Recovery Act: Alpena Biorefinery and Alpena Biorefinery Lignin Separation Final Technical Report

DE-EE0002868 and DE-EE0006120
American Process Inc.

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Contents

1 Executive Summary

The Alpena Biorefinery (AB) was constructed in Alpena, Michigan, at the Decorative Panels International hardboard manufacturing facility. The goal of the AB was to demonstrate a modular, technically successful, and financially viable process of making cellulosic ethanol from woody biomass extract at wood processing facilities. At full capacity, the AB can produce 894,200 gallons per year of cellulosic ethanol and 696,000 gallons per year of aqueous potassium acetate, using extract from northern hardwood and aspen woodchips feedstock. The project objectives and the value proposition of AB promote the national goals of energy independence, greenhouse gas reduction, and green job creation and retention. A successful outcome of the Alpena Biorefinery project has been commercial sales of the first ever cellulosic ethanol RINS generated from woody biomass in the US, under the EPA's Renewable Fuels Standard Program. We believe that American Process is also likely the first company in the world to produce commercial quantities of cellulosic ethanol from mixed forest residue. Life Cycle Analysis performed by Michigan Institute of Technology found that the entire life cycle greenhouse gas emissions from the plant's cellulosic ethanol were only 25 percent that of petroleum-based gasoline. They found the potassium acetate runway de-icer coproduct generates up to 45 percent less greenhouse gases than the production of conventional potassium acetate.

The Alpena Biorefinery project created 31 permanent jobs for direct employees and helped retain 200 jobs associated with the existing Decorative Panels International facility, by increasing its economic viability through significant savings in waste water treatment costs. The AB project has been declared a Michigan Center of Energy Excellence and was awarded a $4 million State of Michigan grant. The project also received New Market Tax Credit financing for locating in an economically distressed community. All other equity funds were contributed by American Process Inc. The facility will remain operational after the demonstration period [redacted]. It will also be available as a pilot-plant "for hire," where third parties can perform trials on emerging biorefinery technologies. Additional capital projects are underway outside of the scope of DOE project [redacted]

2 Schedule

Figure 1 provides a summary of the actual project schedule against baseline. Reasons for deviations from the baseline are discussed throughout this report in the sections corresponding to the individual project tasks.

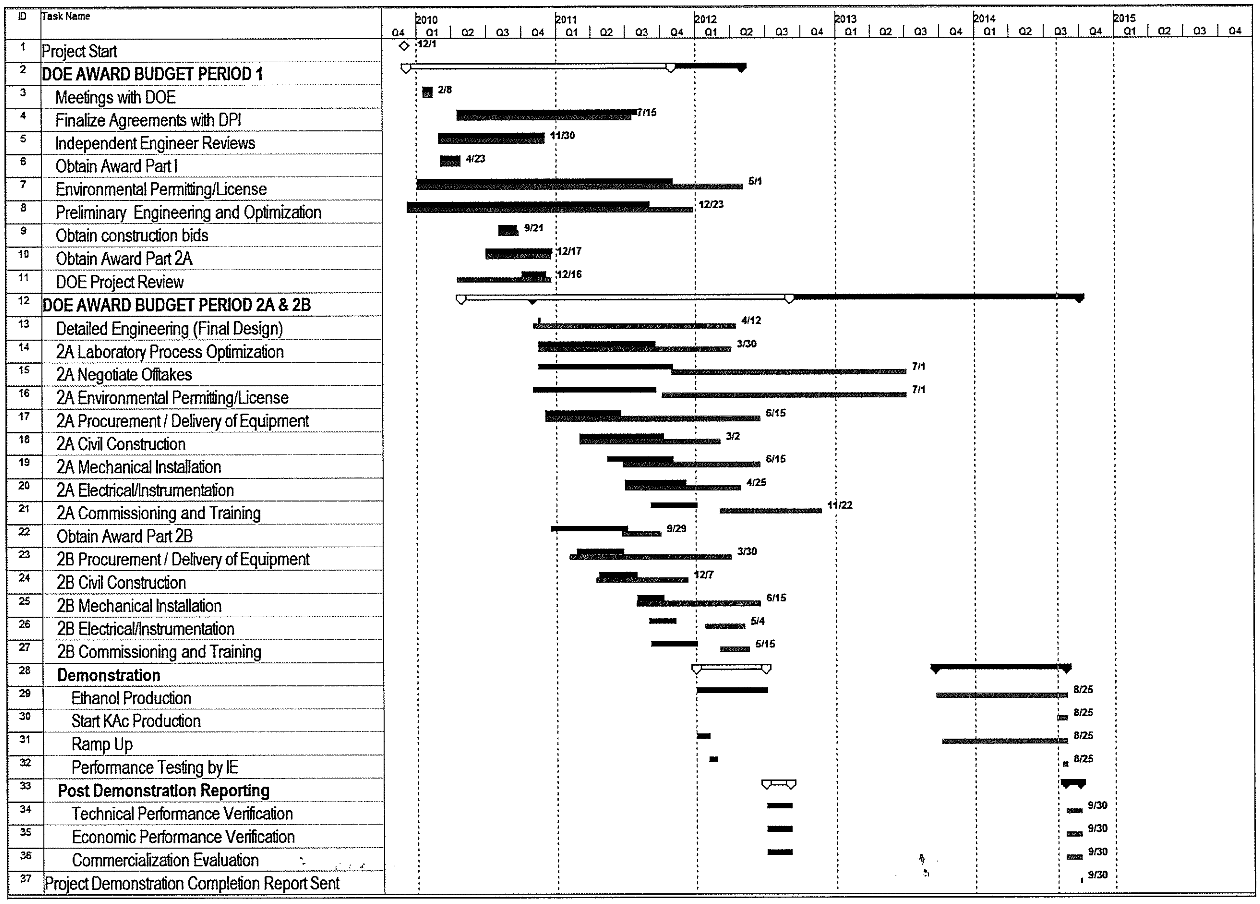


Figure 1. Project Schedule

Key milestones for the project are listed below:

- Start construction: February 25, 2011.
- First start-up: April 2012 – November 2012
- Start-up with lignin removal system: June 3, 2013
- First ethanol sale: Aug 22, 2013
- First cellulosic ethanol RINs generated: April, 18, 2013
- Potassium acetate start-up: July 31, 2014
- DOE Performance Test August 13-23, 2014
- End Demonstration: December 31, 2014

3 Actual Performance versus Objectives

Table 1 below provides a comparison of the actual accomplishments versus the goals of the project.

Project Goal	**Objective achieved?**	**Comments**
Demonstrate concentration, hydrolysis, and simultaneous fermentation of five- and six carbon sugars in a near-scale industrial facility.	Yes	[redacted].
Integration with host wood processing plant	Yes	The Alpena Biorefinery uses the waste stream of a collocated hardboard plant to produce commercial cellulosic ethanol.
Lignin removal	Yes	Sufficient lignin has been removed to allow the facility to operate
Effective removal of inhibitors - sugar platform for other biofuels / biochemicals	Ongoing	The inhibitor level is such that GMO yeast can ferment the hydrolysate to produce ethanol, but the inhibitor level is not yet optimal. A unit operation has been developed for this purpose, and it has been tested extensively at the pilot scale. The engineering for the plant scale installation of the technology is currently in the detail engineering phase.
Continuous co-fermentation of C5 and C6 sugars	Yes	[redacted].
Secure product off-takes	Yes	The cellulosic ethanol, and associated RINS, are being sold for transportation use through Tenaska Commodities, LLC, a multi-commodity marketing and trading company
Co-production of sugars / ethanol with other products /	Onogoing	Due to cost escalation of the potassium hydroxide reactant, API is actively searching for an alternate raw material to supply the potassium. [redacted].

Table 1. Project Accomplishments versus Goals

4 Summary of Project Activities

A summary of the major project activities for the entire period of funding is provided below.

4.1 Project Costing & Financing

Between the release of award and complete construction of the facility, the project cost estimate increased from $25,497,892 to $30,288,867. The key factors impacting this cost increase are listed in Table 2 below. The main reason for increases across engineering and construction was an underestimation of the degree of design changes that would result from the final stages of laboratory optimization for this first of a kind facility.

After commissioning, start-up and demonstration, the total project costs increased to $31,772,568 mainly due to additional hiring and higher than anticipated operations costs. API's final cost share for the project was $13,827,666 or 44%. The cost share was fulfilled by a $4 million grant by the State of Michigan, New Market Tax Credit Financing, and API equity.

API CAPEX	DOE Budget Total	Changes	Total Projected CAPEX	Comments
API Engineering	$ 2,461,289	$ 695,712	$ 3,157,001	Project scope changes
Controls and Instrumentation	$ 848,238	$ 269,545	$ 1,117,783	Project scope changes
Equipment	$ 9,394,000	$ (713,297)	$ 8,680,703	Purchase of selective used equipment
API Soil Disposal		$ 627,000	$ 627,000	Unbudgeted
API Construction	$ 8,690,467	$ 2,752,366	$ 11,442,833	Project scope changes
Environmental/HAZOP/Misc	$ 321,797	$ 79,466	$ 401,263	Lab chemicals and miscellaneous charges omitted from budget
Travel	$ 162,710	$ 46,640	$ 209,350	Under estimated travel costs
Mich Tech and Purdue Ho	$ 593,162	$ (50,000)	$ 543,162	Unable to include $50k spent pre-DOE award
Subtotal	**$ 22,471,663**	**$ 3,707,432**	**$ 26,179,095**	
Contingency	$ 1,077,399	$ (927,399)	$ 150,000	Negotiated lump sum construction cost to complete in February 2012; substantial mechanical completion achieved 4/25/12
TOTAL API CAPEX	**$ 23,549,062**	**$ 2,780,033**	**$ 26,329,095**	
API OPEX	**DOE Budget Total**	**Changes**	**Total Projected CAPEX**	
Operating Personnel (APER)	$ 685,277	$ 142,776	$ 828,053	4 additional operators added to provide necessary coverage
Operating Supplies	$ 1,215,766	$ 1,769,709	$ 2,985,475	Raw material costs approximately double original budget, allowance added for process changes during demonstration period
Insurance, etc	$ 47,787	$ 98,457	$ 146,244	Insurance coverage higher than budgeted to meet requirements of final DPI Agreements
TOTAL OPEX	**$ 1,948,830**	**$ 2,010,942**	**$ 3,959,772**	
Total CAPEX and OPEX	**$ 25,497,892**	**$ 4,790,975**	**$ 30,288,867**	

Table 2. Project Cost INcreases through Construction

In June 2013, API received an additional award from the DOE of $4,536,621 for the engineering and installation of a supplemental lignin removal system to prevent the significant lignin fouling observed during commissioning of the process. API's final cost share for the additional award was $2,007,521or 31%.

4.2 Engineering

Preliminary and detailed engineering for the AB took place between 12/4/2009 and 12/23/2011. Budget and schedule overruns occurred primarily from process changes resulting from the HAZOP review and laboratory process optimization, delays in release of Budget Period 2 funding by DOE, and prolonged State of MI planning reviews.

4.3 Laboratory Optimization

Appendix 1 summarizes the key data obtained in the laboratory during laboratory optimization for the major unit operations shown in the original process design (prior to the lignin separations project) provided in Figure 2.

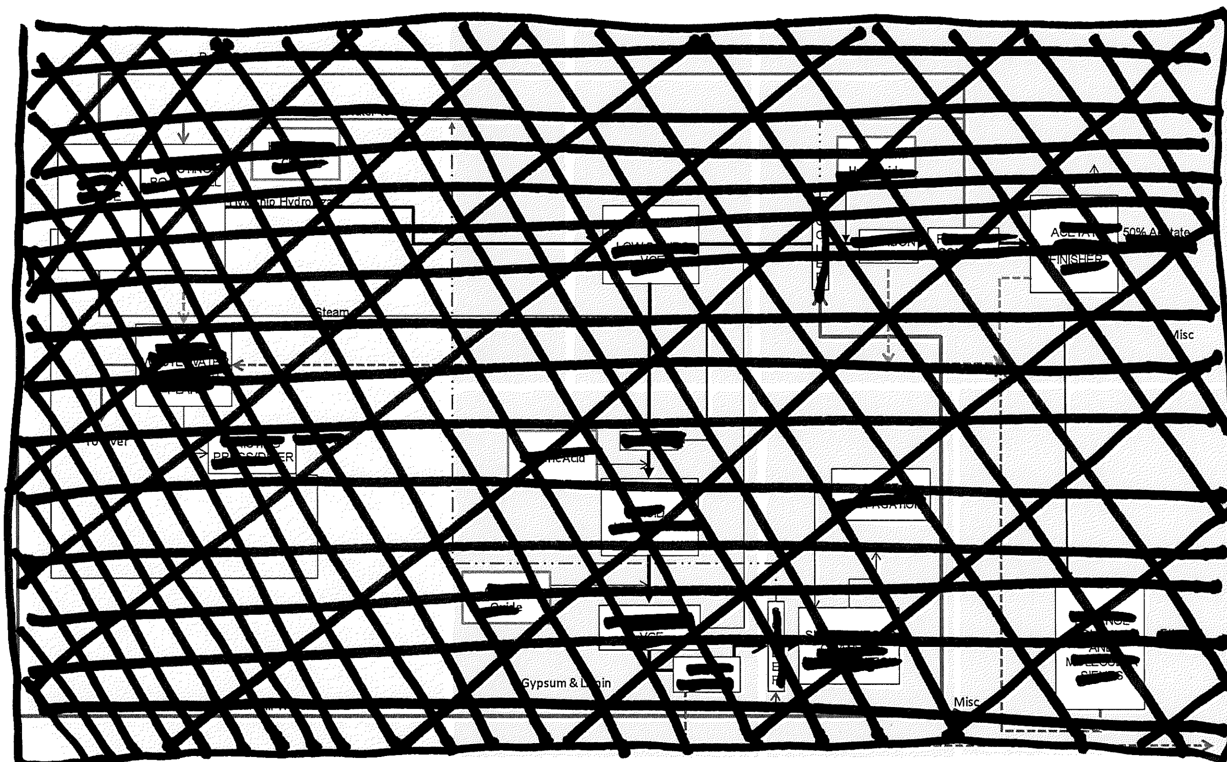


Figure 2. Orignial Process Block Flow Diagram (prior to the lignin separations project)

A process description for the original process design is provided below. Changes to the process design that resulted from the lignin separation project and subsequent process optimization are discussed in subsequent sections.

The biorefinery utilizes a liquid feedstock composed primarily of dilute hemicellulose derived from the steam extraction and washing of wood chips at the collocated DPI hardwood facility. Other constituents include acetic acid, dissolved lignin, and fiber "fines". Feedstock is sourced from DPI's seal chest overflow where it is pumped to the biorefinery utilizing a variable speed pump. Average flow rates to the biorefinery are approximately 320 gpm. The feed is stored at the biorefinery in the Low Solids VCE storage tank. This tank will be located outdoors and provide approximately 8-hours of storage capacity.

From the VCE storage tank, feedstock is pumped to the low solids evaporator [redacted] driven by a [redacted] vapor compressor. The Low Solids Evaporator concentrates the dilute feedstock to approximately 4.2% solids (~150 gpm) and is estimated to remove approximately 75% of the acetic acid.

Following evaporation, feedstock is sent to a hydrolysis reactor to convert the hemicellulose polymers into fermentable sugar monomors. This is accomplished utilizing a continuous dilute acid hydrolysis system, [redacted] Immediately following acid hydrolysis, lime is added to the hydrolyzate via a high shear mixer. The addition of lime halts the reaction process to prevent product degradation and binds with the sulfur ions to generate calcium sulfate (gypsum). Sufficient lime is added to adjust the acidity of the hydrolyzate [redacted].

The High Solids Evaporator concentrates the feedstock to 18.7% solids, or 7% total sugars (~36 gpm). This evaporator is estimated to remove approximately 88% of the acetic acid. The product stream is immediately cooled [redacted] and lime is added [redacted] to neutralize the stream to a pH of 6. The stream is then [redacted] sent to a vacuum belt filter to remove the gypsum formed during neutralization and the lignin precipitated during acid hydrolysis. The solids product produced has approximately 50% moisture content. The cake is discharged via a chute and distribution screw to a bunker located inside the biorefinery building. A truck will ship the solids to landfill.

Filtrate exiting the vacuum belt filter is cooled to 86 °F and sent to fermentation. All vapor produced from VCE evaporation is condensed, collected, and cooled [redacted]. Potassium hydroxide (50%) is added to adjust the pH of the condensed liquid from [redacted] The potassium acetate is then concentrated to 5% utilizing Reverse Osmosis membranes followed by further concentrating in the Potassium Acetate Evaporator to 31.1%. An external Finisher is used to further concentrate acetate to 50-wt% concentration [redacted] Vapor from the Finisher is condensed in a surface condenser and vent condenser.

In fermentation, the individual sugar monomers produced during acid hydrolysis are converted into ethanol using the genetically modified Purdue-Ho *Saccharomyces cerevisiae* yeast strain capable of converting both 5-carbon and 6-carbon sugars into ethanol. The fermentation system was designed to operate in both continuous and batch mode with the capability of yeast recycling when operating in continuous mode.

The Purdue-Ho yeast must initially be propagated from seed culture [redacted] The yeast is then sent to the Yeast Propagation Tank where it is propagated to 15,500 gallons under the same conditions. [redacted]

[redacted]

[redacted]
For continuous fermentation, the yeast is propagated only once during start-up. After continuous fermentation, the yeast is separated from the beer via a stainless steel centrifuge and recycled to the beginning of the fermentation process. The centrifuge is designed to remove the yeast at a concentration of 20% by weight. A portion of the recovered yeast must be discarded to the Beerwell to maintain a constant initial yeast population since some yeast growth takes place during fermentation. The recycled yeast is pumped to the Propagation Tank with fresh nutrients for reactivation from the stationary phase to the growth phase. Reactivation takes approximately four hours. No fermentation happens during the reactivation (or lag) phase. [redacted]

The fermentation temperature is maintained at 86±1°F by circulation through a heat exchanger. Peak heat generation occurs when the yeast achieve their maximum growth rate at ~20 hours. Maintaining fermentation temperature is critical, with every degree rise in temperature giving significant loss of fermentation efficiency and yield. Also, yeast becomes more sensitive to high temperature as alcohol content rises throughout the fermentation. Fermentation is started at a pH of 6.0 and the pH is allowed to decrease during fermentation to a final pH of approximately 5.0.

For each batch fermentation, there is a 72 hour cycle time (18 hour fill time, 66 hours fermentation time[1], 2 hour drain time, and 3 hour CIP time). After fermentation, the yeast is pumped along with the beer directly from the fermenters to the beerwell. The beer is then pumped through a high temperature heat exchanger and yeast destruction chamber designed to kill the Purdue-Ho yeast prior to entering the Beer Column. The dead yeast then collects in the distillation bottoms and is sent to the waste water treatment lagoon.

For continuous fermentation, each fermenter is fed from the preceding fermenter. Fermenter 1 is fed with a combined flow of reactivated yeast from the Propagation Tank and hydrolyzate from the fermentation cooler. Overall average residence time through all four fermenters is 72 hours. A centrifuge following Fermenter 4 separates the yeast from the fermentation broth and recycles it to the Propagation Tank for reactivation.

Carbon dioxide produced during fermentation is routed to a CO_2 scrubber to remove any volatile organic compounds that may be present. Following fermentation, the resulting beer is pumped to the beer well that feeds distillation and dehydration.

[1]Includes all filling time minus 1.0 hour. During batch filling of the fermenters, yeast is added when the fill level in the fermenters reaches a point sufficient to begin operation of the recirculation pump. The time required to reach this level (approximately 5%) is roughly 1 hour.

The beer, containing 2-3% w/w ethanol, is preheated in plate & frame heat exchanger using residual water and solids, called "stillage", from the bottom of the distillation column. Preheated beer enters the top of the first distillation column called the "Beer Column", where solids and a portion of the water are separated and removed from the bottom of the column. The Beer Column is heated directly by using steam as the heating medium.
Vapor ethanol and water leave the top of the Beer Column and enter at the bottom of the second distillation column, or Rectifier Column. In the Rectifier Column, ethanol is concentrated to approximately 95 % v/v ethanol at the top. This stream is an azeotrope and cannot be further purified using standard distillation. A second stream containing predominantly water is collected from the bottom. Bottoms liquid is combine with the feed to the Beer Column. The Rectifier Column is also heated directly by using steam.
Ethanol vapor from the top of the Rectifier Column is partially condensed in the Reflux Condenser, a shell & tube heat exchanger, using Boiler Feed Water and ethanol is collected in the Reflux Receiver. The condensed liquid is returned to the top of the Rectifier Column as reflux. The vapor stream is heated in the Superheater, a shell and tube heat exchanger, using steam and is fed to the Molecular Sieve dehydration vessels. The molecular sieve vessels are designed to remove the remaining water from the ethanol vapor to meet the requirements for fuel grade ethanol. The Molecular Sieve vessels contain alumina-silicate desiccant which selectively adsorbs water from the vapor stream, while allowing the ethanol to pass through. At regular intervals, the molecular sieve vessels are cycled using a series of switching valves. These allow one vessel to be adsorbing water, while the second vessel is desorbing or "regenerating" to prepare it for another adsorption cycle. Adsorption takes place under positive pressure, while regeneration takes place under vacuum. Regeneration vapor from the Molecular Sieves is condensed in the Regen Condenser, a plate & frame heat exchanger, using cooling water. The vacuum is achieved by circulation regeneration liquid through Regeneration Receiver Tank with vacuum pumps.

The regeneration liquid contains approximately 65% w/w ethanol. Regen liquid is returned to Beer Column feed, reprocessed through the distillation system as a means of recovering its ethanol content. Dehydrated ethanol vapor from Molecular Sieve vessels is partially condensed in the Product Condenser, a plate & frame heat exchanger, using cooling water. The product liquid is collected in the Product Receiver. The Product Receiver operates under a slight vacuum, which is provided by the regeneration vacuum pump system. The lower pressure allows any entrained carbon dioxide to be liberated, in order to meet the product acidity specification. Vapor from the receiver is cooled by a plate & frame heat exchanger to condense ethanol entrained with the carbon dioxide. Product liquid is cooled in a plate & frame heat exchanger, filtered and sent to API for storage and shipment. The ethanol product is pumped to one of two shift tanks and analyzed for quality control purposes before being pumped to the final product storage tank. Ethanol product is loaded onto trucks via a load-out system with denaturant being added to the final ethanol product at the load out skid.

4.4 Michigan Technological University Research

David Shonnard, professor of chemical engineering at Michigan Tech, and Susan Bagley, professor emerita of biological sciences, performed fundamental research to inform the process design of the Alpena Biorefinery. Shonnard, who holds the Robbins Chair in Sustainable Use of Materials and heads Tech's Sustainable Futures Institute, and his PhD student, Jifei Liu, compared acid hydrolysis versus enzymatic hydrolysis for hydrolyzing DPI's waste stream to monomer sugars. Enzymatic hydrolysis was less effective and more expensive.

Bagley and her PhD student, Stephanie Groves, then conducted experiments to improve the adaptation of yeast microorganisms to most efficiently ferment the unique mix of 5-carbon sugars produced from the plant's wastewater. Ms. Groves' thesis can be accessed here: http://gradworks.umi.com/35/65/3565320.html.

Shonnard's research group also conducted environmental life cycle assessments of the Alpena Biorefinery, to understand the carbon footprint of its cellulosic ethanol production process and its effects on greenhouse gas emissions. They found that the entire life cycle greenhouse gas emissions from the plant's cellulosic ethanol were only 25 percent that of petroleum-based gasoline, and when the study was based on EPA methodology, even lower.

4.5 Environmental Permitting & Authorizations & Product Specifications

4.5.1 NEPA

American Process prepared and submitted the Environemtnal Assessment, Proposed Action, and Baseline Environmental Conditions reports to the DOE. On September 14, 2010, The DOE issued a Finding of No Significant Impact (FONSI), which, subject to other award conditional provisions, authorized release of DOE's cost-shared funding for the design, construction, and operation of the Alpena Biorefinery.

4.5.2 Permits, Approvals and Licenses

Table 3 lists the permits, approvals, and licenses obtained to operate the biorefinery along with their reporting requirements. The regulatory requirements are onorous for a demonstration facility of its size due to its collocation with DPI, a Title V major source permit holder.

Of note, because API took over ownership and operation of DPI's on-site Wastewater Treatment Plant (WWTP), DPI's original NDPES water permit was divided in two with the treated process water outfall 001 assigned to API. Outfall 002, the permitted outfall for DPI's non-contact turbine cooling water, remained with DPI. Non-contact cooling water is discharged without treatment to Thunder Bay River.

Permit, Approval or License	**Reporting Requirements**
Alcohol Fuel Producer Permit	Annual report of fuel plant activities. Requires certain recordkeeping requirements.
Toxic Substances Control Act	N/A. Requires certain recordkeeping and GMO containment controls.
Hazardous Materials Certificate of Registration	N/A. Reapply annually
Permit to Install	Within 150 days of completion of successful D&D run, Mon-Mact notification of compliance status report is due to agency. By August 31st of each year, first semi-annual MON-MACT compliance report is due to agency. By February 31st of each year, second semi-annual MON-MACT compliance report is due. By May 27th, 2013 the initial semiannual report for Subpart VVa (LDAR) is due. By November 28th of each year, semi-annual VVa compliance report is due. By May 28th of each year, semi-annual VVa compliance report is due. By September 17, 2013, FGAPIFACILITY Compliance report due. Requires certain recordkeeping requirements.
National Pollutant Discharge Elimination System Permit	No later than the 20th day of the month following each month of the authorized discharge period, the permittee shall submit monthly summary and daily data to the department through the e2-Reporting website. On or before March 31 of each year, the permittee shall submit a Pollutant Minimization Plan for Total Mercury status report for the previous calendar year to the Department. Expires on October 1, 2016.
Discharge Monitoring Report-Quality Assurance Study 33	Very receipt of DMR-QA 33 study by March 25th. Coordinate with test lab for testing and data submission. Submit Data Report form to EPA by August 26th.
Limited Alcohol Buyer License	N/A. Reapply annually.
Permit, Approval or License	Reporting Requirements
Agricultural Use	By January 15th of each year, Biotech to submit to API a draft land application Annual Report for review. API to submit final report to the State by Jan 31. Expires on September 14, 2014.
Title III Superfund 311 Report.	Need to submit new report within 90 days of exceeding the threshold for gasoline or ethanol (10,000 lbs).
Title III Superfund 312 Report.	Annual Deadline: March 1, 2013. A complete SARA 312 report must be submitted for all chemicals that exceeded reporting thresholds during the year.
Title III Superfund 313 Form R	Annual Deadline: July 1, 2014.
Integrated Contingency Plan	Official review and evaluation of the ICP must be conducted at least once every 3 years.

RFS2 Program	Reporting through EMTS online system required within 5 business days after assigning, selling, separating, or retiring RINs. Quarterly manual submissions required through EPA's CDX online system for the following reports: RFS2 Activity Report, Renewable Fuel Producer Co-products Report, RFS2 Renewable Biomass Report. Annual RFS2 Production Outlook Report must be submitted manually through EPA's CDX online system. Yearly third-party audit submittal required by May 31st of the year following the calendar year.

Table 3. Alpena Biorefinery Permits, Approvals, and Licenses

The following site environmental and regulatory compliance plans were created for the biorefinery:

- Integrated Contingency Plan
- Renewable Fuels Standard Compliance Plan
- Land Application Operations and Procedures Manual
- Pollutant Minimization Plan for Total Mercury
- Odor Management Plan
- Malfunction Abatement Plan
- Leak Detection and Repair Plan
- Miscellaneous Organic Chemical Manufacturing NESHAP Requirements
- Alpena Biorefinery Management of Wastewaters for Compliance with Miscellaneous Organic NESHAP
- Alpena Biorefinery MCAN Recordkeeping Requirements
- Ho-Purdue Yeast Handling Protocol

4.5.3 Product Specifications

Fuel Ethanol

API's Denatured Fuel Ethanol is a renewable fuel that conforms to the accepted fuel industry's specification for Fuel Ethanol, ASTM-D 4806 Standard Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel. The following table contains the specifications for Fuel Ethanol.

Table 4 Denatured Fuel Ethanol Specifications

Analysis Spec.	Method	Limit	Units
Ethanol Content	ASTM D5501	≥92.1	% volume
Methanol	ASTM D5501	≤0.5	% volume
Solvent-washed Gum	ASTM D381	≤ 5.0	mg/100 ml
Water content	ASTM E203 or E1064	≤/1.0	% volume
Inorganic Chloride	ASTM D7319 or D7328	≤10	mg /l
Copper	ASTM D1688	≤0.10	mg /l
Acidity	ASTM D1613	≤56.0	mg /l as Acetic acid
pHe	ASTM D6423	6.5-9.0	
Sulfur	ASTM D2622, D5453, D3120, or D7039	≤30	mass ppm,
Existent Sulfate	ASTM D7318, D7319, or D7328	≤4	mass ppm,
Denaturant		1.96- 5.0	% volume

Potassium Acetate

Potassium Acetate shall conform to the FAA-approved specification SAE AMS 1435A for a potassium-acetate based deicing/anti-icing fluid in the form of a concentrated liquid.

Table 5 Potassium Acetate Specifications

Analysis Spec.	Method	Limit	Units

Appearance		Fluid shall be homogeneous and uniform in color. If fluid is colored, it shall be blue.	
Flash Point	ASTM D 56 or ASTM D 93	>100 (212)	°C (°F)
Specific Gravity	ASTM D 891	Within ±0.015 of preproduction value	
pH	ASTM E 70	7.0 to 11.5	
Freeze Point	ASTM D 1177	<14.5 (+6) diluted 1:1 by weight with ASTM D 1193 Type IV water	°C (°F)
	ASTM D 1177	Within 4 (7) of the preproduction value	°C (°F)
Sandwich Corrosion	ASTM D 1193	Corrosion not worse than control panels run using ASTM D 1193, Type IV, water.	
Total Immersion Corrosion; AMS 4037 Aluminum Alloy, anodized as in AMS 2470	ASTM F 483	≤0.3 weight change	mg/cm^2 per 24 hours
Total Immersion Corrosion; AMS 4041 Aluminum Alloy	ASTM F 483	≤0.3 weight change	mg/cm^2 per 24 hours
Total Immersion Corrosion; AMS 4049 Aluminum Alloy	ASTM F 483	≤0.3 weight change	mg/cm^2 per 24 hours
Total Immersion Corrosion; AMS 4376 Magnesium Alloy, dichromate treated as in AMS 2475	ASTM F 483	≤0.2 weight change	mg/cm^2 per 24 hours
Total Immersion Corrosion; AMS 4911 or MAM 4911 Titanium Alloy	ASTM F 483	≤0.1 weight change	mg/cm^2 per 24 hours
Total Immersion Corrosion; AMS 5045 Carbon Steel	ASTM F 483	≤0.8 weight change	mg/cm^2 per 24 hours
Low-Embrittling Cadmium Plate	ASTM F 1111	≤0.3 weight change	mg/cm^2 per 24 hours
Hydrogen Embrittlement	ASTM F 519, Type 1a, 1c, or 2a	Fluid shall be nonembrittling	
Stress-Corrosion Resistance	ASTM F 945, Method A	Fluid shall not cause cracks in AMS 4911 or MAM 4911 titanium alloy specimens	

Effect on Transparent Plastics	ASTM F 484	Fluid, at 25 °C ±2, shall not craze, stain, or discolor MIL-PRF-25690 stretched acrylic plastic or MIL-P-83310 polycarbonate plastic	
Effect on Painted Surfaces	ASTM F 502	Fluid, at 25 °C ±2, shall not decrease the paint film hardness by more than two pencil hardness levels or produce any streaking, discoloration, or blistering of paint film	
Effect on Unpainted Surfaces	ASTM F 485	Fluid shall not produce streaking or leave any stains requiring polishing to remove	
Rinsibility		Fluid shall be completely rinsible in tap water	
Runway Concrete Scaling Resistance	ASTM C 672	≤1 for 50 freeze-thaw cycles	
Asphalt Concrete Degradation Resistance	LFV Method 2-98	Reduction in adhesion value of runway asphalt concrete surface shall not be more than 50% of adhesion value of specimens not stored in deicing diluted compound	
Storage Stability	ASTM F 1105	Fluid shall not exhibit separation or an increase in turbidity compared to unaged fluid. Turbidity shall be acceptable if removed by mild agitation.	
Effect on Carbon-Brake Systems	AIR5567	Fluid shall be tested for catalytic oxidation of carbon and date reported for informational purposes only	

4.6 Host Facility Agreements

Several agreements with DPI, the host facility, were put in place that cover the following:

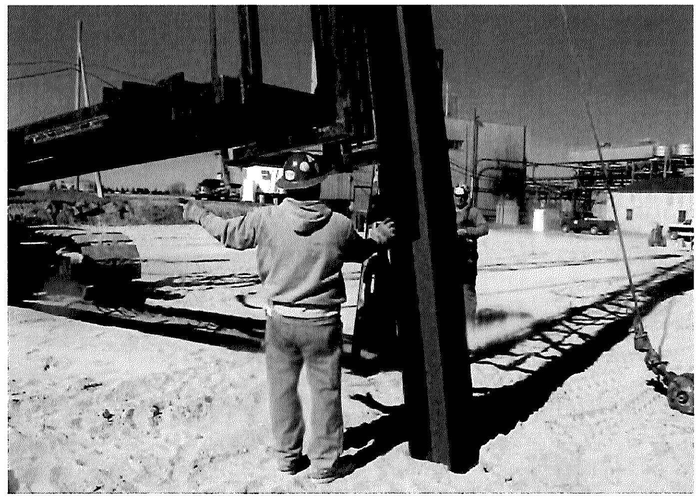
- Lease of land from DPI for biorefinery
- Supply of utilities by DPI to biorefinery
- Waste treatment agreement where DPI provides the sugar containing waste stream to API free of charge and API receives payment for treating DPI's wastewater.

The time and effort devoted to finalizing the agreements was not trivial and should be taken into account during the project scheduling phase for other similar installations.

4.7 Construction

Mechanical completion of the Alpena Biorefinery occured on April 25, 2013, three months behind schedule. Construction was performed by Devere Construction of Alpena, Michigan. The main factors contributing to the construction delays were:

- Delayed release of Phase 2 funding by DOE
- Contaminated soil removal
- Michigan frost laws preventing transportation of construction equipment on state roads
- Prolonged State of MI planning reviews
- Protracted negotiations for DPI Agreements
- Construction scope increase



Lessons learned during construction of the biorefinery include:

- Unit prices for costing construction change orders should be more extensive to reduce need for T&M change orders for those that do not fall into a pre-determined unit category
- Number of scope changes arising out of continued process improvements from ongoing R&D higher than expected after P&IDs "frozen for design"- should be anticipated for future first of a kind demonstration facilities

4.8 Commissioning & Start-up

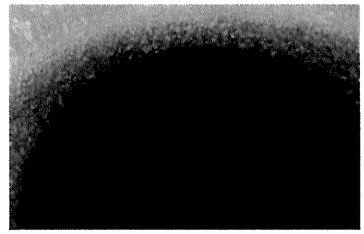
4.8.1 Original Process Start-up / Lignin Separation Project Award

The Alpena Biorefinery experienced a challenging shake-down period primarily due to issues related to handling and separating the lignin that precipitates during acid hydrolysis of DPI's waste stream. American Process Inc. developed a unique solution with universal applications to the challenge of handling the lignin, which is a problem that plagues the entire biorefining industry. DOE issued a noncompetitive award to the project entitled "Alpena Biorefinery Lignin Separation" of $4,536,621 for installation of modifications related to the patent-pending lignin handling process. The start-up challenges and at-scale demonstration of a solution highlight the critical importance of constructing and operating fully-integrated demonstration plants prior to commercialization. As we have seen with condensed lignin, phenomena that are easily managed during laboratory and non-integrated pilot studies under design conditions, can wreak havoc on fully integrated operations under even slight deviations from process design.

It is well known that dissolved lignin condenses, or polymerizes with itself, during acid treatment. For the Alpena Biorefinery, this phenomenon is observed when the dissolved lignin in the hardboard extract feed stream precipitates as a solid during acid hydrolysis. Original process design testing showed this condensed lignin to be light-colored and "fluffy" and easily wiped from a surface that it has adhered to. The condensed lignin formed fine grainy, sandy particles after neutralization [redacted]

However, during shake-down of the biorefinery, the condensed lignin presented a number of serious operational challenges, requiring shut-down and expensive, lengthy system clean-outs. Lignin plugging occurred in the hydrolysis reactor, process lines, and the evaporator (see Figure 3, Figure 4, and Figure 5). The adherence of lignin to the hydrolysis reactor walls caused solids build up in the reactor, which, when subjected to repeated cycles of acid treatment, became large, black, very hard lignin particles that plug the reactor. These large lignin particles then plugged the high solids evaporator distribution box. Serious plugging in the evaporator's lamella was also observed. Finally, shut down of process lines full of precipitated lignin solution allowed condensation into very hard lignin plugs.

•Adherence of lignin to hydrolysis reactor walls causes solids build-up in reactor. Repeated cycles of acid treatment of residual solids enhances degree of condensation-giving large, black, very hard lignin particles.



Hydrolysis reactor full of hard, black lignin

Hard, black lignin from hydrolysis reactor

Figure 3. Alpena Biorefinery Reactor Plugging

•Shut down of the process with lines full of precipitated lignin solution prior to pH adjustment allows condensation into a very hard lignin matrix plug.

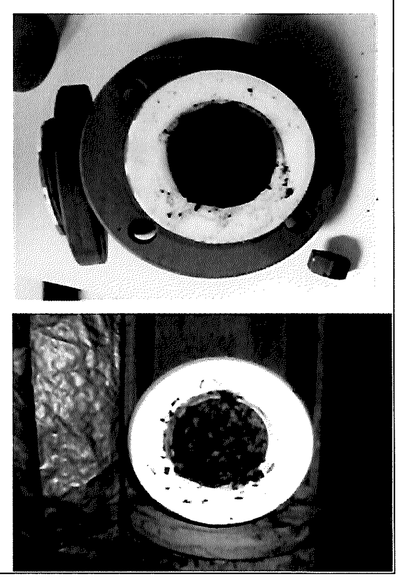


Figure 4. Alpena Biorefinery Pipe Plugging

•Condensation of lignin in reactor into large, hard lignin particles causes lignin plugging of high solids evaporator distribution box.

•Evaporation of lignin solution at less than pH 3 causes condensation and plugging within evaporator lamella.

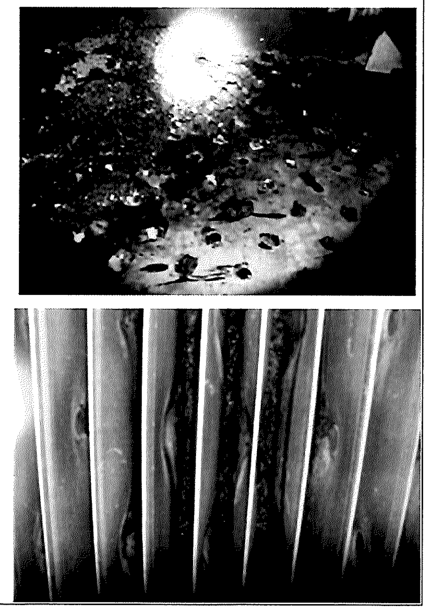


Figure 5. Alpena Biorenery Evaporator Plugging

API has since demonstrated in the lab and at the biorefinery that condensation of lignin into a large, hard matrix and adherence to vessel walls can be prevented [redacted]. During four continuous days of operatio[redacted]o lignin build-up was observed in the reactor [redacted]. API has filed a patent application to protect this significant IP. [redacted]

The proprietary and patent pending technology that forms the basis of the Lignin Separation project encompasses the addition of [redacted] the acid hydrolysis reactor to "detackify" precipitated lignin. The lignin removal and recycle processes train is shown in Figure 6. The major equipment installed at the Alpena Biorefinery for the project includes:

- One 3,000 gallon flash tank
- One 16,000 gallon "Hydrolysis" clarifier
- One 6,300 gallon "Fermentation" clarifier (modification of existing Neutralization Tank)
- One decanting centrifuge

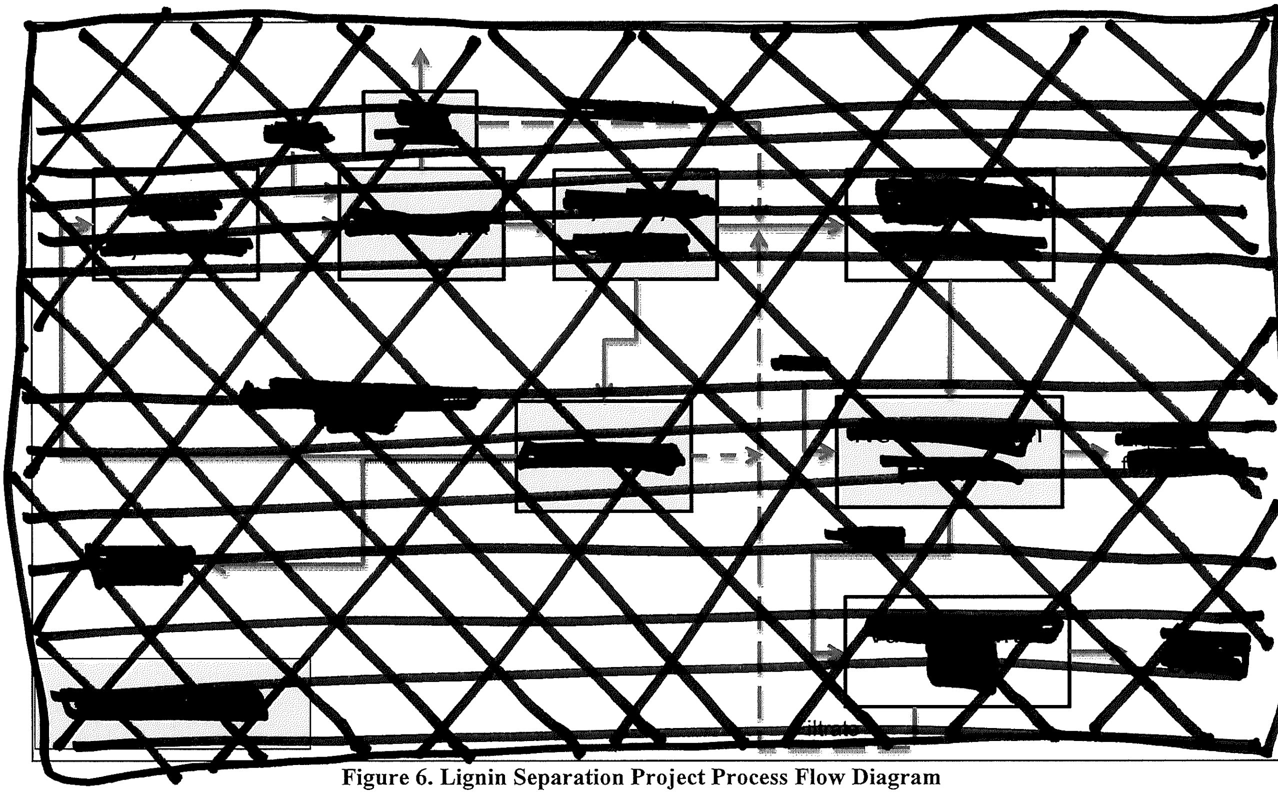


Figure 6. Lignin Separation Project Process Flow Diagram

The first equipment in the new process train is a 3,000 gallon *duplex* stainless steel flash tank installed immediately following the acid hydrolysis reactor. [redacted] The flash tank vents to a condenser which vents to atmosphere. Condensate from the condenser is sent to the process drain. Hydrolyzate from the Flash Tank is sent to a new 16,000 gallon 316 L stainless steel "Hydrolysis" Clarifier equipped with a rake. [redacted]. The clarifier supernatant is sent to a 900 gallon standpipe which feeds the existing High Solids Evaporator. A decanting centrifuge concentrates the solids [redacted] Excess solids are purged to the existing vacuum filter belt bunker. The existing 6,330 gallon Neutralization Tank was modified into a clarifier by the addition of a rake and overflow. This "Fermentation Clarifier" serves to i) act as a lime addition point for neutralization from pH 3 to 6, [redacted] iii) remove additional gypsum formed during the second neutralization. Solids from the clarifier are sent to the existing vacuum filter belt and supernatant is sent to fermentation.

4.8.2 Lignin Separation Project Modifications Start-up

The solids handling equipment installed under the Lignin Separation Project has successfully eliminated downtime associated with process plugging. While start-up trials of the gypsum recycle system where also successful, the need for gypsum recycle to prevent reactor plugging was eliminated by the process sequencing change described below. The modifications have allowed commercial sales of cellulosic ethanol from the biorefinery.

In the original Alpena Biorefinery process design, DPI effluent was concentrated from 2% total solids to 4% solids in the Low Solids Evaporator before entering the hydrolysis reactor. Hydrolysate from the reactor at 2% sugar was then concentrated in the High Solids Evaporator to 7% sugar. During this second evaporation step, the acetic acid produced primarily during hydrolysis was removed to a target concentration of 1.7 g/L.

Currently, DPI's waste stream is concentrated to 10-16% solids before entering hydrolysis reactor by passing through the Low and High Solids Reactor in series. [redacted] The acetic acid produced during hydrolysis will be removed from the hydrolyzate by a stripping system currently being installed outside the scope of the DOE project.

Benefits of resequencing the evaporation stages include:

- Reduction Chemical Cost (~$2M/year)
- Reduction in gypsum disposal
- No Gypsum/Lignin scaling in High Solids Evaporator

The new challenges observed during the second start-up phase and their resolutions are provided in Table 6 below. The equipment modifications listed are being self-funded by API and are out of scope of the DOE project. They are discussed in more detail in a subsequent section of this report.

Cause of downtime	Resolution
Gypsum fouling of the HS section of the evaporator	[redacted]
Stalling of the clarifier underflow, and related pump/piping performance problems	[redacted]
Fermentation capacity	
Inability to propagate yeast at a sufficient rate for present organism- related to out inability to prevent the Crabtree effect because we don't have adequate aeration and metered substrate feed.	Installing new propagation system
Inability to sterilize the propagation equipment (steam to a condition of 121C in a wet environment).	Installing new propagation system
Inability to maintain sterility during propagation (substrate not sterilized, air provided not sterile).	Installing new propagation system
Precipitation of lignin materials in fermentation, which must be manually removed from the ferm after each batch.	Installing new propagation system
We are hydraulically limited in distillation capacity to ~22-27 gal per min due to foam carryover from the top of the beer column to the bottom of the rectifier. This is being addressed with the current round of capital.	Current distillation modifications

Table 6. Phase II Start-up Challenges

4.8.3 Potassium Acetate Coproduct Line

Initial start-up of the potassium acetate line showed hydrolyzate cross-over between the Low and High Solids Evaporator bodies and the Potassium Acetate Evaporator body. As a result of the hydrolyzate cross-over, there were more dark color bodies in the evaporator condensate which required additional optimization of the color-removal / carbon filtration system in terms of carbon media used and residence time. Optimization is currently ongoing.

Repair attempts were made by the vendor evaporator vendor on several occasions to prevent cross-over. Success of the latest repairs will be evaluated during the next potassium acetate production trial.

Due to cost escalation of the potassium hydroxide reactant, API is actively searching for an alternate raw material to supply the potassium. When the evaluation of other raw materials is complete, co-product production trials will resume at the biorefinery.

5 Independent Engineer Performance Review Results

The Independent Engineer Performance Test was performed by Leidos starting on August 13, 2014. The test boundary includes those unit operations highlighted in green, in Figure 7. Those unit operations and streams highlighted in red are outside the test boundary.

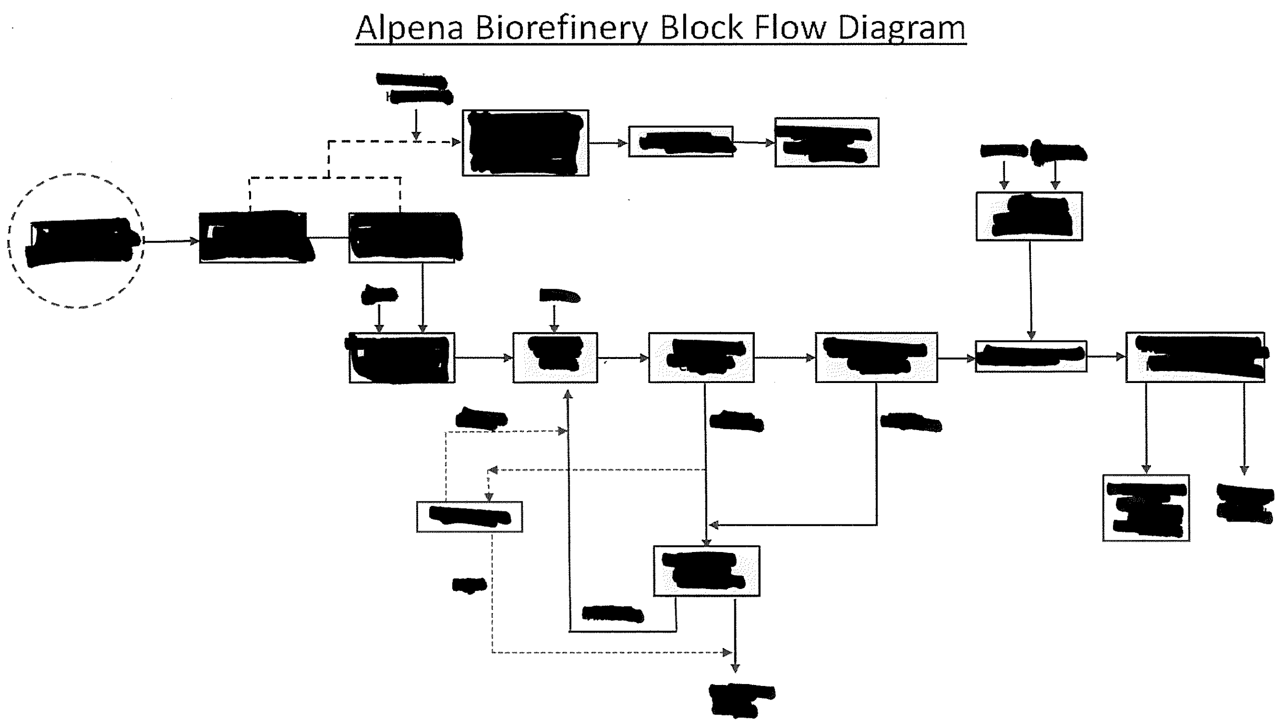


Figure 7. Independent Engineer Test Boundary

The operations of the partner facility, Decorative Panels International (DPI), are outside the test boundary. Likewise the treatment of the stripper column bottoms product (stillage) in the DPI aeration basin is outside the test boundary. Due to the success of the acid hydrolysis agitation scheme, the gypsum/lignin separation centrifuge has not been needed to control the lignin precipitation in the reactor. With the lignin precipitation under control, the operational expense of the gypsum/lignin separation centrifuge can be forgone; therefore, its operation is excluded from the trial.

The test will consist of a single test period. The duration of the test period will be that which is required to produce sufficient hydrolysate to fill all four fermenters, ferment the contents, distil the beer, and dehydrate ethanol. The actual duration is highly dependent upon the run status of the partner facility, DPI, but is anticipated to be 7 to 11 days. During the trial, the potassium acetate process will be run; however, the acetate laden condensate will only be generated while the MVR is operating (approximately 4 days).

5.1 TEST DESIGN

5.2 Test Methods, Procedures, and Reference Conditions

The test will consist of a single period, in which all four fermenters were filled, fermented, then discharged to be distilled, and dehydrated. The duration of the trial was extended because the partner facility (DPI) had a break down which stopped feed to API for a period. However, there was sufficient operational time at steady state to evaluate the performance of the continuous unit operations. The batch operations in the facility were unaffected by the interruption.

The composition of the feedstock material from DPI was low for a period of the trial. However, there was sufficient material of adequate concentration to complete the performance trial and gather meaningful data. The 4 hour feedstock composite concentration is listed in Appendix 4.1 – Lab Results: Infeed, Evaporation, Hydrolysis.

5.3 CALCULATIONS & POINTS OF MEASUREMENT

5.3.1 Points of Measurement

The data sampling points directly applicable to the performance test are contained in Figure 8.

DOE Performance Trail
Alpena Biorefinery Data Sampling Points

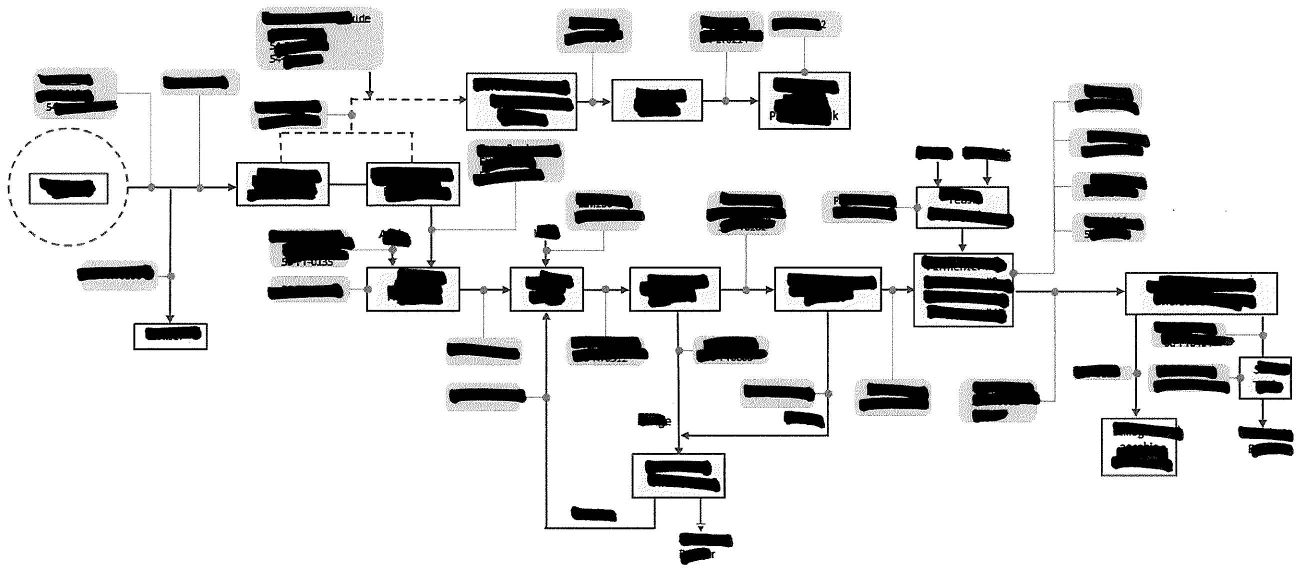


Figure 8. Performance Test Sampling Points

5.3.2 Calculations

The primary drivers of the plant profitability are the feed stock composition, plant throughput, hydrolysis reactor yield, fermentation yield, and the chemical use ratios.

The feed stock composition is outside the control of API but must be considered when evaluating the API plant performance. It affects both the plant power consumption (Evaporator MVR power consumption), and the plant throughput. While some seasonal variation in the sugar profile and saccharide/lignin ratio is to be expected, the primary measurement is the simple, yet reliable, %TS (w/w).

The plant throughput is measured at three locations: the hydrolysis reactor feed, the fermentation feed, and the beer column feed. At these points the volumetric flow rate of the streams are measured, and the concentration of the streams are taken on a regular basis, allowing for a simple yet accurate description of the plant throughput.

After the feedstock composition and plant throughput, the hydrolysis reactor yield has the largest impact on the plant profitability. It is defined as follows:

$$\frac{mass\ monosaccharide\ exiting\ reactor}{mass\ total\ saccharide\ entering\ reactor} \times 100\% = Hydrolysis\ Reactor\ Yield$$

The indicator with the next largest impact on plant profitability is the fermentation yield. It is defined as follows:

$$\left(\frac{(t_f\ fermentation\ ethanol\ concentration)}{t_0\ fermentation\ monosccharide\ concentration \times 0.51}\right) \times 100\% = Fermentation\ Yield$$

The chemical use ratios (mass sulphuric acid / mass total saccharide in feedstock, and mass calcium hydroxide / mass total saccharide in feedstock) have, historically, made a large contribution to the manufacturing cost price. The concentration of the feedstock, prior to hydrolysis has reduced this contribution dramatically. However, it still needs to be tracked.

5.4 Results

5.4.1 Evaporation

As noted, the partner facility (DPI) encountered significant mechanical difficulties during the trial. This, unfortunately, resulted in a period of downtime, when there was no feed from DPI. There was also a period of low concentration feedstock, after DPI came back on line. Appendix 1 contains the concentrations of the DPI feedstock material for the entire trial. The effect of this on API's evaporation process can be seen in the data historian graphic of Figure 9.

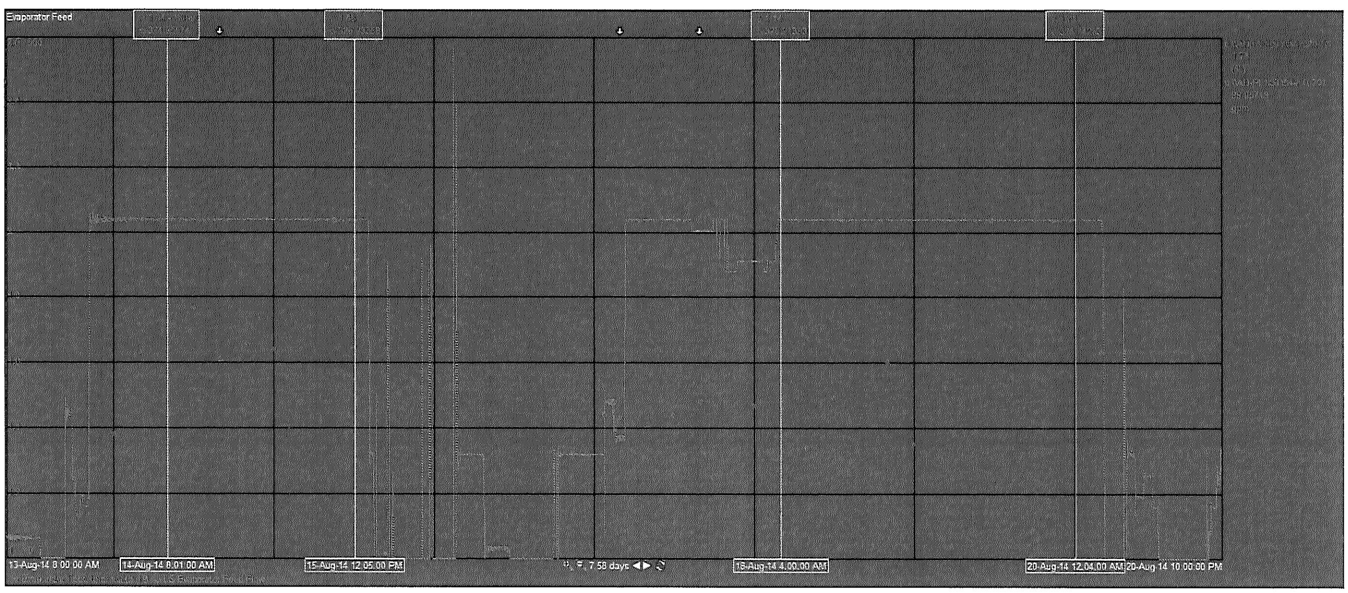


Figure 9. Performance Test Evaporator Results

This feedstock interruption provides us with an opportunity to illustrate the effect that low feedstock concentration has on the API process. From 08:00 14 Aug until 12:30 15 Aug, the feedstock was of a standard concentration. The mechanical issues in DPI then caused useful feed flow to stop for roughly 1.5 days. From 04:00 18 Aug until 24:00 19 Aug, the feedstock was very dilute. Table 7 contains a summary of the evaporator performance during those two time frames

	Standard Feed	Dilute Feed
Start Time	14-08-14 8:00	18-08-14 4:00
End Time	15-08-14 12:30	20-08-14 0:00
Feed Total Saccharide Conc.	0.090 lbm/gal	0.063 lbm/gal
Total Saccharide Flow	1,755 lbm/h	1,228 lbm/h
Evap Feed Flow	325.0 gpm	325.0 gpm
Evap Prod. Flow	36.9 gpm	36.1 gpm
Evaporated	288.1 gpm	288.9 gpm
	140,932 lbm/h	141,106 lbm/h
MVR Avg Amps	255 A	255 A
Voltage	4160 V	4160 V
Average Power	1,472 kWh/h	1,467 kWh/h
Electricity Cost	0.060 USD/kWh	0.060 USD/kWh
Specific Power Consumption	0.84 kWh/lbm sacch	1.19 kWh/lbm sacch
Evaporation MCP Contribution	0.05 USD/lbm sacch	0.07 USD/lbm sacch

Table 7. Performance Test Evaporator Results

The table above represents a practical maximum rate of evaporation that can be achieved. So the dilute feed both reduces plant capacity, and increases the evaporation manufacturing cost price contribution. The dilute evaporator product also increases the hydrolysis, fermentation, and distillation MCP contributions.

5.4.2 Hydrolysis

Of the two periods of time in which API was receiving feed material from DPI, the first period (08:00 14 Aug -12:30 15 Aug) was most representative. The dilute feed of the second period (04:00 18 Aug - 24:00 19 Aug) is of lesser interest, as API rarely encounters extremely dilute feed for days at a time. As such, the performance review of hydrolysis has been written focusing on the period of standard concentration.

Hydrolysis Throughput

The feed flow and concentration coming from the evaporator was sufficient to supply 36 gpm of feed to the reactor (Figure 10) at a total saccharide concentration of 114 g / liter. This equates to a total saccharide flow rate of 2054 lbm / hr. [redacted]

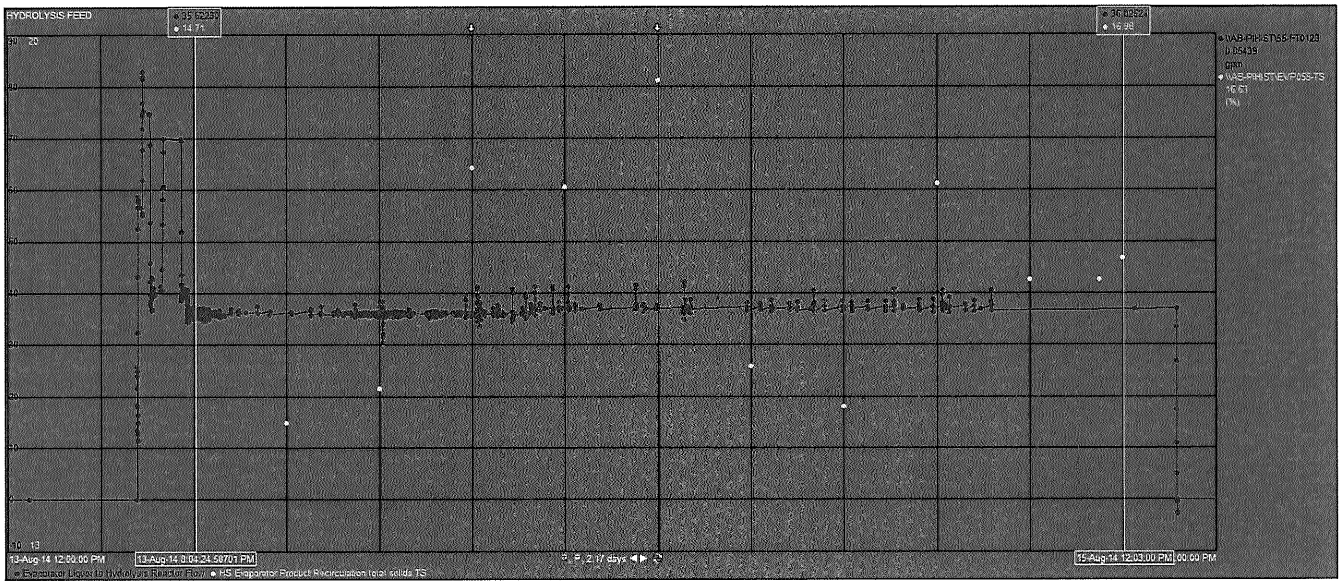


Figure 10. Performance Test Hydrolysis Feedrate

Hydrolysis Yield

During the same period of time, the average total saccharide concentration in the hydrolysis reactor feed (DIG055) was 114 g/liter. The average monosaccharide concentration in the reactor product (HYD030) during this period was 78 g/liter, as can be seen in Figure 11. This equates to an average reactor yield of 68% for the time period.

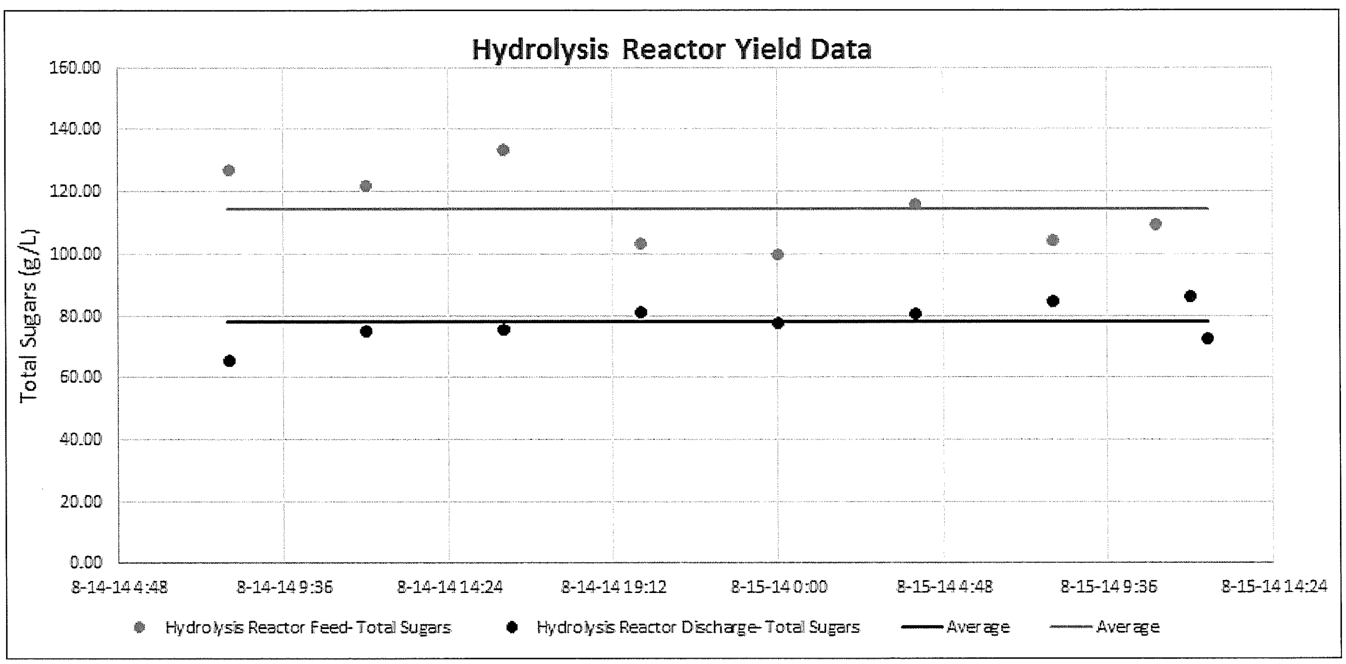


Figure 11. Performance Test Hydrolysis Yield

However, from the 20:00 14 Aug sample round, forward, the hydrolysis reactor yield increases to 78 – 80%. During the 12 hours prior to this period, the reactor temperature was still coming up to temperature as can be seen in figure 6. This explains part of the yield discrepancy; [redacted].

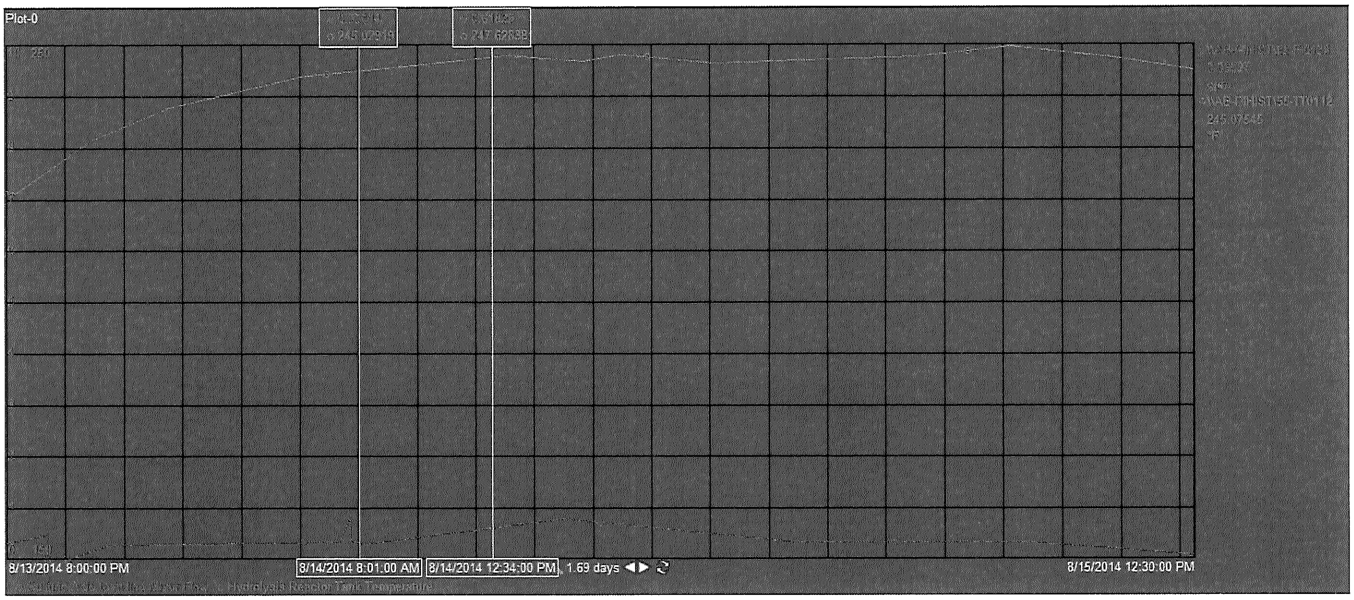


Figure 6

Chemical Use Ratio

The measured use ratio of sulphuric acid during this period of time [redacted]

The measured use ratio of calcium hydroxide during this period of time was [redacted]

5.4.3 Fermentation

Four fermenters were processed during the trial. The fermentation throughput (Table 8) and yield (Table 9) were both low, but representative of the present propagation and fermentation process. Modifications are currently underway to address these issues.

Fermenter	t_0	t_{final}	Total Time	Hydrolysate Volume	Hydrolysate Total Sacch	Throughput Volumetric	Throughput Total Sacch
B0063 (2)	8/21/2014 @ 1800	8/27/2014 @ 1130	5.73 Days	31,616 gal	38 g/L	223 gal/hr	71 lbm/hr
B0064 (1)	8/24/2014 @ 0030	8/28/2014 @ 0700	4.27 Days	36,424 gal	38 g/L	342 gal/hr	108 lbm/hr
B0065 (3)	8/25/201	8/29/201	4	31,425 gal	45 g/L	314 gal/hr	118 lbm/hr

	4 @ 1400	4 @ 1400	Days				
B0066 (4)	8/30/201 4 @ 0700	9/1/2014 @ 1120	2.18 Days	34,272 gal	53 g/L	609 gal/hr	271 lbm/hr

Table 8. Performance Test Fermentation Throughput

Fermenter	EtOH t0 (g/L)	EtOH tf (g/L)	EtOH from cellulose (lbm)	% theoretical Yield
B0063 (2)	4.9 g/L	11.1 g/L	1,957 lbm	43.0%
B0064 (1)	5.1 g/L	10.6 g/L	2,479 lbm	43.1%
B0065 (3)	4.2 g/L	12.1 g/L	2,871 lbm	47.9%
B0066 (4)	5.9 g/L	11.9 g/L	2,388 lbm	34.8%

Table 9. Performance Test Fermentation Yield

5.4.4 Distillation

The average feed flow to distillation was 20.0 gpm. The feed flow is hydraulically limited as a result of carryover from the top of the stripping column to the bottom of the rectifying column. This issue is being addressed.

The average ethanol measured in the beer fed to distillation was 11.1 g EtOH / liter.

The average ethanol measured in the bottoms was 0.14 g EtOH / liter.

6 Cellulosic Ethanol Sales

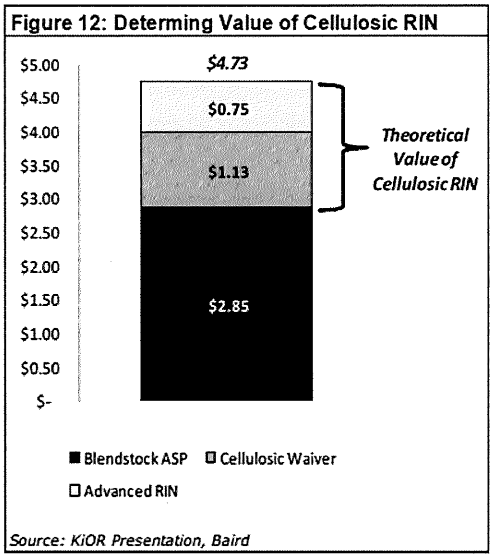
The Alpena Biorefinery has sold 15,750 USG ethanol over four shipments, 8,439 USG cellulosic ethanol, and generated 8,439 RINS in EPA EMTS system. Table 10 provides volumes, pricing, and RINs information for each shipment. All but the first sale have been brokered by Tenaska Commodities, LLC. Tenaska is a multi-commodity marketing and trading company that provides risk management, logistical and supply chain management services to the agriculture and energy industries. In 2013, Tenaska transacted in 53 different agricultural and energy commodities throughout the United

States and internationally.

Date	Conventional Ethanol		Cellulosic Ethanol		RINS Generated	Buyer/Broker	Comments
	USG	$/USG	USG	$/USG			
11/13/2014	4,157	0.75	-	-	-	Carbon Green Bioenergy	[redacted]
4/18/2014	1,325	2.2875	643	3.2825	643	Tenaska Commodities	[redacted]
7/1/2014	3,027	1.6525	2,002	2.6275	2,002	Tenaska Commodities	[redacted]
7/22/2014	3,180	1.4	2,154	1.4	2,154	Tenaska Commodities	[redacted]
9/4/2014	3,317	1.7	2,731	2.63	2,731	Tenaska Commodities	[redacted]
10/31/2014	744	1.43	909	2.35	909	Tenaska Commodities	[redacted]
Total	**15,750**		**8,439**		**8,439**		

Table 10. Alpena Biorefinery Cellulosic Ethanol Sales Summary

Figure 12 provides an example of the pricing mechanism for the cellulosic ethanol and RINs sold.



- Using 4/18/2014 prices, the price buildup was the following:
  - Chicago OPIS+ FOB Woodbury, MI $2.97
  - Corn Ethanol RIN (-)$0.6325
  - Advanced RIN $0.575
  - Cellulosic Waiver $0.42**
  - Total $3.3325

**2013 Waiver price, 2014 price not yet available

Figure 12. Cellulosic Ethanol Pricing Calculation

7 Future Plans Outside Scope of DOE Project

API is currently installing several process modifications to increase the level of production of cellulosic ethanol from the biorefinery. These projects are being self-funded and are outside the scope of the DOE awards.

The projects are:

1. Removal of Fermentation Inhibitors by Steam Stripping
   - Pilot tests demonstrate removal of acetic acid at greater than 50% along with Furfural, 5-HMF, other compounds
   - Will allow using several micro organisms [redacted]n addition of Nancy Ho's yeast which appears to be most resistant to inhibitors.

2. Yeast Propagation System Modifications
   - Sterilization of the Propagator (SIP under pressure (120°C))
   - Use of beet molasses as carbon source
3. Distillation
   - Removal CO_2 prior to "Beer Column"
   - Removal of Methanol after Dehydration

8 Commercialization

The technology developed at the Alpena Biorefinery is being marketed as "GreenPower+" for co-location at pulp mills, biomass power plants, and pellet mills with replication potential of 5.9 billion gallons replication potential by 2022, assuming a 35% market penetration as shown in Table 11 below.

	US Green Power+ Cellulosic Ethanol Production Potential			
	2020 Estimate			
	Annual Outpute Rate		**Ethanol Annual Volume**	
Biomass Power Generation	30	GW	5.3	B gallons
Biomass Pellet Manufacture	10	MMT	270	MM gallons
Pulp Production	1.2	MM BDT	343	MM gallons
Total			**5.9**	**B gallons**

Table 11. Replication Potential

The key value proposition of the GreenPower+ technology is increasing the value of biomass residual biomass at wood processing facilities by conversion to fuels and chemicals, as shown in Figure 13.

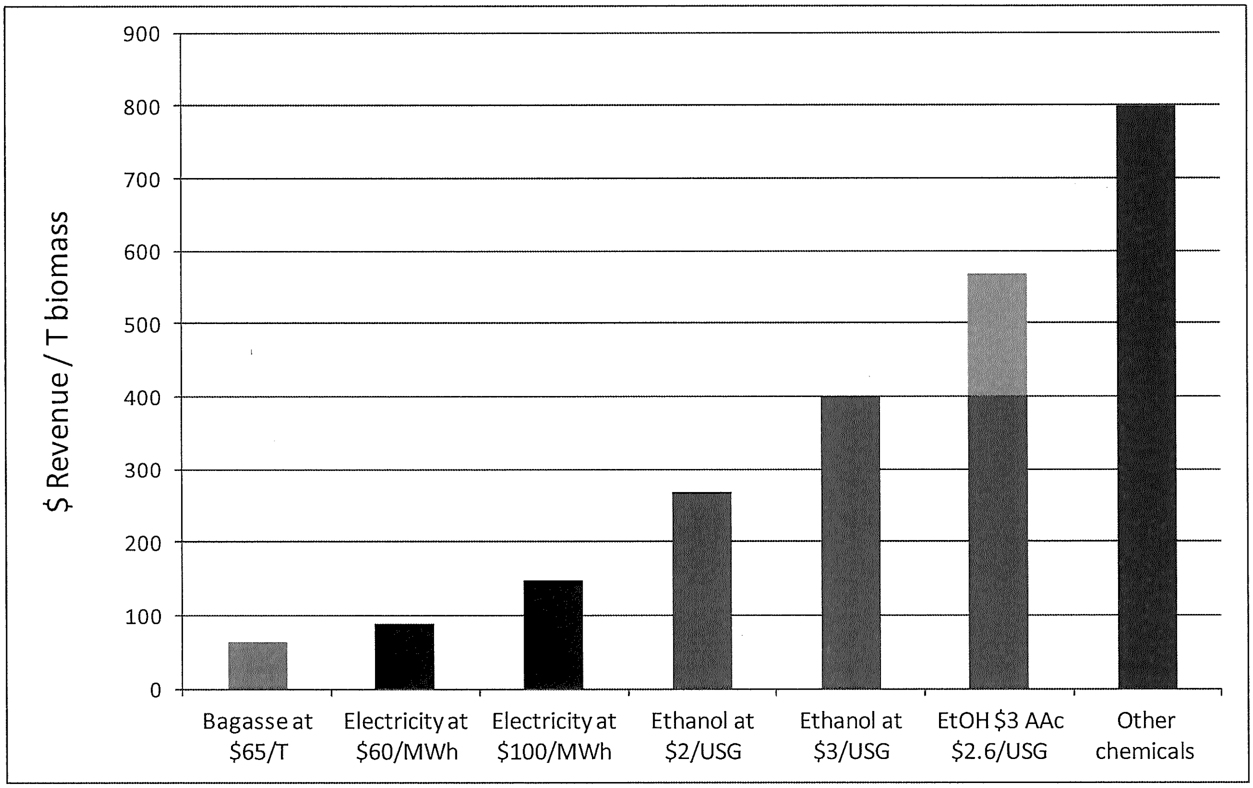


Figure 13. GreenPower+ Value Proposition

The GreenPower+ process is feedstock flexible. It can extract hemicelluloses from any biomass used at a commercial host plant with sugar yields ranging from 10.1-16.8% on OD biomass. As shown in Table 12, depending on feedstock type, the ratio of C5 and C6 sugars and inhibitor concentration will be different. Hardwoods give a majority C5's and high acetic acid (for acetate production). SW gives majority C6 and low acetic acid and can use a non-GMO yeast for ethanol production.

[redacted]	[redacted]											
[redacted]	0.4%	0.8%	[illegible]	1.9%	11.9%	[illegible]	[illegible]	[illegible]	1.7%	[illegible]	[illegible]	[illegible]
[redacted]	[illegible]	12.2%	[illegible]	4.9%	[illegible]	[illegible]	[illegible]	[illegible]	6.1%	[illegible]	[illegible]	[illegible]
[redacted]	[illegible]	0.9%	[illegible]	[illegible]	[illegible]	[illegible]	0.9%	0.0%	[illegible]	[illegible]	[illegible]	0.5%
[redacted]	[illegible]	0.4%	[illegible]	[illegible]	[illegible]	0.4%	[illegible]	0.8%	[illegible]	[illegible]	[illegible]	[illegible]
[redacted]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	2.4%	[illegible]	0.0%	[illegible]	1.6%	[illegible]	[illegible]
[redacted]	[illegible]	[illegible]	**14.8%**	**10.2%**	[illegible]	**15.1%**	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[redacted]	[illegible]	0.3%	[illegible]	0.6%	0.9%	0.7%	[illegible]	[illegible]		[illegible]		
[redacted]	[illegible]	4.3%	[illegible]	1.9%	[illegible]	[illegible]	[illegible]	[illegible]		0.8%		
[redacted]	[illegible]	[illegible]	0.5%	[illegible]	[illegible]	[illegible]	1.3%	0.0%		[illegible]		
[redacted]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	3.7%	0		[illegible]		

Table 12. Hot Water Extraction Results for Various Biomass Sources

A contract for first commercial replication, "GreenBox+", has been secured at the Narampac-Cabano (N-C) corrugating medium mill in Quebec, Canada using a very similar feedstock as the Alpena Biorefinery (mixed hardwood chips including aspen, poplar, birch and maple). The first commercial license of the technology was sold for this installation and API is currently performing the engineering under the project. N-C's 500 t/d hardwood soda chemical pulping process will be retrofitted to API's hot water extraction process for production of corrugating medium and a concentrated hemicelluloses stream as shown in Figure 14.

N-C's project is funded in part through a $14.4 million award from Natural Resources Canada's Investments in Forest Industry Transformation Program. The project is based on laboratory studies performed by API and full-scale mill trials (conducted June through October 2012). Documented successes of the trials include

- Pulp / corrugating medium quality maintained
- 10%+ pulp yield increase, i.e. 10% increase in salable product
- Eliminates pulping chemical costs
- Eliminates Copeland fluidized bed reactor which burns concentrated black liquor

API has also secured contracts for laboratory optimization studies for corrugating mills in Canada, Finland, and Africa. API's investor, [redacted] API is actively marketing for commercial installations in the US.

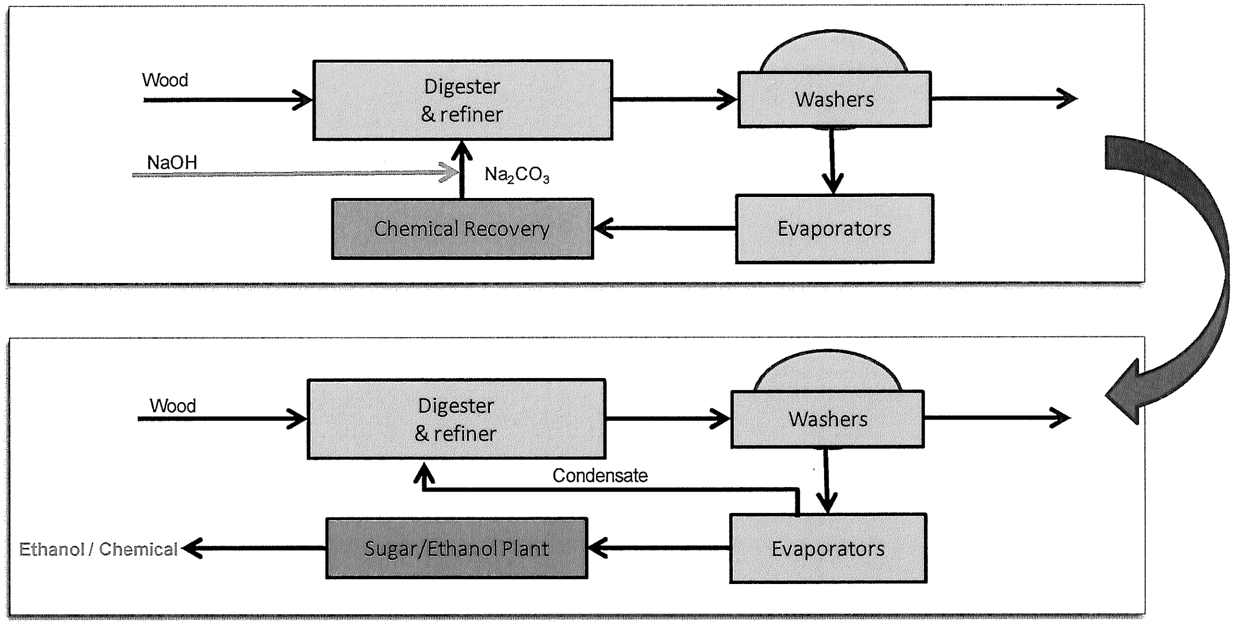
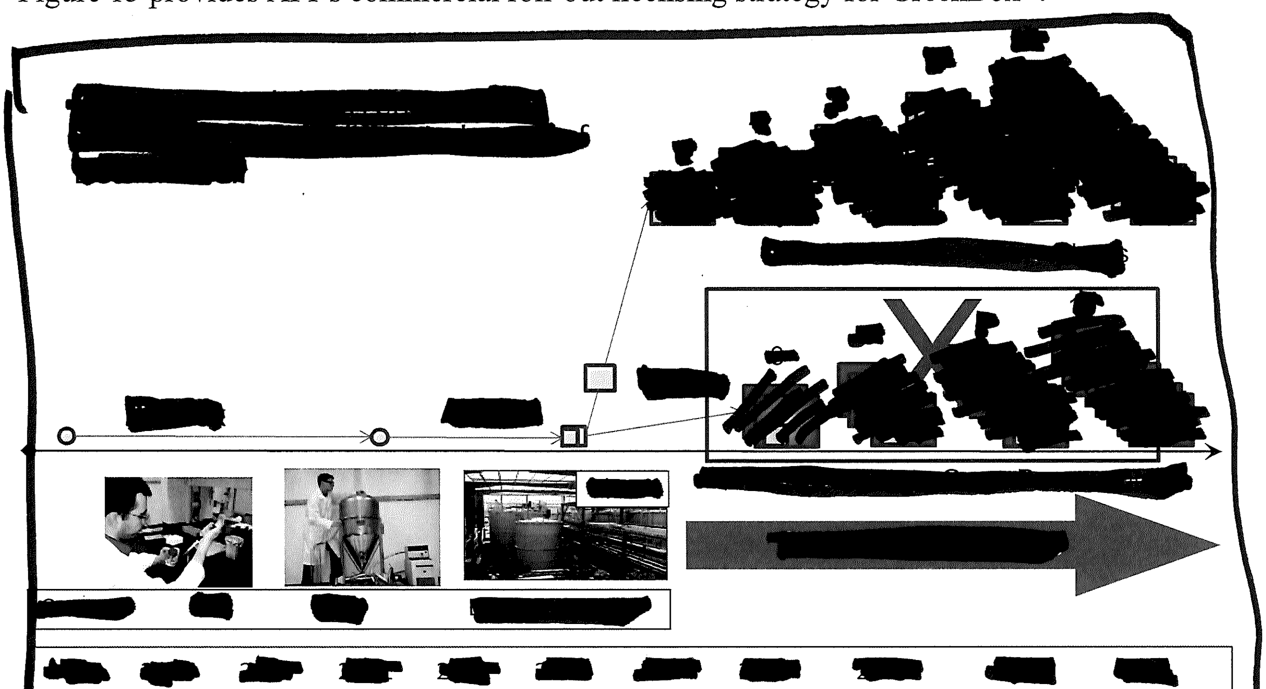


Figure 14. Schematic of First Commerical Installation

Figure 15 provides API's commercial roll-out licensing strategy for GreenBox+.



[redacted]

9 Products Developed Under Award

The following sections present products developed under the award including public releases of results by American Process Inc. or Michigan Institute of Technology.

9.1 Abstracts and Presentations

- Groves S, Liu J, Shonnard DR, and Bagley ST. Comparative metabolomics and physiology of xylose-fermenting yeast adapted to a dilute acid pretreated lignocellulose-containing waste stream. Society of Industrial Microbiology and Biotechnology 2012 Annual Meeting, August 12-16, 2012, Washington DC **(Oral Presentation)**
- Groves S, Liu J, Cary A, Roll A, Shonnard D, Bagley S. Proteomic Analysis of *Pichia stipitis* CBS 6054 adapted to an industrial waste stream. American Society of Microbiology 2012 Annual Meeting, June 16-19, 2012, San Francisco, CA **(*Poster Presentation*)**
- Groves S, Liu J, Cary A, Shonnard D, Bagley S. **Proteomic analysis of** *Scheffersomyces (Pichia) stipitis* **CBS 6054 during xylose, glucose, and mixed sugar fermentations.** American Society of Industrial Microbiology Michigan Branch Meeting Spring 2012, March 23-24, 2012, Mount Pleasant, MI **(Oral Presentation)**
- Groves S, Liu J, Jensen J, Shonnard DR, and Bagley ST. Conversion of an industrial waste stream to biofuels and other value-added products. Michigan Graduate Education Day, March 29, 2012, Lansing, MI **(*Poster Presentation*)**
- Groves S, Liu J, Roll A, Jensen J, Shonnard DR, and Bagley ST. Comparative proteomic analysis of *Scheffersomyces* (*Pichia*) *stipitis* CBS 6054 adapted to a dilute acid pretreated waste stream for improved ethanol yield. Society of Industrial Microbiology and Biotechnology Annual Meeting 2011, July 24-28, 2011, New Orleans, LA **(*Poster Presentation*)**
- Groves S, Liu J, Roll A, Jensen J, Shonnard DR, and Bagley ST. Comparative analysis of *Pichia stipitis* CBS 6054 adapted to a dilute acid pretreated waste stream for improved ethanol yield. American Society of Industrial Microbiology Michigan Branch Meeting Spring 2011, April 8-9, 2011 Grand Rapids, MI **(Oral Presentation)**
- Liu, J., Shonnard, D., Life Cycle Assessments of Ethanol Produced in an Integrated Biorefinery from a Forest Product Industry Wastewater Stream, AIChE 2012 Annual Meeting, Oct. 28-Nov. 2, 2012, Pittsburgh, PA
- Liu, J., Stawicki, D., Wagner, C., Shonnard, D., Groves, S., Bagley, S., Characterization of a Forest Products Waste Effluent for Production of Fermentable Sugars and Co-Products During Hydrolysis, AIChE 2011Annual Meeting, Oct. 16-21, 2011, Minneapolis MN
- Stephanie L. Groves, Jifei Liu, Alex Roll, Jill R. Jensen, David Shonnard and Susan Bagley, Comparative proteomic analysis of *Pichia stipitis* CBS 6054 adapted to a dilute acid pretreated waste stream for improved ethanol yield, Society for Industrial Microbiology, 2011 Annual Meeting and Exhibition, *July 24-28, 2011, New Orleans, LA*
- Liu J, Groves SL, Docsa S, Woldring D, Bagley ST, Shonnard DR. Determination of Optimal Hydrolysis Conditions for Production of Monomer Sugars from an Effluent Obtained from a

Forest Products Wastewater Stream. AICHE 2010 Annual Meeting, Nov. 7-12, 2010, Salt Lake City, UT

9.2 *Student Presentations*

- **Roll A, Groves S, Bagley S. Interaction effects of organic acids on the production of ethanol by** *Scheffersomyces (Pichia) stipitis* **CBS 6054.** American Society of Industrial Microbiology Michigan Branch Meeting Spring 2012, March 23-24, 2012, Mount Pleasant, MI
- **Roll A, Groves S, Bagley S.** The effect organic acids produced from the dilute acid pretreatment of lignocellulose on biomass and ethanol production in *Pichia stipitis* CBS 6054. American Society of Industrial Microbiology Michigan Branch Meeting Spring 2011, April 8-9, 2011 Grand Rapids, MI
- **Hubsky E, Groves S, Bagley S.** Adaptation of *Escherichia coli* KO11 from Dilute Acid Pretreated Industrial Effluent. American Society of Industrial Microbiology Michigan Branch Meeting Spring 2011, April 8-9, 2011 Grand Rapids, MI

9.3 *Publications (In Print)*

- Liu, J., Shonnard, D.R., Life Cycle Carbon Footprint of Ethanol and Potassium Acetate Produced from a Forest Product Wastewater Stream by a Co-located Biorefinery, *Sustainable Chemistry and Engineering*, http://dx.doi.org/10.1021/sc500256y.

9.4 *Publications (In Press)*

- Groves S, Lui J, Shonnard D, Bagley S (2013) Evaluation of hardboard manufacturing process wastewater as a feedstream for ethanol production. J Ind Microbiol Biotechnol 40(7):671-677.

9.5 *Publications (In Preparation)*

- Gleason S, Roll A, and Bagley ST. Interaction Effects of Lignocellulosic Degradation Products on Ethanol Fermentations by *Scheffersomyces stipitis* CBS 6054. *Prepared for publication in Bioresource Technology*
- Gleason S, Liu J, Shonnard DR, and Bagley ST. Evolutionary engineering of *Scheffersomyces stipitis* CBS 6054: Adaptation through repeated batch cultivation on hemicellulose hydrolysate for increased inhibitor tolerance and ethanol yields. *Prepared for publication in the Journal of Industrial Microbiology and Biotechnology*
- Gleason S and Bagley ST. Comparative Proteomic and Metabolomic Analysis of *Scheffersomyces stipitis* CBS 6054 Adapted to a Dilute-acid Hemicellulose Hydrolysate from Hardboard Manufacturing Process Wastewater. *Prepared for publication in the Journal of Industrial Microbiology and Biotechnology*

- Gleason S and Bagley ST. Fermentation Conditions for the Production of Ethanol from Dilute-acid Hemicellulose Hydrolysate to Ethanol by *Scheffersomyces stipitis* CBS 6054. *Prepared for publication in Biomass and Bioenergy as a short communication*
- Gleason SL, Roll AP, and Bagley ST. Evaluation of the Effect of Inhibitors Formed During the Dilute-acid Hydrolysis of Hardboard Manufacturing Process Wastewater on Ethanol Production by *Scheffersomyces stipitis* CBS 6054. P*repared for publication in Journal of Industrial Microbiology and Biotechnology as a short communication*
- Groves, S., Liu, J., Shonnard, D.R., Bagley, S.T. Evaluation of hardboard manufacturing process wastewater as a feedstream for ethanol production, ***Journal of Industrial Microbiology & Biotechnology*, in preparation, March, 2014**.
- Liu, J., Groves, S., Bagley, S.T., Shonnard, D.R., Characterization of a Hardboard Manufacturing Process Wastewater Stream and its Suitability for Conversion to Ethanol and Other Co-products, *BioFPR,* in preparation.
- Liu, J., Gleason, S., Bagley, S.T., Shonnard, D.R., Determination of optimum hydrolysis conditions for converaion of a forest product wastewater effluent to fermentable sugars, *Bioresource Technology*, in preparation.

9.6 Website

A website for the project is located at www.alpenabiorefinery.com. The site contains project progress announcements and construction/operations photos.

9.7 Inventions/Patent Applications, licensing agreements

A list of API's subject inventions to date under the DOE awards is provided in Table 13 below.

Title of Invention	Patent Number or Application Number	Docket No.	Issue Date or Priority Date if Pending	Status
DEICER COMPOSITIONS AND PROCESSES FOR MAKING DEICERS	**US 8,679,364** **United States**	**0033-DIV**	**Mar. 25, 2014**	**Granted**
CORROSION-INHIBITING DEICERS DERIVED FROM BIOMASS	**US 8,845,923** **United States**	**0033-CIP**	**Sept. 30, 2014**	**Granted**
PROCESSES FOR PRODUCING FERMENTABLE SUGARS AND LOW-ASH BIOMASS FOR COMBUSTION OR PELLETS	**US 8,685,685** **United States**	**0034-NPA**	**Apr. 1, 2014**	**Granted**
PROCESSES FOR PRODUCING LOW-ASH BIOMASS FOR COMBUSTION OR PELLETS	USSN 14/173,239 United States	0034-CON	Mar. 19, 2012	Pending

PROCESSES AND APPARATUS FOR PRODUCING FERMENTABLE SUGARS AND LOW-ASH BIOMASS FOR COMBUSTION AT REDUCED EMISSIONS	PCT/US13/32016 Worldwide (PCT)	0034-PCT	Mar. 19, 2012	Pending
PROCESSES AND APPARATUS FOR PRODUCING FERMENTABLE SUGARS AND LOW-ASH BIOMASS FOR COMBUSTION AT REDUCED EMISSIONS	BR 1120130226382 Brazil	0034-BR	Mar. 19, 2012	Pending
CORROSION-INHIBITING DEICERS DERIVED FROM BIOMASS	USSN 14/495,652 United States	0033-CON	Sept. 19, 2011	Pending
DEICER COMPOSITIONS AND PROCESSES FOR MAKING DEICERS	PCT/US12/56007 Worldwide (PCT)	0033-PCT	Sept. 19, 2011	Pending
DEICER COMPOSITIONS AND PROCESSES FOR MAKING DEICERS	BR 1120140064547 Brazil	0033-PCT-BR	Sept. 19, 2011	Pending
PROCESSES FOR PRODUCING FERMENTABLE SUGARS AND ENERGY-DENSE BIOMASS FOR COMBUSTION	USSN 13/829,355 United States	0035-NPA	Mar. 19, 2012	Pending/Allowed
PROCESSES FOR PRODUCING ENERGY-DENSE BIOMASS FOR COMBUSTION	USSN 14/173,332 United States	0035-CON	Mar. 19, 2012	Pending
PROCESSES AND APPARATUS FOR PRODUCING ENERGY-DENSE BIOMASS FOR COMBUSTION AND FERMENTABLE SUGARS FROM THE BIOMASS	PCT/US13/32022 Worldwide (PCT)	0035-PCT	Mar. 19, 2012	Pending
PROCESSES AND APPARATUS FOR PRODUCING ENERGY-DENSE BIOMASS FOR COMBUSTION AND FERMENTABLE SUGARS FROM THE BIOMASS	BR 1120130222565 Brazil	0035-BR	Mar. 19, 2012	Pending
PROCESSES FOR PRODUCING ENERGY-DENSE BIOMASS AND SUGARS OR SUGAR DERIVATIVES, BY INTEGRATED HYDROLYSIS AND TORREFACTION	USSN 13/874,761 United States	0038-NPA	May 2, 2012	Pending

PROCESSES FOR PRODUCING ENERGY-DENSE BIOMASS AND SUGARS OR SUGAR DERIVATIVES, BY INTEGRATED HYDROLYSIS AND TORREFACTION	PCT/US13/39175 Worldwide (PCT)	0038-PCT	May 2, 2012	Pending
PROCESSES AND APPARATUS FOR LIGNIN SEPARATION IN BIOREFINERIES	USSN 13/959,705 United States	0043-NPA	Aug. 6, 2012	Pending
PROCESSES AND APPARATUS FOR LIGNIN SEPARATION IN BIOREFINERIES	PCT/US13/53673 Worldwide (PCT)	0043-PCT	Aug. 6, 2012	Pending
PROCESSES AND APPARATUS FOR PRODUCING FERMENTABLE SUGARS, CELLULOSE SOLIDS, AND LIGNIN FROM LIGNOCELLULOSIC BIOMASS	USSN 14/017,286 United States	0036-NPA	Sept. 4, 2012	Pending
PROCESSES AND APPARATUS FOR PRODUCING FERMENTABLE SUGARS, CELLULOSE SOLIDS, AND LIGNIN FROM LIGNOCELLULOSIC BIOMASS	PCT/US13/58069 Worldwide (PCT)	0036-PCT	Sept. 4, 2012	Pending
PROCESSES FOR PRODUCING LEVULINIC ACID FROM BIOMASS	USSN 14/250,989 United States	0037-NPA	April 11, 2013	Pending
PROCESSES FOR PRODUCING LEVULINIC ACID FROM BIOMASS	PCT/US14/33810 Worldwide (PCT)	0037-PCT	April 11, 2013	Pending

Table 13. Subject Inventions under DOE Award

Please Note: American Process, Inc. has previously elected to retain title to all patent cases herein, in its IP holding company, API Intellectual Property Holdings, LLC. The U.S. Government has been granted a nonexclusive, nontransferable, irrevocable, paid-up, worldwide license in these subject inventions, patent applications and any resulting patents, including any continuation, divisional, reissue, or supplemental application thereof, to practice or to have practiced for or on behalf of the United States throughout the world; and of all other rights acquired by the Government by the patent rights clause in the above-identified contract. This license does not preclude the Government from asserting rights under the provisions of said contract, or from asserting any other rights of the Government with respect to the above-identified subject inventions.

9.8 Project Impact on American Process Inc. Business Development

DOE funding of the Alpena Biorefinery has been instrumental in establishing API as a pioneer in renewable materials, fuels and chemicals from biomass and as a developer of proprietary technologies and strategic alliances in this field to be scaled industrially throughout the world. Since receiving the DOE funding, API has grown from ~20 employees to over 130, secured an equity investor, built two biorefinery demonstration plants in the US, and sold the nations' first cellulosic ethanol from woody biomass.

In addition to the Alpena Biorefinery and its "GreenPower+" technology, API's AVAP™ technology is demonstrated at the "AVAP Biorefinery" in Thomaston, GA. The AVAP Biorefinery produces specialty cellulose products for advanced materials applications and exceptionally pure lignocellulosic sugars for on or off-site conversion into biobased fuels and chemicals. The plant can process three tons of biomass feedstock (dry basis) per day. Beginning in 2012, API has developed an innovative process based on the AVAP technology to produce commercially scalable, low cost nanocellulose. Nanocellulose is a material composed of high-strength nanosized cellulose crystalline rods known as cellulose nanocrystals (CNC) or longer crystalline and amorphous cellulose nanofibrils (CNF). API's nanocellulose has flexibility in final product morphology and surface properties (hydrophilic or hydrophobic) that can service the wide variety of emerging end-use market segments, including composites and foams for automotive, aerospace, and building construction, viscosity modifiers for cosmetics and oil drilling fluids, and high performance fillers for paper, packaging, paints, plastics, and cement. In addition to material performance properties like gelation, shear thinning, exceptionally high strength, and light weight, nanocellulose has a strong sustainability profile. Being made from biomass, it is renewable, biodegradable, compostable, and designed for the environment with a sustainable life cycle carbon footprint.

API's nanocellulose process and products are protected by granted patents patents (such as U.S. Patent Nos. 8,030,039, 8,038,842, 8,268,125, 8,585,863, and 8,685,167) and pending patents (such as U.S. Patent App. Pub. Nos. 2014/0154756, 2014/0154757, and 2014/0155301 and PCT publications WO 2014/085729 and WO 2014/085730) as well as proprietary know-how and trade secrets. In total, API has over 15 issued patents and over 130 patents pending in the biorefinery field. API is currently installing a nanocellulose pilot line at the AVAP Biorefinery for production of the full range of AVAP nanocellulose products by Q2 2015. API has a research and development group composed of engineers and materials scientists that are dedicated to advancing nanocellulose processing and products. The company also partners with industry, academia, and government for nano-scale characterization and development of commercial applications. University and government partners include the US Department of Agriculture's Forest Products Laboratory, the National Institute of Standards and Technology, Georgia Institute of Technology, the Renewable Bioproducts Institute, Clark Atlanta University, University of Maine, University of Alberta and Swinburne University. API's in-house expertise includes technology, product, and process development, engineering, procurement, plant installation, construction, operations, scale-up and commercialization.
In April 2013, the Brazilian company, GranBio, became a shareholder in API. GranBio is a 100% Brazilian-owned company, founded in June of 2011, and is known as an industrial pioneer in biomaterials, biofuels and biochemicals. The Brazilian National Development Bank (BNDESpar) is one of its shareholders.

10 Conclusions / Lessons Learned

American Process Inc.'s CEO, Theodora Retsina, delivered a presentation on the lessons learned from the Alpena Biorefinery Project at DOE's Biomass 2014 Conference. We include her list of lessons learned here for the intended benefit of future DOE project awardees:

1. Leverage existing "across the fence" infrastructure to mitigate:
   - First of a kind risks
   - Small scale of "first of a kind"
2. Engineering time and cost x ~2…..
3. Construction… more….$.
4. By nature of these projects.. "scope cannot be frozen"
5. Cost – contingency planning is needed
6. Financial Risk of "first of a kind" – very high
   - Startup curve
   - Nobody could have prepared us for such a slow startup curve
   - Rebuild – solve scale up issues – DOE assistance
   - Startup – slow startup curve
   - Milestone achieved – started shipments of ethanol
   - Second wave of debottlenecking – 6 months
7. A continuous team thread is needed from R&D to startup to operations
   - R&D scale up from pilot to Demo
   - Choice of equipment / configurations
   - NOBODY REALY KNOWS
8. Location, location, location!
   - Logistics
   - Feedstock
   - Product
   - Supplies / chemicals
   - Personnel
   - Environmental
   - Soil bearing capacity
9. It takes a village, a town - the whole world!
   - No company in the space has all the necessary parts
   - It is worse for smaller companies
   - Chain supply must be secured
   - There must be project management with knowledge of chain supply
   - Forest management and certification, feedstock gathering and procuring, E, P, C, sugar processing, permitting, safety, environmental, GMOs, new equipment, operator training, product certification, product sales, logistics
10. What is the right scale for a first of a kind?
    - Small
      - Does not solve demonstration and scale up needs
      - Smaller CAPEX and OPEX makes impact of "unpredictable events" smaller
      - Has no commercial life after demonstration
    - Large
      - Impact of cost overruns and schedule delays can be crippling

    - Once startup is survived – has independent commercial life.

11. Most Important ! Keep going
    - Many more obstacles
    - Environmental permit issues, capex escalation, IP entanglement, loss of personnel, loss of financing, changes in legislation, uncertainty in markets, uncertainty in fossil fuels prices, failures of others may trip you