OAK RIDGE
NATIONAL LABORATORY
MANAGED BY UT-BATTELLE
FOR THE DEPARTMENT OF ENERGY

NFE-10-03062

Chemical Sciences

CRADA Final Report
For
CRADA Number NFE-10-03062

Method for Improving Separation of Carbohydrates from Wood Pulping Liquors and Wood or Biomass Hydrolysis Liquors

Alicia L. Compere
Oak Ridge National Laboratory

Bruno S. Marcoccia
Domtar Paper Company, LLC
and
James Elliott
General Atomics

Prepared by
Oak Ridge National Laboratory
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FINAL REPORT for Oak Ridge National Laboratory CRADA NFE-10-03062

Industrial Partners: Domtar Paper Company, LLC and General Atomics

Due to the recent retirement of the PI for this project, the CRADA Final Report consists of the Quarterly Reports that detail the work that was completed on this project.

This report will be listed as Protected CRADA Information for five (5) years from August 31, 2012.

Quarterly Progress Report

Project Title: Method for Improving Separation of Carbohydrates from Wood Pulping Liquors and Wood or Biomass Hydrolysis Liquors

ORNL WBS Element: Not yet assigned

Covering Period: Selection to June 30, 2010)
Date of Report: July 14, 2010

Contact: Alicia L. Compere, Physical Sciences Directorate/Chemical Sciences Division, 865-574-4970, compereal@ornl.gov

Corporate Partner(s): To CRADA: Domtar, General Atomics through MTA for product evaluation: AkzoNobel and Novozymes

Project Objective: Work with industrial partners to perform the studies needed to commercialize U.S. patent 7,699,958 for separation of carbohydrates from wood pulping liquors and wood or biomass hydrolysis liquors. These include: 1) selection of the best pulp mill liquor withdrawal sites, 2) additional purification or enzyme hydrolysis required to obtain acceptable sugar feedstocks, 3) and work with partners to optimize the stream and purification methods to provide acceptable feedstocks for algal fuels and industrial chemicals production, and 4) preparation of samples large enough for testing by downstream partners.

Background: This hemicellulose separation was developed with the support of two DOE - EERE projects. One project was developing an advanced wood pulping process and another was developing methods for converting lignin from wood pulping processes into feedstock for carbon fiber production. From the standpoint of a pulp mill operator, this separation process provides a method for substantially increasing both mill throughput and the yield of wood pulp. It also has the potential to improve emissions and overall plant energy efficiency. Because hemicellulose is the major contaminant in lignins produced from biomass and pulping streams, this separation also dramatically improves the ability to produce “clean” or high-purity lignins produced from biomass and pulping streams.

In the last two years, working with commercialization managers, this technology has been discussed with several potential licensees (Domtar, General Atomics, Novozymes, Milliken, Procter and Gamble, AkzoNobel). The ORNL inventors have discussed this in some detail with Domtar (a pulping company with $5.5 billion 2009 sales, >10,000 employees), General Atomics (GA Advanced Process Systems division is a major developer of algal-based fungible, or infrastructure-compatible, fuels funded by DARPA and individual services), and Novozymes (the leading global developer and producer of the enzymes needed to convert hemicellulose into a form suitable as an algal feedstock). Domtar is interested in the hemicellulose stripping process from the standpoints of increased pulp yield, increased mill throughput, higher black liquors solids concentration, and secondary products (algal and chemical feedstocks). General

Atomics would like to evaluate the hemicellulose as a feedstock for heterotrophic algae which they find to be a particularly good feedstock for a military fuel, BioJP-8. Novozymes is interested in extending its cellulosic enzyme market to high pH hemicelluloses. All three companies are interested in working with ORNL to scale and demonstrate hemicellulose-stripping process.
Several factors are driving this interest. First, General Atomics is in moving toward commercialization of their process for producing totally bioderived military fuels. This is driven by two executive orders, 13423 (Bush) and 13514 (Obama), which require all federal agencies, including the DoD, to increase biofuels use dramatically in the next 10 years and by DoD investments to both develop and fully qualify algal biofuels. This process produces feedstock for algae and, additionally, produces the aromatic and cyclic compounds required in aviation fuels but not produced by algae. These are expected to be critical in production of a bioderived replacement for the major military fuel, JP-8. The DoD is the major (93%) federal (2% of U.S.) billion dollar per year consumer of petroleum (133 million barrels – currently ~$10 billion or 0.4 Quad of raw petroleum – in FY2005, as reported by Dimotakis et al., 2006).

Second, Novozymes has made advances which have dramatically lowered cellulosic ethanol enzyme cost. They have also developed suites of enzymes specifically for hemicellulose hydrolysis. This project reflects their interest in broadening their biofuels market. Also, the extensive Novozymes research enzyme collection will permit them to select and supply enzymes specifically for wood pulping liquor hemicellulose hydrolysis.

Third, the combination of currently high wood pulp prices, the Chilean earthquake (which seriously damaged several mills), and forest products deconsolidation (with spinoff and closure of smaller mills) encouraged pulping companies to evaluate a processes for increasing both mill throughput and yield. This trend is expected to continue. This invention increases mill throughput increases by converting the most common mill bottleneck, hemicellulose-lignin liquid crystals which accumulate in and plug the black liquor evaporator, into increased pulp yield or a valuable market product. This invention could permit pulp mills to increase black liquor solids content above 75%, resulting in higher combustion efficiency and lower emissions. This provides significant reductions in mill energy intensity.

These technology developments, regulatory requirements, and environmental factors, provide a unique opportunity to use technology maturation funding to work with industrial partners to demonstrate a technology likely to lead to high-valued commodity product licenses. This project also provides a technical basis for the first set of commercial products and the first license. Our technology could potentially be applied in most chemical pulp mills and, with some further development, in some biomass conversion processes.

Technical Status: Technical activity is expected to start when funding is received.

Technical Plans for Next Quarter: As soon as funding is received, ORNL and the partners expect to start the mill sampling / initial hemicellulose stripping process.

Commercialization Status: Partners have been contacted and the project discussed in some detail. The partners were then provided with copies of modular CRADA and, in the case of AkzoNobel, with a copy of the standard Materials Tranfer Agreement.

Commercialization Plans for Next Quarter: 1) Two partners, Domtar and General Atomics, have expressed an interest in a joint CRADA with ORNL. We will work with the partners and the ORNL Partnerships staff to develop an acceptable statement of work and formally start the eWFO - CRADA process. 2) One partner expressed an interest in working through an MTA. This is appropriate because that partner, AkzoNobel, wants to evaluate hemicellulose sugars as sustainable feedstocks for chemical syntheses.

Invention Disclosures: None

Patents: None

Publications / Presentations: None

Task Schedule

Task Number	Task Description	Task Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
1	Best pulp mill sites for liquor withdrawal					
2	Additional purification or processing required to produce the best sugar mix					
3	Preparation and evaluation of larger samples					
4	Prepare and submit final report					

Project Period: 06/14/10 to mm/dd/yy

Spending Schedule

Current Quarter: 06/14/10 to 06/30/10

Task	Approved Budget	Project Expenditures	
		This Quarter	Cumulative to Date
Task 1 Literature Review	30,000		
Task 2 Prototype Design	35,000		
Task 3 Prototype Fabrication	30,000		
Task 4 Short-Term Testing	5,000		
Total	100,000		
DOE Share	100,000		
Cost Share	105,000		

Cost Share Contributions *

Funding Source	Approved Cost Share		This Quarter		Cumulative to Date	
	Cash	In-Kind	Cash	In-Kind	Cash	In-Kind
AkzoNobel		25,000				
Domtar		30,000				
General Atomics		50,000				
Novozymes		TBD				
Total		105,000				
Cumulative Cost Share Contributions						

SAMPLE

Project Spending and Estimate of Future Spending							
Calendar Year Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative Actual Outlays (Federal + Recipient)**
	Start	12/31/10					
2Q10	06/15/10	06/30/10					
3Q10	07/1/10	09/30/10					
4Q10	10/1/10	12/31/10					
1Q11	1/30/11	03/31/11					
Totals							

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).

Estimates should be provided for the entire project, and actuals should be provided for each quarter as it is completed. Estimates should be updated each quarter.

Quarterly Progress Report

Project Title: Method for Improving Separation of Carbohydrates from Wood Pulping Liquors and Wood or Biomass Hydrolysis Liquors

ORNL WBS Element: Not yet assigned

Covering Period: July 1, 2010 - September 30, 2010
Date of Report: September 30, 2010

Contact: Alicia L. Compere, Physical Sciences Directorate/Chemical Sciences Division, 865-574-4970, compereal@ornl.gov

Corporate Partner(s): To CRADA: Domtar, General Atomics through MTA for product evaluation: AkzoNobel and Novozymes

Project Objective: Work with industrial partners to perform the studies needed to commercialize U.S. patent 7,699,958 for separation of carbohydrates from wood pulping liquors and wood or biomass hydrolysis liquors. These include: 1) selection of the best pulp mill liquor withdrawal sites, 2) additional purification or enzyme hydrolysis required to obtain acceptable sugar feedstocks, 3) and work with partners to optimize the stream and purification methods to provide acceptable feedstocks for algal fuels and industrial chemicals production, and 4) preparation of samples large enough for testing by other downstream partners.

Background: This hemicellulose separation was developed with the support of two DOE - EERE projects. One project was developing an advanced wood pulping process and another was developing methods for converting lignin from wood pulping processes into feedstock for carbon fiber production. From the standpoint of a pulp mill operator, this separation process provides a method for substantially increasing both mill throughput and the yield of wood pulp. It also has the potential to improve emissions and overall plant energy efficiency. Because hemicellulose is the major contaminant in lignins produced from biomass and pulping streams, this separation also dramatically improves the ability to produce “clean” or high-purity lignins produced from biomass and pulping streams.

In the last two years, working with commercialization managers, this technology has been discussed with several potential licensees (Domtar, General Atomics, Novozymes, Milliken, Procter and Gamble, AkzoNobel). The ORNL inventors have discussed this in some detail with Domtar (a pulping company with $5.5 billion 2009 sales, >10,000 employees), General Atomics (GA Advanced Process Systems division is a major developer of algal-based fungible, or infrastructure-compatible, fuels funded by DARPA and individual services), and Novozymes (the leading global developer and producer of the enzymes needed to convert hemicellulose into a form suitable as an algal feedstock). Domtar is interested in the hemicellulose stripping process from the standpoints of increased pulp yield, increased mill throughput, higher black liquors solids concentration, and secondary products (algal and chemical feedstocks). General

Atomics would like to evaluate the hemicellulose as a feedstock for heterotrophic algae which they find to be a particularly good feedstock for a military fuel, BioJP-8. Novozymes is interested in extending its cellulosic enzyme market to high pH hemicelluloses. All three companies are interested in working with ORNL to scale and demonstrate hemicellulose-stripping process.

Several factors are driving this interest. First, General Atomics is in moving toward commercialization of their process for producing totally bioderived military fuels. This is driven by two executive orders, 13423 (Bush) and 13514 (Obama), which require all federal agencies, including the DoD, to increase biofuels use dramatically in the next 10 years and by DoD investments to both develop and fully qualify algal biofuels. This process produces feedstock for algae and, additionally, produces the aromatic and cyclic compounds required in aviation fuels but not produced by algae. These are expected to be critical in production of a bioderived replacement for the major military fuel, JP-8. The DoD is the major (93%) federal (2% of U.S.) billion dollar per year consumer of petroleum (133 million barrels – currently ~$10 billion or 0.4 Quad of raw petroleum – in FY2005, as reported by Dimotakis et al., 2006).

Second, Novozymes has made advances which have dramatically lowered cellulosic ethanol enzyme cost. They have also developed suites of enzymes specifically for hemicellulose hydrolysis. This project reflects their interest in broadening their biofuels market. Also, the extensive Novozymes research enzyme collection will permit them to select and supply enzymes specifically for wood pulping liquor hemicellulose hydrolysis.

Third, the combination of currently high wood pulp prices, the Chilean earthquake (which seriously damaged several mills), and forest products deconsolidation (with spinoff and closure of smaller mills) encouraged pulping companies to evaluate a processes for increasing both mill throughput and yield. This trend is expected to continue. This invention increases mill throughput increases by converting the most common mill bottleneck, hemicellulose-lignin liquid crystals which accumulate in and plug the black liquor evaporator, into increased pulp yield or a valuable market product. This invention could permit pulp mills to increase black liquor solids content above 75%, resulting in higher combustion efficiency and lower emissions. This provides significant reductions in mill energy intensity.

These technology developments, regulatory requirements, and environmental factors, provide a unique opportunity to use technology maturation funding to work with industrial partners to demonstrate a technology likely to lead to high-valued commodity product licenses. This project also provides a technical basis for the first set of commercial products and the first license. Our technology could potentially be applied in most chemical pulp mills and, with some further development, in some biomass conversion processes.

Technical Status: Technical activity is expected to start when funding is received.

Technical Plans for Next Quarter: As soon as funding is received, ORNL and the partners expect to start the mill sampling / initial hemicellulose stripping process.

Commercialization Status:
1) In conjunction with partners, developed a Statement of Work acceptable to all ORNL, Domtar, and General Atomics. 2) Prepared a CRADA/Joint Work Statement template and put it into the eWFO computer system. Revised the submission and obtained approval from Chemical Sciences Division director and financial officer. 3) Developed a list of ORNL background intellectual property. It is attached to the eWFO package. 4) The CRADA partners were unsure of required format for their background intellectual property, so an approved format was developed and provided to them.

5) Requested and obtained partner concurrence with the American Competitiveness Act.

6) Requested formal concurrence with CRADA provisions and the statement of work from both partners. Obtained concurrence from Domtar. Based on discussions, concurrence from General Atomics is expected shortly. Both partners provided "minor edits" to the Joint Work Statement. General Atomics has agreed with Domtar's minor edits.

6) Discussed information needs with Export Control and provided them with copies of the data needed to support their decision.

7) The Materials Transfer Agreement forms for Novozymes (providing enzymes) and AkzoNobel (evaluating carbohydrates as chemical feedstocks) were submitted and each company was sent a sample Materials Transfer Agreement. These are in process

Commercialization Plans for Next Quarter:
1) General Atomics, ORNL, and Domtar would like to complete and sign the agreement in the next couple of weeks. As soon as the agreement is signed and concurrence from ORNL management and DOE is obtained, work is expected to start. 2) An initial plan with Domtar for samples has been prepared.

Invention Disclosures: None

Patents: None

Publications / Presentations: None

Task Schedule

Task Number	Task Description	Task Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
1	Best pulp mill sites for liquor withdrawal					
2	Additional purification or processing required to produce the best sugar mix					
3	Preparation and evaluation of larger samples					
4	Prepare and submit final report					

Project Period: 06/14/10 to mm/dd/yy

Spending Schedule

Current Quarter: 06/14/10 to 06/30/10

Task	Approved Budget	Project Expenditures	
		This Quarter	Cumulative to Date
Task 1 Literature Review	30,000		
Task 2 Prototype Design	35,000		
Task 3 Prototype Fabrication	30,000		
Task 4 Short-Term Testing	5,000		
Total	100,000		
DOE Share	100,000		
Cost Share	105,000		

Cost Share Contributions *

Funding Source	Approved Cost Share		This Quarter		Cumulative to Date	
	Cash	In-Kind	Cash	In-Kind	Cash	In-Kind
AkzoNobel		25,000				
Domtar		30,000				
General Atomics		50,000				
Novozymes		TBD				
Total		105,000				
Cumulative Cost Share Contributions						

Project Spending and Estimate of Future Spending							
Calendar Year Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative Actual Outlays (Federal + Recipient)**
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1Q11	1/30/11	03/31/11					
Totals							

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Quarterly Progress Report

Project Title: Method for Improving Separation of Carbohydrates from Wood Pulping Liquors and Wood or Biomass Hydrolysis Liquors

ORNL WBS Element: Cost center 327100EJ

Covering Period: April 1, 2011 - June 30, 2011

Date of Report: July 18, 2011

Contact: Alicia L. Compere, Physical Sciences Directorate/Chemical Sciences Division, 865-574-4970, compereal@ornl.gov

Corporate Partner(s): To CRADA: Domtar, General Atomics. Through MTA for product evaluation: Novozymes

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solids concentration, and secondary products (algal and chemical feedstocks). General Atomics would like to evaluate the hemicellulose as a feedstock for heterotrophic algae which they find to be a particularly good feedstock for a military fuel, BioJP-8. Novozymes is interested in extending its cellulosic enzyme market to high pH hemicelluloses. All three companies are interested in working with ORNL to scale and demonstrate hemicellulose-stripping process.

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***Technical Status:** The CRADA was signed by the last partner on 28 February and received final approval from Oak Ridge Operations shortly thereafter. The account was

opened on 3/9/2011. Conference calls were used to conduct Kickoff meetings with the staffs of Domtar and with General Atomics within that week.

1. Large (4-5 gallon each) samples from 14 points in the Domtar Kingsport mill were collected, packaged, and sent to ORNL for initial evaluations.

2. The samples were large enough to provide large scale samples for evaluation by General Atomics.

3. These large scale samples are being prepared for hydrolysis and shipment to General Atomics in late July.

4. Domtar mill staff visited in April to discuss sample data. Mill staff wanted review data and also received a complete list of bench scale equipment, supplies and suppliers, and walk through the process. They said that they intend to set up a similar set of process tests using a trainee whom they intended to hire by mid-summer.

5. General Atomics continues to improve its heterotrophic algal culture facility. They are awaiting large scale samples of stripped, purified, hydrolyzed hemicellulose. These will be tested against around 50 cultures to determine whether a viable combination can be found.

6. General Atomics has made significant further developments in its algal test facility. They have also developed a set of factorial screening processes for carbohydrate feedstocks. Samples that met their factorial screening process needs were discussed and that information has been factored into the Domtar sampling plan and Oak Ridge sample evaluation (Task 2).

7. The impact of hemicellulose stripping on pulp mills is being realized. Discussions with Domtar indicate that they are considering putting in a line for this purpose.

Technical Plans for Next Quarter: The ORNL expects to complete its bench work on the project when the large samples are shipped to General Atomics. The ORNL portions of the topical and end of project reports will be written soon. Domtar and General Atomics expect to continue work on evaluation of the data and large samples. Draft topical and program reports will be discussed with Domtar and General Atomics at their planned August visit.

Commercialization Status: CRADA activated during the week of March, 7, 2011.

1. Kickoff teleconferences were held with both Domtar and General Atomics within two days of funding allocation.

2. The Domtar sampling plan meets a CRADA goal to determine whether the volume, purity, and chain length of hemicellulose for feedstock use can be

optimized by taking advantage of the chemical dissolution inherent in each stage of a conventional alkaline pulp mill. We worked out a sample exchange, discussed packaging to meet DOT requirements, and found a chemical carrier which runs between the required locations.

3. The results from these samples will further commercialization by helping us to put limits on the hemicellulose which can be recovered from this mill (Task 1) and by permitting the Domtar staff to start initial evaluations of impact on mill throughput, energy, and chemical balance (Task 1).

4. The General Atomics staff determined the amounts of material that they require in order to perform factorial carbohydrate evaluation tests (Task 2). They also provided estimates of the volumes of feedstocks needed to support heterotrophic algal growth within their system. It was agreed that, to the extent possible, samples will be frozen. Additionally, General Atomics indicated that enzymes (or other reagents) used to hydrolyze hemicellulose need to be inactivated to prevent hydrolysis of algal cell walls and that it would be preferable to reduce salt levels in the samples.

5. Hemicellulose samples from the 14 different Domtar sampling points will be hydrolyzed and, as possible, provided to General Atomics for evaluation. This will help to determine what is required in order to convert these materials into algal feedstocks (Task 2) and provide information on the best ways to prepare feedstock for heterotrophic algae.

6. Outside counsel completed drafts of the 3 patents incorporated into this CRADA as background intellectual property. They have been received for review and should be filed as soon after that as funding permits. This will permit the assigned commercialization manager (Greg Flickinger) and patent staff (Edna Gergel) to move forward on this intellectual property.

Commercialization Plans for Next Quarter:

The General Atomics analysis of the samples should be completed within a couple of months of sample.

A draft topical report covering sample analysis will be prepared. General Atomics project staff and Domtar management expect to visit to review the data and to watch and learn bench-scale versions of the process in early August. The Domtar mill staff will probably also come to that meeting.

ORNL was requested to file patent disclosures on enzyme hydrolysis of stripped hemicellulose. This will be done when the ORNL staff have time.

Invention Disclosures: None

Patents: None, although 3 patents added to the background intellectual property will be reviewed and a decision made as to whether they are filable.

Publications / Presentations: None issued.

Task Schedule

Task Number	Task Description	Task Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
1	Best pulp mill sites for liquor withdrawal	08/2011		06/2011	100	Domtar sent large samples
2	Additional purification or processing required to produce the best sugar mix	10/2011		06/2011	100	
3	Preparation and evaluation of larger samples	01/2012		06/2011	100	
4	Prepare and submit final report	03/2012			35	

Project Period: 03/09/11 to 03/08/12

Spending Schedule

Current Quarter: 03/09/11 to 03/30/11

Task	Approved Budget	Project Expenditures	
		This Quarter	Cumulative to Date
Task 1 Best mill sites for liquor withdrawal	30,000	17,507	30,000
Task 2 Additional purification or processing required to produce the best sugar mix	35,000	30,000	35,000
Task 3 Preparation and evaluation of larger samples	30,000	30,000	30,000
Task 4 Prepare and submit final report	5,000	1,730	1,730
Total	100,000	79,237	96,730
DOE Share	100,000	79,237	96,730
Cost Share	100,000	79,804	97,297

Cost Share Contributions*

Funding Source	Approved Cost Share		This Quarter		Cumulative to Date	
	Cash	In-Kind	Cash	In-Kind	Cash	In-Kind
Domtar		50,000		39,902		48,649
General Atomics		50,000		39,902		48,649
Novozymes		TBD				
Total		100,000				
Cumulative Cost Share Contributions					97,297	

Project Spending and Estimate of Future Spending							
Calendar Year Quarter	**From**	**To**	**Estimated Federal Share of Outlays***	**Actual Federal Share of Outlays**	**Estimated Recipient Share of Outlays***	**Actual Recipient Share of Outlays**	**Cumulative Actual Outlays (Federal + Recipient)**
	Start	03/09/11					
1Q11	03/09/11	03/31/11	18,000	17,493	18,000	17,493	34,986
2Q11	04/01/11	06/30/11	44,000	79,237	44,000	79,804	194,027
3Q11	07/01/11	09/30/11	38,000		38,000		
4Q11	10/01/11	12/31/11					
1Q12	01/01/12	01/01/12					
Totals							

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).

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