its funding arrangements and fabrication of the SCW pressure vessel is scheduled. It is currently estimated that erection of the SCW structural steel supports and platform, and installation of process piping could start in 3Q07 in anticipation of the arrival of the SCW at Port Townsend Paper.

Milestone Status Table:

Steam Cycle Washer for Unbleached Pulp				
Quarter	Task/Milestone	Planned Completion	Actual Completion	Comments
3Q04	-Receive notification of Federal Assistance Award -Begin to set up reporting and accounting formats	-9/20/04 -9/20/04	-9/20/04 -9/30/04	
4Q04	-Complete setting up reporting and accounting formats -Conduct organizational meeting to identify roles, responsibilities, and project timing -Begin permitting process for Steam Cycle Washer project -Start detailed shop drawings and specifications -Establish washer location and tie-ins to existing mill infrastructure	-10/29/04 -11/30/04 -11/1/04 -10/15/04 -12/15/04	-12/17/04 -11/1/04 -1/18/05 -12/04 -meetings held on 11/30/04 & 12/28/04	-21st Century will "invoice mill for all its charges -No Ecology Notice of Construction is required -Alaskan Copper Works is preparing drawings -Hipp Engineering will complete installation details
1Q05	-Complete detailed shop drawings (Finish Design) -Start to write algorithms for process automation -Start development of plant-specific Process and Instrumentation Drawings (P&IDs) -Select support equipment (pumps, motors, valves, instruments, piping, etc) to support P&ID requirements	-3/31/05 -1/31/05 -2/17/05 -12/01/05	 -6/30/05 -3/31/05	- drawings for everything won't be completed until 3Q07. -hired Tim Ooyman, Checkmate Control Systems -used INL and Checkmate for development of P&IDs -budget quotes obtained from several vendors but final selection not complete

Steam Cycle Washer for Unbleached Pulp				
Quarter	Task/Milestone	Planned Completion	Actual Completion	Comments
2Q05	-Complete algorithm development -Complete hydraulic system design and specifications -Complete initial P&IDs -Start to develop mill-specific drawings for piping and foundation fabrication	-6/30/05 -6/10/05 -6/24/05 -5/16/05	 -4Q06 -2Q06 -5/16/05	-Incorporating redline changes into P&ID's is ongoing -Foundation drawing complete and piping drawings are in progress
3Q05	-Bid, evaluate, and award contracts for fabrication and installation -Start fabrication of field piping and washer foundations -All permitting requirements complete -Start installation of foundations, tie-ins, piping, and E&I	-7/12/05 -8/1/05 -9/16/05 -9/19/05	 -1/24/06 -10/3/05	-not yet awarded; will carry into 3Q07 - Most fieldwork will be restarted in 3Q07. -building permits were approved in 1Q06 - piping tie-ins complete; will start foundations in 3Q07.
4Q05	-Assemble and test the Steam Cycle Washer and its associated equipment at fabrication shop (Complete Fabrication) -Complete installation of field piping, tie-ins, and foundations -Write memorandum regarding finished shop test results -Start installation of Steam Cycle Washer at mill	-11/21/05 -12/30/05 -12/16/05 -12/26/05		-Expect this to be delayed to 4Q07 or 1Q08 -will carry over to 1Q08 -can't be completed until 1/Q/08 -delayed to 1Q08
1Q06	-Complete field installation of Steam Cycle Washer including E&I -Train pulp mill personnel on the operation of Steam Cycle Washer -Conduct mechanical and process-technical trial runs -Start development and testing of optimum operating parameters for the Steam Cycle Washer and batch digesters -Write memorandums regarding mechanical trial runs, process- technical trial runs, and commissioning and acceptance of Washer into mill operations	-1/20/06 -2/4/06 -2/25/06 -2/28/06 -3/31/06		-will carry into 1Q08 -expect to occur during 2Q08 -2Q08 and 3Q08 -3Q08 -2Q08

Steam Cycle Washer for Unbleached Pulp				
Quarter	Task/Milestone	Planned Completion	Actual Completion	Comments
2Q06	-Continue development and testing of optimum operating parameters and batch digester recipes for all the common wood species (fir, hemlock, and cedar) -Begin selection and testing of the optimum operating parameters for the paper machine separately for each paper grade produced by the pulp mill -Write a presentation paper regarding the operation of the Steam Cycle Washer using only data that 21st Century Pulp & Paper does not consider being proprietary. -Write a presentation paper regarding unbleached pulp and paper characteristics after processing with the Steam Cycle Washer	-Ongoing -4/4/06 -4/15/06 -6/30/06		-3Q08 and 4Q08 -3Q08 -4Q08 -4Q08
3Q06 & 4Q06	-Continue development and testing of optimum operating parameters and batch digester recipes for all the common wood species (fir, hemlock, and cedar) -Continue selection and testing of the optimum operating parameters for the paper machine separately for each paper grade produced by the pulp mill -Continue to write presentation papers regarding unbleached pulp and paper characteristics after processing with the Steam Cycle Washer -Complete Field Validation	-Ongoing -Ongoing 12/31/06		-1Q08 thru 2Q08 -3Q08 and 4Q08 -4Q08 -4Q08 (2 years behind original schedule)

Plans for Next Quarter:

During 2Q07 21st Century Pulp and Paper efforts will be directed at successfully completing their arbitration process, finalizing their securities offering, and procuring funding for their portion of the project. This is vital to getting this project moving toward completion. This process is finally expected be completed during 2Q07 and we can once again begin fabrication of the SCW. Until 21st Century funding is arranged, refinement of the SCW P&ID's by Checkmate Control Systems will be suspended. Jesse Engineering will continue on hold with fabrication and machining of the SCW internal components pending additional drawings and funding. Efforts of Alaskan Copper to complete final design and completion of fabrication drawings are also not expected to start until 21st Century's funding is in place. Drawings for the hydraulic design could be started by the end of the quarter. INL's efforts in drafting performance auditing test procedures will also continue on hold with minimal activity through 4Q07.

Patents: None

Publications/Presentations: None

Budget Data as of 3/30/07

Port Townsend Paper Corporation - 12943								
Steam Cycle Washer for Unbleached Pulp								
Actual Project to Date Spending and Estimate of Future Spending ($) - DOE and Recipient (PTPC)								
Quarter	From	To	Estimated Federal Share of Outlays	Actual Federal Share of Outlays	Invoices Received for Federal Share of Outlays	Estimated Recipient Share of Outlays	Actual Recipient Share of Outlays	Cumulative
3Q04	7/1/04	9/30/04	109,064	109,064	0	0	0	109,064
4Q04	10/1/04	12/31/04	100,000	100,000	79,234	47,550	47,550	256,614
1Q05	1/1/05	3/31/05	1,050,000	1,050,000	139,813	49,117	49,117	1,355,731
2Q05	4/1/05	6/30/05	0	0	436,971	42,576	42,576	1,398,307
3Q05	7/1/05	9/30/05	232,746	232,746	714,546	204,467	204,467	1,835,520
4Q05	10/1/05	12/31/05	0	0	219,000	402,464	402,464	2,237,984
1Q06	1/1/06	3/31/06	278,190	278,190	150,436	94,970	94,970	2,611,144
2Q06	4/1/06	6/30/06	0	0	0	162,356	162,356	2,773,500
3Q06	7/1/06	9/30/06	0	0	10,000	22,059	22,059	2,795,559
4Q06	10/1/06	12/31/06	0	0	10,000	67,348	67,348	2,862,907
1Q07	1/1/07	3/31/07	0	0	10,000	6,981	6,981	2,869,888
2Q07	4/1/07	6/30/07	0	0		5,000	5,000	2,874,888
3Q07	7/1/07	9/30/07	0	0		2,000,000	2,000,000	4,874,888
4Q08	10/1/07	12/31/07	0	0		1,700,000	1,700,000	6,574,888
1Q08	1/1/08	3/31/08	0	0		800,000	800,000	7,374,888
2Q08	4/1/08	6/30/08	0	0		400,000	400,000	7,774,888
3Q08	7/1/08	9/30/08	0	0		230,000	230,000	8,004,888
4Q08	10/1/08	12/31/08	0	0		193,093	193,093	8,197,981
Totals			1,770,000	1,770,000	1,770,000	6,427,981	6,427,981	8,197,981
								= estimate
						reflects invoice held back per AK request		

Port Townsend Paper Corporation - 12943					
Steam Cycle Washer for Unbleached Pulp					
Actual Project to Date Spending and Estimate of Future Spending ($) - DOE Laboratory Partner (INL)					
Quarter	From	To	Estimated DOE Lab Share of Outlays	Actual DOE Lab Share of Outlays	Cumulative
3Q04	7/1/04	9/30/04	0	0	0
4Q04	10/1/04	12/31/04	0	0	0
1Q05	1/1/05	3/31/05	37,435	37,435	37,435
2Q05	4/1/05	6/30/05	66,340	66,340	103,775
3Q05	7/1/05	9/30/05	29,346	29,364	133,121
4Q05	10/1/05	12/31/05	43,460	43,460	176,581
1Q06	1/1/06	3/31/06	62,500	62,500	239,081
2Q06	4/1/06	6/30/06	27,926	27,926	267,007
3Q06	7/1/06	9/30/06	0	0	267,007
4Q06	10/1/06	12/31/06	13,356	13,356	280,363
1Q07	1/1/07	3/31/07	15,000	15,000	295,363
2Q07	4/1/07	6/30/07	0		295,363
3Q07	7/1/07	9/30/07	15,000		310,363
4Q07	10/1/07	12/31/07	17,168		327,531
1Q08	1/1/08	3/31/08	15,000		342,531
2Q08	4/1/08	6/30/08	15,000		357,531
3Q08	7/1/08	9/30/08	15,000		372,531
4Q08	10/1/08	12/31/08	27,469		400,000
Totals			400,000	295,381	400,000
					= estimate

Summary of Recepient Spending (Port Townsend Paper and 21st Century P&P)

Port Townsend Paper Corporation - 12943								
Steam Cycle Washer for Unbleached Pulp								
Actual Project to Date Spending and Estimate of Future Spending ($) - Recipient								
Quarter	From	To	Current Estimated 21st Century Share of Outlays	Actual 21st Century Share of Outlays	Current Estimated Port Townsend Share of Outlays	Actual Port Townsend Share of Outlays	Total Recipient Share of Outlays	Cumulative
3Q04	7/1/04	9/30/04	0	0	0	0	0	
4Q04	10/1/04	12/31/04	43,260	43,260	4,290	4,290	47,550	47,550
1Q05	1/1/05	3/31/05	20,770	20,770	28,347	28,347	49,117	96,667
2Q05	4/1/05	6/30/05	27,210	27,210	15,366	15,366	42,576	139,243
3Q05	7/1/05	9/30/05	46,575	46,575	157,892	157,892	204,467	343,710
4Q05	10/1/05	12/31/05	146,300	146,300	256,164	256,164	402,464	746,174
1Q06	1/1/06	3/31/06	50,000	50,000	44,970	44,970	94,970	841,144
2Q06	4/1/06	6/30/06	55,000	55,000	107,356	107,356	162,356	1,003,500
3Q06	7/1/06	9/30/06	0	0	22,059	22,059	22,059	1,025,559
4Q06	10/1/06	12/31/06	5,000	5,000	62,348	62,348	67,348	1,092,907
1Q07	1/1/07	3/31/07	0	0	6,981	6,981	6,981	1,099,888
2Q07	4/1/07	6/30/07	0		5,000		5,000	1,104,888
3Q07	7/1/07	9/30/07	1,500,000		500,000		2,000,000	3,104,888
4Q07	10/1/07	12/31/07	1,500,000		200,000		1,700,000	4,804,888
1Q08	1/1/08	3/31/08	200,000		600,000		800,000	5,604,888
2Q08	4/1/08	6/30/08	200,000		200,000		400,000	6,004,888
3Q08	7/1/08	9/30/08	90,000		140,000		230,000	6,234,888
4Q08	10/1/08	12/31/08	115,885		77,208		193,093	6,427,981
Totals			4,000,000	394,115	2,427,981	705,773	6,427,981	6,427,981
			= estimate	under review				
		original	1,255,000		915,000			2,170,000

Development of Screenable Wax Coatings and Water-Based Pressure Sensitive Adhesives

Severtson: University of Minnesota

GO14309

DOE F 4600.3A
(03-94)

U.S. Department of Energy

OMB Control No.
1910-0400

Milestone Log

OMB Burden Disclosure Statement

Program/Project Title: Development of Screenable Wax Coatings and Water-Based Pressure Sensitive Adhesives

Program/Project ID No.: DE-FC36-04GO14309

ID No.	Description	Planned Completion Date	Actual Completion Date	Comments As of (Date)
1	Characterization and removal testing of Franklin label grade water-based PSAs.	10/05	10/05	**Completed as scheduled**
2	Characterization and removal testing of standard wax coatings.	10/05	10/05	**Completed as scheduled**
3	Characterization and removal testing of new model water-based PSAs. Go/No-Go Point *Criterion for proceeding* – Properties identified as those governing fragmentation are confirmed	10/06	10/06	**Completed as scheduled** A meeting in Columbus, OH and a conference call with DOE representatives were held on December 11th, 2006 to review results. All agreed that the project has led to a general understanding of what controls the fragmentation behavior of adhesive.
	Formulation of label grade water-based pressure sensitive adhesives (PSA).			**Several new label grade products are currently being scaled up and will likely be available before the end of the year. The first pilot-scale batch of Covinax 2006 has successfully been completed and is being evaluated.**
	Demonstrate screening of water-based PSAs at pilot scale.			**Six PSAs developed based on results from the model PSA study have been tested at FPL and all of them passed the Protocol Test.**
6	Study on the role of facestock properties in determining removal of PSAs.	10/06	10/06	As described in the previous report, the same relationships found for hot-melts are also found here, but it appears that the presence of the coating package and changes in film properties when placed in liquid also have an impact for water-based systems.
7	Characterization and removal testing of new model wax coatings. Go/No-Go Point *Criterion for proceeding* – Properties identified as those governing removal are confirmed	04/07		The focus currently is on the barrier properties of the clay nanocomposite coatings. Tests for removal will begin again next quarter.
	Demonstrate screenable wax coatings with the same performance as existing formulations			**A contact cell has been built to gauge the permeability. Several problems were identified which are currently being addressed.**
	Development of new benign commercial PS labels. Go/No-Go Point **Criterion for proceeding – Laboratory results confirmed for PS labels at pilot scale**	11/07		**As outlined by Franklin executives during the conference call, they are actively pursuing partnerships to produce benign PS labels to the marketplace.**
10	Study on the role of board properties in determining removal of wax coatings.	08/07		
	Development of new benign commercial treated corrugated containers.	**11/07**		

Budget Data (03/31/07): The actual spending should reflect the money actually spent on the project in the corresponding periods.

Project Spending and Estimate of Future Spending							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays**	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays**	**Actual Recipient Share of Outlays**	**Cumulative**
4Q04	10/1/04	12/31/04	112,872	94,000	35,853	29,140	123,140
1Q05	1/1/05	3/31/05	104,308	98,494	35,854	34,473	256,107
2Q05	4/1/05	6/30/05	50,942	76,506	35,853	41,600	374,213
3Q05	7/1/05	9/30/05	51,090		35,854	48,201	422,414
4Q05	10/1/05	12/31/05	65,796		29,561	28,361	450,775
1Q06	1/1/06	3/31/06	65,800	225,000	29,560	30,374	706,149
2Q06	4/1/06	6/30/06	65,796		29,561	29,784	765,301
3Q06	7/1/06	9/30/06	65,800		29,560	29,368	848,772
4Q06	10/1/06	12/31/06	62,686	60,000	23,670	23,471	931,034
1Q07	1/1/07	3/31/07	49,349	58,880	23,669	23,382	
2Q07	4/1/07	6/30/07	52,688		23,670		
3Q07	7/1/07	9/30/07					
4Q07	10/1/07	11/30/07					
Totals			**747,127***	**612,880**	**332,665***	**318,154**	**931,034**

* Values for the approved budget.

			Approved Spending Plan			**Actual Spent to Date**		
Phase/Budget Period			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	To						
Year 1	10/04	10/05	319,212	143,414	462,626			
Year 2	10/05	10/06	263,183	118,242	381,424			
Year 3	10/06	10/07	210,731	94,676	305,407			
Totals			793,126	356,332	1,149,458			

QUARTERLY PROGRESS REPORT

Project Title: Development of Screenable Wax Coatings and Water-Based Pressure Sensitive Adhesives

Covering Period: January 1st, 2007 through March 31st, 2007

Date of Report: April 27th, 2007

Recipient: Department of Bio-based Products, University of Minnesota, 2004 Folwell Avenue, St. Paul, MN 55108

Award Number: DE-FC36-04GO14309

Subcontractor: United States Department of Agriculture Forest Service, Forest Products Laboratory

Other Partners: Boise Cascade Corp., Franklin International, The International Group, Inc.

Contact: Steven J. Severtson, (612) 625-5265, sever018@umn.edu

Project Objective: The project objective is the design of new water-based pressure sensitive adhesive (PSA) products and wax coatings that are engineered for enhanced removal during the processing of recycled fiber. Research includes the formulation, characterization, and performance measurements of new screenable PSAs and wax treatments, testing of modified paper and board substrates and the design of test methods to characterize the inhibition of adhesive fragmentation and wax comminution and relative removal efficiencies of developed formulations.

Background: The presence of PSAs and wax coatings in recycled paper creates a number of problems for the recycling process including lost production and diminished product quality. This project will focus on the design of adhesives and coatings that are more effectively removed from the papermaking process during furnish screening. These new materials should possess properties that enhance removal without impacting performance.

Work will include the identification of properties that control adhesive fragmentation and coating comminution and use of this information to design new formulations optimized for both removal and product performance. Through an iterative process where the surface and bulk mechanical properties of materials are characterized and compared against repulping behavior and screening removal efficiencies, the properties controlling fragmentation and comminution will be identified. Products will then be reformulated to manipulate these properties and produce commercially feasible, screenable products. In addition to the development of new adhesives and wax coatings, the role of the paper substrates in determining removal efficiencies will be investigated. Treatments such as strength resins, pigment coatings, sizing agents and others will be used to modify the wet and dry strength of paper, interfacial properties and PSA/coating-paper adhesion. The influence of the modifications on the adhesive fragmentation and coating comminution will be determined from monitoring the particle size and morphology during repulping operations and by measuring screening removal efficiencies. It is expected that the results of this work will identify combinations of paper and PSA or board and wax treatments that provide for significant screening removal efficiencies of the adhesive or coating layer.

Status: A summary of results obtained during the reporting period by the University of Minnesota and its partners are as follows:

- Conference calls were held on January 5th, January 8th, January 17th, January 30th, February 12th and March 22nd between L. Gwin (Franklin International), S. Severtson (UM) and J. Guo (UM). These meetings are used to make research plans, clarify and review information conveyed via emails and phone calls and will often involve other participants from Franklin International and/or the University of Minnesota.

- The manuscript entitled "Optimizing the Monomer Composition of Acrylic Water-based Pressure Sensitive Adhesives to minimize their Impact on Recycling Operations" was accepted for publication in Industrial and Engineering Chemistry Research. The reference for this article is Guo, J., Severtson*, S. J. and Gwin, L. E., *Industrial and Engineering Chemistry Research* 46(9), 2753-2759, 2007.

- The major conclusions from this paper are
  - When placed in water, water-based acrylics rapidly swell and their strength is degraded.
  - The ability to retain a level of cohesive strength that is above a threshold value when saturated with water, i.e., a certain minimum wet strength, is required for a PSA film to be environmentally benign.
  - This wet strength was found to be governed primarily by the monomer composition of the adhesive polymer.
  - The combination of vinyl acetate and acrylic acid monomers produces water-based PSAs that readily fragment during paper recycling operations, and vinyl acetate is one of the more problematic monomers for recyclability used in acrylic formulations.
  - Replacing monomers in an adhesive polymer with a more hydrophobic version increases its removal efficiency.
  - More generally, it can be concluded that those changes that enhance wet strength, which may include the inclusion of stronger, more water resistant components can be used to limit the fragmentation of a PSA film during repulping limiting its impact on paper recycling operations.

- A study on the impact of various additives used in the synthesis of adhesive emulsions and their formulation for coating is nearing completion. As discussed previously, the monomer composition appears to be the dominant variable controlling the fragmentation behavior of water-based PSA. More precisely, the residual wet-strength appears to be the most important aspect of these films. However, other components can reduce the removal efficiency of PSA films. For example, paper facestock can have a substantial impact on the fragmentation behavior of the PSA that is attached to it. Studies are currently being carried out in which the additives such as tackifiers and wetting agents are varied in formulations.

The results indicate that for certain types of water-based acrylic PSA, the types and concentration of these additives can control their behavior during recycling operations.

- The first pilot-scale batch of Covinax 2006 developed during this project was produced and is currently being evaluated.

- Laboratory results are being confirmed by the USDA FS Forest Products Laboratory (Madison, WI) using their recyclability protocol. Six PSAs were recently tested, 3 existing commercial products and 3 model systems. All passed as expected. The commercial products were identified through laboratory testing and the new products were designed based on the results of research. Five more adhesive formulations have been submitted, 3 existing commercial and 2 new formulations. These results will be reviewed in the next report.

- Efforts continued this quarter to develop a measurement technique for gauging the permeability of wax coatings. A house built system is currently being used for which a relative humidity gradient of 0 to 90% is possible. Data from this equipment indicates that increasing the concentration of exfoliated organo-clay in paraffin wax substantially reduces its permeability for water-vapor.

- The permeability of PSA films is also being examined with the built equipment. Because of the importance of the interaction with water to their fragmentation behavior, attempts are being made to determine what impact moisture has on the structure and mechanical properties of thin water-based PSA films. In addition, films exposed to moisture and being imaged using various scanning probe techniques.

- A manuscript entitled, “Brittle–Ductile Transitions and the Toughening Mechanism in Paraffin/Organoclay Nanocomposites” was accepted for publication this quarter in *Materials Science and Engineering A*. It will be published next quarter.

- A manuscript entitled, “Properties of Paraffin Wax/Montmorillonite Nanocomposite Coatings” was accepted for presentation at the NSTI 2007 Nanotech Technical Meeting, May 20th-24th in Santa Clara, CA. The paper outlines the performance benefits found for barrier coatings due to the addition of organo-clay to paraffin wax matrices.

Plans for Next Quarter:
The team will focus on Tasks 4 and 5 over the next quarter. We will also begin to explore Task 9. Many of the decisions at this point in the PSA research are driven by business considerations and will involve input from the marketing staff at Franklin. Several new products will be explored as well as current products that can be modified to make them benign based on our research findings. Task 8 has proven to be more challenging than expected, but it is hoped that initial studies on moisture permeability will be completed next quarter. Much effort will also be dedicated towards the publication of several articles reviewing the current results from this project. This information should allow other manufacturers the ability to develop benign products.

Mechatronic Design and Control of a Waste Paper Sorting System for Efficient Recycling

Venditti: North Carolina State University

ID13880

QUARTERLY PROGRESS REPORT

Project Title: Mechatronic Design and Control of a Waste Paper Sorting System for Efficient Recycling

Covering Period: Jan 1, 2007 through Mar 31, 2007, 1st Quarter 2007

Date of Report: April 27, 2007

Recipient: N. C. State University
Dept. of Wood and Paper Science
Raleigh NC 27695-8005

Award Number: DE-FC36-00ID13880 (switched from DE-FC07-00ID13880)

Subcontractors: None

Other Partners: Advanced Sorting Technology, LLC, 3738 Keystone Ave., Nashville, TN 37211
Weyerhaeuser Co., WTC 2E19, PO Box 9777, Federal Way, WA 98063-9777

Contact(s): Dr. Richard A. Venditti (919) 515-6185, richard_venditti@ncsu.edu
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OBJECTIVE

The objective of this research is to develop a sensor network for High Speed Automated Paper type characterization. Past research has led to the development of a high throughput lignin sensor and an experimental concept for a bending stiffness sensor. The possibilities of integrating gloss and color sensors have also been explored. A sorting algorithm purely based on Fuzzy inference systems was attempted during the last quarter. It was found to be inadequate due to the complexity in the data from the sensors which made the development of such a system infeasible. The research performed this quarter involved the testing of a sorting algorithm based on neural networks due their proven success in the fields of pattern recognition and classification.

Introduction

The research performed this quarter was primarily focused on the development of a sorting algorithm based on neural networks. The reason for choosing neural networks was its ability to establish relationships between a known input space and an output space when it is not possible to do so intuitively. Such a network when trained properly would be able to identify the output for any given input that is closely related to the training data's input space. The following sections describe in detail the implementation procedure, the advantages and limitations of this method and also discuss the scope for future improvements.

Neural Networks

Neural networks are systems that try to make use of some of the known or expected organizing principles of the human brain. They consist of a number of independent, simple processors called the neurons. These neurons communicate with each other via weighted connections called the *synaptic weights.* Using a learning rule and a set of examples, such a network can learn to produce target outputs for given input signals by varying the weights of each of the connections. The exact implementation procedure required for accomplishing the tasks mentioned can be found in the material referenced at the end. We have used the Neural Network Toolbox that is built into MATLAB for designing and simulating the network.

Implementation

After carefully evaluating various types of Neural Network Models available and understanding the underlying theory of each of these models, we have chosen the network based on the Radial Basis Function for our application. The reason for doing so is the similarity of the activation function of these type of networks to that of the variation of sensed parameters of different types of paper.

We started out by extracting the required data from the normalized parameter database to create a matrix which will be used to generate various combinations of training vectors and test vectors. The normalized parameter database had details of 224 regions which corresponded to the 63 different samples. Hence the data that was extracted was a 224 x 8 matrix. The columns represented normalized values of lignin, deflection in positive direction, deflection in the negative direction, gloss , red component , green component, blue component and the paper type. The paper types were as follows.

1. *Newsprint*
2. *Uncoated Free Sheet*
3. *Magazine Paper*
4. *Card Stock*
5. *Coated Free Sheet*

The procedure for carrying out each of the simulation runs is as follows. From the 224 x 8 matrix, 204 rows were picked to serve as training data and the remaining 20 rows were used as test data to verify the network design. The training data was classified into the Input Vector which contained the 7 columns representing the normalized sensor data and the Target Output Vector which contained one column representing the paper type. These vectors were given as input to the Neural network design tool, which came up with a network that could best approximate the output space for the given input.

This was followed by testing of the network. The 20 rows of data could be used to test the network to see if it produced the desired output for a completely new input vector. The network design is such that if the input vector is exactly the same as one of the input vectors in the training data, it would identify the output with 100% accuracy. The output expected from the network when it is given any other 1 x 7 input vector containing the sensor data is a number between 1-5 indicating the paper type. We round off the obtained output to the nearest integer and compare it to the desired output. The performance of the network is then evaluated by measuring the number of correct predictions it is able to make from a group of 20 sample inputs. Several such simulations were performed and the results obtained are shown below.

Table 1 – Simulation results of the neural network algorithm

Training Data (sample range)	Test Data (sample range)	Prediction Accuracy
1-199 ; 221 - 224	200 - 220	4.76%
1-159 ; 181 - 224	160 - 180	19.04%
1-139 ; 161 - 224	140 - 160	9.52%
1-119 ; 141 - 224	120 - 140	19.04%
1-99 ; 121 - 224	100 - 120	14.50%
1-39 ; 61 – 224	40 - 60	9.50%

The algorithm performance was found to be poor. Upon careful analyses, it was found that one of the reasons for this could be due to the way in which the target output was presented. Using an output space represented by [1,2,3,4,5] would be prone to errors since the network would try to arithmetically map the output to the input space and hence could lead to an erroneous algorithm. We experimented by replacing this output space with the following one.

Newsprint - *[1 0 0 0 0]*
Uncoated Free Sheet - *[0 1 0 0 0]*
Magazine - *[0 0 1 0 0]*
Card Stock - *[0 0 0 1 0]*
Coated Free Sheet - *[0 0 0 0 1]*

This way the different outputs are decoupled from each other and hence the output space would take this form. When paper type = 3, Output space = [0 0 1 0 0] which is like saying,

Newsprint - *No*
Uncoated Free Sheet - *No*
Magazine Paper - *Yes*
Card Stock - *No*
Coated Free Sheet - *No*

This is equivalent to 5 networks working in parallel to determine whether or not a particular sample belongs to a particular paper type or not. Ideally, only one of the types should get a value close to 1 and the remaining four values should be close to 0, which would be the case if we simulated an input vector from the training data. But when a completely new input vector is simulated, the output would be a 1 x 5 vector with each element varying between 0-1. We choose the element that is closest to 1 and its position in the vector would indicate the identified paper type. Simulations were carried out using this scheme. The data used was exactly the same as that of the previous simulations. The results are as follows.

Table 2 – Simulation results of the neural network algorithm with modified output vector

Training Data (sample range)	Test Data (sample range)	Prediction Accuracy
1-199 ; 221 - 224	200 - 220	28.57%
1-159 ; 181 - 224	160 - 180	19.04%
1-139 ; 161 - 224	140 - 160	14.50%
1-119 ; 141 - 224	120 - 140	19.04%
1-99 ; 121 - 224	100 - 120	28.56%
1-39 ; 61 - 224	40 - 60	23.81%

It can be observed right away that the prediction accuracy has improved. Even though an accuracy of around 25% doesn't look promising, especially when a random guess would be close to 20% accurate. However, a closer observation of the output data suggests that the network is indeed performing very well. On every occasion that the network appeared to fail, the output was something similar to the following.

Target output = 2 ; Predicted Output = 4;
Output Vector = [1.678 0.891 0.234 1.102 0.567]

It can be seen that the output vector element corresponding to the target output was very close to the value 1 (0.891), but there was another element in the vector which was closer to 1 (4 :-- 1.102), which resulted in the algorithm choosing 4 as the predicted output. Hence the network did to some extent come very close to predicting the correct output. The following sections outline the strategies to be adopted in order to improve the efficiency of the algorithm.

Algorithm refinement techniques

- **Complex relationship between lignin and color –** We had assumed that the variation of the lignin sensor output with the variation of the color was similar for all types of paper. Hence we tried to make the network learn the relationship from the training data. This has to be verified by collecting a set of samples that have similar color variations across their surface and measure the variations of the lignin sensor output. If the relationship is indeed different for different types of paper, we would have to come up with some sort of preprocessing algorithm to compute some hybrid parameter that can be used as an input instead of using the raw data from the lignin sensor.

- **Gloss sensor's sensitivity to color –** Similar to the issue addressed for the lignin sensor, even the gloss sensor's sensitivity to color has to be calibrated. The assumption that the effect of color on the gloss sensor output remains constant for all paper types has to be validated or a hybrid variable similar to the one discussed for the lignin sensor has to be developed.

- **Alternative Color Space –** Although representing color in the RGB space would give us the advantage in terms of the number of colors that can be measured and the accuracy with which a particular color can be represented, it does not offer any information that can by itself come close to identifying a particular paper sample's type. This is because the entire range of R,G and B values can be present in all of the samples types and their variation across the samples can be quite arbitrary.

  The LAB color space however is based on the human perception of color and hence has been serving as the industry standard for color representation. Hence, using this color space, one should be able to draw some conclusions based on the variation of the **L**, **a** and **b** components across the samples surface.

RGB to L a* b* conversion

The conversion from RGB space to L a b space is essentially a coordinate transformation. To be able to perform this conversion, the RGB space should be in one of the absolute forms i.e., Adobe RGB, sRGB etc.

The illumination conditions when sRGB values are measured should be known in order for the conversion to work. Since our method of measuring color involves the use of a Digital Single Lens Reflex camera, the images were captured under natural lighting conditions and the camera settings were chosen to use the D65 illumination profile to convert the sensor data into sRGB format.

The conversion is a two step process, which involves converting to the device independent XYZ space and then convert to the L a* b* space. The details of this conversion can be found in the articles referenced below. A Matlab code was developed to implement this algorithm and it was found to be successful in carrying out the conversions. The validity of these conversions was verified by loading the same sample on to the Technidyne Color Touch model at the Wood and Paper Science Laboratories at NC State university and the results were very close to the values produced by the conversion algorithm. These converted values will be used in the design of the neural network.

PLANS FOR NEXT QUARTER:

1. Establish the relationship of Lignin Vs Color and Gloss Vs Color to develop hybrid parameters to be used in the design of the algorithm.

2. Develop a parameter based on the overall variation of the color parameters across the surface to help the color sensor contribute better to the decision making process.

3. Explore the concept of Neuro-Fuzzy systems to see if it can be adapted to this particular application.

REFERENCES

1. Neural networks and simulation methods / Jian-Kang Wu
2. Foundations of neuro-fuzzy systems / Detlef Nauck, Frank Klawonn, Rudolf Kruse
3. http://www.mathworks.com/products/neuralnet/description1.html
4. http://en.wikipedia.org/wiki/Lab_color_space
5. http://en.wikipedia.org/wiki/SRGB

Patents: None

Publications/Presentations:

A Non-Contact Sensor for the Identification of Paper and Board Samples on a High-Speed Sorting Conveyor", M. K. Ramasubramanian, Richard A. Venditti and Kalyan C. Katuri, Appita Journal, in press 2007.

Behavior of Paper on a High-Speed Conveyor Subjected to Air Jet Impingement-A Method for Bending Stiffness Estimation, M. K. Ramasubramanian, R. A. Venditti, K. C. Katuri, Journal of Mechanics of Materials and Structures, in press 2007.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	Design robust industrially worthy lignin sensor	12/03		Completed
2	Develop color tracking system	12/03		Completed. Have opted for RGB measurements that are available.
3	Develop a decision making algorithm	12/05		Completed
4	Evaluate sensing techniques for food pkg, waxed OCC, coated OCC, high adhesive paper	12/05		In progress. The development of a decision making algorithm is part of this.
5	Incorporate and test array of lignin/gloss/color sensors and decision-making algorithm.	12/06		In progress.

NCSU Acct.: 521126						
	Cumulative Thru	Cumulative Thru	Cumulative Thru	Cumulative Thru	Cumulative Thru	
	June 30	June 30	June 30	June 30	March 2007	
	2003	**2004**	**2005**	**2006**	**2007**	**Cumulative thru 03/31/2006**
Total Direct Costs	24,630.81	103,101.39	146,983.27	268,470.84	318,321.93	**318,321.93**
Cost Sharing						
Venditti	329.15	874.77	966.65	5,640.33	4,431.56	12,242.46
Ramasubramanian	355.02	940.85	-	5,056.65	4,593.79	10,946.31
F&A on Cost Sharing	318.14	844.26	449.49	4,974.10	4,196.79	10,782.77
Total Cost Sharing	1,002.31	2,659.88	1,416.14	15,671.08	13,222.14	**33,971.54**
Third Party Cost Sharing	**2003**	**2004**	**2005**	**2006**	**2007**	
Weyerhaeuser(per Letter)	10,000.00	10,000.00	10,000.00	10,000.00	-	40,000.00
Advanced Sorting Technologies(per letter)	10,000.00	10,000.00	10,000.00	-	-	30,000.00
MSS , Inc				10,000.00	-	10,000.00
Total 3rd Party	22,003.00	20,000.00	20,000.00	-	-	**80,000.00**
Total Project Cost						**432,293.47**

Development of Renewable Microbial Polyesters for Cost Effective and Energy-Efficient Wood-Plastic Composites

Thompson: Idaho National Laboratory

Agr id:11428

QUARTERLY PROGRESS REPORT

Project Title: Development of Renewable Microbial Polyesters for Cost Effective and Energy-Efficient Wood-Plastic Composites

Covering Period: January 1, 2007 through March 31, 2007

Date of Report: April 20, 2007

Recipient: Idaho National Laboratory
P.O. Box 1625
Idaho Falls, ID 83415

Project Number: CPS Number: 11428

Project Period: October 1, 2004 – September 30, 2008

Subcontractor: Washington State University
P.O. Box 643140
Pullman, WA 99164-3140

Other Partners: ECO:LOGIC, Inc.
Glatfelter Corporation
Strandex Corporation
University of California-Davis

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Project Objectives:

Objective 1: Determine preferred PHA monomer compositions, PHA/cell debris ratios, and PHA/wood ratios for the production of superior wood-PHA composites

Objective 2: Define feedstock compositional ranges for WTE and PPE for production of PHAs meeting PHA monomer composition ranges specified in Objective 1

Objective 3: Determine the efficacy of supplementing WTE and PPE to improve PHA production from these effluents

Objective 4: Test the material properties of wood-PHA composites produced from waste-derived PHA made and used without extraction or purification

Objective 5: Produce and test wood-PHA composites made from PPE-derived PHA at the pilot-scale.

Background:

This is a collaborative project among the Idaho National Laboratory, Washington State University, the University of California-Davis, Glatfelter Corporation, Strandex Corporation, and ECO:LOGIC Engineering, Inc. The purpose of the project is to develop and produce wood-plastic composites using bacterial polyhydroxyalkanoates (PHA) in place of petroleum-derived plastic. This will be made economical and environmentally friendly by reducing or eliminating costly steps in PHA production. Specifically, this will be accomplished by utilizing PHA produced inherently in wastewater treatment processes, and by utilizing the PHA directly in the composite without removal of the cell debris. The forest products industry will benefit most from this research, both from the perspectives of environment, recycling, and new composite products. The municipal waste treatment industry, as the basis for production of PHAs from effluents, provides the starting point for application to forest products industry effluents and also benefits.

This project is comprised of five tasks. The first four tasks address PHA production, extrusion, and composite properties. Laboratory testing to determine preferred feedstock compositions, together with ease of processing and material properties of composites produced therefrom, will be completed by WSU for pure commercial PHAs (Task 1) and for unpurified effluent-derived PHAs (Task 4). This information will be used to define appropriate effluent feedstocks (Task 2) and optimize supplements (Task 3) to support the preferred composite formulations. Task 5 will include a ca. 1000 lb/h pilot-scale extrusion of wood-WTE-derived PHA composites using the appropriate supplements in the production of the WTE utilized. The project is being completed in three phases. Phase 1 includes determination of PHA compositions and amounts for superior wood-thermoplastic composites. Phase 2 includes determination of feedstock supplements for production of effluent-derived PHAs of the desired monomer compositions. Phase 3 includes pilot-scale testing to identify and mitigate processing issues at the commercial scale.

Status:

Tasks scheduled to be active during this quarter included

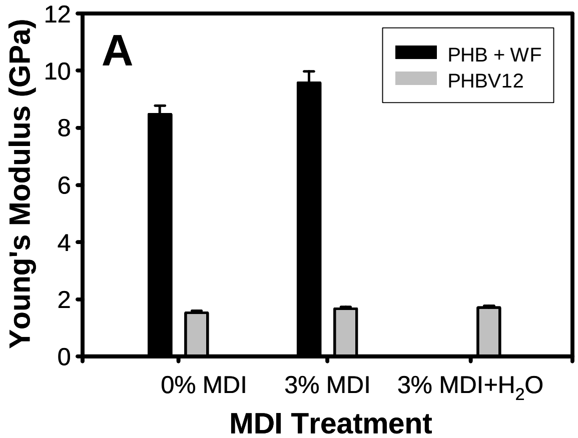
- Task 1, "Purified PHA ± Cell Debris Composite Processing & Material Properties,"
- Task 2, "Effect of Feedstock on PHA Type/Amount,"
- Task 3, "Supplementation of Waste Effluents for Production of PHA," and
- Task 4, "Waste Effluents PHA Composite Processing & Material Properties."

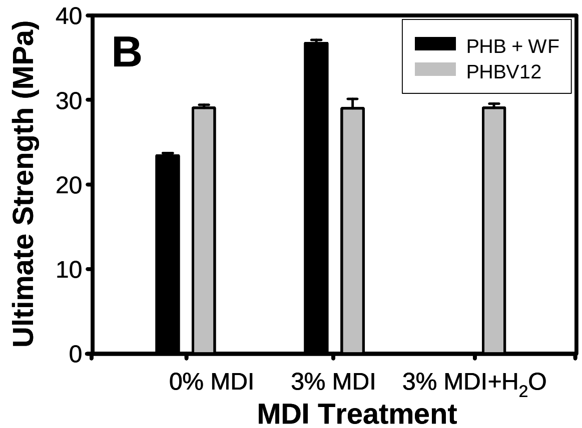
Task 1 and Task 4 work scopes are being performed at Washington State University (WSU). DOE funded Task 2 and Task 3 work scopes are currently being performed at INL, UC-Davis, and Glatfelter.

Task 1 Status:

To date we have investigated the effects of different additives including plasticizer, coupling agent, and compatibilizers on the mechanical and physical properties of the resulting composites. This quarter, we began investigating the influence of wood fiber composition and the presence of residual cellular materials in the polymer. Raw wood fiber is a complicated mixture containing many smaller and potentially volatile molecules besides the biopolymers cellulose, hemicellulose and lignin. In addition, while commercial PHAs are available at high purity, they still contain small amounts of residual protein. Finally, absorbed moisture in the polymer powder and the wood fiber can not be completely avoided. Because of these factors, and also compositional variation from batch to batch, it is neither economical nor practical to investigate the effects of each individual component. Earlier, we demonstrated that 0 to 3 wt% polymeric MDI (pMDI) added as an interfacial adhesion promoter increased the modulus and strength of the composites, and slightly increased the strain at break. These results clearly indicated that the improved and tuned interfacial adhesion would lead to a general improvement of the mechanical properties.

This quarter, we demonstrated that compared to the effect of the coupling reaction between fibers and polymer, the effects on mechanical properties of small variations in moisture content as well as reactions between polymer end groups and the coupling agent (pMDI) are minor. We hypothesize that this is because when effective interfacial adhesion is established at a appropriately high concentration level, small variations in the volatile components may not be very critical to mechanical properties. Figures 1A-C show that the addition of pMDI to neat polymer did not result in an obvious change in mechanical properties. To determine whether residual moisture in the polymer powder or absorption during the processing had an influence, water was added stoichiometrically to the compound formulation on the basis of the added 3 wt% pMDI. Surprisingly, no significant changes in modulus or strength were noted, except that the strain-at-break decreased. In contrast, adding 3 wt% of pMDI significantly increased the mechanical properties of the composites. It is reasonable to attribute such an improvement to the crosslinking between cellulose hydroxyl groups and the polymer end hydroxyl groups.





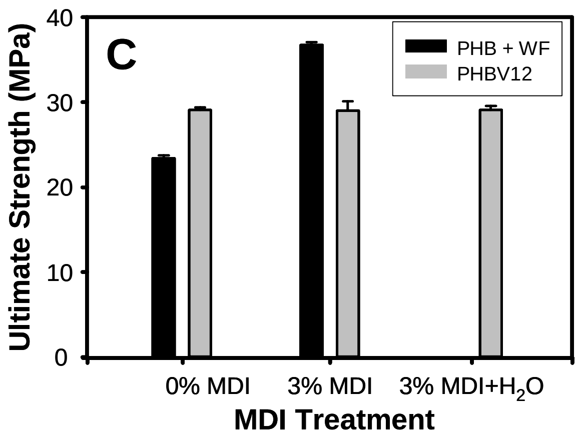


Figure 1. *Comparison of the effects of pMDI on neat PHB and PHB/woodfiber composites.*

We also attempted to study the rheological properties of the composites. However, at 60 wt% of the wood fiber in the composite, steady laminar flow of the composite melt was not obtained in either capillary or dynamic rotational rheometers. Hence, these tests did not yield any meaningful rheological results.

We have achieved significant advancements in the modulus and strength of PHA/wood fiber composites, however, the impact strength remains unsatisfactory. Our results, as well as those of other in the literature, indicate that the impact strength of PHA/wood fiber composites decreases with increasing wood fiber content. As a result, mixing wood fiber together with PHAs further deteriorates the already poor toughness of the PHB and some PHBVs. We believe that significantly increasing the toughness of PHA/wood composites is critical, and this will become an added research goal in Task 4 testing, in addition to the other existing objectives.

Tasks 2 & 3 Status:

During this quarter, Task 2 was completed at UC Davis. Extending our previous results with municipal wastewater treatment systems, experiments were carried out to determine whether PHA production from industrial wastewater can be optimized by varying SRT, HRT or anoxic-oxic cycling. The SRT, HRT, and anoxic-oxic cycling were varied in a 3^2 factorial experiment. The inoculum and wastewater were obtained from an industrial wastewater treatment plant in California. Eight SBRs of 4 L working volume each were simultaneously started and operated independently under the 8 individual conditions. One liter of the reactor volume was decanted each day and replaced with fresh medium. Following stabilization of COD degradation (anticipated at 3 times the SRT), the COD and PHA contents (dry cell weight basis) were measured at regular intervals within the SBR. The factorial design, COD results, and PHA results are summarized in Table 1.

Table 1. *Experimental data & design matrix of PHA production from industrial wastewater*

Parameter \ reactor	A	B	C	D	E	F	G	H
COD removal	26%	30%	82%	77%	13%	33%	77%	66%
Ave. PHA production	2%	17%	31%	25%	0%	8%	36%	17%
SRT (d)	4	12	4	12	4	12	4	12
HRT (d)	1	1	4	4	1	1	4	4
w/Anoxic	0	0	0	0	+	+	+	+

Work at the INL focused on the characterization of operational parameters that influence growth and PHA production of methylotrophic microbial communities enriched from Glatfelter pulp and paper effluents (PPE). Previous results indicate that the foul condensates contain significant quantities of inorganic nitrogen. Although nitrogen is an essential nutrient required for microbial growth, nitrogen limitation in the presence of sufficient carbon has been used to induce microbial PHA production. The influence of C:N ratio on PHA production was examined using the waste activated sludge enrichments, which were fed methanol as the sole carbon and energy source.

Results from the enrichment experiments described in previous quarterly reports demonstrate that secondary waste activated sludge (WAS) from the wastewater treatment plant at the Glatfelter plant in Chillicothe, OH is a viable source of inoculum and can produce methanol-degrading microbial communities. Additionally, dilute foul condensates do not exhibit a toxic effect on the consortia, since microbial biomass (as mixed liquor volatile suspended solids or MLVSS) stabilized, albeit at a lower than initial value, and COD and methanol concentrations decreased over the 24-hour feed cycles indicating microbial consumption. However, data from the enrichment experiments also revealed that the foul condensates are a significant source of nitrogen as well as biologically available carbon. Ammonia released from the wood chips during the kraft pulping process is captured in the condensate collection system along with methanol and other volatile compounds. (Recent analyses of foul condensates from Chillicothe confirm the presence of both methanol and ammonium.). This is an important observation since nutrient status plays a central role in microbial growth and PHA metabolism. Specifically, nitrogen limitation in the presence of excess carbon is a demonstrated means to induce PHA production in microbes capable of hydroxyalkanoic acid synthesis.

The enrichment reactors were run under balanced nutrient conditions to encourage vigorous microbial growth without loss of potential methanol-degrading microorganisms due to nutrient limitations. However, over time ammonium concentrations in the 24-hour decant from the primary out and WAS enrichments reached those of the influent feed (~300 mg/L) indicating an excess of nitrogen. However, the foul condensates contained additional and varying concentrations of ammonium (Figure 2). The excessive nitrogen resulted in a C:N of about 3 in these enrichments. Under these nutrient-sufficient conditions, the reactors did not produce PHA. Therefore, the next step was to investigate the effect of C:N on PHA production.

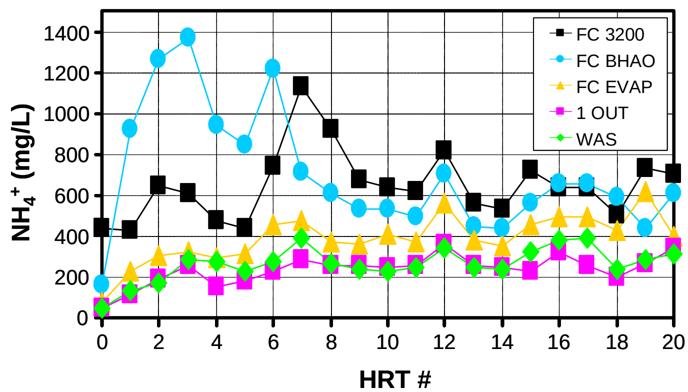
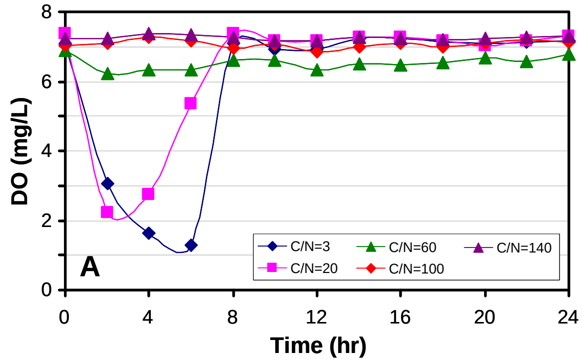
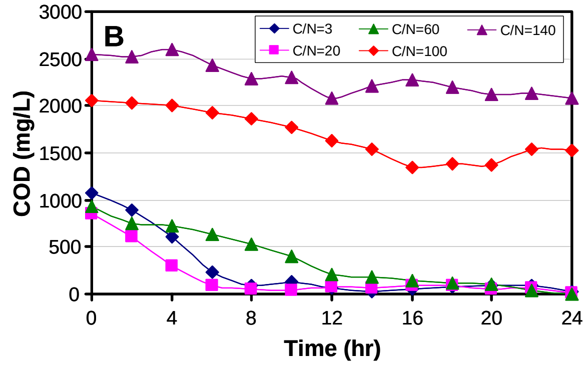


Figure 2. *Variation of the observed soluble NH_4^+ with HRT at the conclusion of each HRT in the enrichment cultures.*

To evaluate the effect of C:N ratio on PHA production, the WAS-only enrichment cultures were re-suspended in a 4L reactor (HRT=SRT= 4 days; C:N=3) and used to create seed materials for the new C:N reactors. When sufficient seed was collected, five reactors were started at increasing carbon to nitrogen ratios (C:N=3, 20, 60, 100, and 140; methanol fixed at 625 mg/L; HRT=SRT=4 days; air flowrate=1.25 vvm). The only difference between the reactors was the concentration of ammonium added in the nutrient medium. Reactors were allowed to stabilize for 6 HRTs before sampling. Reactors were sampled immediately after feeding and again at two hour intervals throughout the feed cycle. Figures 3 A-C show the dissolved oxygen (DO), chemical oxygen demand (COD), and PHA values for the different C:N reactors over the 24 hour feed cycle. COD degradation above 95% occurred in the C:N=3, 20, and 60 reactors indicating rapid biological carbon uptake with a concomitant decrease in soluble oxygen. The reactors run at a C:N=100 and 140 degraded COD by 26 and 18% respectively with very little change in soluble oxygen concentration. PHA was produced in all reactors except for the reactor run at a C:N of 3. Intracellular PHA concentrations peaked between 8 and 14 hours after feeding and declined thereafter indicating utilization of the stored carbon in lieu of available carbon in the media. The reactor run at a C:N=60 produced the greatest quantity of PHA at 13.9% on a dry cell weight basis. These results indicate that a nutrient-limited condition, specifically nitrogen limitation, must be imposed in combination with a transient carbon feeding regime in order to induce PHA storage in these methylotrophic enrichments. Results also demonstrate the need to carefully monitor intracellular PHA concentrations since three of the four PHA-producing reactors consumed intracellular PHA stores before exhausting COD in the media.





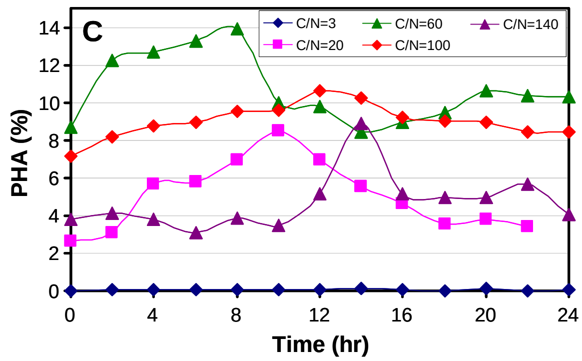


Figure 3. *Variation of* ***(A)*** *Dissolved oxygen (DO),* ***(B)*** *Chemical oxygen demand (COD), and* ***(C)*** *PHA in the C:N reactors.*

Task 4 Status:

Initiation of this task has been hampered by the lack of a source of PHA-free cell debris to utilize for the composite formulations, as well as lack of funds. During this quarter, Glatfelter collected a sizable amount of PHA-free waste activated sludge biosolids from the wastewater treatment system at their Chillicothe, OH mill for our use in composite formulations together with commercial purified PHAs. This material is targeted to be utilized to investigate the properties of the composites with PHB containing cell debris. Because of the strong odor released by the cell wall proteins in these biosolids at composite processing conditions, we will be able to commence reasonably-scaled Task 4 experiments using these biosolids only after we install a ventilation system over the extruders.

Task 5 Status:

To meet the requirements of the project scope and period of performance, it is critical that Task 5 be a schedule-driven task. During this quarter, development of the pilot PHA production system was initiated. David Thompson and William Smith from INL, and Frank Loge from UC

Davis traveled to the Glatfelter mill in Chillicothe, OH in early January to meet with Kathy Wiedeman, Jim Flanders, and Glaffelter facilities staff to identify an onsite location for the pilot system and to discuss the design requirements. A site was identified and a schedule was developed for the pilot system design, development, construction, installation, and operation. The schedule was developed to meet the overall project period of performance and requires that the construction be completed at UC Davis by the end of August 2007. The system must be installed and operational at the Glatfelter mill in Chillicothe by the end of fiscal year 2007 (September 30). While this is a tight schedule, it is necessary in order to allow sufficient time for the composite extrusion elements of Task 5 to be completed within the period of performance for the project. To achieve this, necessary equipment must be procured and delivered to UC Davis by early July. Hence, meeting the PHA pilot system schedule requires that funds be available for the necessary procurements in May 2007. We are currently in the process of determining the total cost of the pilot system equipment, construction and delivery to Chillicothe. It is likely that additional funds in excess of those currently allocated for FY2007 will be required in FY2007 to meet the schedule requirements.

A 3-stage pilot-scale process for the production of PHA-rich biosolids utilizing pulp mill waste effluents has been designed by the UC Davis team and is diagrammed in Figure 4. In the first stage, a specified ratio of the high-strength carbon waste stream (for this project, a foul condensate) and the primary clarifier effluent (used to dilute the foul condensate) is mixed in an

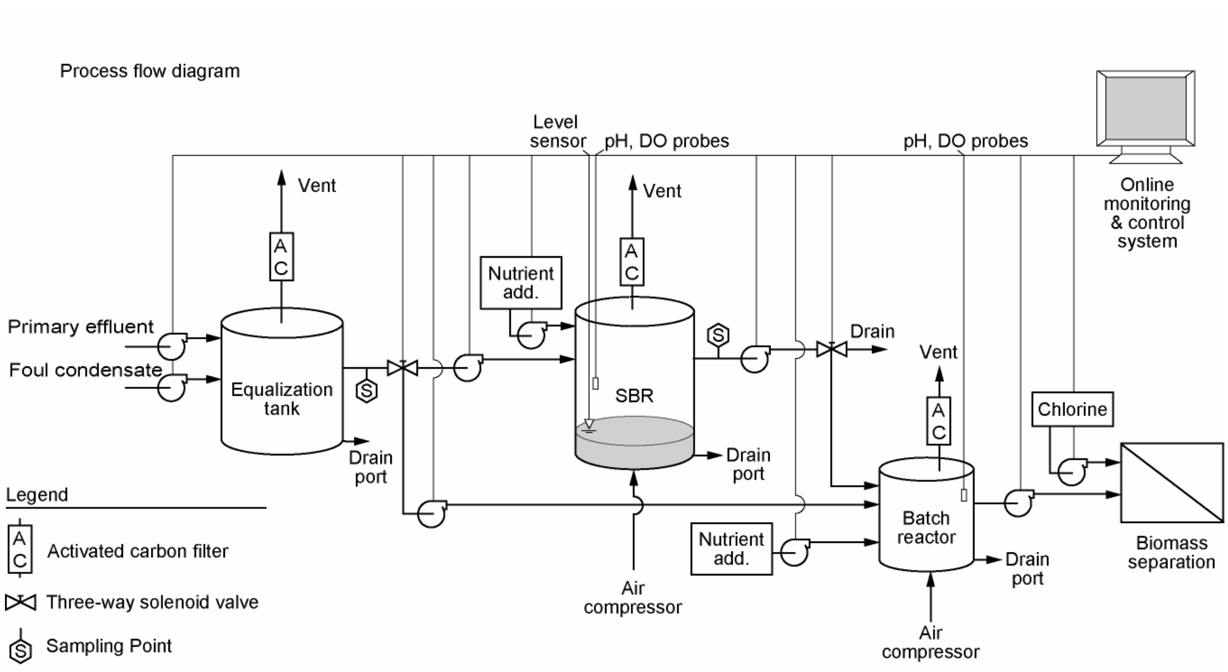


Figure 4. *Preliminary design of the pilot scale PHA production process*

equalization tank to obtain the appropriate substrate and nutrient loadings. In the second stage, selective pressure (i.e. feast-famine dynamic feeding) is imposed on the activated sludge to enrich microorganisms capable of PHA accumulation. Subsequently, the selected/acclimated microbes are fed with a higher substrate load for PHA enrichment in the third stage. Biomass will be harvested daily. This design is undergoing review at INL, Glatfelter, and UC Davis.

Plans for Next Quarter:

For Task 1, we expect to complete the planned Task 1 research, which includes the remainder of the thermal and dynamic mechanical analyses of the composites with different additives and from different processing conditions.

Task 2 research was completed during the last quarter. No further tests are planned for Task 2.

For Task 3, work will focus on the characterization of the effects of C:N ratio on growth and PHA production in the methylotrophic microbial enrichments when they are fed mixtures of specific pulp and paper mill effluents from the Glatfelter Plant in Chillicothe, OH. Foul condensate from the 3200 tank will be used as the primary carbon and nitrogen source; effluent from the primary waste water treatment process will be used as the aqueous growth medium and source of soluble micronutrients. The mixture of 3200 tank foul condensate and primary wastewater will be tested at C:N ratios of 3, 20, 60, 100, and 140. Since methanol is the primary carbon source for these enrichments and constitutes approximately 80% of the COD in the combined foul condensates, the highest C:N ratios will be achieved through methanol amendment. Side-stream batch reactors will be used to investigate the influence of F:M ratio on PHA production in enrichments run at the various C:N ratios.

Task 4 work at WSU remains on hold due to insufficient funds availability. As we expect to complete Task 1 work during the next quarter, we will commence Task 4 late during the next quarter. We expect to install the ventilation system over the extruder before proceeding to work with significant amounts of the PHA-free biosolids obtained from Glatfelter. It is critical that funds be available for this task as its composite formulation data are a prerequisite to perform the pilot scale extrusion tests in Task 5 during the next fiscal year.

For Task 5, the next step requires finalization of the design, procurement of the equipment for the system, and construction and installation of the pilot PHA-production system at the Glatfelter mill. As stated above, the necessary equipment must be procured and delivered to UC Davis by early July. Hence, meeting the project schedule (and the PHA pilot system schedule) requires that funds be available for the necessary procurements in May 2007. We are currently in the process of determining the total cost of the pilot system equipment, construction and delivery to Chillicothe. UC Davis, Glatfelter, and INL have identified several pieces of equipment that can be loaned by each institution for the construction of the unit, however, significant procurements will still be necessary during the next quarter to construct and install a working and controllable pilot system at the Glatfelter mill. It is likely that additional funds in excess of those currently allocated for FY2007 will be required in FY2007 to meet the schedule requirements. Hence, the extent of work completed on this task during the next quarter will depend largely on funds availability to purchase the necessary equipment. To complete the project in the approved period of performance, it will be necessary to have the system installed and ready to be used to enrich large quantities of WAS at the Glatfelter mill by the end of fiscal year 2007 (September 30). In the interim, the UC Davis team will focus on finalizing (a) the design of the pilot-scale system and (b) equipment and cost estimate. Procurement will begin in May for the equipment to construct the pilot unit.

Patents:

No patents resulting from the project were awarded or applied for during this quarter.

Publications/Presentations:

No publications were published or presentations made during this quarter.

Milestone Status Table (as of March 2007 accounting month end):

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.0	**Task 1 – Purified PHA ± Cell Debris Composite Processing & Material Properties**			
1.1	Physical & rheological properties of PHAs and WFRTCs defined	3/31/06		Ongoing; delayed due to funding shortfalls
1.2	Composite processing/mechanical properties of pure PHA WFRTCs completed	7/31/06	6/30/06	
1.3	Wood/purified PHA composites with integrated cell debris produced having MOR ≥ 1500 psi and MOE ≥ 0.20 Mpsi	8/4/06	12/31/05	Met and exceeded
2.0	**Task 2 – Effect of Feedstock on PHA Type/Amount**			
2.1	WTE survey of several waste treatment facilities completed	7/1/06	6/30/06	Further tests useful but not critical
2.2	Enriched paper mill inoculum source and/or ATCC Sphaerotilus culture ready for testing	11/30/06	11/1/06	Five enriched mill sources
2.4	PHA produced from a PPE source by indigenous or inoculated laboratory cultures at ≥ 1 wt% of the dry cell mass	4/1/07	6/5/06	Met and exceeded
2.3	Unsupplemented PHA from PPE completed	8/1/07	3/31/07	
3.0	**Task 3 – Supplementation of Waste Effluents for Production of PHA**			
3.1	PPE supplements & production criteria for pilot test defined	12/15/07		
3.2	In situ PPE process requirements for pilot test defined	12/15/07		
4.0	**Task 4 – Waste Effluents PHA Composite Processing & Material Properties**			
4.1	Material & properties defined for waste-PHA composites	815/07		Delayed due to funding shortfalls
4.2	Wood/purified PHA composites with integrated cell debris produced having MOR ≥ 2000 psi and MOE ≥ 0.25 Mpsi	8/15/07		Delayed due to funding shortfalls
4.3	Basic processing conditions defined for pilot test	10/15/07		Delayed due to funding shortfalls
4.4	Formulations identified for pilot extrusions	4/1/08		
5.0	**Task 5 – Pilot-scale Extrusion Testing of Waste Effluents PHA Composites**			
5.1	Pilot test plan completed	12/15/07		Additional funds will likely be required in FY2007 to meet the Task 5 schedule
5.2	Supplemented or unsupplemented PPE biosolids produced for pilot extrusions	2/15/08		
5.3	Pilot extrusions completed	7/15/08		
5.4	Project completion and transition planned to technology demonstration phase	7/31/08		
5.5	Final Report delivered to DOE	9/30/08		

Budget Data (as of March 2007 accounting month end):

Phase / Budget Period			Approved Spending Plan			Actual Spent to Date		
			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	**To**						
Yr 1	10/1/04	9/30/05	$262,000	$305,411	$567,411	$260,204	$334,364	$594,568
Yr 2	10/1/05	9/30/06	$225,000[a]	$459,726	$684,726	$224,382	$293,090	$517,472
Yr 3	10/1/06	9/30/07	$550,514[b]	$482,006	$1,032,520	$ 110,103	$ 76,910	$187,013
Yr 4	10/1/07	9/30/08	$402,486	$137,400	$539,886			
Yr 5								
		Totals	$1,440,000	$1,384,543	$2,824,543	$594,689	$704,364	$1,299,053

a An amount of $1,797 was carried over from FY2005 to FY2006 and is not included in this figure.
b An amount of $2,417 was carried over from FY2006 to FY2007 and is not included in this figure.

Rapid, Low Temperature Electron, X-Ray, and Gamma Beam Curable Resins

Griffith: Oak Ridge National Laboratory

Agr id:10276

Quarterly Progress Report
DE-AC05-00OR22725

QUARTERLY PROGRESS REPORT

Project Title: Rapid, Low-Temperature Electron, X-ray, and Gamma Beam-Curable Resins

Covering Period: January 1, 2007 through March 31, 2007.

Date of Report: April 30, 2007

Recipient: Oak Ridge National Laboratory
Managed by UT Battelle, L.L.C for the Department of Energy
Post Office Box 2008
Oak Ridge TN 37831-6150

Award Number: DE-AC05-00OR22725

Subcontractors: P. M. Winistorfer, Department of Wood Science and Forest Products, Virginia Tech

G. F. Dorsey, W. W. Moschler, Jr., T. G. Rials, and D. P. Harper, Tennessee Forest Products Center, University of Tennessee

Other Partners: J. B. Eppner, Dow Chemical Company, 2301 N. Brazosport Blvd., Freeport, TX 77451.

L. A. Parks and Cheng Song, SteriGenics International, Inc., 7695 Formula Place, San Diego, CA 92121-2418.

A. G. Landers, J. M. Huber Corporation, Engineered Woods, P. O. Box 670, Commerce, GA 30529.

J. Fyie, TrusJoist, a Weyerhaeuser Business,2910 East Amity Road, P. O. Box 8449, Boise, ID 83707-2449.

Contact(s): William L. Griffith, (865) 574-4970, griffithwl@ornl.gov

Project Team: Project Team: Drew Ronneburg, DOE program manager; Wood and Wood Composites Task Group (mentors).

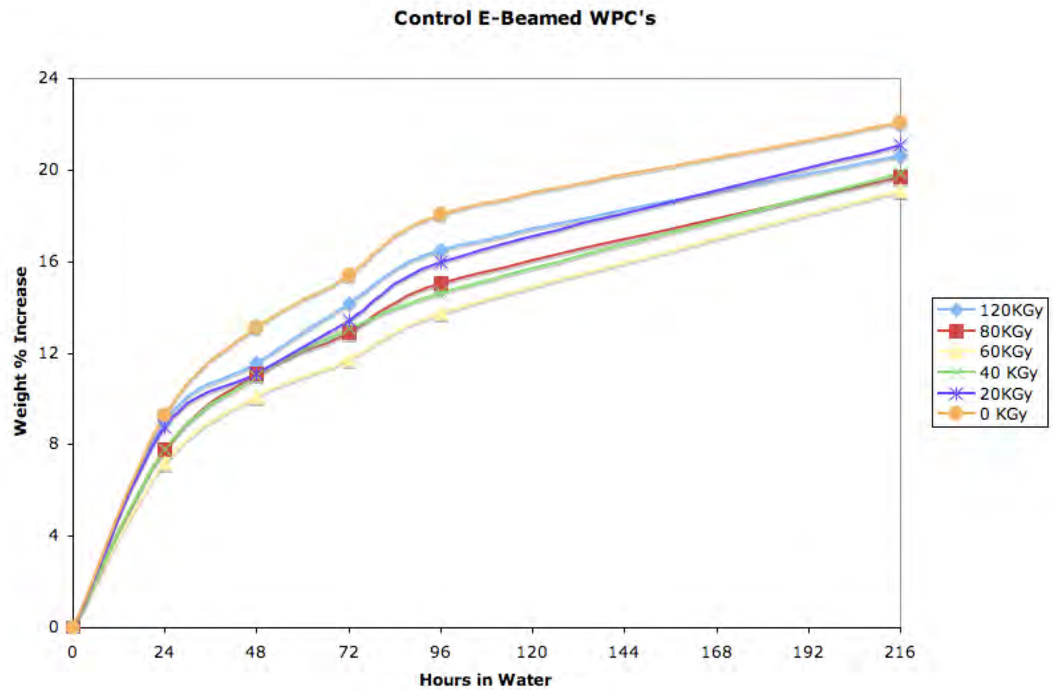
Background: Approximately 50% of all wood used today is some type of glued-wood assembly. The manufacture of most glued-wood assemblies requires process heat. Process heat is required to dry the parent wood material, assist in consolidation of the product (flat-pressed panel products) and polymerize and cure the resin system. Glued-wood products range from structural laminated beams and flat-pressed panels to furniture assemblies and non-structural wooden assemblies. Many of these products are referred to as wood composites. The moisture content of the wood materials must be reduced to low levels and then be carefully controlled to ensure efficient resin polymerization and eventual resin curing and to avoid generation of excessive steam vapor pressure internal to the product. Drying the wood furnish materials and controlling the substrate moisture content is a major consumer of energy in the manufacturing plant. "Hot-pressed" wood panels such as oriented strand board, medium density fiberboard (mdf) and particleboard can be mismanufactured due to lack of moisture control.

Development of rapid, low-temperature electron beam-curable resin systems offers an **energy savings potential to the wood composites industry of 65 Trillion Btu/yr at full market penetration.** The lower curing temperatures (from 450 °F to 250 °F) possible with beam-curing systems also offer the potential of reducing unit capital costs and doubling throughput. The **lower curing temperatures can also decrease process emissions by reducing organic volatiles**.

Status:
The global objective for this project is to produce new adhesives for wood composites that can be cured at low temperatures, with little drying of raw materials, and virtually instantaneously. Development of these new adhesives offers tremendous energy savings over more commonly used methods to cure wood adhesives and has previously been extensively reported. In this past quarter, we have made significant progress in three key areas important to the adoption of these new adhesive systems: 1) have begun quantifying the degree of interaction of target adhesive systems with the wood substrates, 2) have formulated new adhesive systems that take advantage of the benefits of thermal and e-beam cure without sacrificing much thermal energy, 3) are determining the optimum formulation and dose for novel extruded wood-polymer composites. In addition, a wood composites trade journal article was published this quarter promoting the use of e-beam curable adhesive technologies in wood composites.

Dynamic mechanical analysis (DMA) has been performed on wood strands saturated with an acrylic (R67) and a methacrylate (R46) e-beam curable formulation. The adhesives were subjected to e-beam doses from 0 to 120 kGy. The adhesive have displayed an ability to swell wood, which gives a qualitative indication of molecular interaction. We have run a series of “dry” and submersion DMA tests. The DMA specimens were submerged in a bath of ethylene glycol in order to plasticize the wood polymers. This allows one to view the entire range of the glass transition of lignin without thermal degradation. Some shifts in the wood lignin peak have been observed. We are currently attempting to determine if this is a molecular interaction (e.g. molecular coupling), or if this is an additive effect caused by the glassy transition point (Tg) of the adhesive. In addition, dry DMA specimens have been run to determine the impact of wood and e-beam dose on the adhesive. The transitions in the wood are comparatively small and high in temperature compared to the adhesives when not sufficiently plasticized.

New combined cure systems have been formulated and evaluated. These new adhesive systems demonstrate an efficacy for adhering wood and improved toughness within service temperature ranges. Small composites have been manufactured and tested via dynamic mechanical analysis, calorimetry, and infrared spectroscopy. These new adhesive formulations exhibit thermoplastic behavior while retaining e-beam curing functionality after multiple heating/cooling cycles. The dynamic mechanical properties appear to be favorable for wood-composite applications.


Control E-Beamed WPC's
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12
16
20
24
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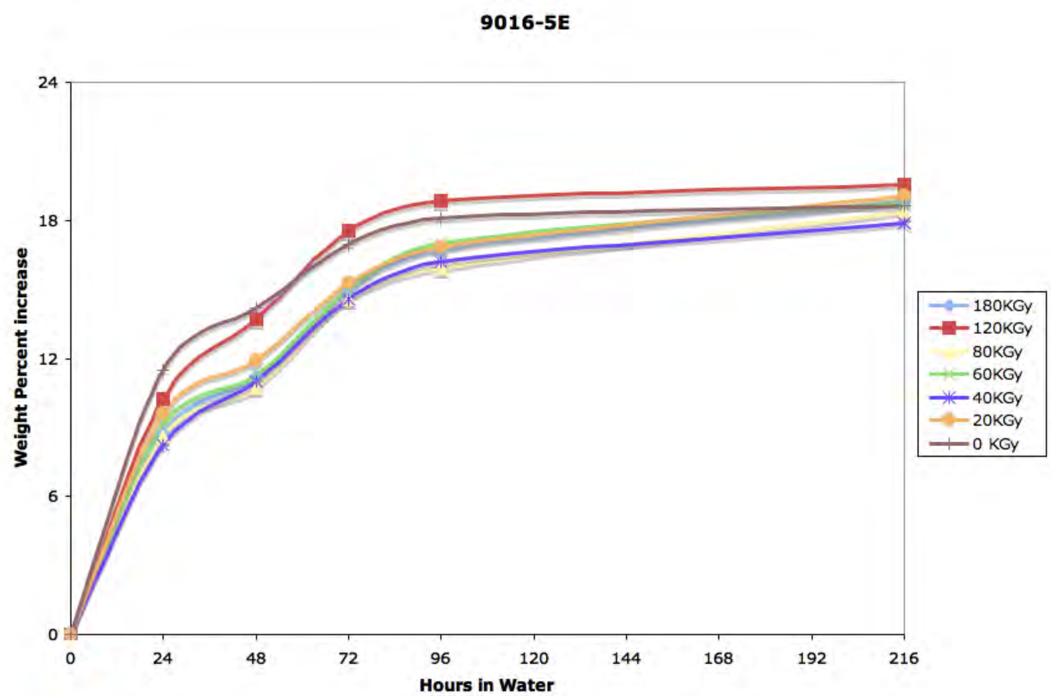
9016-5E
Weight Percent increase
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6
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168
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216
180KGy
120KGy
80KGy
60KGy
40KGy
20KGy
0 KGy

Extruded wood-high density polyethylene (HDPE)-metal acrylate (9016) or metal methacrylate (708) composites were produced and irradiated at doses between 0 – 180 kGy. Water sorption studies are being carried out to determine the composites resistance to water uptake (a primary application concern). Preliminary results indicate that e-beam treatment slows the uptake of moisture. Further, as shown in figures, formulations not containing an e-beam curable additive appear to be retaining water at a higher rate from 120 hrs to 216 hrs. However, these results are still preliminary.

Plans for Next Quarter:. Editing of the Five year report will be completed. Additional commercial prototypes will be prepared. Final preparations for the technology workshop are expected to be finalized. Trade publications describing the new technology are to be published. Presentations will be made ANTEC2007 and the SAMPE Baltimore 2007 meetings.

Patents: None

Publications/Presentations: Papers were accepted for presentation and publication at the ANTEC2007 and the SAMPE Baltimore 2007 meetings. A publication entitled E-beam Curable Resins will appear in the April 2007 issue of the Specialty Wood Journal, p43.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1.1	Survey existing wood adhesive systems	09/30/02	09/30/02	
1.2	Evaluation of initial resin systems	12/31/02	12/31/02	
1.3	Develop test protocol for block-tests	12/31/02	11/05/02	
1.4	Initiate adhesion studies	03/31/03	03/01/03	
1.5	Ethylenic and acetylinic bond systems	09/30/03	09/30/03	
1.6	Downselect promising resin systems	05/01/04	05/01/04	
1.7	Properties of glued wood assemblies	08/30/04	08/30/04	
1.8	Evaluation of process energy balance	09/30/04	09/30/04	
1.9	Initiate testing of large sections	01/01/05	12/23/04	
1.10	Evaluate alternative beam application methods including X-rays	09/30/05	09/30/05	
1.11	Select large-scale systems	01/01/06	01/01/06	
1.12	Draft Five-year Project Report	12/31/06	12/31/06	Issue prior to Workshop
1.13	Technology Workshop	09/30/07		Revised

Budget Data (as of 3/31/07:

			Approved Spending Plan			Actual Spent to Date		
Phase / Budget Period			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	To						
Year 1[a]	10/01/01	9/30/02	200,000	71,000	271,000	116,000	65,000	181,000
Year 2	10/01/02	9/30/03	210,000	91,000	311,000	158,000	91,000	249,000
Year 3	10/01/03	9/30/04	220,000	91,000	321,000	262,000	91,000	353,000
Year 4[a]	10/01/04	9/30/05	230,000	91,000	331,000	21,000	91,000	112,000
Year 5[a]	10/01/05	9/30/06	240,000	71,000	240,000	59,000	71,000	123,000
Year 6[b]	10/01/06	9/30/07		30,000		15,000	15,000	30,000
Totals			1,000,000	445,000	1,445,000	616,000	429,000	1,048,000

[a] We received $150,000 in funding during FY 2002; $95,000 in FY2005; and $40,000 in FY2006.
[B] Additional $30,000 industrial cost share for FY2007.

Novel Isocyanate-Reactive Adhesives for Structural Wood-Based Composites

Frazier: Virginia Tech

GO14307

As of May 15, 2007, the PI has not submitted a status report for the quarter ending March 31, 2007.

Biological Air Emissions Control for an Energy Efficient Forest Products Industry of the Future

Jones: Texas A&M

GO14310

QUARTERLY PROGRESS REPORT

Project Title: **Biological Air Emissions Control for an Energy Efficient Forest Products Industry of the Future**

Covering Period: **January 1, 2007 through March 31, 2007**

Date of Report: **April 29, 2007**

Recipient: **South Texas Environmental Institute**
Texas A&M University-Kingsville
Department of Environmental Engineering, MSC 213
Kingsville, Texas 78363

Award Number: **DE-FC36-04GO14310**

Subcontractors: **Bio•Reaction Industries LLC**
18500 SW Teton Avenue
Tualatin, OR 97062

Other Partners: **Stimson Lumber Company**
Forest Grove, OR

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Project Objectives

The gaseous emissions from the hardboard mill presses at lumber plants such as that of the Stimson Lumber Company contain both volatile and condensable organic compounds (VOC and COC, respectively), as well as fine wood and other very small particulate material. In applying bio-oxidation technology to these emissions Texas A&M University-Kingsville (TAMUK) and BioReaction (BRI) evaluated the potential of this equipment to resolve two (2) control issues which are critical to the industry:

- First, the hazardous air pollutant (HAP) emissions (primarily methanol and formaldehyde) and
- Second, the fine particulate and COC from the press exhaust which contribute to visual emissions (opacity) from the stack.

In a field test in 2006, the biological treatment technology met the HAP and COC control project objectives and demonstrated significantly lower energy use (than regenerative thermal oxidizers (RTOs) or regenerative catalytic oxidizers (RCOs), lower water use (than conventional scrubbers) all the while being less costly than either for maintenance. The project was continued into 2007-2008 to assist the commercial partner in reducing unit size and footprint and cost, through added optimization of water recycle and improved biofilm activity.

Q1 2007 Status

To further enhance the technology's commercialization and reduce technology startup costs, allowing for broader implementation by the forest products industry, additional field testing and laboratory testing is ongoing to reduce the unit footprint and potentially reduce costs for the industrial clients. This testing will be focused on developing increased optimization parameter criteria and performance evaluation before the implementation of MACT guidelines by EPA, which were postponed until mid 2008.

The lab scale biofiltration system has been constructed and started up specifically targeting improving removal efficiency for α-pinene and formaldehyde contaminants in air flows. This mixture exemplifies the challenges for the industry to optimally remove both a moderately soluble and a sparingly soluble contaminant mixture with reduced retention times and smaller unit sizes. Analytical methods for accurate detection of both compounds are being developed with new GC columns and temperature programs.

The biofilter media used in the process must provide suitable conditions for microbial growth and maintain high porosity to allow air to flow easily. The media provides support and contact between the gas phase contaminants and active microbial cultures attached as biofilms on the media's surface. The transformations in the biofilm and the media during optimal biofiltration operations are not well understood. The main aim of this part of the project was to characterize the biofilm formed on the media during the biofiltration process using Fourier Transform Infrared Spectroscopy (FT-IR) and the FT-IR Microscope, and also attempt to correlate the results with the performance data of the biofiltration process for VOCs.

An infrared spectrum represents a fingerprint of the sample with absorption peaks corresponding to the bonds of the molecules making up the material. The size (height) of the peak in the sample is a direct indication of the amount of material present. Engineered biomedia samples from a field scale operation at the Stimson Lumber Plant in Oregon, and samples from the biofiltration experiment conducted in the laboratory at the Texas A&M University-Kingsville were analyzed for the identification of organic compounds present. The FT-IR bench scale and microscope were used to analyze the media samples from the field and lab biofiltration experiments, to evaluate the degradation of α-pinene. The fingerprint of the biofilms on the biofilter media at various stages in the biofiltration process was correlated with the performance of the biofilters. It was discovered that there were qualitative differences in the biofilms in the top and the bottom bed of the biofilters, and the performance of the sections. The bench (Attenuated Total Reflectance) unit and the microscope (Transmission) results were compared during the characterization of the biofilm, and both found to be useful. This characterization of the media and biofilm are being evaluated a second time in the lab experiments to focus on improvements in biofilm quality and coverage which may lead to improved contaminant removal performance.

A peer reviewed article describing the project field test at Stimson Lumber in 2006 was revised and re-submitted to the *Biochemical Engineering Journal* for publication during this quarter. Another article outlining the results of biological treatment unit field test and describing the energy saving features of the technology appeared in *FDM Asia*, a publication targeting forest products manufacturers in the Pacific Rim and Asian markets.

Bio•Reaction Industries (BRI) has been actively working within the U.S. Wood Products industry marketing the biological treatment technology at trade shows and with client visits over the past year. During the past six months Bio•Reaction Industries has been actively marketing the technology in the U.S. and abroad, both in Europe and Asia. Several applications have been quoted in Asia and potential applications are currently being explored in Europe. Both Asian and European activities are in concert with industrial engineering partners on those continents, respectively. In North America several units have been sold, one 160,000 acfm unit for the wood products industry is under construction and the contract for a 30,000 acfm unit for controlling paint emissions has been signed. Additional marketing effort is being directed toward Ethanol Production, controlling emissions from the "spent mash" dryers, used as animal feed. In addition, a presentation was made at the Water Environment Federation annual conference, October 2006, and another is scheduled for the Air & Waste Management Association annual conference in June 2007.

Q2 2007 Plans

The project partners have developed a design for another field pilot test for the Stimson Lumber Company emissions application, in which the opacity reduction capability of the technology will be evaluated and optimized through trickling water recycle and flow rate or retention time variation. This evaluation will utilize a smoke and particulate testing meter with a stack piece, rather than relying solely on operator observations. This testing is being planned for the end of the 2nd Quarter 2007.

The optimization experiments for enhanced biofilms targeting pinene and formaldehyde removals in the influent air streams will be continued in the TAMUK laboratories to be used in the process model for design improvement evaluation.

The research group is evaluating the development of new media with improved porosity and biofilm coverage which may reduce the cost of the field scale implementation of the technology.

Patents: None this Quarter

Publications/Presentations:

Santos, S., Jones, K., and Boswell, J. (2005) "Biological Treatment of Air Emissions for Forest Product Industry Applications," presented at the NSF sponsored CREST-RESSACA Conference on Emerging Technologies for a Sustainable Environment, October 20-21, South Padre Island, Texas.

Santos, S., Jones, K., Baliwala, L., Abdul, R., Boswell, J. and Cochran, J. (2006) Treatment of Wet Process Hardboard Plant Emissions by a Pilot Scale Biological System, published in the Proceedings of the 2006 USC-TRG Conference on Biofiltration for Air Pollution Control, Long Beach, California, October 18-20.

Jones, K., Santos, S., Baliwala, L., Boswell, J. and Paca, J. (2006) Biological air emissions control for α-pinene and formaldehyde for a forest product industry application with a coupled biotrickling filter and biofilter system, in Proceedings of 17th International Congress of Chemical and Process Engineering CHISA 2006, Prague, Czech Republic, 27–31 August 2006.

Jones, K. (2007) Saving Energy, Saving Earth, *FDM Asia*, v19, n3, April 2007.

Jones, K., Boswell, J., Santos, S., Cochran, J., and John, P. (2007) Treatment of Wet Process Hardboard Plant VOC Emissions by a Pilot Scale Biological System, submitted for publication to *Biochemical Engineering Journal*.

Milestone Status Table:

ID	Tasks/Milestones				
No.		Planned Completion	Actual Completion	Comments	
1.	Bench Scale and Field Scale Unit Design	12/31/04	12/31/04		
2.	Field Pilot Unit Construction	6/30/05	8/15/05		
3.	Field Pilot Unit Startup	7/31/05	8/15/05		
4.	Preliminary Pilot Scale Testing and Field Testing Plan Development	10/1/05	8/15/05		
5.	Optimization of Biotrickling Filter	12/31/05	3/15/06	Field unit 1	
6.	Optimization of Biofilter Section	12/31/05	3/15/06	Field unit 1	
7.	Develop process models	2/28/06	8/31/07	continued	
8.	Optimize water reuse	2/28/06	8/31/07	continued	
9.	Characterize microbial biofilm	2/28/06	8/31/07	continued	
10.	BF Tech Product marketing	12/31/08		Ongoing	
11.	Final Report Phase I Publication	9/1/06		Revised – in review	
12.	Optimization of Bench BTF	12/31/07			
13.	Optimization of Bench BF	12/31/07			
14.	Final Report Phase II Publication	9/1/08			

Budget Data:

Texas A&M University – Kingsville Award DE-FC36-04GO14310 **Project Spending and Estimate of Future Spending**							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays****	**Cumulative**
	Start		Note 1		Note 1		
3Q04	9/1/04	9/30/04	0	0	0	0	
4Q04	10/1/04	12/31/04	4,642	4,642	2,000	2,000	
1Q05	1/1/05	3/31/05	3,922	3,922	3,500	3,500	
2Q05	4/1/05	6/30/05	18,955	18,955	15,000	15,000	
3Q05	7/31/05	9/30/05	25,649	25,649	20,000	20,000	
4Q05	10/1/05	12/31/05	26,696	26,696	17,265	17,265	
1Q06	1/1/06	3/31/06	95,220	95,220	34,235	34,235	
2Q06	4/1/06	6/30/06	15,892	15,892			
3Q06	7/31/06	9/30/06	1,753	1,753			
4Q06	10/1/06	12/31/06	16,497	16,497			
1Q07	1/1/07	3/31/07	14,988	14,988			
2Q07	4/1/07	6/30/07	20,000		15,000		
3Q07	7/1/07	9/30/07	20,000		15,000		
4Q07	10/1/07	12/31/07	20,000		15,000		
1Q08	1/1/08	3/31/08	20,000		15,000		
2Q08	4/1/08	6/30/08	20,000		15,000		
3Q08	7/1/08	9/30/08	18,486		10,000		
Totals			**342,700**	**209,226**	**177,000**	**92,000**	

Federal Share = $192,729 (9/1/04-8/31/06) + $149,971(9/1/06-8/31/08)
* Update quarterly
**Estimated for 2006
General Note: DOE Laboratory partner spending should not be included in the above table. If a DOE Laboratory is a partner, report their spending and spend plan information in the table below (use separate tables if multiple DOE Laboratories are involved).
General Note: The information in this table should be consistent with the information provided in section 10 of the quarterly financial status reports (SF269 or SF269A).
Note 1: Leave blank. Only the actual DOE/Cost Share amounts spent through 6/30/04 are needed.
Note 2: Amount for this quarter and subsequent quarters should be updated as necessary on a quarterly basis. Estimates need to be provided for the entire project. If spending for a given quarter is different than estimated, then the remaining quarter's estimates should be updated to account for the difference. Total DOE and Cost Share amounts should be the same as the Award amount.

Low VOC Drying of Lumber and Wood Panel Products

Banerjee: IPST at Georgia Tech

ID13439

PROGRESS REPORT

Project Title: Implementing Strategies for Drying and Pressing Wood Without Emissions Controls

Authors: Rallming Yang, Sujit Banerjee
IPST at Georgia Tech

Covering Period: January 1, 2007 to March 31, 2007

Date of Report: April 21, 2007

Recipient: Institute of Paper Science and Technology

Award Number: DE-FCO7-96ID13439

Other Partners: Lawrence Otwell (Georgia-Pacific)

Contact: Sujit Banerjee (404) 894-9709; s.banerjee@ipst.edu

Project Objective: The objective of this project is to devise strategies for sizing down control devices needed for treating VOC emissions from dryers and presses for wood products such as OSB and veneer.

Background: Our previous work has brought us to the point where we can dry wood full-scale for brief periods without emissions controls. We now need to do this consistently and without adversely affecting throughput and production economics. Our first objective is to identify the mechanisms of release of some of these HAPs, to identify second-order variables that affect HAPs generation, and to develop and field-demonstrate a comprehensive strategy. Our second goal is to reduce resin use during pressing through droplet control. An ancillary goal is to determine the feasibility of applying urea as a fine mist to the mat in order to quench formaldehyde emissions.

Status:

- Contacting green flakes with dilute sodium hydroxide or hydrogen peroxide prior to drying increases the contact angle of water or MDI resin on the dried flake. The drying rate is unaffected, at least with NaOH treatment.
- Treating flakes with a dilute suspension of silicon oil prior to drying reduces the contact angle of bonding resin but increases the contact angle of water.

Changing the resin-wood contact angle by pretreating flakes

In the previous quarter we found that brief contact of wood flakes with dilute NaOH improved the surface properties of the flakes, which could lead to lower resin use. We have pursued this approach with several chemicals in addition to NaOH. Briefly, green wood flakes were cut into two 1.5" x 0.5" halves. One was dipped into the solution used for conditioning the flakes; the other was dipped into water as a control. The excess water was shaken off and the wood was dried for 30 minutes at 150°C to an MC of <5%. The weights of the flakes before and after treatment, and after drying were recorded. The contact angles of water, PF and MDI resin were measured on the dry flakes with a Dynamic Contact Angle Analyzer model FTÅ 200 from First Ten Angstroms. The contact angle of water was determined, the flake was then redried for 10 minutes at 150^{o}C to remove the added water, and then used again for the MDI measurement.

<u>NaOH treatment</u>

Contact angles were measured after 5 seconds and 20 seconds after placing the droplet on the surface of the flake. Seven data points were collected. A t-test was carried out to determine if the differences in contact angles between the treated flakes and their controls were significant. The results in Table 1 demonstrate that water contact angle is significantly increased. The change for MDI is much less significant, probably because the angle is low to begin with. Changes in the surface pH of the flakes after NaOH treatment were also measured with a pH microprobe after the surface was wetted. As shown in Table 2, the surface pH increased substantially.

<u>Treatment with hydrogen peroxide</u>

Flakes were dipped in 0.5 and 2% hydrogen peroxide and dried. The contact angle of water and MDI was then measured on the same flake, which was redried between measurements. The results reported in Table 3 show that the contact angle of an MDI droplet is raised substantially upon 0.5% peroxide treatment. The effect on a water droplet is less significant, but increasing the peroxide concentration to 2% leads to a large increase in the water contact angle, confirming the general trend.

<u>Treatment with silicon oil (Poly-dimethylsiloxane)</u>

Silicon oil was prepared as a stable 0.5% emulsion in water with a variety of surfactants including ethyltrimethyl ammonium bromide, polyethylene glycol, and glycerol added at 0.2% by weight. The mixtures were stirred and then sonicated for 15 minutes. The emulsions were stable at room temperature over 68 hours.

Table 4 is summarizes contact angle results using polyethylene glycol. Results for the two other surfactants were very similar. Silicon oil treatment *increases* the contact angle of water, but *decreases* that of MDI, which contrasts with behavior observed with sodium hydroxide and hydrogen peroxide where the contact angle of water and MDI changed in the same direction. The silicon oil result is important because it simultaneously sizes the flake while at the same time increasing resin spread.

Table 1: Effect of NaOH on contact angle.					
MC (per-cent)	NaOH (percent)[1]	5s contact angle		20s contact angle	
		treated	control	treated	control
water droplet					
200	0.09	90.0	87.7	59.0	59.1
185	0.08	91.9	95.3	78.4	57.2
190	0.07	93.3	95.0	72.9	51.2
168	0.05	83.6	88.0	64.8	57.4
162	0.04	88.0	89.8	63.1	60.7
209	0.07	88.5	89.1	69.1	56.3
185	0.06	90.1	92.5	67.5	52.4
184	0.08	88.4	86.2	53.8	51.5
138	0.07	86.9	86.8	39.3	45.0
129	0.04	86.5	88.8	43.6	47.1
98	0.03	85.6	87.5	45.5	44.3
164	0.06	84.8	85.9	45.8	54.2
t-test		0.028		0.043	
MDI droplet					
152	0.07	55.3	55.5	21.9	20.7
200	0.06	49.1	53.1	23.0	23.0
186	0.10	51.3	55.5	27.0	27.7
155	0.05	53.6	54.9	22.2	20.1
139	0.05	54.8	56.3	24.1	19.5
133	0.06	54.8	53.1	22.9	20.1
190	0.06	50.2	55.5	24.5	21.0
133	0.06	50.8	51.2	18.9	21.6
143	0.05	53.3	51.0	16.8	18.4
175	0.08	56.3	53.6	16.9	20.7
t-test		0.14		0.28	
[1]od wood basis.					

Table 2: Effect of NaOH on surface pH.						
NaOH (%)	0 (control)			0.1	0.1	1
surface pH	4.89	4.95	4.91	7.01	6.89	8.98

Table 3: Influence of hydrogen peroxide on dried wood flakes contact angles.							
MC (%)	H_2O_2 (%) od basis	5 sec contact angle			20 sec contact angle		
		treated	control	t-test	treated	control	t-test
water droplet							
2.0% H_2O_2							
227	1.38	97.6	105	0.008	75.0	92.6	0.001
133	1.14	94.0	98.2	0.032	81.9	85.2	0.024
162	0.76	84.6	99.1	0.000	72.2	94.1	0.000
0.5% H_2O_2							
128	0.22	86.1	87.6	0.113	64.7	62.8	0.17
184	0.68	87.1	89.6	0.067	65.6	64.3	0.22
157	0.57	87.8	88.9	0.209	59.5	66.0	0.12
MDI droplet (2.0% H_2O_2)							
148	0.43	57.6	61.6	0.043	18.3	23.7	0.005
193	1.78	55.9	59.2	0.031	15.7	18.7	0.010
138	0.86	55.4	60.8	0.001	18.6	23.2	0.000

Table 4. Influence of silicon oil on wood flakes contact angles.							
Run	silicone % o.d	5 sec contact angle			20 sec contact angle		
		treated	control	t-test	treated	control	t-test
water droplet							
1	0.53	78.4	62.4	0.000	67.0	51.6	0.000
2	0.58	72.6	47.1	0.000	61.3	22.4	0.000
3	0.44	76.4	66.4	0.000	63.9	45.7	0.000
4	0.29	93.0	81.5	0.000	78.8	62.0	0.000
5	0.31	88.5	84.0	0.000	81.4	66.5	0.000
6	0.41	83.1	77.9	0.002	64.5	55.8	0.000
MDI droplet							
1	0.53	30.5	32.7	0.050	15.1	19.5	0.002
2	0.58	28.9	28.2	0.156	15.2	14.8	0.321
3	0.44	28.4	34.3	0.000	15.4	21.6	0.079
4	0.29	31.8	34.7	0.001	16.6	22.3	0.000
5	0.31	27.8	32.5	0.017	14.6	19.1	0.001
6	0.41	30.8	35.1	0.000	14.6	16.2	0.014

The following experiment was requested by Georgia-Pacific. The effect of silicon oil on powdered resin was studied by forming a 1-g 4.9 cm^2 pellet from powder resin (145C64) from GP in a pellet press. The pellet was cut in half with a pair of scissors. One half was placed in the middle of a flake dipped in 0.5% silicon oil in surfactant/water; the other was placed on the

Table 5. Effectof silicon treatment on resin spread.		
run	treated flakes area (cm^2)	control flakes area (cm^2)
1	5.18	4.50
2	1.58	1.23
3	1.43	1.19
4	0.86	0.78
5	0.51	0.51
6	0.97	0.97
7	0.73	0.77
8	0.86	0.78
9	0.51	0.51
10	1.10	0.86
11	1.23	1.06
12	0.86	0.85
13	1.32	1.33
t-test	0.03	

control flake. The flakes were placed side by side on a 6.0" x 6.0" piece of felt covered with a blotter and hot pressed at 25 psi for 7 minutes. The temperatures of the upper and lower platens were 100 and 170°C, respectively. The pressed flakes with the melted resin were then photocopied, and the resin images cut and weighed. The results in Table 5 show that silicon treatment increases resin spread by 9%.

Effect of treatment on drying rate

It seemed possible that the treatments could affect the drying rates of the flakes. Control and NaOH-treated flakes were suspended inside an oven and the changes of flake surface temperature during drying were imaged with an infrared camera. The results of one of several experiments are illustrated in Figure 1. No major differences were seen.

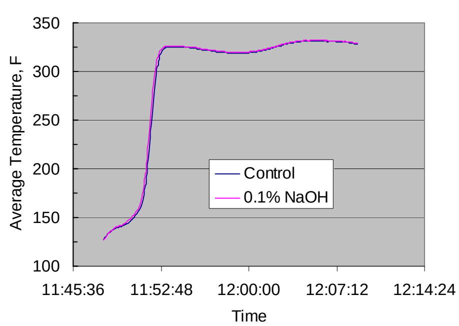

Figure 1: Temperature profiles of control and NaOH-treated flakes during drying.

ID Number	Task/Milestone Description	Planned Completion	Percent Completion	Actual Completion	Comments
1	Identify factors that lead to HAPs generation during drying				
1.1	Lab work	4.04	100	6.04	
1.2	OSB mill trials (rotary dryers)	5.05	100	3.04	
1.3	Veneer mill trials	12.04	100	6.03	
1.4	OSB mill trials (conveyor dryers)	5.05	100	12.04	
2	Integrate items identified above into strategies for reducing emissions during drying				
2.1	Lab work				1
	(a) Construct lab-scale flaker for high-speed imaging of fines production	6.05	100	12.05	
	(b) Devise lab tests for generating input parameters for model	12.05	100	3.06	
	(c) Model fines production from flaker	3.06	100	6.06	
	(d) Pilot trials at LP, Nashville	9.05	100	6.05	
2.2	OSB mill trials				
	Skippers (rotary dryer)	6.06	-		2
	Dudley (rotary dryer)	3.06	-		2
	Guntown (conveyor dryer)	12.05	100		3
	Fordyce (rotary dryer)	9.05	100		
2.3	Veneer mill trials	12.04	100	6.03	
2.4	Work with flaker vendor for broad commercialization	3.07	0		
3	Extend above strategies to pressing OSB and veneer	10.04	100		4
4	Reduce press HAPs by applying urea and resin as a fine aerosol		100		5
4.1	Lab work with urea/resin	12.05	90		
4.2	Effect on product properties	6.06	40		
4.3	Pilots	12.06	30		
5	Studies on knife corrosion				
5.1	Rate studies and characterization	3.05	100	12.04	
5.2	Application of coatings	6.05	100	12.04	
5.3	Field trials	9.05	-		6
6	Economic analysis	12.06	20		
7	Final report	3.07			

Comments

1. Flaking was found to be the principal cause of fines that lead to HAPs upon drying and this element of the project was developed further. An understanding of the process is needed so that the results from the field (which are presently successful) can be applied with confidence.
2. Promulgation of the wood MACT rule was delayed by EPA and the mill trials were pushed back by Georgia-Pacific.
3. Sampling must be done across at least four seasons so that the effect of wood temperature can be modeled.
4. Work on pressing was terminated, and the resources applied to drying where a bigger return is anticipated. This was done upon the advice of our industry advisory board.
5. Work on this item was delayed so that the dryer aspect of the work could be accelerated as discussed above.
6. The Norbord mill does not want to run a field trial because of safety reasons.

Project Spending and Estimate of Future Spending DE-FCO7-96ID13439							
Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative**
2Q07	1/1/07	3/31/07		1,846391			
Totals				1,84,6391		651,059	
Approved (committed)			**1,998,007 (1,867,383)**				

Index of Award CID Numbers

Index of Award CID Numbers