Inventions and Innovations Final Report
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Completing Pre-Pilot Tasks To Scale Up Biomass Fractionation Pretreatment Apparatus From Batch To Continuous

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Executive Summary:

PureVision Technology, Inc. (PureVision) was the recipient of a $200,000 Invention and Innovations (I&I) grant from the U. S. Department of Energy (DOE) to complete pre-pilot tasks in order to scale up its patented biomass fractionation pretreatment apparatus[1] from batch to continuous processing. The initial goal of the I&I program, as detailed in PureVision's original application to the DOE, was to develop the design criteria to build a small continuous biomass fractionation pilot apparatus utilizing a retrofitted extruder with a novel screw configuration to create multiple reaction zones, separated by dynamic plugs within the reaction chamber that support the continuous counter-flow of liquids and solids at elevated temperature and pressure. Although the ultimate results of this 27-month I&I program exceeded the initial expectations, some of the originally planned tasks were not completed due to a modification of direction in the program. PureVision achieved its primary milestone by establishing the design criteria for a continuous process development unit (PDU). In addition, PureVision was able to complete the procurement, assembly, and initiate shake down of the PDU at Western Research Institute (WRI) in Laramie, WY during August 2003 to February 2004. During the month of March 2004, PureVision and WRI performed initial testing of the continuous PDU at WRI.

Project Description/Background:

During 1999 and 2000, PureVision's Chief Scientist Dr. Dick Wingerson conceived a novel technology that would have the potential to economically separate lignocellulosic materials into product streams for processing into energy and industrial products. During this same time and under the direction of Dick Wingerson, initial lab scale proof-of-concept testing was performed at Hazen Research, Inc. in Golden, CO. In 2001, Dr. Wingerson directed the bench scale batch fractionation research and development activities of processing corn stover at WRI in Laramie, WY. Based on promising data produced at WRI, PureVision established plans to scale up its new biomass pretreatment innovation that included submitting an I&I proposal to DOE.

One of the ultimate goals that the PureVision team continues to pursue is to produce a purified cellulose stream from biomass on a continuous basis. The bench scale biomass fractionation technology program at WRI produced a purified cellulose product with greater than 97% purity. The PureVision team believes that producing purified cellulose that can be enzymatically hydrolyzed will be a critical step necessary to achieve economical conversion of biomass into sugars and industrial products in biorefineries.

To mitigate major equipment design, development and testing efforts, it was determined by Dr. Wingerson that an off-the-shelf extruder apparatus was needed to efficiently proceed with efforts of progressing from batch to continuous processing. Extrusion equipment is typically used in the plastics and food processing industries. Since 2001 PureVision has had technical communications with extrusion equipment manufacturers

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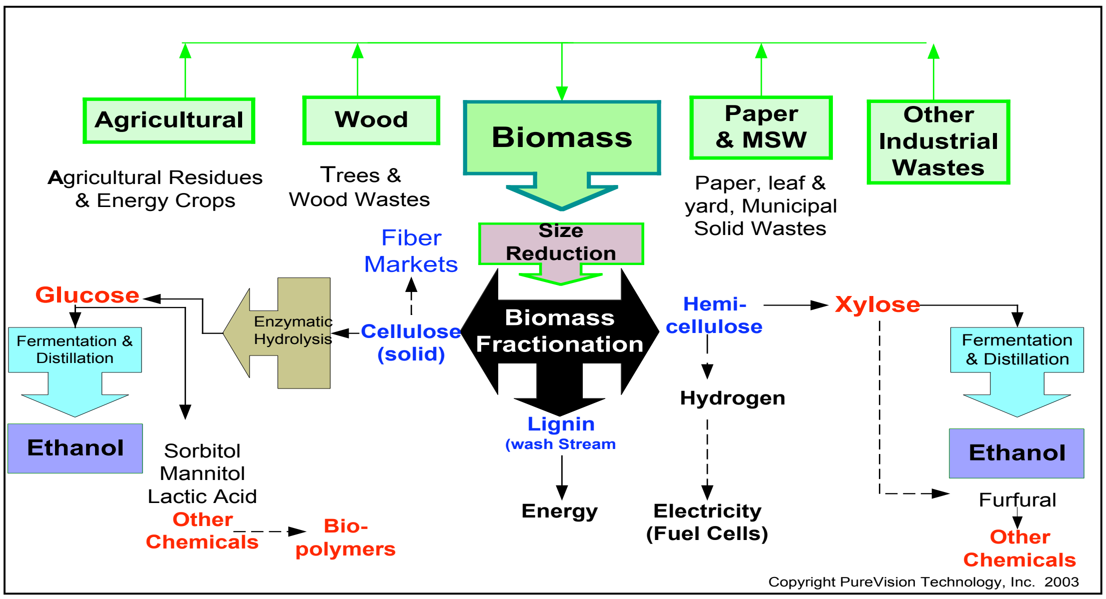
[1] U.S. Patent #6,419,788 B1, "Method of Treating Lignocellulosic Biomass to Produce Cellulose", granted to Dr. Richard C. Wingerson and assigned to PureVision Technology, Inc., July 16, 2002 and U.S. Patent #6,620,292 B2, "Cellulose Production from Lignocellulosic Biomass", granted to Dr. Richard C. Wingerson and assigned to PureVision Technology, Inc., September 16, 2003.

and has identified and communicated with some of the world's most recognized extrusion manufacturers from around the world.

Progressing from batch processing of biomass to continuous processing is a goal that must be achieved by PureVision in order to ultimately be successful in the marketplace. A small continuous pilot-scale apparatus should provide important engineering and design data for a large scale pilot plant and ultimately for a commercial demonstration biorefinery and will provide the basis for performing formal processing and economic assessments of converting diverse biomass feedstocks into resources at commercial biorefineries. The successful scale-up and implementation of the PureVision reactive fractionation biomass pretreatment technology could result in the economical processing of abundant biomass into resources, thereby enhancing national commerce, productivity and global competitiveness while minimizing adverse environmental impacts.



Flow diagram of the proposed PureVision biomass pretreatment fractionation technology and biorefinery.

Project Description/Goals:

The project description and the primary focus of this I&I project was to demonstrate continuous processing of biomass and to ultimately generate sufficient data to design a continuous biomass fractionation reactor. PureVision expected to demonstrate the ability to process corn stover on a continuous basis and be able to separate the biomass into three fractions. The first fraction was planned to be a wash liquor stream containing primarily water and most of the hemi-sugars. The second fraction was targeted to be an alkali wash liquor stream containing primarily water, lignin and the remaining of the hemi-sugars. The third fraction was planned to be a relatively purified cellulose product. Once a purified cellulose product was produced, PureVision had proposed to then enzymatically hydrolyze the cellulose into glucose.

Specific goals of the I & I program included the following:

1. Conduct pilot testing at an extruder manufacturer's pilot testing facility to process corn stover in a continuous reactor whereby dynamic plugs would be formed within the reaction chamber creating a high-pressure reaction zone.

2. Perform true counterflow of liquids washing the corn stover inside the continuous reaction chamber.
3. Extract the hemicellulose and lignin fractions from the corn stover within the reaction chamber, leaving a relatively pure cellulose product leaving the reaction chamber.
4. Characterize the wash liquor streams containing the hemicellulose and lignin fractions.
5. Characterize the cellulose fraction.
6. Perform enzymatic hydrolysis on the cellulose fraction.
7. Analyze the data from the above six tasks and develop the design criteria to build a small continuous pilot plant to convert biomass into primarily hemicellulose, lignin and cellulose fractions.

To summarize, the ultimate goal of this I&I project was to perform continuous pilot testing using a subcontractor's pilot plant in order to develop the costing and design criteria for a small continuous biomass pretreatment pilot plant. The research focused on determining the necessary elements, procedures and costs in retrofitting off-the-shelf plastic extrusion equipment into biomass processing equipment.

I & I Program Begins: One of the largest and historically most successful extruder manufacturers in the world is Coperion Corporation, formally Werner-Phliterer. Based in Germany and with a U.S. office and pilot-testing laboratory in New Jersey, many technical discussions took place between PureVision and Coperion Corporation between 2001 and 2002. Based on these early discussions, plans to perform pilot testing at Coperion's New Jersey laboratories were initiated. With the promise of a formal pilot-testing program, a technology team was assembled by PureVision to initiate the PureVision technology scale-up program. (This scale-up program was the focus of the Invention and Innovations application that was submitted to DOE.) After samples of corn stover were sent to Coperion and after the senior engineering staff from Germany became involved with the PureVision project, in August of 2002 Coperion determined that the PureVision counter-flow process would never work utilizing Coperion's extrusion equipment. This was about the same time PureVision was given the authority by DOE to began the I&I program.

Discouraged by the experience from Coperion and at the same time determined, PureVision continued on its path and focused on identifying another extruder manufacturer with experience in the pulp and paper industry. PureVision was able to find one such company, Clextral Corporation, based in Firminy, France. Clextral also has a U.S. presence with a sales office and a pilot-testing laboratory located in Tampa, Florida. In August 2002, PureVision began technical discussions with Clextral. After communications between DOE and PureVision regarding a change of subcontractor, formal plans were developed to visit the Tampa facility and meet with Clextral's engineering staff including a senior process engineer specializing in pulp and paper who would be coming from their headquarters in France.

Approximately one month after this I&I program began, Dr. Wingerson and Ed Lehrburger of PureVision visited Clextral's Tampa pilot testing laboratory and had extensive meetings over the two-day visit. Like Coperion, Clextral was also convinced that PureVision's counter-flow concept and technology could not work utilizing the

Clextral extrusion equipment. Due to the fact that Clextral had never tried to perform the unconventional type of testing and equipment configuration that Dr. Wingerson (the innovator of the fractionation technology) was suggesting, Clextral finally agreed to continue with the program. This two-day visit led to the initiation of a formal pilot testing program set up between PureVision and Clextral.

After sending various size samples of size-reduced corn stover to Tampa and Firminy, France, and after running this corn stover into the Tampa pilot testing feeder and extruder, it was finally determined what the appropriate particle size to which the stover had to be reduced for the Clextral pilot testing equipment. There were no costing or modeling data generated regarding what the extra costs were going to be at a commercial scale to have the corn stover appropriately size reduced. This two-month effort of trying to feed corn stover into an extruder led to a one-day pilot test run in France with only Clextral's engineers and staff performing the tests with the goal of achieving counterflow within the extruder. The results from this one-day test were not favorable.

After the unsuccessful one-day test run, it was determined by PureVision that Dr. Wingerson would need to be present during the next series of pilot tests.

At that point of the PureVision I&I program it was determined by Clextral that the pilot testing program could only continue if the tests took place at Clextral's headquarters, located in Firminy, France. The decision to evaluate the PureVision reactive fractionation concept in France was made because of the superior equipment and human resources available in Firminy as opposed to the satellite pilot testing facility in Tampa. As pilot testing in France was not part of the initial I&I program, formal arrangements were made between PureVision (Ed Lehrburger) and DOE (Andy Trenka and Eric Hass) to modify the original plan and then continue with the new plan to perform tests overseas. This new plan also included making the arrangements to transport via airplane approximately 1,000 kilograms of size-reduced Iowa corn stover to Firminy, France.

During the week of January 20-24 of 2003, Dr. Wingerson was present and participated in the second series of cold-flow tests of the continuous processing of corn stover utilizing Clextral's extrusion pilot testing equipment. The Clextral engineering staff directed the first three days of pilot testing with input from Dr. Wingerson. After a number of attempts, Dr. Wingerson and the Clextral staff demonstrated the formation of two dynamic dams or plugs that could retain pressures of at least 1,000 psi in the space between the plugs even as corn stover was continuously added to and removed from the plugs by the action of the extruder screws. This high-pressure zone is critical for high temperature continuous processing and represented a major technical success for the PureVision technology scale-up program. Although the ultimate PureVision reactive fractionation design calls for the formation of three dynamic plugs within the one reactor, the goal of achieving two dynamic plugs was the target during this second pilot testing session.

After many unsuccessful attempts of achieving true counter-flow by the Clextral engineers and on the last day of the testing sessions in France, under the direction of Dr. Wingerson, true counter-flow was accomplished utilizing extruder equipment perhaps for the first time in history. This was a great achievement and milestone for the PureVision I&I program and PureVision's overall technology advancement program.

Although true counter-flow was achieved during the testing session on the last day, it was only for a brief time before the liquids discharge port became clogged with corn stover fines. This situation presented a new processing challenge.

Drawing on what had been learned in France, a confidential technical report was prepared on February 6, 2003 by Dr. Wingerson and submitted to DOE (Andy Trenka and Eric Hass) by PureVision (Ed Lehrburger) at the DOE Golden field office on February 10, 2003. This confidential report is attached and marked as Appendix D.

Request to Modify the Direction of the I&I Program:

Two major successes resulted from the series of pilot tests in France. The first was proving that two dynamic plugs could be formed inside of the continuous processing chamber of an extruder to create a segregated high-pressure reaction zone. The second success was demonstrating that true counterflow of liquids could be achieved while maintaining pressures in the high-pressure zone within the extruder. The PureVision development work at Firminy also identified two significant technical challenges presented by the reactive fractionation technology that were greater than anticipated. Both of these challenges are highlighted below in the following paragraph.

The first and perhaps the greatest challenge was to devise a method to successfully separate the liquids flowing out of the reactor while keeping the solids moving, intact and under pressure. It was determined that this new solids/liquids separation processing challenge was going to require a new direction of the development program. A new design and engineering scheme would be needed on developing liquid extraction ports. This would mean a sizeable additional investment would be required for developing the needed apparatus to support liquid counter-flow at elevated temperature and pressure. The second challenge was to try and design an apparatus that could successfully allow the solids to be removed from the downstream end of the extruder while trying to maintain pressure inside the extruder. A new technical plan was going to have to be developed in order to firm up the design and costing of a solids discharge valve that would be adapted to the extruder.

The successes achieved during the first one-week testing session in Firminy, France in combination with large, non-recoverable costs anticipated from continuation of the I&I program led PureVision to seriously re-think its I&I scale-up program. PureVision gave careful consideration to modify the I&I program. The new direction is summarized below.

Instead of continuing with the program at Clextral's pilot testing facility in France, PureVision wanted to build a small continuous process development unit (PDU) in the U.S. With its own PDU, PureVision believed it would be in a much stronger position to be able to overcome all remaining processing obstacles. PureVision wanted the new focus to be directed toward designing, procuring and building a PDU. It was thought that PureVision would be in a much stronger position to then develop and perfect a solids/liquids separation system and a solids discharge port having its own PUD to work with.

PureVision had initially planned for and budgeted two additional one-week pilot testing sessions to take place in Tampa over the course of the I&I program. After the first one-week session in France, Clextral wanted these additional one-week pilot testing sessions to take place at their headquarters in France and not in Tampa. Performing the pilot tests in France would have ended up costing significantly more money compared to carrying out pilot testing in Tampa for obvious reasons. For this and a variety of other important reasons, PureVision made a formal oral request for a no-cost extension and a request to modify the direction of the I&I program. This request was detailed during a meeting between Ed Lehrburger, Andy Trenka and Eric Hass on February 10, 2003. On February 13, 2003 PureVision submitted a written request and corresponding budget modifications to Andy Trenka and Eric Hass to modify the direction of the I&I program. Although the general scope of the program did not change, the research and development direction did change.

Originally, the principal deliverable for the PureVision I&I program was to develop the design criteria to build a continuous pilot plant or process development unit (PDU). With the original plan of conducting three one-week pilot testing sessions, production of barrel-size quantities of liquid fractions containing derivatives of lignin and hemicellulose was anticipated, and this material was to be characterized and evaluated for its commercial potential. The new direction was to focus on the design, procurement, retrofit, shakedown and testing of a PDU. The decision to pursue this new direction was made following PureVision achieving its ultimate goal of the original I&I scope. This project modification led to adding new tasks as well as deleting some of the original tasks. The new change in direction cancelled the plans to continue with performing pilot testing at Clextral. This also resulted in not being able to produce barrel-size quantities of liquid fractions containing derivatives of lignin and hemicellulose and performing the analyses on the wash liquor streams.

At the time the request to change direction of the I&I program was made, PureVision had planned to be able to produce smaller samples of the wash liquor streams and also be able to produce a purified cellulose product that could be enzymatically hydrolyzed. Two weeks after the request to alter the direction of the program was submitted, DOE accepted the new direction per telephone conversation between Ed Lehrburger and Andy Trenka.

The heart of the PDU was envisioned to be an off-the-shelf extruder. The PureVision target was to purchase a used extruder for the primary reason of cost-savings. After eight weeks of looking extensively at the new and used twin-screw extruder market, PureVision located a used extruder that fit most of Dr. Wingerson's criteria. Within one week of performing due diligence on that particular extruder, it was sold to another party.

In addition to searching for used extruders, PureVision looked into purchasing new extruders. PureVision pursued three options in this regard. One was to purchase a new 25mm extruder from Clextral that would be manufactured in France. The second option was to purchase a 25mm extruder from a German manufacturer. The third option was pursued with a domestic extruder manufacturer based in Lebanon, Oregon. This company, Entek Extruders (Entek), was a relatively small subsidiary of a major company and had not been in the business of manufacturing extruders for very long. However, after many conversations and exchanges of technical documentation, Dr. Wingerson, Dennis Gertenbach (an engineer from Hazen Research, Inc.) and Ed Lehrburger spent two days visiting with Entek. The PureVision team discovered that Entek was a U.S.-based world-class extrusion manufacturing company with a state-of-the-art product line and a top-notch pilot-testing laboratory.

After many technical discussions and meetings, Ed Lehrburger engaged Entek to manufacturer a custom 27mm extruder for the following reasons:

- The extruder was the appropriate size, was versatile and had the correct metallurgy.
- The price negotiated was not much more than purchasing a used extruder after the costs of retrofitting a used extruder was taken into consideration.
- The purchase price Entek worked out was the best deal PureVision was able to find anywhere for a new extruder.
- Entek displayed a favorable corporate culture and expressed a strong willingness to work closely with PureVision throughout the pre-commercialization and commercialization stages of developing PureVision biorefineries.

In September of 2003, the Entek extruder was delivered to PureVision at the WRI facilities in Laramie, Wyoming. In addition to the extruder, there were many other PDU components that Dr. Wingerson had ordered from various U.S. venders that were delivered to the WRI facility to be retrofitted and connected to the extruder. These other components included pumps, piping, valves, sensors, instrumentation, heat exchangers, etc. The retrofitting of the PDU took place from September to November of 2003. Dr. Wingerson was the PDU designer and hands-on retrofitter, who at the time was also residing in Laramie and working as a WRI "visiting scientist."

Major Technical Challenges Addressed:

During the course of this I&I program, there were a number of significant technical challenges PureVision identified in progressing from a batch process to a continuous process. In summary, these challenges included:

1. Feeding biomass into the PDU while maintaining adequate processing pressures within the PDU;
2. Generating two dynamic plugs within the PDU to create a high-pressure zone able to maintain pressure under steady state processing conditions;
3. Performing true counter-flow, with solids going one direction and liquids going the other direction inside the PDU;
4. Extracting solids at the downstream end of the PDU after liquid counter-flow washing;
5. Extracting liquids after counter-flow washing of the biomass inside the PDU without plugging the liquid discharge port(s) with fines;
6. Performing steam explosion on the cellulose product utilizing two solid discharge valves and maintain pressures inside the PDU while successfully discharging solids at the downstream end of the PDU;
7. Performing steady state counterflow biomass fractionation at desired processing temperatures and pressures; and
8. Producing a purified cellulose product that is highly reactive and amenable to enzymatic hydrolysis.

One of the first technical challenges addressed was to develop a biomass feeding system to consistently and accurately feed biomass into the PDU. A number of feeding devices were tried and tested. Eventually, after much trial and error, two feeders were used for the ultimate application. Dr. Wingerson designed a custom crammer feeder that was later built by Hazen Research Inc. and an off-the-shelf loss-of-weight feeder was purchased and mounted upstream from the crammer feeder. After two modifications were made to the crammer feeder, a working feeding system had been achieved that could consistently feed and accurately measure the biomass entering the PDU.

During the program and most significantly during the month of March 2004, significant development efforts took place at WRI under the direction of Dr. Wingerson. Corn stover was first sized-reduced using a hammer mill with a three-eights inch screen. The corn stover was then processed through the PDU at ambient temperatures. During this one-month series of tests, major technical challenges were overcome including numbers 1 through 4 listed above. With reference to challenge number 2 (formation of two dynamic plugs that created a high- pressure zone), modifications to the layout of the extruder screws allowed two plugs to be formed to retain pressures from 200 psi to over 1,000 psi. These experiments demonstrated that maintenance of plugs takes power from the screws. Plugs should be no tighter than required, and optimization may be different for different feedstocks and operating conditions. Because of the complexity of the situation, the focus was on creating counter-flow of liquids utilizing only one reaction zone within the two dynamic plugs that were formed. Trying to make two reaction zones with three dynamic plugs within the extruder appeared to be too ambitious of a goal given the limitations of the PDU equipment, the time and money that was available to complete the I&I program.

The most significant remaining processing challenge (number 5 above) had been overcome during the last two weeks of March (see Confidential Appendix E). This milestone has now provided the PureVision development team with very important data that has led to a new design of a new liquids discharge port.

Although shakedown was initiated and the PDU ran for most of the month of March 2004, there were two primary factors that put a hold on continuing the operation of the PDU. First, after Dr. Wingerson overcame the problem of not being able to discharge liquids from the PDU, it would take at least six months for a custom liquids discharge apparatus to be designed and built. Second, PureVision exhausted its I&I budget and ran out of time and money before it could complete the lofty goals of running the PDU at optimal operating pressures and temperatures and producing a cellulose product to be used in initial hydrolysis testing. It therefore became impossible to generate meaningful wash liquor streams for analysis and to operate and test the solids discharge valves that had been designed, built by Hazen Research, Inc. and purchased for the PDU. This situation also made it impossible to obtain meaningful data for mass and energy balances and to perform preliminary economic modeling that would have provided some kind economic evaluation data at running a commercial biorefinery. PureVision anticipates these remaining tasks (number 6, 7 and 8 above) will be undertaken during the last two months of 2004 and during 2005.

Summary, Conclusions and Plans for Future Development Work:

PureVision's DOE-funded I&I program resulted in the design, fabrication, and initial testing of a unique continuous apparatus that in the future should be able to fractionate biomass into its major components. Although this targeted milestone was achieved, there were still significant technical questions that remain unanswered including the results of ramping up the PDU to proper operating pressures and temperatures, the characterization of the wash liquor streams and the ultimate purity of the cellulose. These remaining technical questions will hopefully be answered in the near future.

The ultimate goal of the PureVision I&I program was to develop the design criteria to build a continuous biomass-processing reactor. The goal was established without knowing that once the reactor was built according to the design everything would work as planned. After successfully demonstrating innovative processing techniques and after overcoming significant processing challenges, the PureVision team was able to build, shakedown and operate a continuous biomass reactor at WRI's bioprocessing laboratory in Laramie, WY.

After operating the PDU for approximately one month, there are now believed to be no significant unresolved technical barriers with the PureVision biomass reactive fractionation conversion technology. The most questionable features (such as feeding the biomass into the reactor, the formation of pressure retaining dynamic plugs to create at least one reaction chambers, the counter-flow of solids and liquids, and the separation of liquids with fines from solids at the liquid discharge ports) have been successfully demonstrated in the PDU. An apparatus for releasing cellulose product in a steam explosion has been fabricated at Hazen Research, Inc. for the PDU, but it has not been tested or optimized. And although processing pressures reached 1,000 psi in the reaction zone, PureVision was not able to ramp up processing temperatures and corresponding pressures to targeted levels due to timing, mechanical and budget constraints. The remaining major technical challenges are expected to be those associated with scale-up. As summarized by Dr. Wingerson, the innovator who conceived this unique biomass fractionation technology, "At this point, there appear to be no showstoppers."

In the end, PureVision believes it has successfully completed its Inventions and Innovations biomass fractionation scale-up program. The initial goal was to develop the design criteria to build a small continuous biomass fractionation apparatus utilizing an off-the-shelf extruder with a novel screw configuration to create multiple reaction zones, separated by dynamic plugs that support the continuous counter-flow of liquids and solids at elevated pressure. In many respects, the eventual outcome of this I&I program far exceeded the initial expectations by not only developing the design criteria for a continuous process development unit, but the PureVision team was able to procure, build, shakedown and perform initial cold-flow testing of the PDU. Although the primary goal of coming up with the design specifications for building a continuous biomass fractionation reactor was achieved, many of the other initial I&I program goals were not achieved including:

1. Not performing assays on the wash liquor streams;
2. Not operating the continuous biomass fractionation reactor at targeted processing temperatures and pressures;
3. Not producing a purified cellulose product;
4. Not performing enzymatic hydrolysis on a purified cellulose product; and
5. Not conducting economic modeling on the PureVision process of converting biomass into solid and liquid fractions.

Although there remain many unknowns about the ultimate commercial success of the PureVision reactive fractionation technology, the PureVision team has built and is currently operating a unique process development unit to advance the state of the art in economically converting lignocellulosic biomass into value-added product streams. The PDU is currently set up at Hazen Research, Inc. in Golden, CO and is now available for future research and development efforts to perform reactive fractionation on diverse biomass feedstocks.

Dr. Wingerson designed a new solids/liquids separation system that was later built. PureVision purchased this new apparatus. After initial testing of the new solids/liquids separation system in October 2004, additional separation problems continued and Dr. Wingerson refined the design and further modified the apparatus. As of December 15, 2004 the PDU and is up and running under cold-flow conditions at the Hazen Research, Inc. campus in Golden, CO.

From September 2004 to the present, the PureVision development team has been working on a new design of the solids discharge port located at the downstream end of the extruder. Based on new testing results, the original solids discharge system that was build by Hazen now has to be modified. Work is currently underway by the PureVision development team to modify and perfect the solids discharge system.

The PDU has yet to be operated at targeted elevated temperatures. This work must await the fabrication (now completed) of the redesigned liquid discharge ports and the re-design of the solids discharge system (currently underway). Optimization of operating conditions (temperatures, flows, reagents, etc.) and characterization of fractionation products will then be initiated.

PureVision has been notified of winning two DOE financial assistance awards. With the financial assistance that is expected to come as a result of these awards, PureVision plans to operate the PDU in 2004 and 2005 processing different feedstocks including corn stover and softwood. Among the planned research projects are fractionation of corn stover and related hydrolysis and wash liquor studies under USDA proposal # 03 1803-001, *Demonstration of a PureVision Biorefinery*. The second pending DOE-funded program will focus on converting loblolly pine woodchips into pulp and paper products under DOE solicitation #DE-PS36-03GO93015. These and other PureVision biomass conversion programs are expected to result in a new design for a scaled-up biomass fractionation prototype to be followed by a commercial demonstration biorefinery.

Subcontractors:

- Clextral, Firminy France and Tampa, Florida. Initial testing of biomass feeding into the extruder was undertaken at Clextral's Tampa facility followed by pilot scale testing of running corn stover through Clextral's testing equipment in Firminy, France.
- Hazen Research, Inc., Golden, CO. Designed, custom-built and retrofitted various components of the PDU.
- Western Research Institute, Laramie, WY. Location of assembly, shakedown and testing of PDU and assisted with retrofit and operations of running the PDU.
- Entek Extruders, Lebanon, Oregon. Extruder manufacturer and location of the design and testing of liquid discharge systems.

The entities below were initially planned to be subcontractors. Due to the change of direction of the PureVision I&I program, the following subcontractors were not hired to perform the following tasks:

- Coperion Corporation was planned to be hired to host the biomass-testing program utilizing Coperion's extruder equipment.

- Colorado University was planned to be hired to assist with the enzymatic hydrolysis of the cellulose produced from the PureVision process.
- Colorado State University was planned to be hired to assist with the enzymatic hydrolysis of the cellulose produced from the PureVision process.
- Membrane Technology and Research, Inc. was planned to be hired to conduct wash liquor stream separation and purification.

Patents: PureVision's biomass fractionation technology is patented in the U.S. The two patents conceived by Dr. Wingerson are:

Patent #6,419,788 B1, "Method of Treating Lignocellulosic Biomass to Produce Cellulose, granted to Dr. Richard C. Wingerson, July 16, 2002 and US 6,620,292 B2, "Cellulose Production From Lignocellulosic Biomass, granted to Dr. Richard C. Wingerson and assigned to PureVision Technology, Inc., September 16, 2003.

PureVision is also planning to apply for two new patents, one in the area of forming dynamic plugs separating reaction zones within the extruder and the other in the liquids discharge systems that has been designed and tested by PureVision. These two new patents will implement features set forth in the original two Wingerson patents.

Cost-Share Partner: PureVision was the only entity providing cost share for this I&I program. During the coarse of this I&I program, PureVision provided approximately 35% of the cost share broken down as follows:

Total Costs Charged To Inventions and Innovations Project	$307,134.40	
Total Funds Provided by U.S. Department of Energy	$200,000.00	
Total Amount of Cost Share Provided by PureVision		$107,134.40
Total Percentage of Cost Share Provided by PureVision		**35%**

Other Reports Submitted Regarding the I&I Program: 17 progress reports and a mid-term report have been prepared and delivered to DOE during this I&I program. Copies of any of these reports will be made available upon request.

Publications and Presentations: The following presentation was made as a partial result of the I&I program that included an abstract that was accepted for the World Renewable Energy Conference VIII, September 1-3, 2004 in Denver, Colorado, and presented by Ed Lehrburger.

An Emerging Biorefining Platform:
Lignocellulosic Biomass Processing Using Reactive Fractionation Technology

Richard Wingerson Ph.D., Ed Lehrburger, Carl Lehrburger
PureVision Technology, Inc., 511 N. McKinley, Ft. Lupton, CO USA 80621

PureVision's reactive fractionation is an emerging platform technology with the potential of converting diverse biomass inputs into fibers, sugars, fuels and industrial products in an economic manner. The primary business opportunity created by this innovation is to develop biorefineries that will benefit worldwide commerce and many

diverse industries including agriculture, forestry, biotech, waste management, transportation and energy.

Summary

A new biomass processing regime referred to as **Reactive Fractionation** or the "PureVision process" has demonstrated the ability to fractionate lignocellulosic materials into pure, reactive cellulose, hemicellulose and lignin.[1] The PureVision reactive fractionation method employs sequential, counterflow washing of biomass with water, dilute alkali, and alkali oxidizer at elevated temperature followed by steam explosion in a single continuous reactor. The process yields liquid fractions containing lignin, xylose and extractives and a solid fraction of highly reactive and essentially pure cellulose. This new technology shows promise in reducing both the use of reagents and the generation of unwanted by-products when employed as a pretreatment for processing lignocellulosic materials into sugars and fiber from diverse biomass including wood and agricultural residues. During 2002 and 2003 PureVision investigated biomass fractionation chemistry at bench scale (15 grams) in a washed bed reactor. The purified cellulose was hydrolyzed enzymatically with encouraging results. This work resulted in the design and construction of PureVision's process development unit (PDU) that came on line in January 2004. During 2004 PureVision is scaling up the innovation from batch to continuous processing.

It has long been recognized that pretreatment is necessary for a lignocellulosic biomass to be amenable to economical enzymatic hydrolysis. As a result, the primary deterrent for economic utilization of lignocellulose as a source of sugars in a biorefinery has been the high cost of acids and enzymes required to hydrolyze cellulose into sugars. Historically, biomass treatment and processing leading to sugar production has emphasized acid processes.[ii] Extensive work has also been done using hot water treatments with the goal to minimize reagent use.[iii,iv,v,vi] Steam explosion has been a major area of investigation.[vii] Research has also been reported using biomass treatments with alkali[viii] and oxidation[ix]. Alkali treatment of biomass is also used in the production of paper (Kraft pulping). Both the production of wood alcohol (using acid hydrolysis) and the Kraft process for paper pulp are noted for their consumption of chemicals and the environment impact of their wastes.

For the past 50 years, and especially for the past 20 years, there has been a growing interest in the use of lignocellulosic biomass as a source of sugar for the production of fuel ethanol and other chemicals. Acid hydrolysis has a history going back over 100 years for the production of wood alcohol, but the cost of reagents and waste disposal makes this approach unattractive today. The mainstream effort has focused on the most direct path to hydrolysis with subsequent cleanup of products as necessary for particular applications. Enzymatic hydrolysis is seen as a way to minimize waste disposal problems, but "pretreatment" of feedstock is necessary to increase the susceptibility of the biomass to enzymatic attack. The PureVision approach employs reactive fractionation to purify cellulose for enzymatic hydrolysis requiring fewer enzymes and as a means to separate/recover hemicellulose and lignin.

New Biomass Processing Platform

PureVision Technology, Inc. (PureVision) has focused its process development efforts at economically producing a reactive, purified cellulose stream that will undergo enzyme hydrolysis requiring a minimum of enzymes.[x] In the PureVision process, biomass fractionation is achieved in aqueous media (water and alkali) at elevated temperatures (up to 235ΥC).

Counterflow washing in multiple stages (yielding liquid fractions containing dissolved extractives, xylose, and lignin) is followed by steam explosion to yield a solid fraction of essentially pure cellulose. These reactions are carried out in a pressurized extruder apparatus.

The PureVision fractionation technology fundamentally differs from mainstream pretreatment approaches. Biomass is fed into a counter-flow reactor which produces purified cellulose and "wash liquor streams" containing the other biomass components.

The commercial rationale is that the cellulose fiber itself will find high value markets, and the purified cellulose is easily hydrolyzed to a purified glucose sugar that requires little downstream cleanup for fuel or chemical applications.

The PureVision approach has a much shorter history and has received far less funding than the mainstream biomass conversion technologies. None-the-less, the technical feasibility has been conclusively demonstrated. Cold-flow tests with water and three feedstocks (hammer milled corn stover, hammer milled wheat straw, and softwood planer-mill shavings) have been carried out under a U.S. Department of Energy (DOE) contract.[xi] These tests during early 2004 utilizing PureVision's PDU have demonstrated a continuous throughput of feedstock as well as a liquid/solids separation after counter-flow of liquids with solids.

Beginning in mid-2004, PureVision will utilize the PDU to evaluate continuous processing of corn stover. The experience obtained and the data generated using the continuous 10-kg/hour PDU will be applied in design, engineering, modeling, costing and assembly of a continuous 53mm prototype that will replace the 27mm PDU (scheduled for retirement) in 2005.

Reactive fractionation wash liquor streams contain a mix of sugars, lignin, protein, and other extractives. Commercial success of the reactive fractionation technology, as well as other biorefining approaches, will depend on economic utilization of the lignin, hemicellulose, mobilized cellulose, extractives, and fines that represent approximately two thirds of lignocellulosic feedstocks.

Cellulose is a biopolymer composed primarily of glucose. The reactive fractionation process emphasizes cellulose purity as a key feature of treating lignocellulosic materials. Previous PureVision bench-scale research on corn stover identified processing conditions to produce a cellulose product with minimum lignin content, identified the processing conditions to produce hemicellulose-rich and lignin-rich wash liquor streams, and documented the amenability of the cellulose to enzyme hydrolysis.[xii] Subsequent bench studies using wheat straw resulted in cellulose with greater than 97% of the lignin and greater than 99% of the hemicellulose removed. The reactive cellulose can be used both as a source of pulp for paper and non-paper markets and as feedstock for producing glucose for numerous applications including ethanol production.

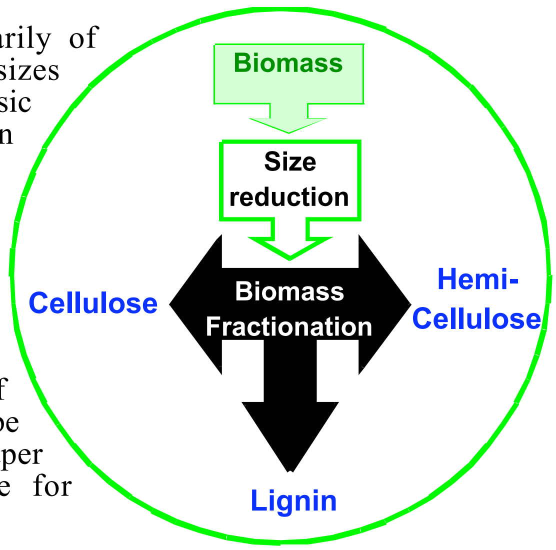


Figure 1: Biomass Fractionation

Hemicellulose is a biopolymer composed primarily of 5-carbon sugars that is mobilized in the wash liquor stream. 5-carbon hemicellulose sugars are raw materials for producing chemicals, consumer products, industrial products and energy. Products such as xylose and xylose derivatives, ethanol, bio-plastics, hydrogen and other chemicals can be produced from 5-carbon sugars.

Lignin: The wash liquor also contains the lignin that can be used as an energy source and/or be converted into valuable product streams that include use as adhesives, dyes, a binder for asphalt and higher-value building/construction applications.

Bio-Sugar Production

The enzyme industry is reducing the cost of cellulase enzymes used to produce sugars from biomass. Novozymes A/S, the world's largest manufacturer of enzymes today, has reported a twelve-fold enzyme cost reduction for the conversion of biomass into sugars for fuel ethanol production under its U.S. Department of Energy subcontract.[xiii] Novozymes has announced an overall reduction in the costs of cellulase enzymes for hydrolysis from above $5 (U.S.) to below $0.30 (U.S.) per gallon of ethanol, a twenty-fold decrease.[xiv]

The reactive quality of fractionated cellulose could further reduce the amount of enzymes needed for conversion into glucose. Others have shown net enzyme consumption with lignin free cellulose to be reduced by as much as a factor of five compared to that needed to hydrolyze biomass after conventional pretreatments that leave a significant fraction of residual lignin.[xv]

Lignocellulosic feedstock supplies in the United States are vast and virtually untapped for producing fermentation sugars. For example, the domestic ethanol industry could double corn grain ethanol production without new sources of grain by using corn stover to produce ethanol.

The reactive fractionation process also offers a technology to process waste wood into valuable products including ethanol. It is estimated there are presently 80 million tons of waste wood available annually for use as feedstocks in bio-refineries. The solid waste management industry is another potential beneficiary of the new technology, since over half of the materials entering solid waste disposal facilities are lignocellulosic. Establishing biorefineries at existing landfills could provide value-added products, reduce waste and conserve landfill space.

Biomass Derived Hydrogen using reactive fractionation

There are great challenges facing the emerging hydrogen production and fuel cell industry. The reactive fractionation technique could improve the overall economics for producing fuel cell-grade hydrogen (H_2) from biomass-derived ethanol, glucose and xylose. The chemical intermediates or bio-fuels produced in the PureVision process can be subsequently converted into H_2-rich gas by catalytic steam reforming followed by water-gas shift reaction. Advances by Pennsylvania State University researcher Dr. Chunshan Song indicate use of low-temperature catalytic oxidative steam reforming of methanol, ethanol and hydrocarbon fuels is encouraging for medium term fuel cell applications.[xvi] Ethanol, aqueous glucose and xylose can be easily transported to neighborhood hydrogen reforming stations to provide H_2 as transportation fuel or to large-scale H_2 production and generation facilities to provide heat and electricity. In both cases there is no need to transport H_2.

The reactive fractionation-to-hydrogen process might consist of three major components: (1) catalytic low-temperature oxidative steam reforming (OSR) of ethanol, glucose and xylose under ambient pressure to produce H_2-rich gas; (2) a single-stage oxygen-assisted water-gas shift (OWGS) reaction for the downstream CO clean-up; and (3) adsorptive removal of CO_2 under ambient conditions using novel molecular baskets for CO_2 capture. Reactive fractionation technology potentially offers lower cost and nearer term pathway for integrating renewable, zero-carbon emission H_2 into applications for both fuel cell-powered vehicles and electricity distributed generation devices.

Pulping Applications

PureVision's fractionation process has demonstrated at bench-scale the ability to improve the fiber quality of wheat straw over conventional pulping processes.[xvii] The resulting wheat straw pulp was determined to be of sufficient quality to meet hard wood pulp and pulp substitute technical specifications. Other pulp grades were identified as candidate markets including semi bleached newsprint and potentially higher grade dissolving pulps.[xviii] This indicates reactive fractionation could allow for the production of fiber from agricultural residues for pulp and paper markets using mini-mills or agri-biorefineries located near feedstock supplies.[xix]

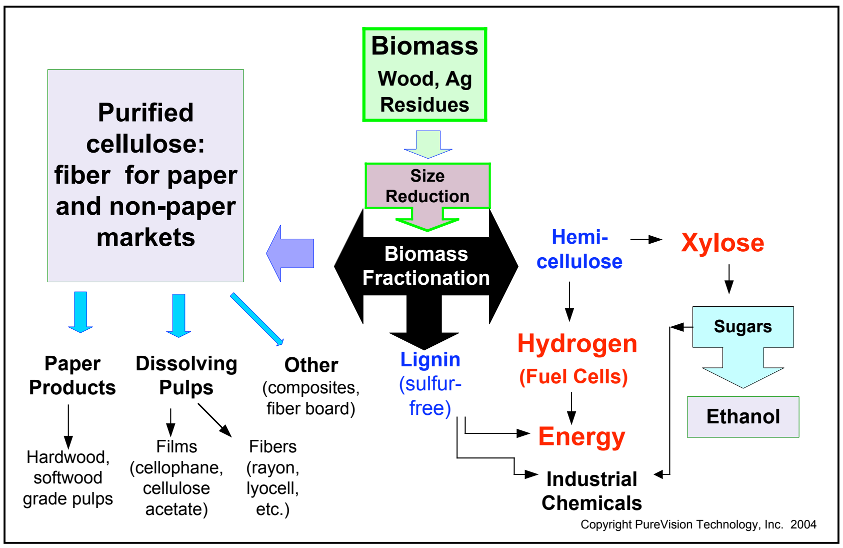
In addition to creating new sources of fiber from agriculture, the reactive fractionation technology potentially could replace the Kraft process for making fibers and paper products from wood. Because the PureVision technology does not use acid reagents to remove lignin and hemicellulose, the resulting fibers should be desired for pulp and paper products.

Currently, the sulfate or the Kraft process is the preferred chemical pulping process that is used to convert approximately 70% of the world's wood into pulp and wood-based products. Replacing the Kraft process with the PureVision process will substantially reduce or eliminate caustic effluent discharges by employing a closed loop pulping system. In addition to producing a cellulose fraction, hemicellulose and lignin fractions will also be produced. (PureVision is presenting a separate paper on the pulping applications of reactive fractionation at WREC VII.[xx])

Figure 3: Reactive Fractionation for Pulping Schematic

Biorefinery Economic Drivers

PureVision is proposing to commercialize the reactive fractionation technology as the core biorefining process for a "sugar platform" that will produce a wide range of bio-products and chemicals from diverse lignocellulosic materials including wood, waste wood, municipal solid waste and agricultural residues. Presently enzymatic hydrolysis of lignocellulosic biomass has been too costly to widely apply to conventional industries such as ethanol manufacturing. Two significant economic variables favorably affecting the commercial economics of the reactive fractionation technology are the declining cost of enzymes for hydrolysis and the dramatic increase in the selling price of ethanol and gasoline, now at a historical high (April 2004). A looming economic driver may become raising corn prices in coming years resulting from increasing demand from China and diminishing worldwide yields due to draught, demand for food and global warming.[xxi] Utilizing corn stover would approximately double the ethanol that can be produced from a corn crop without diverting more grain from food production.



Utilizing exponential scaling laws for the cost of labor and of various pieces of process equipment, investigators have concluded that the optimum capacity of biorefineries should be as much as several thousand dry tons of feed per day.[xxii],[xxiii] In the case of reactive fractionation processing, small units being mass-produced have their advantages. We envision hundreds and eventually thousands of commercial biorefineries, perhaps associated with farmers' co-ops. Standardization would enhance the learning curve and improve reliability. Larger plants can be co-located adjacent to existing ethanol plants. Smaller plants can be highly automated to minimize manpower requirements. Plants can be scattered perhaps 50 miles apart thereby reducing impacts on rural roads and other infrastructure. The concept of a rural community growing food for consumption and export while converting the leftover biomass to produce transportation fuel, electricity and other products is truly a global vision. A rural biorefinery would contribute to community, regional and national economic stability by producing food, transportation fuel, bio-products and electricity utilizing a 100% domestic labor pool. The PureVision reactive fractionation process offers an opportunity to maximize recovery of lignocellulosic materials in biorefineries. Anticipated improvement in biorefining economics and efficiency will benefit both sugar and fiber production. Commercialization of the technology could enhance biorefining economics, open up new opportunities for farmers, and increase national security by providing bio-products that can replace imported oil.

(References are at end of document)

Please submit any questions or comments regarding this Final Report to:

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The following tables are being provided as a supplement to this Final Report:

Appendix A

Final Task Schedule

Appendix B

PureVision I&I Task Schedule

Task Number	Task Description	Task Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
1	Design new screw configuration, retrofit, operate and collect data on Clextral 53mm pilot testing extruder	04/18/02	02/20/03	04/21/03	100%	Due to the limited success of this task and due to identifying significant scale-up problems, a program modification was initiated by PureVision and then approved during Feb. of 2003. See task 7
2,3	Collect data on liquid separation from hemi-sugar stream (task 2) and lignin stream (task 3)	04/18/03	10/15/03	N/A	0%	These tasks were revised per Feb. '03 program change. See task 7
4	Determine BTU value of purified lignin fraction	04/18/03	N/A	N/A	0%	This task was deleted during Feb. '03 and replaced with task 7
5	Perform enzymatic hydrolysis on cellulose fraction and collect data	04/30/03	11/31/03	N/A	0%	Unable to complete this task—ran out of money and time to perform testing at elevated temperatures.
6	Complete preliminary design/costing of pretreatment pilot plant	04/20/03	05/30/03	05/30/03	100%	Finalized design of continuous process development unit (PDU). Ultimate costing data was not determined until the PDU was built.
New Task 7	Purchase pretreatment pilot plant (PDU) equipment, retrofit, shakedown, operate PDU for 1 month and collect data	N/A	10/31/03	03/31/04	100%	This was a new task as of Feb. '03. All the components to build PDU were procured. The PDU was retrofitted, shaken down and operated for 1 month with new challenges identified.
8	Project Management & Reporting	09/18/03	05/18/04	12/15/04	100%	Final report and billing completed

Final Spending Schedule

Project Period: 08/18/02 to 12/1/04

Task	**Approved Budget**	**Final Project Expenditures**
Task 1. Design, retrofit, operate and collect data on Clextral twin screw extruder	$ 71,000	$ 144,123
Tasks 2 & 3. Collect data on liquid separation from hemi-sugar stream (task 2) and lignin stream (task 3)	50,000	-
Task 4. Determine BTU value of purified lignin fraction	4,000	-
Task 5. Perform enzymatic hydrolysis on cellulose fraction and collect data	17,000	-
Task 6. Complete preliminary design/costing of pretreatment pilot plant	34,000	12,620
New Task 7. PureVision Lab Work (procure, retrofit, shakedown and operate continuous process development unit)	42,224	115,153
Task 8. Project Management & Reporting	15,575	35,238.40
Total	**$** 233,799	**$** 307,134.40
DOE Share	200,000	200,000
Cost Share	**$** 33,799	**$** 107,134.40

Appendix C

Final Cost Share Contributions

Expenses and Cost Share Contributions After Program Change

Funding Source	Approved Cost Share		Final Contributions	
	Cash	In-Kind	Cash	In-Kind
PureVision Technology, Inc.	$-	$33,799	$-	$107,134.40
Total	$-	$33,799	$-	$107,134.40
Cumulative Cost Share Contributions				**$107,134.40**

Appendix D

(The following is CONFIDENTIAL information in red)

Appendix E

(The following is CONFIDENTIAL information in red)

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[xiv] "Novozymes and NREL report further progress in biomass-to-ethanol project", April 26, 2004. http://www.novozymes.com/cgi-bin/bvisapi.dll/press/press.jsp?id=28895&lang=en
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