# Final Report

**Prepared for:**

**DEPARTMENT OF ENERGY**

Use of Bark-Derived Pyrolysis Oils as a Phenol Substitute in Structural Panel Adhesives

(Wood Adhesive Formulations from Bark Derived Phenols)

DOE Cooperative Agreement No. DE-FC 36-00GO10597

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# EXECUTIVE SUMMARY

The quantities of underutilized wood and bark residues in North America, including forest and mill fiber, is enormous. Presently, a certain amount of this material is used as fuel for industrial process heat and power production, but the vast majority is stockpiled or landfilled. Energy prices remain relatively low in the USA and Canada, and energy-from-wood projects are capital intensive, with the result that there has not been a significant move by the industry nor by government utilities to sponsor broad-scale power generation form this vast potential resource.

One option, however, to effectively process wood and bark residues involves a Bio-Refinery concept, where value-added chemicals are produced, and any “leftover” biomass material is then directed to heat and/or power production. This Bio-Refinery concept is analogous to petroleum refining, where crude oil is refined to an array of valuable petro-chemical and light distillate fuel products, and the low-value barrel “bottoms” are used for fuel oil applications. In Ensyn’s Bio-Refinery configuration, wood residues are converted to core chemical products – resins, polymers and certain carbon materials – and the residual byproducts are used for energy. More specifically, wood residues are converted to a liquid “bio-oil” (and some carbon), and the core chemical products are efficiently and economically extracted from this liquid. The remaining liquid is then used to fuel boilers or engines for heat and power production.

This Bio-Refinery concept is particularly applicable and attractive to the large “integrated” forest companies such as Louisiana Pacific (L-P), Weyerhaeuser Company and Tembec. Since these companies generate significant quantities of wood and bark residues, they have a significant “in-house” demand for heat and power, and they are the principal customers for the resins that are used in their OSB and plywood panel mills. It is for this reason that all three of the above integrated forest companies have participated in this present project, the main focus of which was to develop commercial phenol formaldehyde (“PF”) resin systems with bark-derived and wood-derived phenolics as a principal ingredient. PF resins are the principal adhesives used in North America for OSB and plywood panel manufacture.

Initially, based on a proposal submitted in 2000, Louisiana Pacific Corp. and Ensyn Group Inc. secured financial help from DOE to pursue the development and large-scale demonstration of waste bark utilization in the production of wood adhesives. The program involved a number of participants, with strong complementary expertise, including:

1. Integrated Forest Products Companies: Louisiana Pacific, Weyerhaeuser and Tembec
2. A Commercial Bio-Refinery Technology Provider: Ensyn Renewables Inc. and its “Rapid Thermal Processing (RTP™) biomass conversion technology
3. Three of the Major PF North American Resin Producers: Dynea, Georgia Pacific and Tembec)
4. World-Leading Biomass Conversion, Wood Panel and Resin System Expertise: National Renewable Energy Laboratory (NREL) and Forintek

Each of these participants contributed specialized expertise and business experience toward the common goals. Individual tasks were set up for all participants and progress of these were reported periodically at scheduled meetings. This report contains all information made available for the record and the first part of it summarizes Ensyn’s interpretation of the key developments and results.

The principal outcome of this program is the world’s first commercial-scale production of an acceptable Phenol-Formaldehyde (PF) resin with a significant content (up to 50% of phenol displacement) of bark-derived and wood-derived phenolics. These phenol substitutes are identified as Natural Resin ("NR"), a "green" or "eco" resin known commercially as MNRP. During the course of this project the following general goals were accomplished:

1. Ensyn produced an array of Natural Resin products under controlled conditions from both bark and "white" wood in both lab-scale and commercial RTP™ equipment. These Natural Resins were solid flakes or powders which, over time, were optimized for specific PF resin formulations
2. NREL monitored the character and chemical properties of the NR products and related the properties to those of phenol and to anticipated performance in a PF resin system. NREL provided valuable insight into the science of NR production and product optimization
3. The resin companies, specifically Dynea, Georgia Pacific and Tembec, developed commercial resin systems using Natural Resin (MNRP) to displace up to 50% phenol in PF OSB resins. In the eventual OSB commercial mill trials, a commercial resin system using MNRP to displace 40% phenol was successfully tested.
4. The integrated forest companies (Louisiana Pacific, Weyerhaeuser and Tembec) collectively provided representative bark and wood residues for RTP™ conversion, supplied expertise and lab trials for resin tests, provided analytical services, made commercial OSB facilities available for mill trials, and interfaced with the resin producers for formulation development
5. The MNRP Natural Resin formulation was certified as an environmentally preferred product and granted use of the EcoLogo™ trademark
6. Forintek, Tembec and Weyerhaeuser labs tested the resin in OSB panels. Even at 40% phenol substitution, the MNRP resins were always at least equivalent to control PF resins in performance (i.e., no statistical difference), and in may cases exceeded control PF resin performance
7. In the process to achieve industry's acceptance by "TECO" certification, three successful commercial OSB mill trials were carried out at a commercial Tembec mill. TECO has confirmed that certification has been granted to the natural resin boards, but their final report was not available in time to be included in this final DOE report. During the course of the three mill trials, approximately 50,000 OSB panels were made with Ensyn's EcoLogo™ MNRP resin system, at a level of 40% phenol substitution.
8. Sensory tests were conducted by Weyerhaeuser in Washington and by the Energy Dept. (Canmet) in Ottawa, and conclusively indicated that there was no difference between MNRP OSB panels and commercial control PF OSB panels, even at 40% phenol displacement. These tests were conducted since MNRP alone possesses a slightly smoky aroma, not the "benzene-ring" odor that is typical of phenol. The Weyerhaeuser and Energy Dept. (Canmet) tests clearly showed that this slight smoky aroma did not survive in the final panel product.

The project evolved significantly over the course of the work. Initially, the focus was on the utilization of bark residues, since bark is typically less valuable than wood residues for commercial purposes, and much of it is greatly underutilized during both forest and mill operations. Furthermore, L-P was primarily interested in finding value-added uses for their large bark inventory. Ensyn produced bio-oil and MNRP from bark of different tree species supplied by Louisiana Pacific Corp. The bio-oil was generated using bench-scale, pilot-scale and commercial-scale RTP™ plants. The bench and pilot scale runs explored the effect of processing conditions such as reactor temperature while the National Renewable Energy Laboratory studied chemistry and the qualitative changes in the relevant products. Prototype resin formulations were generated using blends of MNRP and PF stocks by participating resin producers (Dynea and Georgia Pacific Resins Inc.). These were then used to bind sample OSB panels to be tested for qualities such as strength and resistance to ambient effects. Much needed information was gathered by this work and many tests showed acceptable or near acceptable results. The program participants were encouraged by these results anticipating good technical feasibility of the intended bark utilization. The wax content of MNRP produced from certain bark species was an issue that emerged during the program, and Ensyn had to adjust conversion and recovery processing to overcome this obstacle. Other bark species, as identified in the body of this report, had relatively low wax contents, and no problems were encountered in these instances.

The program focus was redirected from bark to forest and mill residues in 2001 when Louisiana Pacific experienced a significant reorganization and downsizing, and its key R&D project personnel were either re-assigned to operational positions or laid off from the company. At this time, Weyerhaeuser opted into the project as LP's replacement. In 2002 and 2003, Tembec was involved in mill trials of NR resin substitute from whitewood, with the NR eventually certified for commercial use. Ensyn produced a large batch of MNRP for the commercial mill run. OSB panels produced with the blend of MNRP-containing PF resin eventually proved to exceed some of the quality parameters typical for the PF resin alone. Ensyn further produced MNRP from two other wood residues supplied by Weyerhaeuser and the PF resin formulations from these were used to produce and test OSB panels at Forintek. Consistent with the Tembec experience, the results showed acceptable qualities of the sample boards.

To explore potential benefits of byproducts formed during RTP™ conversion of wood residues to bio-oil, Ensyn conducted a series of by-product char steam activation experiments. The work was completed in 2003. Results showed that a reasonable quality of activated char is obtainable to boost the economic feasibility of NR production.

The whole program was successfully completed in mid 2003. Material balance accounts indicate that in excess of 20 wt% of NR (MNRP) is obtainable on the moisture and ash free basis depending on the origin of bark-containing wood residues. Blends of MNRP and PF where MNRP can typically substitute 50 wt% phenol in PF, yield a resin formulation with acceptable bonding (adhesive) quality for OSB production. The economic benefits of substituting natural crude/gas derived components of PF with one derived from a renewable resource further enhance the environmental benefits of utilizing materials that are otherwise wasted. The combination of benefits suggests this approach as one of the future technological solutions in the wood processing industries where Ensyn actively pursues further business development.

# BACKGROUND

The background information in this section of the Final Report summarizes the commercial and scientific rationale for the work performed by Ensyn, Weyerhaeuser, Louisiana Pacific, and participants in this program. More detail in this regard can be found in the proposal document submitted to DOE in 2000. The data generated during this program clearly demonstrates the commercial and technical viability of the work, and is published in this report both for immediate use and for enhancing future development. It will be shown in this report that Ensyn's Rapid Thermal Processing (RTP™) pyrolysis technology, is presently the most suitable one for the conversion of bark and wood residues to phenolic products for use in wood adhesive formulations. Dramatic benefits, both economic and environmental, that can be realized by the implementation of RTP™ in this context, are currently the goal of Ensyn's business efforts.

## Bark as a waste stream

Estimates for bark waste streams generated in North America range in several tens of million tons per year. Softwood bark waste is estimated at almost 40 million tons per year. Wood product mills generate a significant amount of wood fines and bark that are not well suited for producing premium structural panels. A typical Oriented-Strand Board (OSB panels) mill may generate as much as 200,000 tons/year of bark and wood fines, and the OSB industry alone generates a total of approximately 4 million tons/year of bark. While some of the fines and bark can be used as a thermal energy supply in the mill, most OSB mills generate a net excess of bark and/or wood fines.

The excess bark for a single wood product mill is in the range of 5,000-50,000 tons/year, with many large OSB mills producing over 30,000 tons/year of excess bark. Additional excesses of bark exist in dimensional lumber mills, plywood mills, and other wood product facilities. Louisiana Pacific (L-P) alone generates an estimated 400,000 tons per year of bark that it is unable to use as fuel within its mills.

## Structural panels, resins and natural resins

Louisiana Pacific Corp. (L-P, the principal manager of this program until 2001) is the world's largest manufacturer of OSB. Weyerhaeuser Corp., the second largest producer, is close to LP in sales and production. Together, they supply about 50% of the North American OSB market. LP's core business is the manufacture of structural panels for the home building industry. OSB is the construction industry's preferred panel product, and the OSB industry is therefore growing, partly at the expense of plywood, and partly to satisfy generally increasing demand for structural wood panels. Unlike plywood, OSB can be produced from relatively small diameter trees, allowing for the sustainable utilization of today's forest resources.

Resin is a generic term used to describe both natural and synthetic glues, which derive their adhesive properties from their inherent ability to polymerize in a consistent and predictable fashion. The vast majority of modern industrial resins is synthetic, and is normally derived from petroleum and natural gas. Phenol formaldehyde (PF) resin is one of the main classes of synthetic resins in terms of production volume and total sales. The principal market application is for use as an adhesive binder in man-made wood products.

PF resin, because of its resistance to moisture, has a particular value for use in external (outdoor) or damp environments. It is therefore, the leading adhesive used for the manufacture of plywood, OSB and wafer board. As its name suggests, the principal ingredients in PF resins are phenol and formaldehyde. However, the finished product is actually a mixture of PF, caustic, and water. Assorted fillers, extenders and dispersion agents may then be added for specific adhesive applications. The OSB panel production requires up to 5 wt % of PF resin content.

The formaldehyde ingredient in PF resin is derived from methanol normally produced from natural gas. The phenol ingredient is typically manufactured from benzene (BTX crude refinery streams) and propylene via a cumene intermediate. When mixed together in water and with caustic (such as sodium hydroxide) added as a catalyst, phenol and formaldehyde undergo a condensation reaction to form ortho- or para-methylolphenol. The resultant PF resin, as shipped to market, is a dark brown liquid intermediate polymer, which polymerized by cross-linking. It is then cured in the final board, laminate or other product without catalyst simply by heating, at which time the final polymerization and cross-linking take place via further condensation reactions.

A non-fossil alternative for phenol, relevant to this current research program, is lignin. Lignin is the natural polymer that holds wood and bark fibers together and gives wood its strength. It can be extracted from wood and bark either intentionally or as a byproduct using a number of varying thermal and chemical processes, and its properties, when isolated and purified, depend largely on the process by which it was extracted. In general, lignin consists of a network of structures that are similar to those of PF resins. Lignin-based adhesive formulations have been tested for use within plywood, particleboard and fiberboard manufacture, but has experienced very limited commercial success. The addition of polymeric lignin to PF formulations has been found to prematurely gel the PF resin thereby reducing shelf life, limiting permeation of the lignin-PF resin into the wood, and producing an inferior mechanical bond. It is important to note that the lignins that are isolated and recovered from biomass, and which have been tested in resin formulations, are not identical to the natural lignin present in the original biomass, but are altered somewhat by the recovery process. Some examples of recovered lignin that has been tested in PF resin formulations are Kraft lignin, and other lignosulphonates. The reactivity of these lignins as a result of its complex high-mass structure is severely limited. However, if the proper conversion and recovery technology is applied, the lignin-like products derived by pyrolytic distillation can maintain certain desirable phenolic properties, and can remain chemically quite active. As a result, they are quite useful in PF resins, not as cheap fillers but as active phenol replacers.

### Suitability of excess bark for conversion by RTP™

Integration of bark pyrolysis as a unit operation in the overall process of producing wood products, especially OSB, does not require a special feedstock supply system because of the on-site availability of the feedstock bark. This application also ensures a balanced demand for the co-products, as explained later, to heat the OSB or plywood press.

In addition to the resources listed previously, it is estimated that another 4-6 million tons per year of underutilized biomass is generated during the harvesting operations for L-P’s OSB mills alone. Weyerhaeuser also reports the availability of enormous quantities of wood fibre residues from forest, mill and paper operations. While originally the main objective of this program was to demonstrate the integration of bark pyrolysis in L-P’s OSB mill operations, the technology can efficiently convert various wood wastes that are associated with harvesting and mill operations. The proximity of OSB mills to the tree harvesting operations makes these mills a logical location for a primary conversion process, converting biomass (rich in bark) into a renewable chemicals in the form of a high density, easily-transportable liquid bio-oil.

## Ensyn's RTP™ technology, product natural resin (MNRP) and byproducts

Ensyn's core technology is the Rapid Thermal Processing or RTP™, a proprietary process for the fast distillation of wood and other biomass via rapid thermal conversion (also referred to as pyrolytic distillation, and not to be confused with gasification). During RTP™ processing, wood, bark or other biomass is converted to high yields of product liquid and by-product char and gas. The immediate liquid product from biomass (termed "bio-oil") can be used directly as a fuel in boilers to produce process heat and process steam, in engines to produce power, and/or as a raw material for the production of various chemicals. The RTP™ technology is described in a number of patents and its application relies on know-how and skills acquired during its development and commercial use. A simplified Process Flow Diagram of the RTP™ process is shown in Figure 1.

The RTP™ technology was developed in the 1980s and was a breakthrough in biomass thermal conversion to bio-oil. It is still the only commercially proven fast pyrolysis process for industrial biomass conversion. The process is not severe. It takes place at atmospheric pressure without the use of catalysts, and at conversion temperatures that are about half of those required for combustion. The transport and feeding of the wood into the conversion unit, the conversion itself, and the recovery of the products, are simple and safe operations. Good economy is attainable at a size commensurate to the supply of feedstock. The first commercial RTP™ plant was commissioned in 1989 in Wisconsin. Since then, Ensyn has built and commissioned six additional commercial RTP™ plants, all of which are presently operating. The largest of these were installed in Rhinelander, Wisconsin in 1996 and 2002, and are capable of processing about 100 dry tons per day.

RTP™ converts hardwood to about 74% bio-oil 14% char and 12% gas. Depending on the biomass feedstock composition, the bio-oil liquid yield increases slightly with an increase in the cellulose content of the wood feedstock, and decreases somewhat with an increase in lignin content (note that cellulose and lignin are the principal structural components of wood). Both phenol and energy contents are greater in the case of wood feedstocks with higher lignin content, and since bark is typically high in lignin content, it gives higher yields of phenolic products than "white" wood residues.

The RTP™ bio-oil from wood and bark residues contains a highly reactive depolymerized lignin, the base for the natural resin (NR). To generate NR, the bio-oil is further processed through separation and evaporation steps such that non-resin chemicals and undesirable components (i.e., acids, volatiles, etc.) are removed, and the melting/softening point of the final NR product is thereby adjusted. The NR so produced by Ensyn is a phenolic black granular solid, and is marketed commercially as "MNRP". MNRP is delivered to the resin manufacturer where it is dissolved in the other components of resin production as a substitute for either some of the phenol component of a phenol-containing formaldehyde resin or for both the phenol and formaldehyde components of the resin, or as a substitute within urea formaldehyde type resins. It can typically substitute up to 50% of phenol in PF resins.

To use wood and other biomass residues in the context of their conversion to MNRP by RTP™, Ensyn's processing facilities would be integrated into wood products and resin manufacturing steps. A general illustration of the integrated manufacturing scheme is shown in Figure 2.

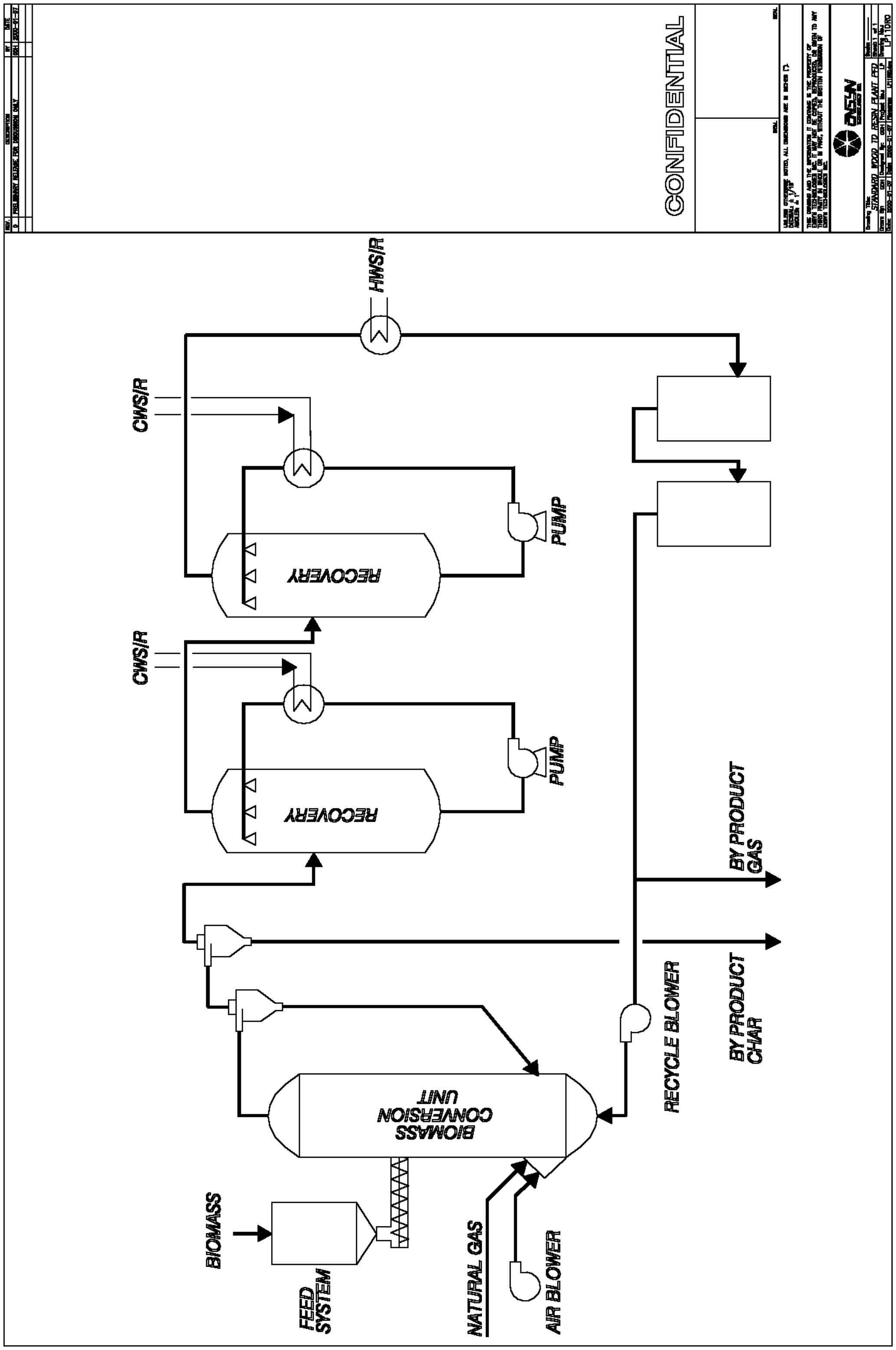


**Figure 1 - Simplified process flow diagram for Ensyn's RTP$^{TM}$**

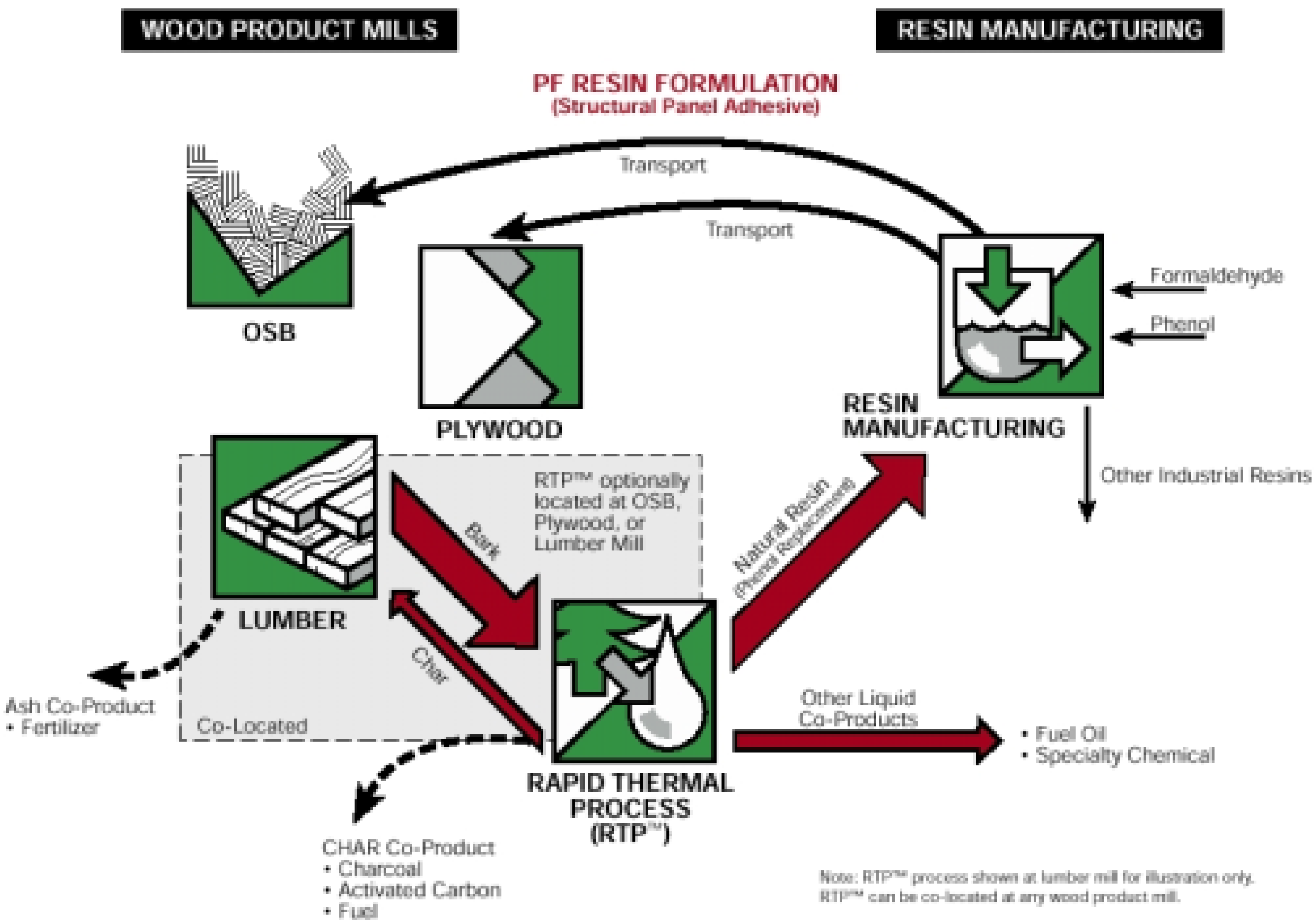


**Figure 2 - RTP™ processing integration into wood products and resin manufacturing**

# OBJECTIVES AND SCOPE

The main objective of this program was to pilot the world's first commercial-scale production of an acceptable PF resin containing natural resin (NR) ingredients, for use as an adhesive in OSB and plywood panel products. Natural Resin products, specifically MNRP are not lignin "fillers". They are chemically active, natural phenolics that effectively displace significant amounts of phenol in PF resins, and which are extracted from bark-derived and wood-derived bio-oils.

Other objectives included the enhancement of the economics of NR (MNRP) production by optimizing the production of certain RTP™ byproducts, particularly char and activated carbon. The options were to activate the char for use in waste-water and/or stack gas purification. The preliminary results indicate that RTP™ carbon may ultimately serve as a feedstock for activated carbon synthesis, as a fuel to be used within the wood product mill, or a fuel for an electrical power generating facility. Incorporation of the char as an industrial heat source for use in mill operations was L-P's initial intention for the carbon, and was also of interest to Weyerhaeuser as they stepped into in the project.

## Original Scope (2000)

Initially, a team of participants organized by L-P and Ensyn worked out a plan to achieve the above objectives that can be summarized as follows:

- produce demonstration scale quantities of a biomass-derived phenol substitute (i.e., NR or MNRP) at one of Ensyn's R&D plant facilities from three species of bark supplied by L-P; Southern Yellow Pine, Aspen/Poplar and Fir-Spruce softwood
- integrate the bark-derived NR phenol substitutes into commercial resin formulations, and evaluate these formulations in lab scale manufacture of plywood and OSB samples
- produce commercial scale batches of biomass-derived NR phenol substitute at one of Ensyn's commercial facilities, from one or more species of bark (supplied by L-P)
- manufacture commercial batch quantities of phenol-formaldehyde (PF) adhesive resins by both Neste and GPRI, two of the three largest resin suppliers in North America
- produce sufficient quantities of plywood and OSB structural panels at one or more of L-P's commercial mills, and certify the panel products by approved product certification laboratories
- integrate the use of char co-product in one of the combustion units of L-P

## Revised Scope (2001)

The program progressed successfully to more than half completion when Louisiana-Pacific (L-P) underwent a considerable corporate reorganization and the technology division was significantly downsized. As a result, L-P's development resources were withdrawn from this and a majority of L-P's other development projects. The downsizing was not related in any way to the progress nor to the results of the Natural Resin project, and L-P's commercial OSB Division operation has retained a strong interest in Ensyn's NR technology. Subsequently, Ensyn received DOE's approval to take over the team leadership, and Weyerhaeuser agreed to join the project as the OSB/plywood industry partner, in place of L-P. Since Weyerhaeuser had already been moving toward the commercial use of natural resins from “white” wood residues, and since the DOE program was generally consistent with technical and business goals of prior Weyerhaeuser-Ensyn work, only some minor revisions were needed. The revised program was subsequently approved with the essential changes noted:

- use white wood-derived in place of bark-derived NR in mill trials, since this was found comparable to bark-based NR; Weyerhaeuser generates an abundance of mixed wood/bark residues, and Ensyn had generated significant NR data from the RTP™ processing of white wood
- produce additional bench-scale and large-scale batches of NR from material supplied by Weyerhaeuser
- obtain additional NR laboratory characterizations
- produce commercial OSB panels with NR content (Tembec) for TECO certification
- perform wood-based and bark-based NR comparisons (end-user acceptance tests)
- generate char byproduct from wood residues and prepare steam-activated carbon for evaluation

## PARTICIPANTS AND FUNDING/COSTS SHARE

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| | Phase I Actual $ | Phase II Budget $ | Phase II Actual $ | Total Costs Actual $ | Contribution % |
|---|---|---|---|---|---|
| **DOE** | 763,227 | 445,236 | 445,236 | 1,208,463 | 51 |
| **Ensyn** | 206,957 | 163,724 | 201,274 | 408,231 | 17 |
| **L-P** | 90,296 | - | - | 90,296 | 4 |
| **NREL** | 200,000 | - | - | 200,000 | 8 |
| **GP** | 46,937 | 56,335 | 1,081 | 48,018 | 2 |
| **Dynea** | 63,522 | 73,419 | - | 63,522 | 3 |
| **Weyerhaeuser** | - | 173,400 | 173,400 | 173,400 | 7 |
| **Tembec** | - | 82,000 | 179,000 | 179,000 | 8 |
| **Forintek** | - | - | - | - | 0 |
| **Total** | 1,370,939 | 994,114 | 999,991 | 2,370,930 | 100 |

# WORK PLANNED AND COMPLETED

The original plan consisted of three phases. The tasks of Phases I and II involved small-scale feed preparation and natural resin (MNRP) production runs. Combined with laboratory and resin manufacturer tests, these were meant to evaluate the bark source suitability and optimal operating conditions for MNRP production. Ensyn produced the MNRP in its bench scale and pilot scale facilities in Greely, Ontario. All tasks originally included in Phase I and in Phase II were completed by the end of 2001.

The original Phase III tasks involved the large-scale production of MNRP batches from the selected bark stock supplied by L-P. Ensyn planned to produce these at one of its commercial plants in Wisconsin. Dynea and Georgia Pacific were then to prepare natural resin (NR) formulations (also identifiable as “green” PF resins) having the bark-derived MNRP as one of its components. Using the green resin as wood adhesive, L-P planned to manufacture OSB panels for subsequent tests and panel certification. Ensyn did produce a 600 lb batch of MNRP using the RTP™ system in Manitowoc, Wisconsin and its product recovery equipment in Greely, Ontario in 2001. After reorganization and downsizing of Louisiana-Pacific, and the introduction of Weyerhaeuser into the project in 2001 (as noted in the previous sections) the original Phase III tasks were revised. Since the original Phases I and II were somewhat modified (expanded as needed) while the project progressed throughout 2001, and since they both exclusively relate to processing bark materials, the relevant data and observations are now reported under Phase I.

Since L-P’s bark residues were of little relevance to Weyerhaeuser’s involvement, Ensyn committed to produce two large-scale batches of MNRP from mill residue (sawdust). These batches were produced in Rhinelander, Wisconsin. ARC Resins (now Tembec) produced the green PF resin formulation and Tembec manufactured TECO-certified OSB panels. The revised work had additional tasks involving lab tests of MNRP, char and activated char produced from wood residues obtained from Weyerhaeuser. Ensyn used its bench-scale and lab equipment to perform these tasks. A comparison of both bark-derived and wood-derived MNRP was also included, and was performed by Ensyn using its MNRP database. Samples of PF resins, that contained MNRP from Weyerhaeuser’s forest and mill residues were produced by Dynea. These were subsequently used to manufacture OSB panel prototypes at Forintek where the effect of MNRP on OSB quality was compared with that of Dynea’s standard PF resin. All work, not exclusively related to bark-derived material is now reported under Phase II.

The bark feedstock preparation for the work related to Phase I was done by L-P. This involved green bark drying, grinding and sieving. L-P also prepared for Particulate Matter (PM) and Volatile Organic Compound (VOC) emission tests during both the drying and board pressing steps to determine what, if any, air pollution control devices would be required on a commercial unit.

All planned work was completed by mid 2003. The results and outcome of individual tasks are reported under each Phase in the following sections. This part of the Final Report mainly summarizes the results and provides Ensyn’s interpretations when deemed desirable. Relevant details and supporting documents are appended.

## Phase I – Results related to bark materials

Specific objectives in this phase included the bench and pilot-scale RTP™ processing of three different species of bark (Aspen, Spruce Pine Fir, and Southern Yellow Pine) at different RTP™ process temperatures to enable the production of natural resin (NR) samples with good adhesive properties. Nine initial bench scale runs were originally targeted at three different temperatures. Ensyn's operating experience and know-how of wood processing helped selecting the temperature range. Small quantity samples produced in the bench-scale unit (BSU) went to NREL for analyses and analytical method development purposes. The process flow schematic for the BSU is shown in Appendix A1. The bark feedstock and processing conditions for the subsequent large-scale work were then assessed based on Ensyn's operating and NREL's lab results.

Selected bark feedstock data

In the summer of 2000 Louisiana-Pacific provided Ensyn with samples of three different bark species,

- Southern Yellow Pine bark ("SYP"),
- Aspen/Poplar bark ("Aspen"), and
- Spruce Pine Fir bark ("SPF").

The "as-received" samples contained some extremely small particles (i.e., "fines"), the conversion of which can cause processing problems in the small BSU unit (note that fines are not a concern in Ensyn's commercial operations). The fines were therefore removed by sieving prior to RTP™ conversion. The properties of "as fed" bark samples (to the BSU) were analyzed at Ensyn's lab and are shown in Table 1.

**Table 1 - Feedstock bark analyses**

| Analysis | | Aspen | SYP | SPF | **SPF |
|---|---|---|---|---|---|
| | | ECN 239 | ECN 240 | ECN 243 | **ECN 247 |
| | | | | | |
| Bulk density @ 25°C | g/mL | 0.419 | 0.392 | 0.22 | 0.238 |
| Moisture content | wt % | 10.2 | 13.8 | 9.2 | 10.9 |
| Ash | wt % | 5.55 | 1.63 | 1.94 | 2.06 |
| HHV | MJ/kg | 17.6 | 17.5 | 18.9 | 19.3 |
| Lignin (Klason) content | wt % | 43.5 | 50.2 | 47.6 | 47.6 |
| Cellulose | wt % | 20.9 | 21.2 | 21.9 | 24.2 |
| Hemicellulose | wt % | 19.0 | 13.7 | 13.5 | 14.1 |
| Wood Extractives (resin) | wt% | 3.0 | 2.1 | 10.5 | 16.3 |
| Carbohydrates | wt % | 39.9 | 34.9 | 35.4 | 38.3 |

** Additional feedstock was required to complete the work with this species. These analyses were obtained from the second shipment of Spruce Pine Fir bark from Louisiana Pacific.

Initial BSU runs and results

The initial production involved 22 runs on the BSU to provide sufficient quantities of MNRP samples. Thirteen small batch samples of MNRP were produced from the three bark species. Incorporated into the 22 runs was the production of two larger samples (5 – 10 lbs. each) from the Spruce Pine Fir bark. All NR samples were shipped to the NREL laboratory. The larger NR samples produced from Spruce Pine Fir bark were shipped to the resin manufacturers, GP and Dynea (Neste). A summary of the BSU runs to produce MNRP samples is shown in Table 2.

As seen in Table 2 the reactor temperatures were varied from 500ºC-615ºC and the BSU was operated for several hours at a throughput of approximately 2 to 3 pounds per hour. Some runs were repeated to obtain sufficient product quantities for subsequent analyses and evaluation. MNRP yields were determined for each run based on the analytical data for both the feedstock and the MNRP product. Analyses included

- ash content of the feedstock and products
- trace elemental analysis
- Higher Heating Value (HHV)
- byproduct gas analysis
- feedstock basic analysis (lignin, cellulose, hemicellulose content)
- bulk density

MNRP samples were shipped to NREL for evaluations based on characteristics identified by the resin manufacturing companies. The NREL evaluations were in turn used to identify operating conditions to produce a satisfactory quality MNRP. The MNRP was subsequently produced and delivered to the resin manufacturers for NR/PF (green) resin formulation.

**Table 2 – Bench scale unit run summary**

| Run No. | Reactor Temperature, ºC | Feedstock Bark | Feed Sample ECN No. | Date |
|---|---|---|---|---|
| | | | | |
| 1A | 550 | Aspen | 239 | 7/31/00 |
| 2A | 550 | Aspen | 239 | 8/2/00 |
| 3A | 500 | Aspen | 239 | 8/11/00 |
| 4A | 500 | Aspen | 239 | 8/15/00 |
| 5A | 500 | Aspen | 239 | 8/17/00 |
| 6A | 600 | Aspen | 239 | 8/22/00 |
| 7A | 600 | Aspen | 239 | 8/24/00 |
| 8A | 600 | SYP | 240 | 8/28/00 |
| 9A | 550 | SYP | 240 | 8/29/00 |
| 10A | 500 | Aspen | 239 | 8/30/00 |
| 11A | 550 | Aspen | 239 | 8/31/00 |
| 12A | 500 | SYP | 240 | 9/5/00 |
| 13A | 550 | SYP | 240 | 9/6/00 |
| 14A | 600 | SPF | 243 | 9/14/00 |
| 15A | 550 | SPF | 243 | 9/18/00 |
| 21A | 575 | SPF | 243 | 10/26/00 |
| 22A | 575 | SPF | 243 | 10/27/00 |
| 24A | 575 | SPF | 243 | 11/1/00 |
| 25A | 575 | SPF | 247 | 11/3/00 |
| 26A | 575 | SPF | 247 | 11/6/00 |
| 27A | 575 | SPF | 247 | 11/9/00 |
| 34A | 615 | Aspen | 239 | 12/13/00 |

Three of the 13 small batches of MNRP samples were produced to exhibit low acidity and low molecular weight properties to assist in the MNRP product solubility with the manufacturers' proprietary PF resin components. Ensyn then produced two 5-pound samples of SPF bark-derived MNRP, which were shipped to Dynea and GP. NREL received small quantities of these samples for analyses. Small samples of MNRP from Aspen (run 34A) were sent to Dynea, GP and NREL. The analytical data summaries and details for the samples sent to NREL, Dynea/NESTE and GP can be found in Appendices A2 and A3.

At a project team meeting on December 8, 2000, the resin companies indicated that the 5-lb samples had been adequate for analytical and preliminary resin formulation purposes, but requested that larger samples (1 pail or approximately 15 lbs.) would be required to produce suitable quantities of final resin formulations. The larger (pail-sized) samples were produced in the pilot-scale system.

Examples of mass balance are shown in Table 3 and Table 4 for the runs 25A and 34A. Run 25A used Spruce Pine Fir bark as feedstock at a reactor temperature 585°C whereas run 34A used Aspen bark as feedstock with at 615°C.

**Table 3 - Mass Balance of Experimental Run 25A – SPF Bark Feedstock**

| **INPUT Moisture and Ash Free (MAF)** | | |
|---|---|---|
| | Weight (grams) | wt % |
| SPF Bark Feedstock | 4323.28 | 100 |
| **OUTPUT Moisture and Ash Free (MAF)** | | |
| NR | 860.5 | 26.34 |
| Liquid Product (Non-resinous) | 25.88 | 7.92 |
| Pyrolytic Water (by difference) | 566.03 | 17.33 |
| Gas | 704.0 | 21.55 |
| Char | 877.48 | 26.86 |

**Table 4 - Mass Balance of Experimental Run 34A - Aspen Bark Feedstock**

| **INPUT Moisture and Ash Free (MAF)** | | |
|---|---|---|
| | Weight (grams) | wt % |
| Aspen Bark Feedstock | 5053.74 | 100 |
| **OUTPUT Moisture and Ash Free (MAF)** | | |
| NR | 409.21 | 10.5 |
| Liquid Product (Non-resinous) | 723.56 | 18.56 |
| Pyrolytic Water (by difference) | 829.92 | 21.29 |
| Gas | 1159.17 | 29.73 |
| Char | 776.54 | 19.92 |

The difference in NR yields is attributable to the combined effects of feedstock properties and processing conditions. Specifically, SPF bark contains more bio-oil precursors with more extractives and less ash as compared to Aspen. The Aspen run temperature was already high enough to cause undesirable over cracking to byproduct gas in place of the liquid product.

Additional bench and RTP-4 pilot-scale work

The main reason for using the pilot-scale RTP™ was to obtain larger batches of bark-derived MNRP prior to large-scale production. Eventually, Ensyn conducted three pilot and four additional bench-scale runs. In preparation, the front-end feedstock delivery system had to be modified. A modified surge bin and a conveyor were installed early in 2001. Photographs in Appendix E depict the installation progress of the surge bin and feedstock handling system.

The process flow diagram for the pilot unit (PSU) also referred to as RTP-4 is shown in Appendix A4. Overall yields for the PSU runs are schematically interpreted. Table 5 and Table 6 highlight selected operational, feedstock and product data.

**Table 5 – Feedstock, operating and product data for additional BSU runs**

| Property and Run Parameters | | Run 44A | Run 45A | Run 48A | Run 49A |
|---|---|---|---|---|---|
| Feedstock Bark | | Aspen | SPF | Aspen | Aspen |
| Particle Mesh Size | | -8 +325 | -8 +325 | -20 +325 | -20 +325 |
| Moisture Content | wt % | 6.7 | 6.7 | 9.0 | 9.0 |
| Ash Content | wt % | 3.34 | 3.34 | 5.58 | 5.58 |
| Amount fed | kg | 2.677 | 2.842 | 3.254 | 3.267 |
| Run Temperature | °C | 531 | 539 | 514 | 507 |
| Run Time | min | 90 | 90 | 120 | 90 |
| Liquid (bio-oil) Yield (maf)* | wt % | | 55.7 | | 59.6 |
| Gas Yield (maf)* | wt % | | 17.5 | | 18.8 |
| Char Yield (maf)* | wt % | | 18.2 | | 21.7 |
| Total Recovered (maf)* | wt % | | 91.5 | | 100.1 |
| Bio Oil Lignin Content | wt % | 45.1 | 45.7 | | 40.6 |
| MNRP yield (maf)* | wt % | 22.4 | 22.8 | | 23.1 |

* calculations based on moisture and ash free basis

The average water content in the aspen-derived product bio-oil was about 20 wt % whereas it was only 11 wt % in that from SPF (analyses at Ensyn and elsewhere). The unrecovered portion in the material balance is assumed to reflect losses of some of the moisture along with volatile organic components.

For successful PSU operation a start-up liquid (lignin free bio-oil) was used for internal condensing of the product bio-oil from the product vapor. This ensured rapid cooling of product vapor, recovery and preservation of reactive chemical compounds. Although the start-up liquid enabled smooth operation of the PSU, it can reduce recovery efficiency, thus lowering the overall MNRP yield.

**Table 6 – Feedstock, operating and product data for PSU runs**

| Property and Run Parameters | | Run 269 | Run 270 | Run 271 |
|---|---|---|---|---|
| Feedstock Bark | | Aspen | Aspen | Aspen |
| Bulk Density @ 25°C | g/mL | 0.398 | 0.398 | 0.339 |
| Particle Mesh Size | | -1/4 +50 | -1/4 +50 | -1/4 +50 |
| Moisture Content | wt % | 4.8 | 4.87 | 2.9 |
| Ash Content | wt % | 4.67 | 4.67 | 3.61 |
| HHV | MJ/kg | 20.2 | 20.2 | |
| Lignin (Klason) Content | wt % | 45.1 | 45.1 | 43.7 |
| Cellulose | wt % | 24.3 | 24.3 | 20.8 |
| Hemicellulose | wt % | 18.0 | 18.0 | 18.4 |
| Wood Extractives (resin) | wt % | 8.7 | 8.7 | 3.6 |
| Carbohydrates | wt % | 42.3 | 42.3 | 39.2 |
| Amount fed | kg | 500.4 | 694.5 | 1501.8 |
| Run Temperature | °C | 600 | 533 | 529 |
| Run Time | min | 717 | 785 | 1938 |
| Liquid (bio-oil) Yield (maf)* | wt % | 42.7 | 61.5 | 51.0 |
| Gas Yield (maf)* | wt % | 19.7 | 13.9 | 12.7 |
| Char Yield (maf)* | wt % | 20.9 | 23.6 | 24.9 |
| Total Recovered (maf)* | wt % | 83.3 | 98.9 | 88.6 |
| MNRP yield (maf)* | wt % | 11.6 | 14.1 | 15.4 |

* calculations based on moisture and ash free basis

### Resin characterizations

The work performed at the National Renewable Energy Laboratory (NREL) for this project had four components,

- analysis of MNRP samples produced by Ensyn
- analysis of the effects of temperature on composition of bark pyrolysis products
- analysis of the effects of temperature on composition of mill and forest residue pyrolysis products
- identifying tools for process monitoring of the biomass feedstocks

Samples of Aspen bark-derived bio-oil at high temperature (615ºC) and low temperature (530ºC) analyzed by GPC showed that the high temperature samples contained a larger number of low MW species, a characteristic expected to be favorable to economical resin formulations. The GPC analyses of these two oils are shown in Figure 2.

All of the samples supplied by Ensyn were subjected to chemical analyses with Gas Chromatography-Mass Spectroscopy (GC-MS), Nuclear Magnetic Spectroscopy (NMR) and Gel Permeation Chromatography (GPC). In combination these tools provided information about the number of reactive sites on the average phenolic ring and the number of non-branching phenolic rings, e.g., difunctional and monofunctional molecules.

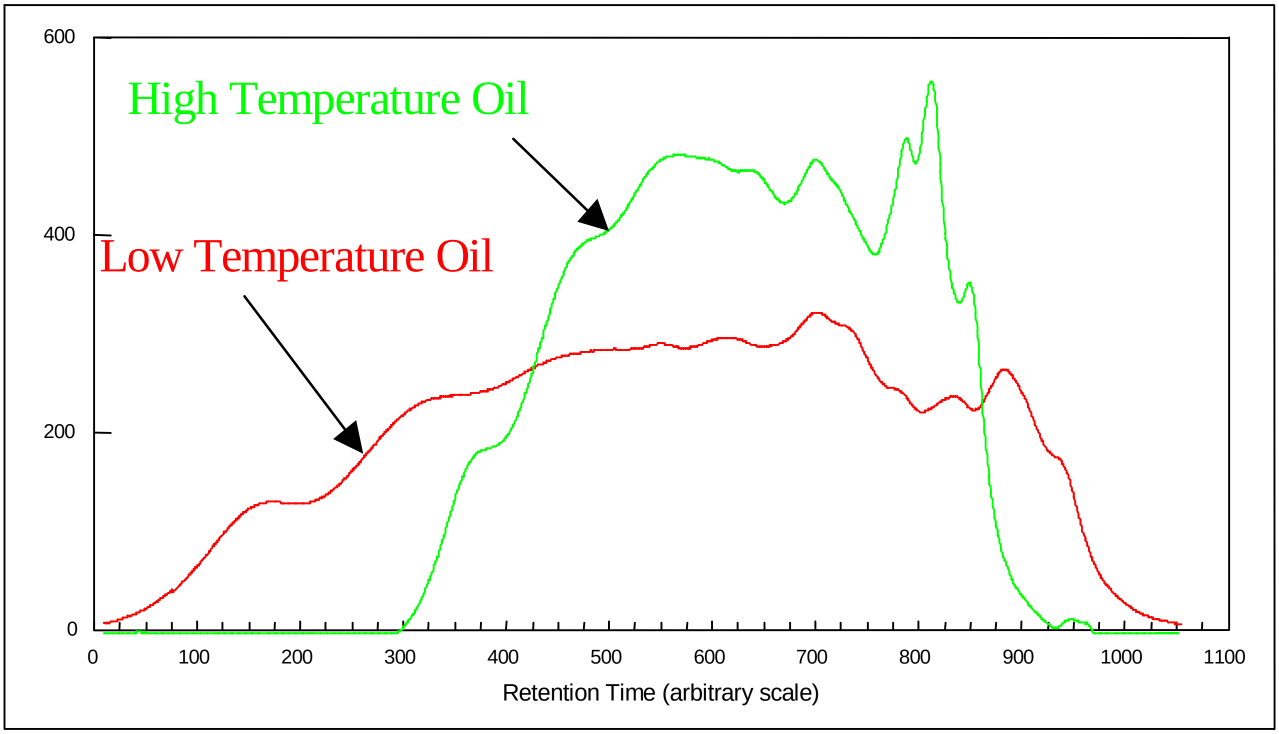



**Figure 3 – Molecular weight distribution by GPC**

A detailed report by NREL is appended (Appendix B). The portion of the NREL report pertinent to the Phase I can be summarized as follows:

- There was a clear trend towards increasing functionality with increasing pyrolysis temperature.
- The molecular weight distribution of the processed MNRP was relatively insensitive to the bark origin and pyrolysis temperature.
- The softwood barks produced a relatively high concentration of trifunctional and difunctional phenolics, which was expected given their moderate levels of tannins and polyphenolic extractives.

The three bark sources, Aspen, SYP and SPF were all subjected to pyrolysis at a series of different temperatures and the pyrolysis products were monitored with molecular beam mass spectrometry (MBMS) and modeled (to apply to RTP bio-oil) using Projection to Latent Structures (PLS).

- In the pyrolysis products, an increase in the relative concentration of phenol, catechol and guaiacol and a loss in the relative concentration of lignin dimers as the temperature increased was consistent across all three bark sources.
- The effects on the pyrolysis products caused by the different sources of bark were much greater than the effects of temperature.

Resin and other quality tests

MNRP-containing NR/PF resin formulations were developed at Dynea and GP using their proprietary procedures. L-P manufactured sample OSB boards to test the adhesive quality of the formulations. Figure 3 exemplifies this development on testing the internal bond strength of MNRP-containing NR/PF from Aspen bark produced by RTP at high temperature.

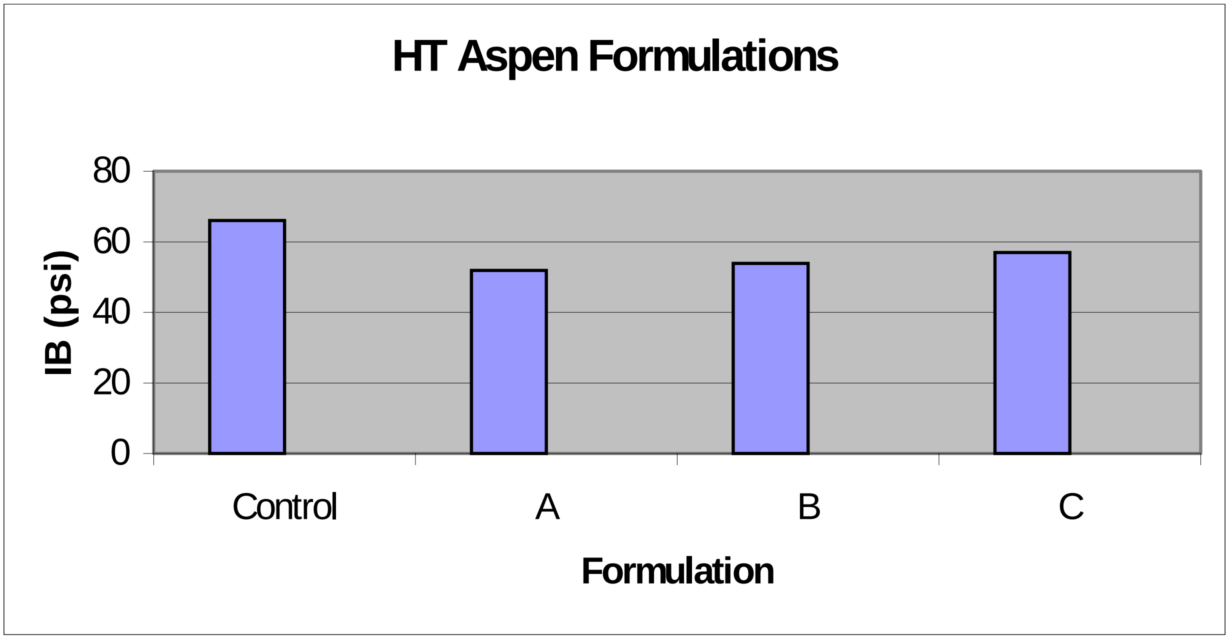



**Figure 4 – Preliminary testing of NR/PF formulations**

From SPF and Aspen bark-derived MNRP ACM (ACM Wood Chemicals Group) prepared prototype NR resin formulations and L-P manufactured sample boards for quality tests. The results of these tests are detailed in Appendix C. The results were used in support of decisions and further plans as stated previously.

GP initiated PF resin formulation with SYP bark derived pyrolysis oil. The initial formulation effort resulted in an unstable resin. High carbohydrate and wax content in the bark-derived pyrolysis oil were identified as the cause for resin instability. Dynea evaluated numerous samples of bark-derived MNRP in resin formulations, many of which were found unsuitable due to problems associated with insolubles and/or thixotropy. Adjustments were made to the Ensyn process to eliminate these problems and the final sample of MNRP was found suitable to proceed with the planned mill trial. The MNRP for the trial resin production was received but the trial had to be cancelled as a result of the L-P reorganization and downsizing. In 2001, Dynea and GP issued reports on their resin formulation tests and L-P supplemented these reports in a document with comparative summaries and graphs. The summary is shown in Appendix D. Although some quality parameters of the formulations were slightly inferior to the control PF resin, the general acceptability of the parameters was very encouraging in terms of potential commercial use.

The trend to a more reactive MNRP with increasing pyrolysis temperature is beneficial in terms of the product chemical composition but is offset by decreasing the overall yield of the MNRP.

Furthermore, resin manufacturers reported a thixotropic phenomenon that was only present in the SPF derived NR product. Based on the BSU experience and NREL results L-P and the rest of the participants selected Aspen bark for the pilot plant runs and large-scale batches at a conversion temperature of approximately 530°C.

L-P contracted an environmental testing firm in June 2001 to evaluate volatile organic compounds (VOC) and particulate matter (PM) emissions during the bark drying phase of aspen and southern yellow pine bark (SYP) processing. The drying, preparation and testing took place at L-P's Two Harbors research facility using a 500 lb/h rated single pass dryer. Collection of samples took place over three days at durations of eight hours steady state operations. Results indicate emission abatement controls would be required in drying processes for both the aspen and SYP bark.

An analysis of the VOC emission data is presented in Table 7 that takes into consideration wood use on a production basis of a million square feet (MSF) for nominal 3/8" thickness OSB. Typical hardwood OSB mills use 1.5 tons of green wood for every MSF produced (slightly higher for softwood mills). Table 7 incorporates calculated emission factors from the bark drying test and historical data from OSB industry environmental testing.

**Table 7 – Dryer emission test results**

| Wood Species | Production Wood Usage (Ton green wood/ MSF 3/8") | Emission Factor (Lbs. VOC/ OD Ton) | VOC Generation (Lbs./ MSF 3/8") | | |
|---|---|---|---|---|---|
| | | | Total | Cumulative | % Increase |
| Aspen | 1.50 | 3.55 | 2.56 | 2.95 | 16% |
| Bark | 0.38 | 2.30 | 0.40 | | |
| SYP | 1.60 | 7.87 | 6.04 | 6.54 | 8% |
| Bark | 0.40 | 2.70 | 0.50 | | |

* Test dryer operating conditions: Inlet/Outlet Temperature 550-600/250ºF and 4 % final bark moisture

Particulate matter testing followed EPA Methods 5 and 202 for condensable materials. On a production basis an Aspen and SYP bark drying process would produce 0.38 and 0.71 pounds of PM per MSF (3/8"), respectively. Of the total PM for each bark species, 62% (Aspen) and 31% (SYP) came from condensable materials. The differences in results of the two species could be contributed to a number of factors including wood-fiber structural difference or pre-processing prior to drying. In this trial the SYP bark had been previously processed with a hammer mill whereas the Aspen came straight from de-barking equipment. Both PM and VOC environmental controls would be required at the stated emission levels.

### Summary and conclusions reached at the completion of Phase I

NR samples were successfully produced in Ensyn's RTP™ process from three representative bark materials; Southern Yellow Pine (SYP), Spruce/Pine/Fir (SPF) and Aspen. These materials were readily processed in the bench-scale unit (BSU) and high quality natural resin (MNRP) samples were produced.

MNRP was made following all the required conditions with the three species of bark feedstock. Processing runs followed Standard Operating Procedures to obtain reliable mass balance figures. Both BSU and PSU systems worked smoothly in converting bark to obtain bio-oil. The MNRP product was then separated out of the bio-oil. Samples were prepared for characterizations performed at NREL. Structural differences found in the various samples were linked to both the original source and the processing conditions. NR/PF formulations, with MNRP substituting up to 50 % phenol component, were generated at Dynea and GP. In one case resin manufacturers reported a thixotropic phenomenon that was only present in the SPF derived MNRP product. Selected NR formulations were then applied in manufacturing prototype OSB panels that were subsequently subjected to a series of acceptability tests. The tests evaluated adhesive quality of the NR/PF formulations. Both L-P and resin manufacturers were encouraged by the results indicating that this commercial utilization of bark residues is technically feasible.

Decisions taken by the group from this segment of the research program provided the directives for the next phase. L-P decided that the next phase would concentrate on the Aspen bark feedstock. This conclusion was reached by considering the work and recommendations delivered by all parties. Aspen bark feedstock processed at 530°C was chosen for large batch production based on the economics, excess material, and MNRP quality and yields.

### Large-scale Aspen bark-derived MNRP batch and supplementary small samples

To fulfill the requirements of this task a 26 tpd commercial RTP™ processing plant located in Manitowoc was selected.

A large quantity of dried and sized Aspen bark was delivered to the facility and processed in September 2001. Product liquids were collected while byproduct char and gas were burned in the facility byproduct fuel burners. Product liquids were shipped to Ensyn's research facility in Greely, Ontario and further processed to produce more than 500 lbs of MNRP for commercial testing. As explained in the introductory part of this section the large batch of MNRP has not been further utilized.

In the same period of time (September-October 2001) Ensyn completed two supplementary BSU runs to supply two samples of 150 g MNRP to GP. One sample was derived from SYP bark (Run 51A at 520°C) and the other from Aspen bark (Run 52A at 520°C).

## Phase II – Large-Scale Work, Additional Bench-Scale Products and Activated Char

### Production and application of large-scale batches of MNRP

As outlined previously, the large batches of MNRP were produced processing mill residue at the Rhinelander RTP™ plant. These production runs took place in 2002 and 2003, producing more than one ton of MNRP for mill trials. The final "green" formulation (MNRP at 40% phenol substitution in a PF resin) was subsequently produced and applied to OSB panel manufacture at ARC Resins/Tembec. Three full mill trials were conducted in 2002 and 2003 producing approximately 50,000 OSB boards. Tembec reported no operational difficulties producing the MNRP-based resin in their industrial kettles or using it commercially in the OSB mill operations. Tembec sent the appropriate number of commercial panels to TECO for certification. TECO subsequently confirmed that the MNRP panels had passed certification.

Plant data associated with the large-scale MNRP production are appended (Appendix A6), and summarized in Table 8.

**Table 8 - Feedstock, operating and product data for the large-scale production**

| Property and Run Parameters | | Rhinelander |
|---|---|---|
| Whitewood (Oak, Maple, Ash) Saw Dust | | -1/4 Mesh |
| Moisture Content | wt % | 5 – 6 |
| Ash Content | wt % | 0.5 |
| Feed Rate (total) | lb/hr | 2900 |
| Feed Rate (organics) | lb/hr | 2741 |
| Liquid (bio-oil) Yield (maf)* | wt % | 72 |
| Organics in Liquid | wt % | 86 |
| Water in Liquid | wt % | 14 |
| MNRP Yield (maf)* | wt % | 20 |
| Separated Liquid Yield (maf)* | wt % | 52 |
| Organics in Sep. Liquid | wt % | 80 |
| Water in Separated Liquid | wt % | 20 |
| Gas Yield (maf)* | wt % | 14 |
| Char Yield (maf)* | wt % | 14 |

* calculations based on moisture and ash free basis

The available OSB board quality test data as reported by Tembec can be found in Appendix F. The panel quality parameters for the MNRP-containing boards are shown comparatively with those using only the standard PF resin before and after the MNRP-containing panels were produced. The comparative results indicate statistically significant deviations for some parameters (the two reference boards inclusive), however, the MNRP-containing boards exceeded all quality parameters tested. The first commercial application of wood biomass to the production of wood adhesives was thus successfully completed. There were no problems with the OSB panel manufacture and “Green” NR formulations containing the MNRP were found suitable for the purpose. Sample boards are available for exhibition/demonstration purposes.

Additional bench-scale runs and evaluations of the relevant MNRP products

This work involved MNRP generation and OSB application of the “forest residue” and “planar shavings” materials supplied by Weyerhaeuser in 2003. Ensyn carried out further bench-scale runs to produce MNRP from the two samples. The product MNRP was sent to Dynea where three batches of resin formulations were generated. One was a standard “control” PF resin for comparison purposes whereas the two other resins contained MNRP (as 50 wt % phenol substitute) produced by Ensyn from the two residues supplied by Weyerhaeuser. These resins were then given to Forintek Canada Corp. where OSB boards were manufactured and their evaluations performed.

The same bench-scale unit (BSU) as that involved in Phase I was used to produce the necessary quality of MNRP. The pertinent run and analytical information is incorporated in the Appendix A3 and selected data are shown in Table 9.

**Table 9 - Feedstock, operating and product data for Weyerhaeuser forest and mill residues**

| Property and Run Parameters | | Run 84A | Run 85A | Run 86A | Run 87A |
|---|---|---|---|---|---|
| Feedstock Forest or Mill Residue | | Forest | Mill | Forest | Mill |
| Particle Mesh Size | | -8+325 | -8+325 | -8+325 | -8+325 |
| Moisture Content | wt % | 6.1 | 5.8 | 6.1 | 5.8 |
| Ash Content | wt % | 2.5 | 0.3 | 2.5 | 0.3 |
| Run Temperature | °C | 520 | 520 | 520 | 520 |
| Liquid (bio-oil) Yield (maf)* | wt % | 61.1 | 70.9 | 65.0 | 70.8 |
| Gas Yield (maf)* | wt % | 12.7 | 14.9 | 14.8 | 16.6 |
| Char Yield (maf)* | wt % | 10.9 | 9.2 | 10.8 | 7.8 |
| Total Recovered (maf)* | wt % | 86.5 | 97.2 | 92.4 | 97.7 |
| MNRP yield (maf)* | wt % | 19.3 | 20.5 | | |

* calculations based on moisture and ash free basis

The OSB test results (Forintek – Project 4139-1) are reported in Appendix G. The objective was to investigate the bonding quality of the two MNRP-containing resins in comparison with a Dynea control PF resin. For this purpose twelve (12) OSB panels were generated as detailed in Appendix G, by applying four different press times to the three resin formulations in the OSB panels. All OSB panels were manufactured using 3 wt % resin solids add-on rate. Tests included Board Density, Internal Bond Strength (IB), Modulus of Rupture (MOR) and Elasticity (MOE), Thickness Swell and Water Absorption after 24 hour water soak, and Flexure tests after 2 hour water boil. The variation in these quality parameters related to the control and MNRP-containing resins is shown in Table 10.

**Table 10 – Ranges for quality parameters**

| Resin | Density, lb/cu.ft. | IB, psi | MOR, psi | MOE, kpsi | Thickness Swell after 24 h boil, % | Water Uptake after 24 h boil, % | MOR after 2 h boil, psi | MOE after 2 h boil, kpsi |
|---|---|---|---|---|---|---|---|---|
| Control | 39.4-46.0 | 37-55 | 4400-4500 | 640-700 | 15-18 | 33-36 | 650-1020 | 87-111 |
| NR-1 * | 41.7-43.5 | 41-53 | 3700-4200 | 630-710 | 14-17 | 34-39 | 390-630 | 55-82 |
| NR-2** | 41.1-44.1 | 41-47 | 3700-5000 | 610-730 | 15-20 | 33-36 | 610-760 | 70-90 |

* Resin containing MNRP from planar shavings
**Resin containing MNRP from forest residue

The comparison and range of levels indicate that in spite of variations reported (see Appendix G), similar adhesive quality was reached with each resin sample. It thus can be further assumed that a natural resin formulation (NR) of acceptable quality can be reached producing MNRP from either of the Weyerhaeuser residues processed.

During Phase II, the project team decided to address an important marketing issue that could have a potential effect on market acceptance of MNRP panels. MNRP resins do not exhibit the “phenolic” odor which is typical of standard PF resins. Instead they have a very slight aroma which is best associated with that of a BBQ grill. The smoky components in the MNRP resins, however, do react and effectively disappear during polymerization, when MNRP is formulated into a PF resin that is cured in the panel matrix During the course of Phase II, Weyerhaeuser personnel highlighted the importance of verifying that there would be no discernable difference between the sensory perception of control PF resin panels and MNRP resin panels. This issue was raised since other non-related work, performed several years previously and using “raw” volatile pyrolytic liquids in PF resin formulations, had indicated the possibility of an undesirable odor in the final panel products.

Ensyn and its OSB/plywood colleagues had never experienced an undesirable odor in MNRP panels, since MNRP is isolated as a solid product from the raw pyrolytic liquid, and the volatiles are (by definition) removed in the MNRP recovery process. Furthermore, as stated above, any minor amounts of residual volatiles react and disappear during curing. Nevertheless, the project team decided that it would be best to prove this out using a sensory test protocol. Sensory tests were conducted on MNRP OSB boards at Weyerhaeuser (Washington State) and the Department of Energy (Canmet, Ottawa). In all cases, there was no statistical difference between the control and MNRP panels, and there was no negative sensory perception associated with the MNRP panels. In fact, the Weyerhaeuser tests would seem to indicate a slightly favorable assessment of the aroma of the MNRP boards. The results of the Weyerhaeuser tests are given in Appendix H.

### Activated char

Char byproduct can be readily used as a fuel with a typical HHV value of 23 MJ/kg. Alternatively, this char could be utilized more cost effectively by conversion to activated char (carbon). Ensyn’s bench-scale activated carbon unit and work conducted on the activation of char using the raw pyrolysis char byproduct obtained from wood residues is reported in Appendix A8. About 40 wt % of activated char with the adsorption value of 700-900 mg/g (Iodine Number analysis) can be produced from the pyrolytic char. Activated carbon of higher adsorption value can be reached at lower yields.

### Bark-derived MNRP application in comparison with that from Weyerhaeuser residues

During Phase II NREL was engaged in testing Weyerhaeuser “forest residue” and “planar shavings” samples. Details can be found in Appendix B. Using py-MBMS and NIR spectroscopy NREL researched the chemical composition of the pyrolysis vapors derived from bark and the other materials. This research indicated significant differences between bark and the other residues. However, as resin represents only a part of the product vapor, it is important to narrow the comparison down to the MNRP.

All information available to Ensyn at the time of compilation of this report indicates that the MNRP produced from bark offers adequate properties to be utilized for its intended application. The main difference in the quality of the bark-derived MNRP is its high wax content. The wax content is associated with the wax-producing chemical environment within the original bark, which is always more abundant in bark than in related wood. Part of the wax is transferred via pyrolysis and separation steps to the MNRP. When applied using established methods to produce panels the wax contained in this bark-derived MNRP becomes detrimental to the final adhesive bonding strength. The forest and mill residues that had only some bark content in them provided MNRP of acceptable OSB application results.

Feedback from the resin companies utilizing the southern yellow pine bark based MNRP in particular, indicated that a thixotropic effect was observed during testing. Upon further review of the MNRP it was noted that a waxy component from the bark extractives remained in the MNRP during normal post processing of the bio-oil. Through simple extractive techniques this waxy component was isolated from the bio-oil and no further thixotropic effects were observed in the finished resin formulations. Thus the RTP™ application to mainly bark-containing feedstock would benefit from separating the wax component from the bio-oil. The most efficient method to do this can be found through selection and testing. It is furthermore possible to speculate on the subsequent use of the wax such as in the panel manufacture itself.

# SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

In 2000 and 2001, Ensyn produced bio-oil and MNRP from bark of different tree species supplied by Louisiana Pacific Corp. Dynea and Georgia Pacific Resins successfully integrated the MNRP into PF resin formulations, and the resultant boards were tested and compared with PF control boards. Weyerhaeuser joined the project in 2001, and focused their interest on MNRP derived from mill and forest wood residues (i.e., sawdust, shavings, etc.). This MNRP was successfully incorporated into a commercial PF resin system by Tembec and used in three large-scale OSB mill trials that produced about 50,000 commercial OSB panels. Testing by Tembec, Forintek, Weyerhaeuser and TECO indicated that there was no significant difference between commercial MNRP resin formulations and commercial PF control resins.

During the “Phase I” L-P work, MNRP was extracted from bark-derived “bio-oil”, produced via Ensyn’s RTP™ process. This bio-oil was generated using bench-scale, pilot-scale and commercial-scale RTP™ plants. The bench and pilot scale runs explored the effect of processing conditions such as reactor temperature while the National Renewable Energy Laboratory studied chemistry and the qualitative changes in the relevant products. Prototype resin formulations were generated using blends of MNRP and phenol formaldehyde (PF) stocks by participating resin producers (Dynea and Georgia Pacific Resins Inc.). These were then used to bind sample Oriented Strand Board (OSB) panels to be tested for qualities such as strength and resistance to ambient effects. Much needed information was gathered by this work and many tests showed acceptable or near acceptable results. The program participants were encouraged by these results anticipating good technical feasibility of the intended bark utilization.

One problem associated with certain bark feedstocks was the high wax content, that was inevitably transferred to the MNRP formulation, and which had a negative influence on the final physical characteristics of the resin formulation. Not all bark materials contained significant quantities of wax, and this was never a problem with “white” wood residues. However, certain species were quite waxy, and in these cases, wax removal from the pyrolytic liquid prior to MNRP production is desirable. It is technically feasible to remove wax from the bio-oil simply as one of the refining steps in MNRP production. A smaller content of bark in candidate feedstocks (such as forest and mill residues) can be accommodated without wax separation.

The program focus was redirected from bark to forest and mill residues in 2001 when Louisiana Pacific experienced a significant reorganization and downsizing, and its key project personnel were re-assigned or laid off from the company. In 2002 and 2003, Tembec was brought into the project to conduct commercial mill trials using MNRP from these wood sources. As a result of these mill trials, wood-derived MNRP PF resins were certified by Tembec for commercial use. Ensyn produced a large batch of MNRP for the commercial mill run. Tests of OSB panels produced with the blend of MNRP-containing PF resin indicated that the parameters typical for the standard commercial PF resins can be reached and some even exceeded. Ensyn further produced MNRP from two other wood residues supplied by Weyerhaeuser and the PF resin formulations from these were used to produce and test OSB panels at Forintek. Consistent with the Tembec experience, the results showed commercially acceptable specifications for the sample boards.

To explore potential benefits of byproducts formed during RTP™ conversion of wood residues to bio-oil, Ensyn conducted a series of by-product char steam activation experiments. The work was completed in 2003. Results showed that a reasonable quality of activated char is obtainable to boost the economic viability of MNRP production.

The whole program was successfully completed in the mid 2003. Material balance accounts indicate that in excess of 20 wt% of the natural resin substitute (MNRP) is obtainable on the moisture and ash free basis depending on the origin of bark-containing wood residues. Blends of

MNRP and PF where MNRP can typically substitute 50 wt% phenol in PF, yield a resin formulation with acceptable bonding (adhesive) quality for OSB production. The economic benefits of substituting natural crude/gas derived components of PF with one derived from a renewable resource further enhance the environmental benefits of utilizing materials that are otherwise wasted. The combination of benefits suggests this approach as one of the future technological solutions in the wood processing industries where Ensyn actively pursues further business development.

The program was successful, as highlighted below:

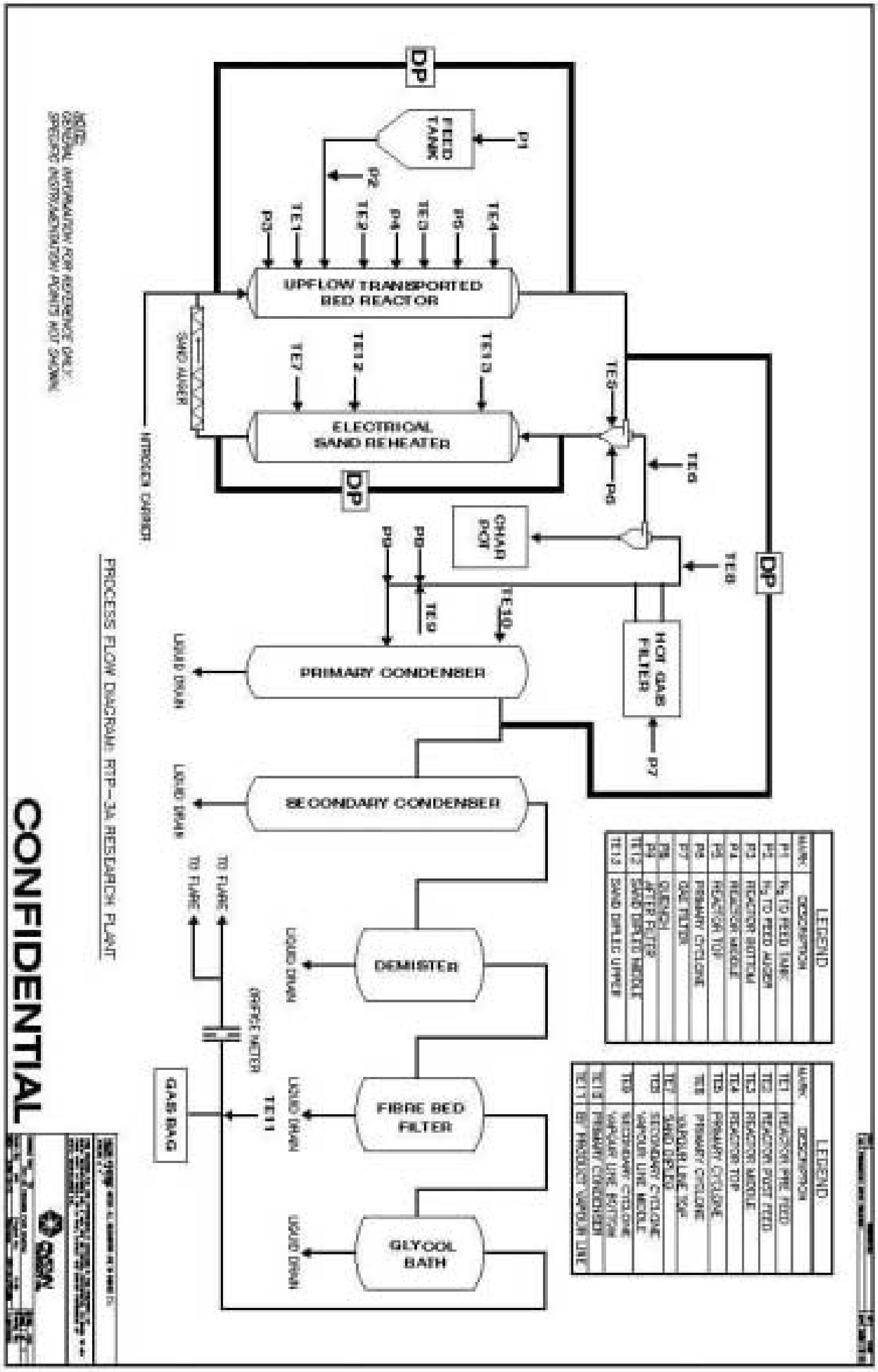
1. A commercial Natural Resin system, which displaced up to 50% phenol in PF resins, was developed by industrial resin producers
2. Successful mill trials were carried out using wood-derived MNRP at 40% phenol substitution – approximately 50,000 commercial OSB panels were manufactured using this Natural Resin system
3. The commercial MNRP OSB panels, with 40% phenol substitution in the PF resin, were granted industry's acceptance by TECO certification
4. The MNRP resin was granted environmentally-preferred "EcoLogo™" certification
5. The science of MNRP production, analyses and characterization was advanced and used to optimize MNRP production in commercial RTP™ systems
6. Carbon byproducts were characterized and optimized

There are several recommendations for future work:

1. Continue work with NREL and the resin manufactures to increase phenol substitution beyond 50%, with 100% phenol substitution as a final goal
2. Continue investigations on bark processing, to evaluate recovery techniques and commercial application for the waxy byproducts, and to optimize bark-derived MNRP formulations
3. Conduct full mill trials with bark-derived MNRP resin formulations (note that such formulations were successfully developed in the lab by the resin producers, but full-scale mill trials using bark-derived MNRP were not conducted)
4. Develop the technology to produce, recover and isolate other value-added byproducts from RTP™ bio-oil, including polymers, co-polymers and cross-linkers. This would further enhance Bio-Refinery economics and provide for even broader utilization of bark and wood residues
5. Continue byproduct carbon development work to the point where commercial specs are met, and efficient, economic market acceptance is achieved

# APPENDIX A1

**Bench Scale Unit (RTP-3A) Configuration – Process Flow Diagram**


DP
FEED TANK
UPFLOW TRANSPORTED BED REACTOR
ELECTRICAL SAND REHEATER
SAND AUGER
CHAR POT
HOT GAS FILTER
PRIMARY CONDENSER
SECONDARY CONDENSER
DEMISTER
FIBRE BED FILTER
GLYCOL BATH
GAS BAG
LIQUID DRAIN
TO FLARE
LEGEND
PROCESS FLOW DIAGRAM: RTP–3A RESEARCH PLANT
CONFIDENTIAL

# APPENDIX A2

**Phase I, Initial Bench-Scale Production: Certificates of Analysis - Summaries**

A Sample Certificate of Analysis (shipped with MNRP samples to NREL)

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September 27, 2000

# CERTIFICATE OF ANALYSIS

Ship to: NREL
1617 Cole Rd.
Golden, CO
80401
USA
Attn: Stephen Kelley
Telephone: (303) 384-6123
Fax:
Email:

Content: MNRP-B(58)T
Batch #: S202240800EN
Ship: 162g
Ship Date: August 24, 2000
Analytical Request: 262

This sample was produced from the RTP3A run 2A using an Aspen feedstock @ 550°C.

| **Sample** | **Melting Point (°C)** | **Acetone / THF /DMSO Insolubles (wt %)** | **Ash Content (wt %)** | **Acid (weak) Content (dry wt %)** | **Acid Number (mg KOH/ g)** | **Bulk Density @ 25°C (g/mL)** |
|---|---|---|---|---|---|---|
| Spec. Range | (58±2) | <3.00 | <0.60 | <3.0 | New Parameter (spec not defined) | Typical 0.75 |
| MNRP-B(58)T | 57 | N/A | N/A | N/A | N/A | N/A |

**Sieve Analysis:** **(N/A)**
(U.S. Sieve #)

| +8 | -8+10 | -10+100 | -100+200 | -200 | Total |
|---|---|---|---|---|---|
| Zero Tolerance | (Max. 10 wt %) | (Min. 70 wt %) | (Max. 20 wt %) | (Max. 1 wt %) | |
| | | | | | 100 |

**Note: N/A stands for insufficient sample available.**

**Summary for Samples Shipped to NREL**

| Sample | Melting Point (°C) | Ash Content (wt %) | Run no. | Feedstock Bark | Run Reactor Temp. (°C) | Amount Shipped (kg) | Date Shipped | Batch no. |
|---|---|---|---|---|---|---|---|---|
| MNRP-S202 | 57 | - | 2A | Aspen | 550 | 0.162 | 08-24-00 | S202240800EN |
| MNRP-S203 | 57 | 1.02 | 7A | Aspen | 600 | 1 | 09-01-00 | S203010900EN |
| MNRP-S204 | 59 | 0.40 | 8A | SYP | 600 | 1 | 09-01-00 | S204010900EN |
| MNRP-S205 | 59 | 0.14 | 9A | SYP | 550 | 2 | 09-08-00 | S205090800EN |
| MNRP-S206 | 57 | 1.26 | 10A | Aspen | 500 | 1 | 09-13-00 | S206130900EN |
| MNRP-S206 | 58 | 0.43 | 11A | Aspen | 550 | 1 | 09-15-00 | S206140900EN |
| MNRP-S207 | 39 | 0.41 | 11A | Aspen | 550 | 1 | 09-15-00 | S207140900EN |
| MNRP-S208 | 56 | 0.94 | 11A | Aspen | 550 | 1 | 09-15-00 | S208140900EN |
| MNRP-S209 | 58 | - | 12A | SYP | 550 | 1 | 09-15-00 | S209150900EN |
| MNRP-S210 | 59 | 0.23 | 12A | SYP | 550 | 1 | 09-15-00 | S210150900EN |
| MNRP-S211 | 56 | 0.28 | 13A | SYP | 500 | 1 | 09-15-00 | S211150900EN |
| MNRP-S214 | 56 | 0.46 | 14A | SPF | 600 | 1 | 09-22-00 | S214220900EN |
| MNRP-S215 | 58 | 1.09 | 15A | SPF | 550 | 1 | 09-22-00 | S215220900EN |

# CERTIFICATE OF ANALYSIS

(Internal)

Content: MNRP-B(58) T 1010
Analytical Request: 310

**RTP3A Run #: 25A**

| **Sample** | **Melting Point (°C)** | **Acetone / THF / DMSO Insolubles (wt %)** | **Ash Content (wt %)** | **Acid (weak) Content (dry wt %)** | **Acid Number (mgKOH/g )** | **Bulk Density @ 25°C (g/mL)** |
|---|---|---|---|---|---|---|
| Spec. Range | (58±2) | <3.00 | <0.60 | <3.0 | New Parameter (Spec not defined) | Typical 0.75 |
| MNRP-B(58) T | 58 | 0.27 | 0.16 | 1.4 | 39 | 0.63 |

**Sieve Analysis:**
(U.S. Sieve #)

| +8 | -8+10 | -10+100 | -100+200 | -200 | Total |
|---|---|---|---|---|---|
| Zero Tolerance | (Max. 10 wt %) | (Min. 70 wt %) | (Max. 20 wt %) | (Max. 1 wt %) | |
| 0.0 | 9.7 | 87.8 | 2.3 | 0.2 | 100 |

MNRP Samples to NESTE Chemicals

December 7, 2000

# CERTIFICATE OF ANALYSIS

Ship to: Richard Adams
NESTE Chemicals
5865 McGaughlin Road
Unit 3
Mississauga, Ontario
L5R 1B8
Canada
Telephone: (905)712-0900 ext 283
Fax: (905)712-0902

Content: MNRP-B(58) T 1010
Batch #: 220171100EN(SPF)
Ship: 5 lbs
Ship Date: Nov. 17, 2000
Analytical Request: 310

| Sample | Melting Point (°C) | Acetone / THF / DMSO Insolubles (wt %) | Ash Content (wt %) | Acid (weak) Content (dry wt %) | Acid Number (mgKOH/g ) | Bulk Density @ 25°C (g/mL) |
|---|---|---|---|---|---|---|
| Spec. Range | (58±2) | <3.00 | <0.60 | <3.0 | New Parameter (Spec not defined) | Typical 0.75 |
| MNRP-B(58) T | 56 | 0.14 | 0.19 | 1.1 | 42 | 0.72 |

**Sieve Analysis:**
(U.S. Sieve #)

| +8 | -8+10 | -10+100 | -100+200 | -200 | Total |
|---|---|---|---|---|---|
| Zero Tolerance | (Max. 10 wt %) | (Min. 70 wt %) | (Max. 20 wt %) | (Max. 1 wt %) | |
| 0.0 | 9.7 | 87.8 | 2.3 | 0.2 | 100 |

January 9, 2001

# CERTIFICATE OF ANALYSIS

Ship to: Richard Adams
NESTE Chemicals

5865 McGaughlin Road
Unit 3
Mississauga, Ontario
L5R 1B8
Canada
Telephone: (905)712-0900 ext 283
Fax: (905)712-0902

Content: MNRP-B(58) T 1010
Batch #: S228221200EN
(Aspen)
Ship: 15g
Ship Date: December 22, 2000
Analytical Request: 337

| **Sample** | **Melting Point (°C)** | **Acetone / THF/ DMSO Insolubles (wt %)** | **Ash Content (wt %)** | **Acid (weak) Content (dry wt %)** | **Acid Number (mgKOH/g )** | **Bulk Density @ 25°C (g/mL)** | **Remassing (wt %)** |
|---|---|---|---|---|---|---|---|
| Spec. Range | (58±2) | <3.00 | <0.60 | <3.0 | New Parameter (Spec not defined) | Typical 0.75 | Ideal spec: 0 |
| MNRP-B(58) T | 56 | 0.33 | 0.78 | 0.3 | 12 | 0.70 | To follow |

**Sieve Analysis:**
(U.S. Sieve #)

| +8 | -8+10 | -10+100 | -100+200 | -200 | Total |
|---|---|---|---|---|---|
| Zero Tolerance | (Max. 10 wt %) | (Min. 70 wt %) | (Max. 20 wt %) | (Max. 1 wt %) | |
| 0.0 | 0.1 | 97.2 | 2.5 | 0.2 | 100 |

MNRP Samples to Georgia Pacific

December 7, 2000

# CERTIFICATE OF ANALYSIS

Ship to: World L.-S. Nieh
Georgia-Pacific Resins Inc.
2883 Miller Road
Decatur, GA
30035
USA
Telephone: (770) 593-6852
Fax: (770) 322-9973

Content: MNRP-B(58) T 1010
Batch #: S2222211000EN (SPF)
Ship: 5lbs
Ship Date: November 22, 2000
Analytical Request: 310

| SAMPLE | Melting Point (°C) | Acetone / THF Insolubles (wt %) | Ash Content (wt %) | Acid (weak) Content (dry wt %) | Acid Number (mgKOH/g ) | Bulk Density @ 25°C (g/mL) |
|---|---|---|---|---|---|---|
| Spec. Range | (58±2) | <3.00 | <0.60 | <3.0 | New Parameter (Spec not defined) | Typical 0.75 |
| MNRP-B(58) T | 56 | 0.14 | 0.19 | 1.1 | 42 | 0.72 |

**Sieve Analysis:**
(U.S. Sieve #)

| +8 | -8+10 | -10+100 | -100+200 | -200 | Total |
|---|---|---|---|---|---|
| Zero Tolerance | (Max. 10 wt %) | (Min. 70 wt %) | (Max. 20 wt %) | (Max. 1 wt %) | |
| 0.0 | 9.7 | 87.8 | 2.3 | 0.2 | 100 |

January 9, 2001

# CERTIFICATE OF ANALYSIS

Ship to: World L.-S. Nieh
Georgia-Pacific Resins Inc.

2883 Miller Road
Decatur, GA
30035
USA
Telephone: (770) 593-6852
Fax: (770) 322-9973

Content: MNRP-B(58) T 1010
Batch #: S2282212000EN
(Aspen)
Ship: 15g
Ship Date: December 22, 2000
Analytical Request: 337

| SAMPLE | Melting Point (°C) | Acetone / THF/DMSO Insolubles (wt %) | Ash Content (wt %) | Acid (weak) Content (dry wt %) | Acid Number (mgKOH/g ) | Bulk Density @ 25°C (g/mL) | Remassing (wt %) |
|---|---|---|---|---|---|---|---|
| Spec. Range | (58±2) | <3.00 | <0.60 | <3.0 | New Parameter (Spec not defined) | Typical 0.75 | Ideal Spec: 0 |
| MNRP-B(58) T | 56 | 0.33 | 0.78 | 0.3 | 12 | 0.70 | To follow |

**Sieve Analysis:**
(U.S. Sieve #)

| +8 | -8+10 | -10+100 | -100+200 | -200 | Total |
|---|---|---|---|---|---|
| Zero Tolerance | (Max. 10 wt %) | (Min. 70 wt %) | (Max. 20 wt %) | (Max. 1 wt %) | |
| 0.0 | 0.1 | 97.2 | 2.5 | 0.2 | 100 |

# APPENDIX A3

**Certificates of Analysis - Details**

Run 2A
Feedstock ECN #239 Aspen
Reactor Temperature 550°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.419 |
| Moisture Content | T208 om-94 | wt % | 10.2 |
| Ash Content | In-house | wt % | 5.55 |
| HHV | D240-92 | MJ/kg | 17.6 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.2 |
| U.S Sieve # -8+12 | In-house | wt % | 0.6 |
| U.S Sieve # -12+14 | In-house | wt % | 1.1 |
| U.S Sieve # -14+20 | In-house | wt % | 9.2 |
| U.S Sieve # -20+30 | In-house | wt % | 13.3 |
| U.S Sieve # -30+35 | In-house | wt % | 15.3 |
| U.S Sieve # -35+50 | In-house | wt % | 27.1 |
| U.S Sieve # -50 | In-house | wt % | 33.2 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 43.5 |
| Cellulose Content | T203 om-93 | wt % | 20.9 |
| Hemicellulose Content | T203 om-93 | wt % | 19.0 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 3.0 |
| Carbohydrates | T212 om-93 | wt % | 39.9 |

| Product Liquid (Whole Oil) | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | wt % | - |
| Acetone/THF Insoluble (solids) | In-house | wt % | 1.70 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.13** |
| Carbon dioxide | In-house GC | vol % | **1.99** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **1.99** |
| Methane | In-house GC | vol % | **0.58** |
| Ethane | In-house GC | vol % | **0.06** |
| Ethylene | In-house GC | vol % | **0.12** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.01** |
| Propylene | In-house GC | vol % | **0.06** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.01** |
| Isobutylene | In-house GC | vol % | **0.00** |
| T-2-Butene | In-house GC | vol % | **0.01** |
| C-2-Butene | In-house GC | vol % | **0.00** |
| 1,3-Butadiene | In-house GC | vol % | **0.01** |
| I-Pentane | In-house GC | vol % | **0.00** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **0.00** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.03** |
| Oxygen | In-house GC | vol % | **1.43** |
| Nitrogen | In-house GC | vol % | **92.19** |
| High Heating Value | Calculated | MJ/kg | |

Run 7A
Feedstock ECN #239 Aspen
Reactor Temperature 600°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.419 |
| Moisture Content | T208 om-94 | wt % | 10.2 |
| Ash Content | In-house | wt % | 5.55 |
| HHV | D240-92 | MJ/kg | 17.6 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.2 |
| U.S Sieve # -8+12 | In-house | wt % | 0.6 |
| U.S Sieve # -12+14 | In-house | wt % | 1.1 |
| U.S Sieve # -14+20 | In-house | wt % | 9.2 |
| U.S Sieve # -20+30 | In-house | wt % | 13.3 |
| U.S Sieve # -30+35 | In-house | wt % | 15.3 |
| U.S Sieve # -35+50 | In-house | wt % | 27.1 |
| U.S Sieve # -50 | In-house | wt % | 33.2 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **1.69** |
| Carbon dioxide | In-house GC | vol % | **4.15** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.01** |
| Carbon monoxide | In-house GC | vol % | **4.75** |
| Methane | In-house GC | vol % | **1.66** |
| Ethane | In-house GC | vol % | **0.20** |
| Ethylene | In-house GC | vol % | **0.30** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.03** |
| Propylene | In-house GC | vol % | **0.20** |
| I-Butane | In-house GC | vol % | **trace** |
| N-Butane | In-house GC | vol % | **0.01** |
| 1-Butene | In-house GC | vol % | **0.05** |
| Isobutylene | In-house GC | vol % | **0.02** |
| T-2-Butene | In-house GC | vol % | **0.02** |
| C-2-Butene | In-house GC | vol % | **0.01** |
| 1,3-Butadiene | In-house GC | vol % | **0.05** |
| I-Pentane | In-house GC | vol % | **trace** |
| N-Pentane | In-house GC | vol % | **0.01** |
| 3-ME-1-Butene | In-house GC | vol % | **0.01** |
| 1-Pentene | In-house GC | vol % | **0.02** |
| 2-ME-1-Butene | In-house GC | vol % | **0.01** |
| T-2-Pentene | In-house GC | vol % | **0.01** |
| C-2-Pentene | In-house GC | vol % | **0.01** |
| 2-ME-2-Butene | In-house GC | vol % | **0.01** |
| C6+ | In-house GC | vol % | **0.20** |
| Oxygen | In-house GC | vol % | **0.50** |
| Nitrogen | In-house GC | vol % | **85.33** |
| High Heating Value | Calculated | MJ/kg | |

Run 8A
Feedstock ECN #240 SYP
Reactor Temperature 600°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.392 |
| Moisture Content | T208 om-94 | wt % | 13.8 |
| Ash Content | In-house | wt % | 1.63 |
| HHV | D240-92 | MJ/kg | 17.5 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+12 | In-house | wt % | 1.0 |
| U.S Sieve # -12+14 | In-house | wt % | 1.3 |
| U.S Sieve # -14+20 | In-house | wt % | 16.3 |
| U.S Sieve # -20+30 | In-house | wt % | 14.3 |
| U.S Sieve # -30+35 | In-house | wt % | 15.3 |
| U.S Sieve # -35+50 | In-house | wt % | 25.9 |
| U.S Sieve # -50 | In-house | wt % | 25.9 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 50.2 |
| Cellulose Content | T203 om-93 | wt % | 21.2 |
| Hemicellulose Content | T203 om-93 | wt % | 13.7 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 2.1 |
| Carbohydrates | T212 om-93 | wt % | 34.9 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.92** |
| Carbon dioxide | In-house GC | vol % | **2.17** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.01** |
| Carbon monoxide | In-house GC | vol % | **5.37** |
| Methane | In-house GC | vol % | **1.08** |
| Ethane | In-house GC | vol % | **0.09** |
| Ethylene | In-house GC | vol % | **0.33** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.01** |
| Propylene | In-house GC | vol % | **0.13** |
| I-Butane | In-house GC | vol % | **0.02** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.02** |
| Isobutylene | In-house GC | vol % | **0.01** |
| T-2-Butene | In-house GC | vol % | **0.01** |
| C-2-Butene | In-house GC | vol % | **0.01** |
| 1,3-Butadiene | In-house GC | vol % | **0.03** |
| I-Pentane | In-house GC | vol % | **0.01** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **0.00** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.08** |
| Oxygen | In-house GC | vol % | **0.74** |
| Nitrogen | In-house GC | vol % | **87.26** |
| High Heating Value | Calculated | MJ/kg | |

Run 9A
Feedstock ECN #240 SYP
Reactor Temperature 550°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.392 |
| Moisture Content | T208 om-94 | wt % | 13.8 |
| Ash Content | In-house | wt % | 1.63 |
| HHV | D240-92 | MJ/kg | 17.5 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+12 | In-house | wt % | 1.0 |
| U.S Sieve # -12+14 | In-house | wt % | 1.3 |
| U.S Sieve # -14+20 | In-house | wt % | 16.3 |
| U.S Sieve # -20+30 | In-house | wt % | 14.3 |
| U.S Sieve # -30+35 | In-house | wt % | 15.3 |
| U.S Sieve # -35+50 | In-house | wt % | 25.9 |
| U.S Sieve # -50 | In-house | wt % | 25.9 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 50.2 |
| Cellulose Content | T203 om-93 | wt % | 21.2 |
| Hemicellulose Content | T203 om-93 | wt % | 13.7 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 2.1 |
| Carbohydrates | T212 om-93 | wt % | 34.9 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.41** |
| Carbon dioxide | In-house GC | vol % | **2.59** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **trace** |
| Carbon monoxide | In-house GC | vol % | **4.11** |
| Methane | In-house GC | vol % | **0.88** |
| Ethane | In-house GC | vol % | **0.07** |
| Ethylene | In-house GC | vol % | **0.16** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.02** |
| Propylene | In-house GC | vol % | **0.08** |
| I-Butane | In-house GC | vol % | **0.01** |
| N-Butane | In-house GC | vol % | **0.01** |
| 1-Butene | In-house GC | vol % | **0.02** |
| Isobutylene | In-house GC | vol % | **0.01** |
| T-2-Butene | In-house GC | vol % | **0.01** |
| C-2-Butene | In-house GC | vol % | **0.02** |
| 1,3-Butadiene | In-house GC | vol % | **0.03** |
| I-Pentane | In-house GC | vol % | **0.01** |
| N-Pentane | In-house GC | vol % | **0.02** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **0.00** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.05** |
| Oxygen | In-house GC | vol % | **2.08** |
| Nitrogen | In-house GC | vol % | **88.98** |
| High Heating Value | Calculated | MJ/kg | |

Run 10A
Feedstock ECN #239 Aspen
Reactor Temperature 500°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.419 |
| Moisture Content | T208 om-94 | wt % | 10.2 |
| Ash Content | In-house | wt % | 5.55 |
| HHV | D240-92 | MJ/kg | 17.6 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.2 |
| U.S Sieve # -8+12 | In-house | wt % | 0.6 |
| U.S Sieve # -12+14 | In-house | wt % | 1.1 |
| U.S Sieve # -14+20 | In-house | wt % | 9.2 |
| U.S Sieve # -20+30 | In-house | wt % | 13.3 |
| U.S Sieve # -30+35 | In-house | wt % | 15.3 |
| U.S Sieve # -35+50 | In-house | wt % | 27.1 |
| U.S Sieve # -50 | In-house | wt % | 33.2 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 43.5 |
| Cellulose Content | T203 om-93 | wt % | 20.9 |
| Hemicellulose Content | T203 om-93 | wt % | 19.0 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 3.0 |
| Carbohydrates | T212 om-93 | wt % | 39.9 |

| Secondary Cyclone Char | | | |
|---|---|---|---|
| **Mass Balance Analysis (2 weeks)** | | | **into cyclone** |
| Moisture | In-house | wt % | 2.87 |
| Ash | In-house | wt % | 27.61 |
| Carbon | D5373 | wt % | 56.11 |
| Hydrogen | D5373 | wt % | 2.50 |
| Nitrogen | D5373 | wt % | 0.41 |
| Sulfur | D4239C | wt % | 0.07 |
| High Heating Value (ash free) | D240-92 | MJ/kg | 25.0 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.04** |
| Carbon dioxide | In-house GC | vol % | **3.55** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **2.14** |
| Methane | In-house GC | vol % | **0.66** |
| Ethane | In-house GC | vol % | **0.08** |
| Ethylene | In-house GC | vol % | **0.07** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.02** |
| Propylene | In-house GC | vol % | **0.04** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.01** |
| Isobutylene | In-house GC | vol % | **trace** |
| T-2-Butene | In-house GC | vol % | **trace** |
| C-2-Butene | In-house GC | vol % | **trace** |
| 1,3-Butadiene | In-house GC | vol % | **0.01** |
| I-Pentane | In-house GC | vol % | **0.01** |
| N-Pentane | In-house GC | vol % | **trace** |
| 3-ME-1-Butene | In-house GC | vol % | **0.01** |
| 1-Pentene | In-house GC | vol % | **trace** |
| 2-ME-1-Butene | In-house GC | vol % | **0.01** |
| T-2-Pentene | In-house GC | vol % | **trace** |
| C-2-Pentene | In-house GC | vol % | **trace** |
| 2-ME-2-Butene | In-house GC | vol % | **0.01** |
| C6+ | In-house GC | vol % | **0.26** |
| Oxygen | In-house GC | vol % | **1.84** |
| Nitrogen | In-house GC | vol % | **89.21** |
| High Heating Value | Calculated | MJ/kg | |

Run 11A
Feedstock ECN #239 Aspen
Reactor Temperature 550°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.419 |
| Moisture Content | T208 om-94 | wt % | 10.2 |
| Ash Content | In-house | wt % | 5.55 |
| HHV | D240-92 | MJ/kg | 17.6 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.2 |
| U.S Sieve # -8+12 | In-house | wt % | 0.6 |
| U.S Sieve # -12+14 | In-house | wt % | 1.1 |
| U.S Sieve # -14+20 | In-house | wt % | 9.2 |
| U.S Sieve # -20+30 | In-house | wt % | 13.3 |
| U.S Sieve # -30+35 | In-house | wt % | 15.3 |
| U.S Sieve # -35+50 | In-house | wt % | 27.1 |
| U.S Sieve # -50 | In-house | wt % | 33.2 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 43.5 |
| Cellulose Content | T203 om-93 | wt % | 20.9 |
| Hemicellulose Content | T203 om-93 | wt % | 19.0 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 3.0 |
| Carbohydrates | T212 om-93 | wt % | 39.9 |

| Secondary Cyclone Char | | | |
|---|---|---|---|
| **Mass Balance Analysis (2 weeks)** | | | **into cyclone** |
| Moisture | In-house | wt % | 2.11 |
| Ash | In-house | wt % | 69.26 |
| Carbon | D5373 | wt % | 21.72 |
| Hydrogen | D5373 | wt % | 0.98 |
| Nitrogen | D5373 | wt % | 0.13 |
| Sulfur | D4239C | wt % | 0.06 |
| High Heating Value (ash free) | D240-92 | MJ/kg | 25.3 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.84** |
| Carbon dioxide | In-house GC | vol % | **3.70** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.02** |
| Carbon monoxide | In-house GC | vol % | **3.19** |
| Methane | In-house GC | vol % | **1.08** |
| Ethane | In-house GC | vol % | **0.12** |
| Ethylene | In-house GC | vol % | **0.19** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.03** |
| Propylene | In-house GC | vol % | **0.11** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.01** |
| 1-Butene | In-house GC | vol % | **0.03** |
| Isobutylene | In-house GC | vol % | **0.02** |
| T-2-Butene | In-house GC | vol % | **0.01** |
| C-2-Butene | In-house GC | vol % | **0.01** |
| 1,3-Butadiene | In-house GC | vol % | **0.02** |
| I-Pentane | In-house GC | vol % | **0.00** |
| N-Pentane | In-house GC | vol % | **0.01** |
| 3-ME-1-Butene | In-house GC | vol % | **0.01** |
| 1-Pentene | In-house GC | vol % | **0.01** |
| 2-ME-1-Butene | In-house GC | vol % | **0.01** |
| T-2-Pentene | In-house GC | vol % | **0.01** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.03** |
| C6+ | In-house GC | vol % | **0.15** |
| Oxygen | In-house GC | vol % | **0.38** |
| Nitrogen | In-house GC | vol % | **88.89** |
| High Heating Value | Calculated | MJ/kg | |

Run 12A
Feedstock ECN #240 SYP
Reactor Temperature 550°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.392 |
| Moisture Content | T208 om-94 | wt % | 13.5 |
| Ash Content | In-house | wt % | 1.63 |
| HHV | D240-92 | MJ/kg | 17.5 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+12 | In-house | wt % | 1.0 |
| U.S Sieve # -12+14 | In-house | wt % | 1.3 |
| U.S Sieve # -14+20 | In-house | wt % | 16.3 |
| U.S Sieve # -20+30 | In-house | wt % | 14.3 |
| U.S Sieve # -30+35 | In-house | wt % | 15.3 |
| U.S Sieve # -35+50 | In-house | wt % | 25.9 |
| U.S Sieve # -50 | In-house | wt % | 25.9 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 50.2 |
| Cellulose Content | T203 om-93 | wt % | 21.2 |
| Hemicellulose Content | T203 om-93 | wt % | 13.7 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 2.1 |
| Carbohydrates | T212 om-93 | wt % | 34.9 |

| Secondary Cyclone Char | | | |
|---|---|---|---|
| **Mass Balance Analysis (2 weeks)** | | | **into cyclone** |
| Moisture | In-house | wt % | 1.84 |
| Ash | In-house | wt % | 52.83 |
| Carbon | D5373 | wt % | 32.10 |
| Hydrogen | D5373 | wt % | 1.40 |
| Nitrogen | D5373 | wt % | 0.18 |
| Sulfur | D4239C | wt % | 0.14 |
| High Heating Value (ash free) | D240-92 | MJ/kg | 24.2 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.48** |
| Carbon dioxide | In-house GC | vol % | **1.84** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **3.37** |
| Methane | In-house GC | vol % | **0.75** |
| Ethane | In-house GC | vol % | **0.06** |
| Ethylene | In-house GC | vol % | **0.16** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.01** |
| Propylene | In-house GC | vol % | **0.07** |
| I-Butane | In-house GC | vol % | **trace** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.01** |
| Isobutylene | In-house GC | vol % | **trace** |
| T-2-Butene | In-house GC | vol % | **trace** |
| C-2-Butene | In-house GC | vol % | **trace** |
| 1,3-Butadiene | In-house GC | vol % | **0.01** |
| I-Pentane | In-house GC | vol % | **trace** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **trace** |
| 1-Pentene | In-house GC | vol % | **trace** |
| 2-ME-1-Butene | In-house GC | vol % | **trace** |
| T-2-Pentene | In-house GC | vol % | **trace** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **trace** |
| C6+ | In-house GC | vol % | **0.07** |
| Oxygen | In-house GC | vol % | **0.86** |
| Nitrogen | In-house GC | vol % | **90.53** |
| High Heating Value | Calculated | MJ/kg | |

Run 13A
Feedstock ECN #240 SYP
Reactor Temperature 500°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.392 |
| Moisture Content | T208 om-94 | wt % | 13.5 |
| Ash Content | In-house | wt % | 1.63 |
| HHV | D240-92 | MJ/kg | 17.5 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+12 | In-house | wt % | 1.0 |
| U.S Sieve # -12+14 | In-house | wt % | 1.3 |
| U.S Sieve # -14+20 | In-house | wt % | 16.3 |
| U.S Sieve # -20+30 | In-house | wt % | 14.3 |
| U.S Sieve # -30+35 | In-house | wt % | 15.3 |
| U.S Sieve # -35+50 | In-house | wt % | 25.9 |
| U.S Sieve # -50 | In-house | wt % | 25.9 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 50.2 |
| Cellulose Content | T203 om-93 | wt % | 21.2 |
| Hemicellulose Content | T203 om-93 | wt % | 13.7 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 2.1 |
| Carbohydrates | T212 om-93 | wt % | 34.9 |

| Product Liquid (Whole Oil) | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | wt % | 39.5 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.26** |
| Carbon dioxide | In-house GC | vol % | **2.87** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.01** |
| Carbon monoxide | In-house GC | vol % | **2.98** |
| Methane | In-house GC | vol % | **0.61** |
| Ethane | In-house GC | vol % | **0.05** |
| Ethylene | In-house GC | vol % | **0.07** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.01** |
| Propylene | In-house GC | vol % | **0.05** |
| I-Butane | In-house GC | vol % | **trace** |
| N-Butane | In-house GC | vol % | **trace** |
| 1-Butene | In-house GC | vol % | **0.01** |
| Isobutylene | In-house GC | vol % | **0.01** |
| T-2-Butene | In-house GC | vol % | **0.00** |
| C-2-Butene | In-house GC | vol % | **trace** |
| 1,3-Butadiene | In-house GC | vol % | **0.01** |
| I-Pentane | In-house GC | vol % | **trace** |
| N-Pentane | In-house GC | vol % | **trace** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.01** |
| 2-ME-1-Butene | In-house GC | vol % | **trace** |
| T-2-Pentene | In-house GC | vol % | **trace** |
| C-2-Pentene | In-house GC | vol % | **trace** |
| 2-ME-2-Butene | In-house GC | vol % | **0.01** |
| C6+ | In-house GC | vol % | **0.29** |
| Oxygen | In-house GC | vol % | **1.00** |
| Nitrogen | In-house GC | vol % | **90.59** |
| High Heating Value | Calculated | MJ/kg | |

| Secondary Cyclone Char | | | |
|---|---|---|---|
| **Mass Balance Analysis (2 weeks)** | | | **into cyclone** |
| Moisture | In-house | wt % | 1.82 |
| Ash | In-house | wt % | 19.14 |
| Carbon | D5373 | wt % | 65.47 |
| Hydrogen | D5373 | wt % | 2.94 |
| Nitrogen | D5373 | wt % | 0.35 |
| Sulfur | D4239C | wt % | 0.06 |
| High Heating Value (ash free) | D240-92 | MJ/kg | 25.4 |

Run 14A
Feedstock ECN #243 SPF
Reactor Temperature 600°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.220 |
| Moisture Content | T208 om-94 | wt % | 9.2 |
| Ash Content | In-house | wt % | 1.94 |
| HHV | D240-92 | MJ/kg | 18.9 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.3 |
| U.S Sieve # -8+12 | In-house | wt % | 2.9 |
| U.S Sieve # -12+14 | In-house | wt % | 7.8 |
| U.S Sieve # -14+20 | In-house | wt % | 23.5 |
| U.S Sieve # -20+30 | In-house | wt % | 9.1 |
| U.S Sieve # -30+35 | In-house | wt % | 7.2 |
| U.S Sieve # -35+50 | In-house | wt % | 21.5 |
| U.S Sieve # -50 | In-house | wt % | 27.7 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 47.6 |
| Cellulose Content | T203 om-93 | wt % | 21.9 |
| Hemicellulose Content | T203 om-93 | wt % | 13.5 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 10.5 |
| Carbohydrates | T212 om-93 | wt % | 35.4 |

| Product Liquid (Whole Oil) | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | wt % | 44.9 |
| Acetone/THF Insoluble (solids) | In-house | wt % | - |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.73** |
| Carbon dioxide | In-house GC | vol % | **2.21** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.01** |
| Carbon monoxide | In-house GC | vol % | **3.78** |
| Methane | In-house GC | vol % | **0.83** |
| Ethane | In-house GC | vol % | **0.10** |
| Ethylene | In-house GC | vol % | **0.32** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.02** |
| Propylene | In-house GC | vol % | **0.16** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **trace** |
| 1-Butene | In-house GC | vol % | **0.04** |
| Isobutylene | In-house GC | vol % | **0.02** |
| T-2-Butene | In-house GC | vol % | **0.01** |
| C-2-Butene | In-house GC | vol % | **0.01** |
| 1,3-Butadiene | In-house GC | vol % | **0.04** |
| I-Pentane | In-house GC | vol % | **0.01** |
| N-Pentane | In-house GC | vol % | **trace** |
| 3-ME-1-Butene | In-house GC | vol % | **0.01** |
| 1-Pentene | In-house GC | vol % | **0.02** |
| 2-ME-1-Butene | In-house GC | vol % | **0.01** |
| T-2-Pentene | In-house GC | vol % | **0.01** |
| C-2-Pentene | In-house GC | vol % | **trace** |
| 2-ME-2-Butene | In-house GC | vol % | **0.01** |
| C6+ | In-house GC | vol % | **0.27** |
| Oxygen | In-house GC | vol % | **1.23** |
| Nitrogen | In-house GC | vol % | **89.13** |
| High Heating Value | Calculated | MJ/kg | |

| Secondary Cyclone Char | | | |
|---|---|---|---|
| **Mass Balance Analysis (2 weeks)** | | | **into cyclone** |
| Moisture | In-house | wt % | 1.45 |
| Ash | In-house | wt % | 21.81 |
| Carbon | D5373 | wt % | 66.08 |
| Hydrogen | D5373 | wt % | 2.34 |
| Nitrogen | D5373 | wt % | 0.40 |
| Sulfur | D4239C | wt % | 0.05 |
| High Heating Value (ash free) | D240-92 | MJ/kg | 22.5 |

Run 15A
Feedstock ECN #243 SPF
Reactor Temperature 550°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.220 |
| Moisture Content | T208 om-94 | wt % | 9.2 |
| Ash Content | In-house | wt % | 1.94 |
| HHV | D240-92 | MJ/kg | 18.9 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 0.3 |
| U.S Sieve # -8+12 | In-house | wt % | 2.9 |
| U.S Sieve # -12+14 | In-house | wt % | 7.8 |
| U.S Sieve # -14+20 | In-house | wt % | 23.5 |
| U.S Sieve # -20+30 | In-house | wt % | 9.1 |
| U.S Sieve # -30+35 | In-house | wt % | 7.2 |
| U.S Sieve # -35+50 | In-house | wt % | 21.5 |
| U.S Sieve # -50 | In-house | wt % | 27.7 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 47.6 |
| Cellulose Content | T203 om-93 | wt % | 21.9 |
| Hemicellulose Content | T203 om-93 | wt % | 13.5 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 10.5 |
| Carbohydrates | T212 om-93 | wt % | 35.4 |

| Product Liquid (Whole Oil) | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | wt % | 45.5 |
| Acetone/THF Insoluble (solids) | In-house | wt % | - |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.19** |
| Carbon dioxide | In-house GC | vol % | **1.57** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **1.86** |
| Methane | In-house GC | vol % | **0.36** |
| Ethane | In-house GC | vol % | **0.05** |
| Ethylene | In-house GC | vol % | **0.12** |
| Acetylene | In-house GC | vol % | **trace** |
| Propane | In-house GC | vol % | **0.02** |
| Propylene | In-house GC | vol % | **0.07** |
| I-Butane | In-house GC | vol % | **trace** |
| N-Butane | In-house GC | vol % | **0.01** |
| 1-Butene | In-house GC | vol % | **0.03** |
| Isobutylene | In-house GC | vol % | **0.02** |
| T-2-Butene | In-house GC | vol % | **0.01** |
| C-2-Butene | In-house GC | vol % | **trace** |
| 1,3-Butadiene | In-house GC | vol % | **0.01** |
| I-Pentane | In-house GC | vol % | **trace** |
| N-Pentane | In-house GC | vol % | **trace** |
| 3-ME-1-Butene | In-house GC | vol % | **trace** |
| 1-Pentene | In-house GC | vol % | **0.02** |
| 2-ME-1-Butene | In-house GC | vol % | **trace** |
| T-2-Pentene | In-house GC | vol % | **trace** |
| C-2-Pentene | In-house GC | vol % | **trace** |
| 2-ME-2-Butene | In-house GC | vol % | **0.01** |
| C6+ | In-house GC | vol % | **0.15** |
| Oxygen | In-house GC | vol % | **2.70** |
| Nitrogen | In-house GC | vol % | **90.04** |
| High Heating Value | Calculated | MJ/kg | |

| Secondary Cyclone Char | | | |
|---|---|---|---|
| **Mass Balance Analysis (2 weeks)** | | | **into cyclone** |
| Moisture | In-house | wt % | 1.28 |
| Ash | In-house | wt % | 48.87 |
| Carbon | D5373 | wt % | 50.79 |
| Hydrogen | D5373 | wt % | 2.02 |
| Nitrogen | D5373 | wt % | 0.34 |
| Sulfur | D4239C | wt % | 0.10 |
| High Heating Value (ash free) | D240-92 | MJ/kg | 20.4 |

Run 34A
Feedstock ECN #239 Aspen
Reactor Temperature 612°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.306 |
| Moisture Content | T208 om-94 | wt % | 5.8 |
| Ash Content | In-house | wt % | 5.46 |
| HHV | D240-92 | MJ/kg | 19.4 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | |
| Cellulose Content | T203 om-93 | wt % | 20.9 |
| Hemicellulose Content | T203 om-93 | wt % | 19.0 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 7.6 |
| Carbohydrates | T212 om-93 | wt % | 39.9 |
| **Mass Balance Analysis** | | | |
| Carbon | D5291 | wt % | 46.25 |
| Hydrogen | D5291 | wt % | 6.09 |
| Nitrogen | D5291 | wt % | 0.23 |
| Sulfur | D1552 | wt % | 0.09 |

| Product Liquid (Whole Oil) | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | wt % | 50.2 |
| Acetone/THF Insoluble (solids) | In-house | wt % | 0.35 |
| Ash Content | In-house | wt % | 0.36 |
| Carbon | D5291 | wt % | 43.29 |
| Hydrogen | D5291 | wt % | 9.68 |
| Nitrogen | D5291 | wt % | 0.46 |
| Sulfur | D1552 | wt % | 0.23 |
| **Aqueous phase 55.63 wt %** | | | **(aqueous)** |
| Density @ 25°C | D1298 | g/mL | 1.038 |
| PH | In-house | | 3.9 |
| Water Content | E 203-96 | wt % | 89.3 |
| Acetone/THF Insoluble (solids) | In-house | wt % | 0.09 |
| NRP (dry) | In-house | wt % | 0.30 |
| Ash Content | In-house | wt % | 0.24 |
| Carbon | D5291 | wt % | 8.91 |
| Hydrogen | D5291 | wt % | 10.07 |
| Nitrogen | D5291 | wt % | 0.02 |
| Sulfur | D1552 | wt % | 0.04 |
| **Heavies phase 44.37 wt %** | | | **(heavies)** |
| Water Content | E 203-96 | wt % | 1.2 |
| Acetone/THF Insoluble (solids) | In-house | wt % | 0.68 |
| Ash Content | In-house | wt % | 0.51 |
| Carbon | D5291 | wt % | 86.39 |
| Hydrogen | D5291 | wt % | 9.20 |
| Nitrogen | D5291 | wt % | 1.02 |
| Sulfur | D1552 | wt % | 0.46 |

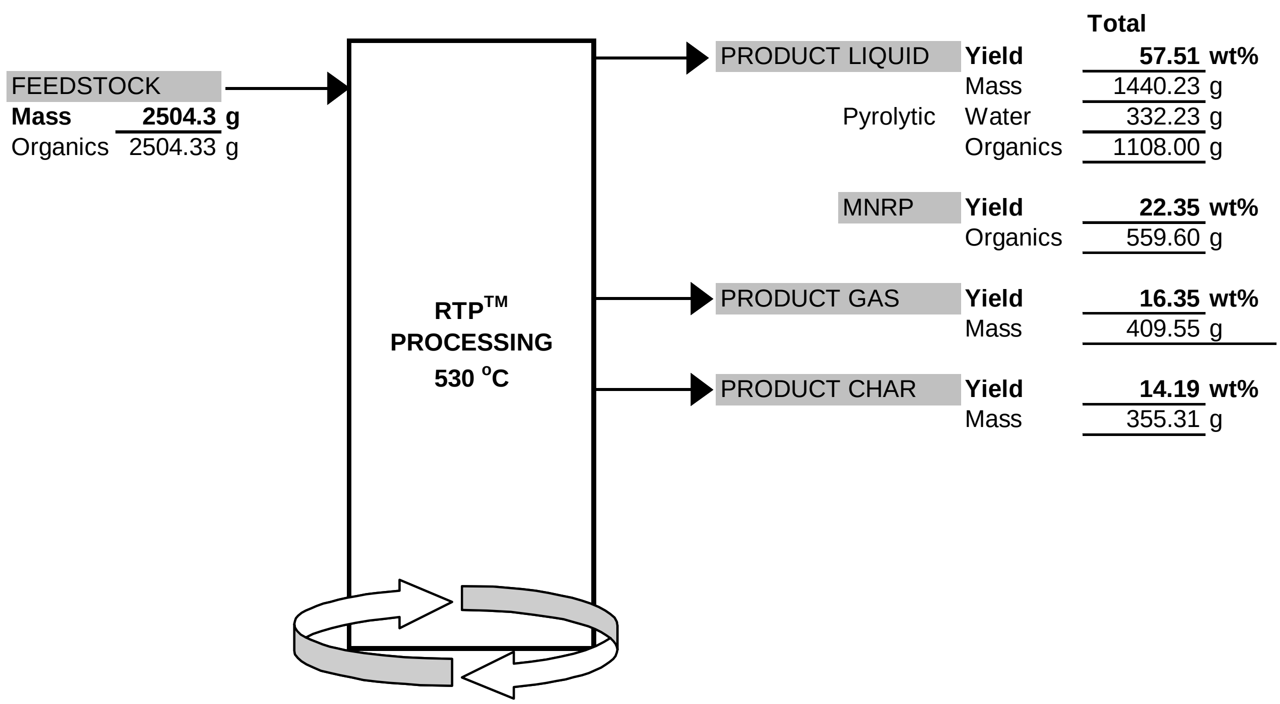
| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.53** |
| Carbon dioxide | In-house GC | vol % | **2.38** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **2.95** |
| Methane | In-house GC | vol % | **0.96** |
| Ethane | In-house GC | vol % | **0.11** |
| Ethylene | In-house GC | vol % | **0.33** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.02** |
| Propylene | In-house GC | vol % | **0.15** |
| I-Butane | In-house GC | vol % | **0.01** |
| N-Butane | In-house GC | vol % | **trace** |
| 1-Butene | In-house GC | vol % | **0.04** |
| Isobutylene | In-house GC | vol % | **0.01** |
| T-2-Butene | In-house GC | vol % | **0.01** |
| C-2-Butene | In-house GC | vol % | **0.01** |
| 1,3-Butadiene | In-house GC | vol % | **0.04** |
| I-Pentane | In-house GC | vol % | **0.01** |
| N-Pentane | In-house GC | vol % | **trace** |
| 3-ME-1-Butene | In-house GC | vol % | **0.01** |
| 1-Pentene | In-house GC | vol % | **0.01** |
| 2-ME-1-Butene | In-house GC | vol % | **trace** |
| T-2-Pentene | In-house GC | vol % | **0.01** |
| C-2-Pentene | In-house GC | vol % | **trace** |
| 2-ME-2-Butene | In-house GC | vol % | **trace** |
| C6+ | In-house GC | vol % | **0.07** |
| Oxygen | In-house GC | vol % | **4.82** |
| Nitrogen | In-house GC | vol % | **85.52** |

| Secondary Cyclone Char | | | |
|---|---|---|---|
| **Mass Balance Analysis (2 weeks)** | | | **into cyclone** |
| Moisture | In-house | wt % | 0.25 |
| Ash | In-house | wt % | 70.49 |
| Carbon | D5373 | wt % | 43.23 |
| Hydrogen | D5373 | wt % | 1.31 |
| Nitrogen | D5373 | wt % | 0.24 |
| Sulfur | D4239C | wt % | 0.06 |
| **Mass Balance Analysis (2 weeks)** | | | **into oil** |
| Moisture | In-house | wt % | 4.99 |
| Ash | In-house | wt % | 11.51 |
| Carbon | D5373 | wt % | 68.30 |
| Hydrogen | D5373 | wt % | 4.55 |
| Nitrogen | D5373 | wt % | 1.98 |
| Sulfur | D4239C | wt % | 0.34 |

## RTP-3A™ MATERIAL BALANCE FLOWSHEET (MAF) R44A

***(ASPEN)***

| *Mass Balance Closure* | 88% |
| --- | --- |



Run 44A
Feedstock ECN #257 Aspen
Reactor Temperature 530°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Moisture Content | T208 om-94 | wt % | 3.2 |
| Ash Content | In-house | wt % | 3.25 |
| U.S Sieve # +8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+14 | In-house | wt % | 3.6 |
| U.S Sieve # -14+18 | In-house | wt % | 29.4 |
| U.S Sieve # -18+20 | In-house | wt % | 21.3 |
| U.S Sieve # -20+35 | In-house | wt % | 25.0 |
| U.S Sieve # -35+70 | In-house | wt % | 10.5 |
| U.S Sieve # -70+100 | In-house | wt % | 6.1 |
| U.S Sieve # -100+200 | In-house | wt % | 3.6 |
| U.S Sieve # -200+325 | In-house | wt % | 0.5 |
| U.S Sieve # -325 | In-house | wt % | 0.0 |

| Collectables | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | wt % | 33.5 |
| Acetone/THF Insoluble (solids) | In-house | wt % | 0.03 |
| Ash Content | In-house | wt % | 0.09 |
| Acetone Wash Water Content | E 203-96 | wt % | 1.3 |

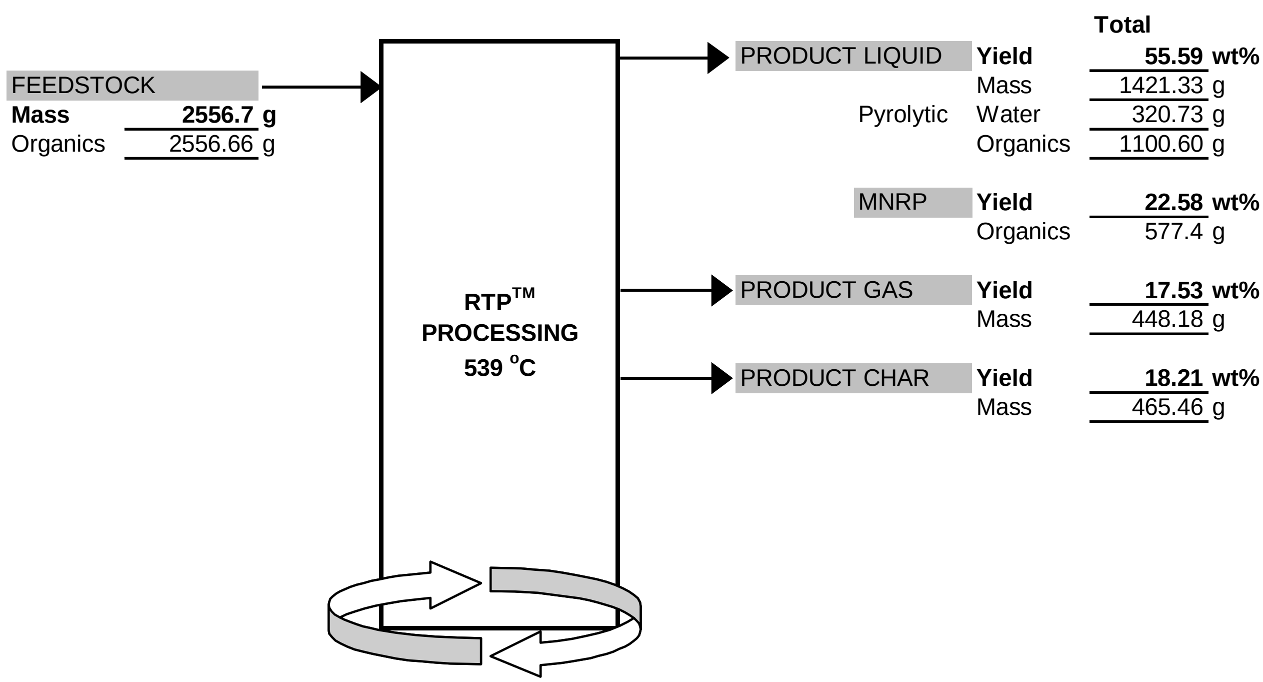
| MNRP-(68) T | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Acetone/THF Insoluble (solids) | In-house | wt % | 9.98 |
| Ash Content | In-house | wt % | 4.58 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.31** |
| Carbon dioxide | In-house GC | vol % | **1.47** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **1.59** |
| Methane | In-house GC | vol % | **0.43** |
| Ethane | In-house GC | vol % | **0.04** |
| Ethylene | In-house GC | vol % | **0.09** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.01** |
| Propylene | In-house GC | vol % | **0.05** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.01** |
| Isobutylene | In-house GC | vol % | **trace** |
| T-2-Butene | In-house GC | vol % | **trace** |
| C-2-Butene | In-house GC | vol % | **trace** |
| 1,3-Butadiene | In-house GC | vol % | **0.01** |
| I-Pentane | In-house GC | vol % | **0.00** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **trace** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **trace** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.05** |
| Oxygen | In-house GC | vol % | **0.32** |
| Nitrogen | In-house GC | vol % | **95.15** |

## RTP-3A™ MATERIAL BALANCE FLOWSHEET (MAF) R45A

***(Spruce Pine Fir)***

| | |
|---|---|
| ***Mass Balance Closure*** | **91.33** |



Run 45A
Feedstock ECN #253 SPF
Reactor Temperature 539°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Moisture Content | T208 om-94 | wt % | 6.7 |
| Ash Content | In-house | wt % | 3.34 |
| U.S Sieve # +8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+14 | In-house | wt % | 0.3 |
| U.S Sieve # -14+18 | In-house | wt % | 22.7 |
| U.S Sieve # -18+20 | In-house | wt % | 15.7 |
| U.S Sieve # -20+35 | In-house | wt % | 29.4 |
| U.S Sieve # -35+70 | In-house | wt % | 15.2 |
| U.S Sieve # -70+100 | In-house | wt % | 12.7 |
| U.S Sieve # -100+200 | In-house | wt % | 3.5 |
| U.S Sieve # -200+325 | In-house | wt % | 0.5 |
| U.S Sieve # -325 | In-house | wt % | 0.0 |

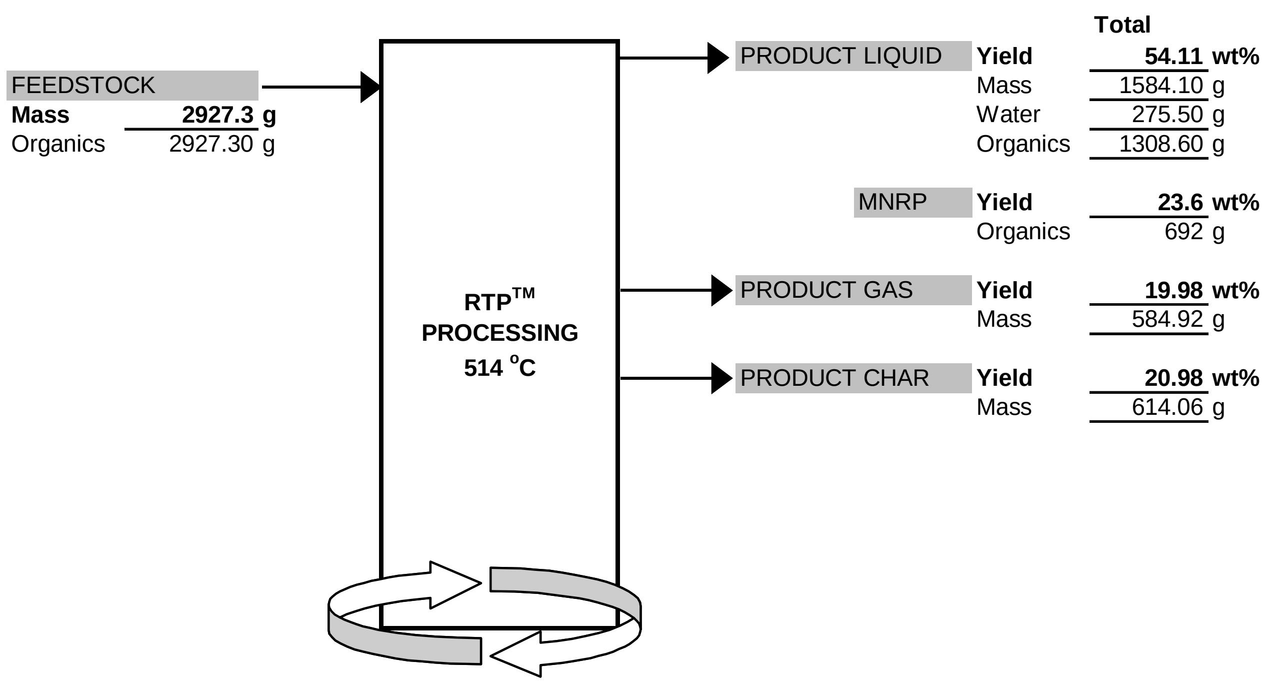
| MNRP-(68) T | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Acetone/THF Insoluble (solids) | In-house | wt % | 10.80 |
| Ash Content | In-house | wt % | 3.61 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | 0.33 |
| Carbon dioxide | In-house GC | vol % | 1.54 |
| Hydrogen Sulfide | In-house GC | vol % | 0.00 |
| (M) Carbonyl Sulfide | In-house GC | vol % | 0.00 |
| Carbon monoxide | In-house GC | vol % | 2.45 |
| Methane | In-house GC | vol % | 0.52 |
| Ethane | In-house GC | vol % | 0.04 |
| Ethylene | In-house GC | vol % | 0.13 |
| Acetylene | In-house GC | vol % | 0.00 |
| Propane | In-house GC | vol % | 0.01 |
| Propylene | In-house GC | vol % | 0.06 |
| I-Butane | In-house GC | vol % | trace |
| N-Butane | In-house GC | vol % | 0.00 |
| 1-Butene | In-house GC | vol % | 0.01 |
| Isobutylene | In-house GC | vol % | 0.01 |
| T-2-Butene | In-house GC | vol % | trace |
| C-2-Butene | In-house GC | vol % | 0.00 |
| 1,3-Butadiene | In-house GC | vol % | 0.01 |
| I-Pentane | In-house GC | vol % | trace |
| N-Pentane | In-house GC | vol % | 0.00 |
| 3-ME-1-Butene | In-house GC | vol % | trace |
| 1-Pentene | In-house GC | vol % | trace |
| 2-ME-1-Butene | In-house GC | vol % | trace |
| T-2-Pentene | In-house GC | vol % | 0.00 |
| C-2-Pentene | In-house GC | vol % | 0.00 |
| 2-ME-2-Butene | In-house GC | vol % | 0.00 |
| C6+ | In-house GC | vol % | 0.06 |
| Oxygen | In-house GC | vol % | 0.48 |
| Nitrogen | In-house GC | vol % | 94.74 |

## RTP-3A™ MATERIAL BALANCE FLOWSHEET (MAF) R48A

***(ASPEN)***

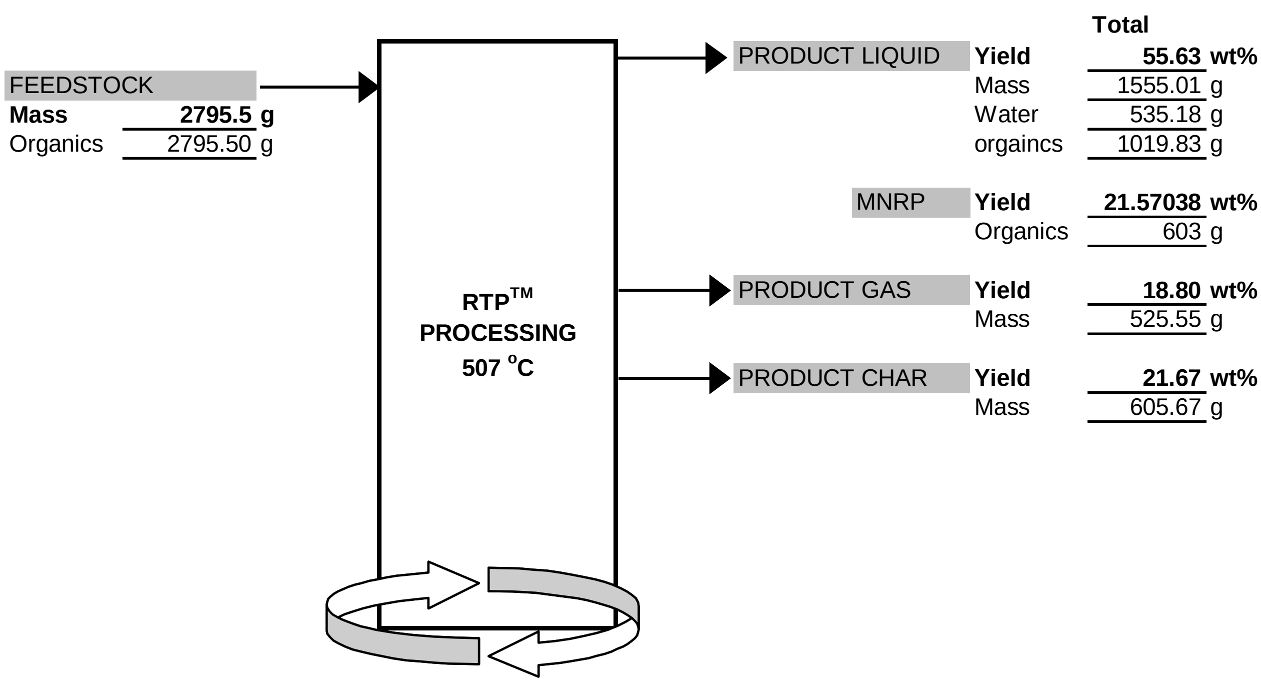
| | |
|---|---|
| ***Mass Balance Closure*** | **95.07** |



## RTP-3A™ MATERIAL BALANCE FLOWSHEET (MAF) R49A

***(ASPEN)***

| *Mass Balance Closure* | **96** % |
| --- | --- |



Run 49A
Feedstock ECN #239 Aspen
Reactor Temperature 507°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Moisture Content | T208 om-94 | wt % | 9.2 |
| Ash Content | In-house | wt % | 5.23 |
| U.S Sieve # +8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+14 | In-house | wt % | 3.4 |
| U.S Sieve # -14+18 | In-house | wt % | 19.9 |
| U.S Sieve # -18+20 | In-house | wt % | 19.8 |
| U.S Sieve # -20+35 | In-house | wt % | 33.5 |
| U.S Sieve # -35+70 | In-house | wt % | 11.5 |
| U.S Sieve # -70+100 | In-house | wt % | 6.0 |
| U.S Sieve # -100+200 | In-house | wt % | 4.0 |
| U.S Sieve # -200+325 | In-house | wt % | 1.0 |
| U.S Sieve # -325 | In-house | wt % | 1.0 |

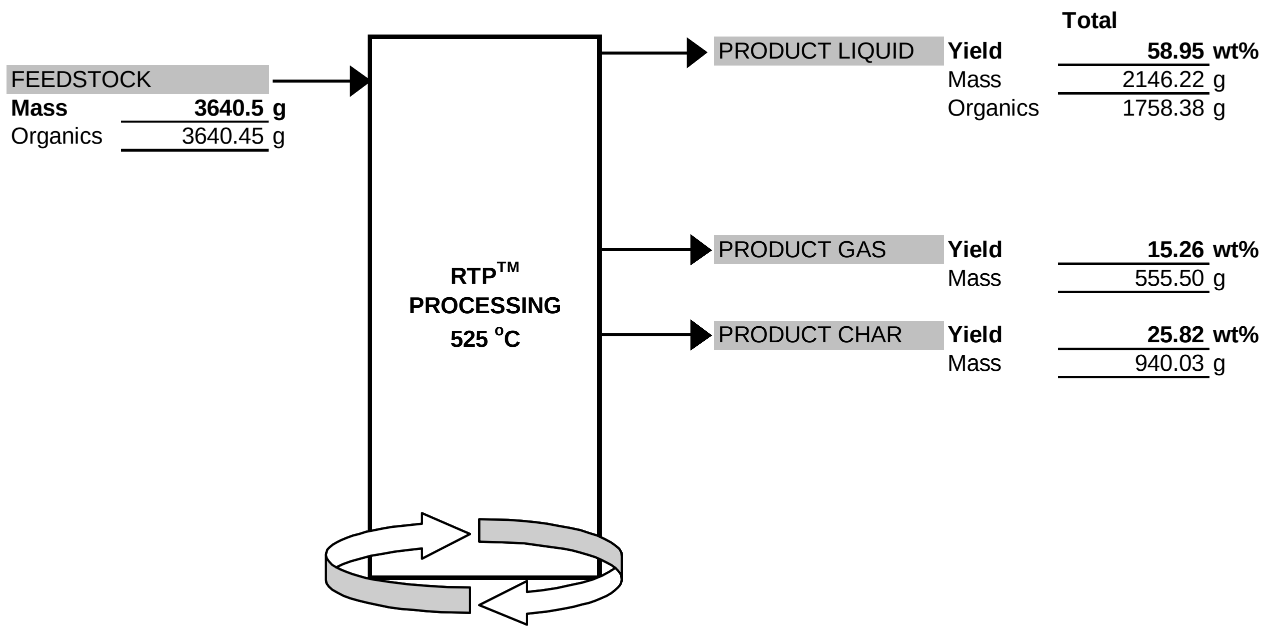
| MNRP-(68) T | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Acetone/THF Insoluble (solids) | In-house | wt % | 14.88 |
| Ash Content | In-house | wt % | 5.08 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | 0.40 |
| Carbon dioxide | In-house GC | vol % | 2.50 |
| Hydrogen Sulfide | In-house GC | vol % | 0.00 |
| (M) Carbonyl Sulfide | In-house GC | vol % | 0.00 |
| Carbon monoxide | In-house GC | vol % | 1.63 |
| Methane | In-house GC | vol % | 0.59 |
| Ethane | In-house GC | vol % | 0.06 |
| Ethylene | In-house GC | vol % | 0.06 |
| Acetylene | In-house GC | vol % | 0.00 |
| Propane | In-house GC | vol % | 0.01 |
| Propylene | In-house GC | vol % | 0.04 |
| I-Butane | In-house GC | vol % | 0.00 |
| N-Butane | In-house GC | vol % | trace |
| 1-Butene | In-house GC | vol % | trace |
| Isobutylene | In-house GC | vol % | trace |
| T-2-Butene | In-house GC | vol % | trace |
| C-2-Butene | In-house GC | vol % | trace |
| 1,3-Butadiene | In-house GC | vol % | 0.01 |
| I-Pentane | In-house GC | vol % | 0.00 |
| N-Pentane | In-house GC | vol % | 0.00 |
| 3-ME-1-Butene | In-house GC | vol % | trace |
| 1-Pentene | In-house GC | vol % | trace |
| 2-ME-1-Butene | In-house GC | vol % | trace |
| T-2-Pentene | In-house GC | vol % | trace |
| C-2-Pentene | In-house GC | vol % | 0.00 |
| 2-ME-2-Butene | In-house GC | vol % | 0.00 |
| C6+ | In-house GC | vol % | 0.05 |
| Oxygen | In-house GC | vol % | 0.20 |
| Nitrogen | In-house GC | vol % | 93.98 |

## RTP-3A™ MATERIAL BALANCE FLOWSHEET (MAF) R51A

(Southern Yellow Pine)

| *Mass Balance Closure* | 100.04 |
|---|---|



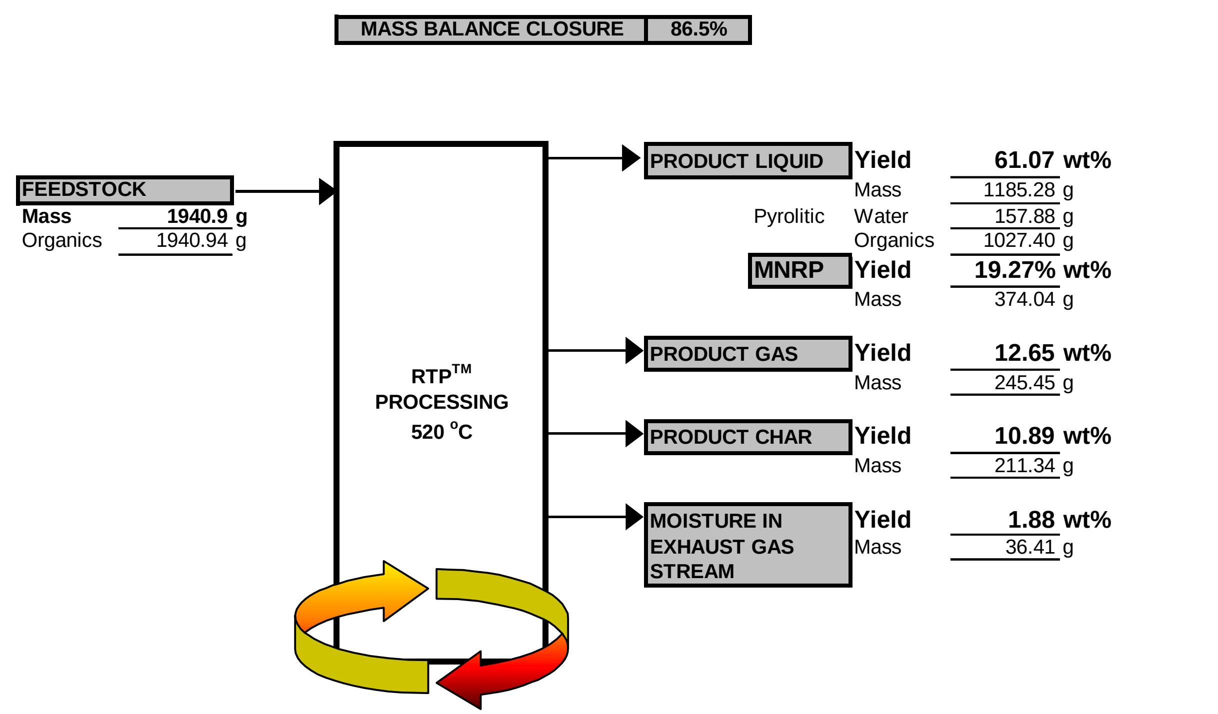
Run 51A
Feedstock ECN #270 SYP
Reactor Temperature 525°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Moisture Content | T208 om-94 | wt % | 9.9 |
| Ash Content | In-house | wt % | 2.23 |
| Bulk Density @ 25°C | D5291 | wt % | 0.276 |
| U.S Sieve # +8 | In-house | wt % | 0.3 |
| U.S Sieve # -8+14 | In-house | wt % | 1.2 |
| U.S Sieve # -14+18 | In-house | wt % | 28.5 |
| U.S Sieve # -18+20 | In-house | wt % | 24.3 |
| U.S Sieve # -20+35 | In-house | wt % | 25.2 |
| U.S Sieve # -35+70 | In-house | wt % | 12.6 |
| U.S Sieve # -70+100 | In-house | wt % | 5.0 |
| U.S Sieve # -100+200 | In-house | wt % | 2.5 |
| U.S Sieve # -200+325 | In-house | wt % | 0.3 |
| U.S Sieve # -325 | In-house | wt % | 0.1 |

| MNRP-B(58) T | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Acetone/THF Insoluble (solids) | In-house | wt % | 1.08 |
| Ash Content | In-house | wt % | 0.20 |
| Bulk Density | D1298-85 | g/Ml | 0.551 |
| Wax Content | In-house | wt % | 11.6 |
| Acid Number | In-house | mg/g | 39 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.18** |
| Carbon dioxide | In-house GC | vol % | **1.45** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **1.28** |
| Methane | In-house GC | vol % | **0.34** |
| Ethane | In-house GC | vol % | **0.02** |
| Ethylene | In-house GC | vol % | **0.04** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.03** |
| Propylene | In-house GC | vol % | **0.02** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **trace** |
| Isobutylene | In-house GC | vol % | **trace** |
| T-2-Butene | In-house GC | vol % | **trace** |
| C-2-Butene | In-house GC | vol % | **0.01** |
| 1,3-Butadiene | In-house GC | vol % | **trace** |
| I-Pentane | In-house GC | vol % | **trace** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **0.00** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.02** |
| Oxygen | In-house GC | vol % | **1.17** |
| Nitrogen | In-house GC | vol % | **95.50** |

## RTP-3A™ MATERIAL BALANCE FLOWSHEET (MAF) R84a (Forest Residue)




| Element | Feedstock (g) |
|---|---|
| C | 1094.8 |
| H | 137.9 |
| N | 6.6 |
| O-by diff | 831.3 |

| Liquid (g) | Char (g) | Gas (g) | Sat.Water (g) |
|---|---|---|---|
| 575.5 | 159.8 | 102.5 | #REF! |
| 98.9 | 6.4 | 6.6 | 4.0 |
| 5.8 | 0.6 | 0.0 | 0 |
| 634.5 | 44.7 | 136.3 | 32.40 |

| Balance |
|---|
| 77% |
| 84% |
| 96% |
| 98% |

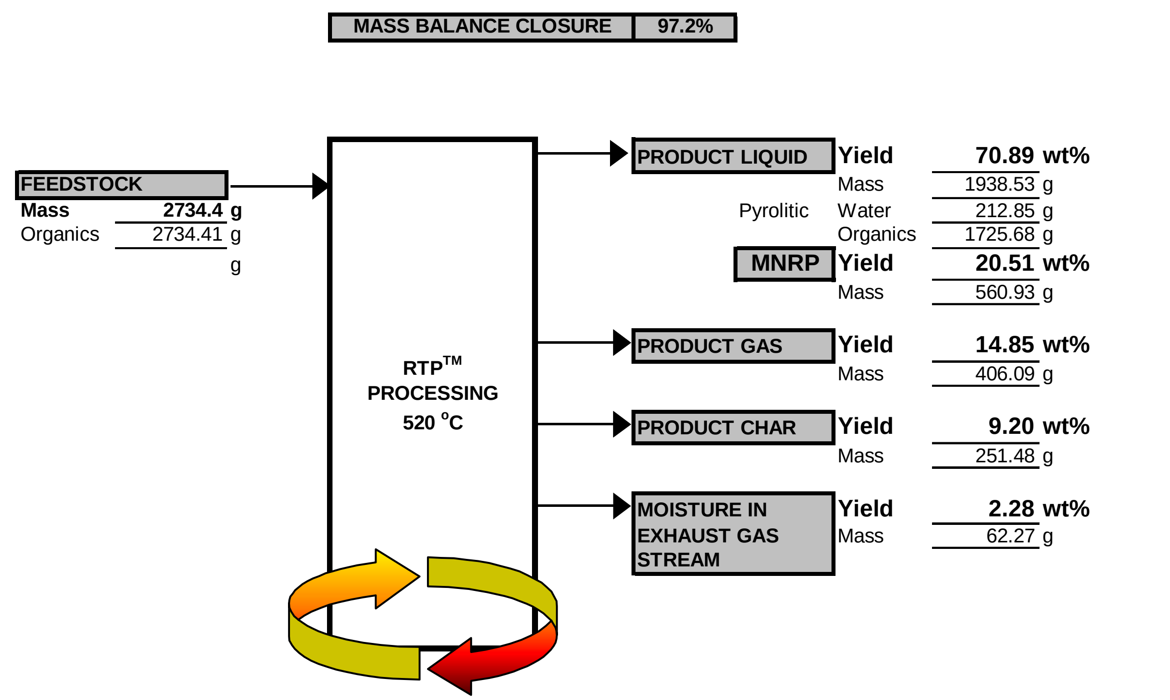
Run 84A
Feedstock ECN #308 Forest Residue
Reactor Temperature 520°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Moisture Content | T208 om-94 | wt % | 6.10 |
| Ash Content | In-house | wt % | 2.54 |
| Carbon | D5291 | wt % | 51.53 |
| Hydrogen | D5291 | wt % | 6.49 |
| Nitrogen | D5291 | wt % | 0.31 |
| U.S Sieve # +8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+14 | In-house | wt % | 1.2 |
| U.S Sieve # -14+18 | In-house | wt % | 19.2 |
| U.S Sieve # -18+20 | In-house | wt % | 27.3 |
| U.S Sieve # -20+35 | In-house | wt % | 29.2 |
| U.S Sieve # -35+70 | In-house | wt % | 17.7 |
| U.S Sieve # -70+100 | In-house | Wt % | 3.5 |
| U.S Sieve # -100+200 | In-house | Wt % | 1.5 |
| U.S Sieve # -200+325 | In-house | Wt % | 0.4 |
| U.S Sieve # -325 | In-house | Wt % | 0.0 |

| Composite Product Liquid | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | Wt % | 5.06 |
| Acetone/THF Insoluble (solids) | In-house | Wt % | 0.39 |
| Ash Content | In-house | Wt % | 0.059 |
| Carbon | D5291 | Wt % | 57.88 |
| Hydrogen | D5291 | Wt % | 7.47 |
| Nitrogen | D5291 | Wt % | 0.35 |
| Acid Number | In-house | mg/g | 45.40 |
| High Heating Value | D240-92 | MJ/kg | 24.32 |
| pH Value | In-house | | 2.77 |
| Bulk Density | D1298-85 | g/Ml | 1.08 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.12** |
| Carbon dioxide | In-house GC | vol % | **0.82** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **1.49** |
| Methane | In-house GC | vol % | **0.31** |
| Ethane | In-house GC | vol % | **0.02** |
| Ethylene | In-house GC | vol % | **0.06** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.01** |
| Propylene | In-house GC | vol % | **0.05** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.00** |
| Isobutylene | In-house GC | vol % | **0.00** |
| T-2-Butene | In-house GC | vol % | **0.00** |
| C-2-Butene | In-house GC | vol % | **0.00** |
| 1,3-Butadiene | In-house GC | vol % | **0.00** |
| I-Pentane | In-house GC | vol % | **0.00** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **0.00** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.03** |
| Oxygen | In-house GC | vol % | **0.07** |
| Nitrogen | In-house GC | vol % | **96.10** |

## RTP-3A™ MATERIAL BALANCE FLOWSHEET (MAF) R85a (Mill Residue)




| Element | Feedstock (g) |
|---|---|
| C | 1485.8 |
| H | 190.3 |
| N | 7.6 |
| O-by diff | 1218.0 |

| Liquid (g) | Char (g) | Gas (g) | Sat.Water (g) |
|---|---|---|---|
| 991.7 | 205.9 | 169.0 | #REF! |
| 171.9 | 7.3 | 10.3 | 6.8 |
| 8.3 | 0.5 | 0.0 | 0.0 |
| 933.1 | 37.8 | 226.9 | 55.4 |

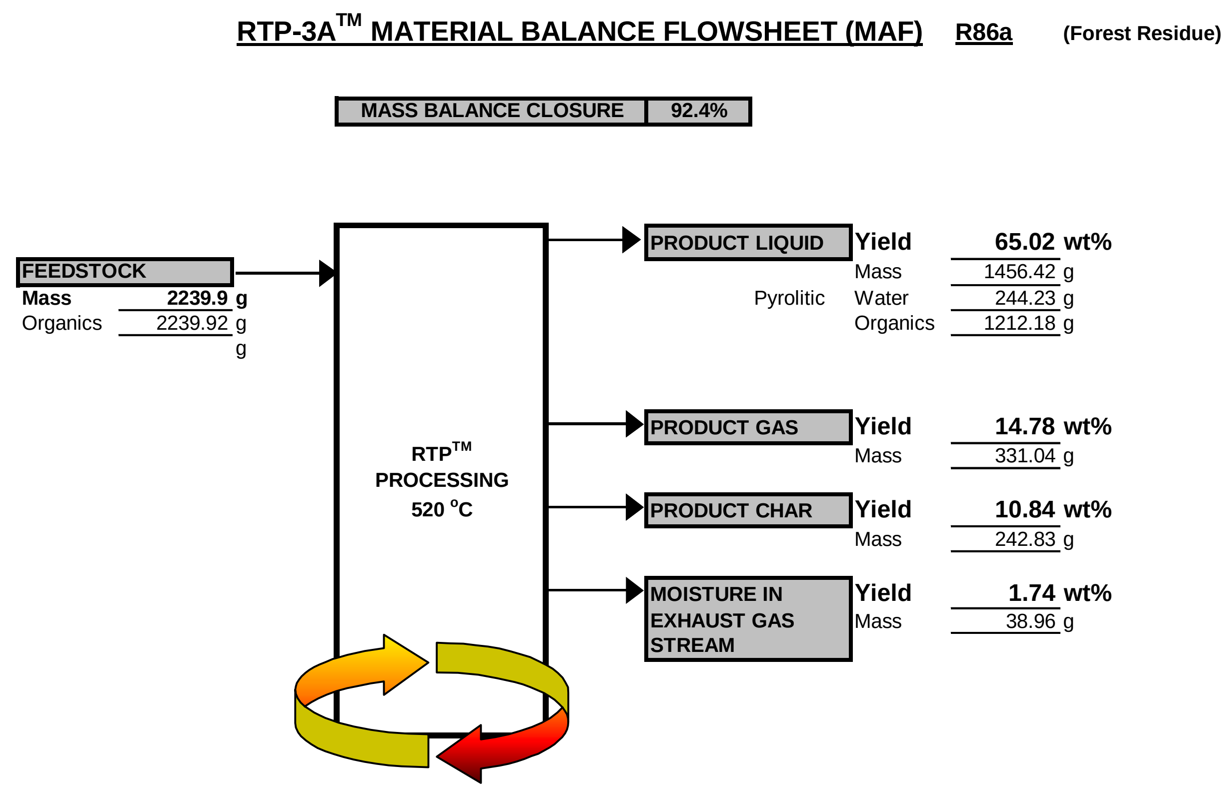
| Balance |
|---|
| #REF! |
| 103% |
| 117% |
| 103% |

Run 85A
Feedstock ECN #287 Mill Residue (Planar Shavings)
Reactor Temperature 520°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Moisture Content | T208 om-94 | wt % | 5.75 |
| Ash Content | In-house | wt % | 0.30 |
| Carbon | D5291 | wt % | 51.05 |
| Hydrogen | D5291 | wt % | 6.54 |
| Nitrogen | D5291 | wt % | 0.26 |
| U.S Sieve # +8 | In-house | wt % | 0.8 |
| U.S Sieve # -8+14 | In-house | wt % | 0.0 |
| U.S Sieve # -14+18 | In-house | wt % | 3.5 |
| U.S Sieve # -18+20 | In-house | wt % | 11.1 |
| U.S Sieve # -20+35 | In-house | wt % | 58.0 |
| U.S Sieve # -35+70 | In-house | wt % | 22.8 |
| U.S Sieve # -70+100 | In-house | Wt % | 3.0 |
| U.S Sieve # -100+200 | In-house | Wt % | 0.9 |
| U.S Sieve # -200+325 | In-house | Wt % | 0.0 |
| U.S Sieve # -325 | In-house | Wt % | 0.1 |

| Composite Product Liquid | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | Wt % | 6.52 |
| Acetone/THF Insoluble (solids) | In-house | Wt % | 1.05 |
| Ash Content | In-house | Wt % | 0.220 |
| Carbon | D5291 | Wt % | 57.42 |
| Hydrogen | D5291 | Wt % | 7.94 |
| Nitrogen | D5291 | Wt % | 0.38 |
| HA content | GC | Wt % | n/a |
| High Heating Value | D240-92 | MJ/kg | 22.57 |
| Bulk Density | D1298-85 | g/Ml | 1.10 |
| pH Value | In-house | | 2.34 |
| Acid Number | In-house | mg/g | 48.58 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.15** |
| Carbon dioxide | In-house GC | vol % | **0.74** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **1.56** |
| Methane | In-house GC | vol % | **0.29** |
| Ethane | In-house GC | vol % | **0.02** |
| Ethylene | In-house GC | vol % | **0.06** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.00** |
| Propylene | In-house GC | vol % | **0.03** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.00** |
| Isobutylene | In-house GC | vol % | **0.00** |
| T-2-Butene | In-house GC | vol % | **0.00** |
| C-2-Butene | In-house GC | vol % | **0.00** |
| 1,3-Butadiene | In-house GC | vol % | **0.00** |
| I-Pentane | In-house GC | vol % | **0.00** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **0.00** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.03** |
| Oxygen | In-house GC | vol % | **0.31** |
| Nitrogen | In-house GC | vol % | **96.19** |




| Element | Feedstock (g) | Liquid (g) | Char (g) | Gas (g) | Sat.Water (g) | Balance |
|---|---|---|---|---|---|---|
| C | 1263.4 | 777.1 | 204.5 | 141.2 | 0 | 89% |
| H | 159.1 | 134.9 | 7.1 | 9.4 | 4.3 | 98% |
| N | 7.6 | 5.3 | 0.8 | | 34.68 | 80% |
| O-by diff | 809.8 | 539.0 | 30.5 | 180.4 | 0 | 93% |

Run 86A
Feedstock ECN #308 Forest Residue
Reactor Temperature 520°C

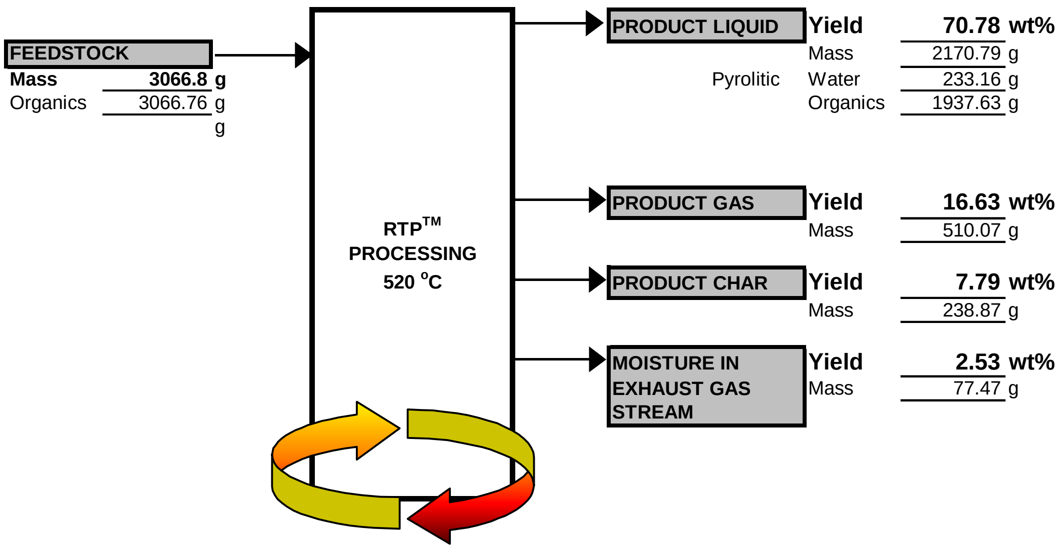
| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Moisture Content | T208 om-94 | wt % | 6.10 |
| Ash Content | In-house | wt % | 2.54 |
| Carbon | D5291 | wt % | 51.53 |
| Hydrogen | D5291 | wt % | 6.49 |
| Nitrogen | D5291 | wt % | 0.31 |
| U.S Sieve # +8 | In-house | wt % | 0.0 |
| U.S Sieve # -8+14 | In-house | wt % | 1.2 |
| U.S Sieve # -14+18 | In-house | wt % | 19.2 |
| U.S Sieve # -18+20 | In-house | wt % | 27.3 |
| U.S Sieve # -20+35 | In-house | wt % | 29.2 |
| U.S Sieve # -35+70 | In-house | wt % | 17.7 |
| U.S Sieve # -70+100 | In-house | Wt % | 3.5 |
| U.S Sieve # -100+200 | In-house | Wt % | 1.5 |
| U.S Sieve # -200+325 | In-house | Wt % | 0.4 |
| U.S Sieve # -325 | In-house | Wt % | 0.0 |

| Composite Product Liquid | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | Wt % | 10.43 |
| Acetone/THF Insoluble (solids) | In-house | Wt % | 0.05 |
| Ash Content | In-house | Wt % | 0.038 |
| Carbon | D5291 | Wt % | 57.42 |
| Hydrogen | D5291 | Wt % | 7.94 |
| Nitrogen | D5291 | Wt % | 0.38 |
| Acid Number | In-house | mg/g | 58.03 |
| High Heating Value | D240-92 | MJ/kg | 22.57 |
| pH Value | In-house | | 2.42 |
| Bulk Density | D1298-85 | g/Ml | 1.01 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.14** |
| Carbon dioxide | In-house GC | vol % | **0.98** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **1.91** |
| Methane | In-house GC | vol % | **0.41** |
| Ethane | In-house GC | vol % | **0.03** |
| Ethylene | In-house GC | vol % | **0.08** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.01** |
| Propylene | In-house GC | vol % | **0.05** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.01** |
| Isobutylene | In-house GC | vol % | **0.00** |
| T-2-Butene | In-house GC | vol % | **0.00** |
| C-2-Butene | In-house GC | vol % | **0.00** |
| 1,3-Butadiene | In-house GC | vol % | **0.00** |
| I-Pentane | In-house GC | vol % | **0.00** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **0.00** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.05** |
| Oxygen | In-house GC | vol % | **0.10** |
| Nitrogen | In-house GC | vol % | **95.25** |

**RTP-3A™ MATERIAL BALANCE FLOWSHEET (MAF)** **87a** **(Mill Residue)**

| MASS BALANCE CLOSURE | 97.7% |
|---|---|




| Element | Feedstock (g) |
|---|---|
| C | 1666.4 |
| H | 213.5 |
| N | 8.5 |
| O-by diff | 1178.4 |

| Liquid (g) | Char (g) | Gas (g) | Sat.Water (g) |
|---|---|---|---|
| 1172.7 | 199.7 | 215.8 | 0 |
| 179.3 | 7.2 | 14.0 | 8.5 |
| 6.5 | 0.4 | | |
| 812.4 | 31.4 | 280.4 | 68.94541 |

| Balance |
|---|
| 95% |
| 98% |
| 82% |
| 101% |

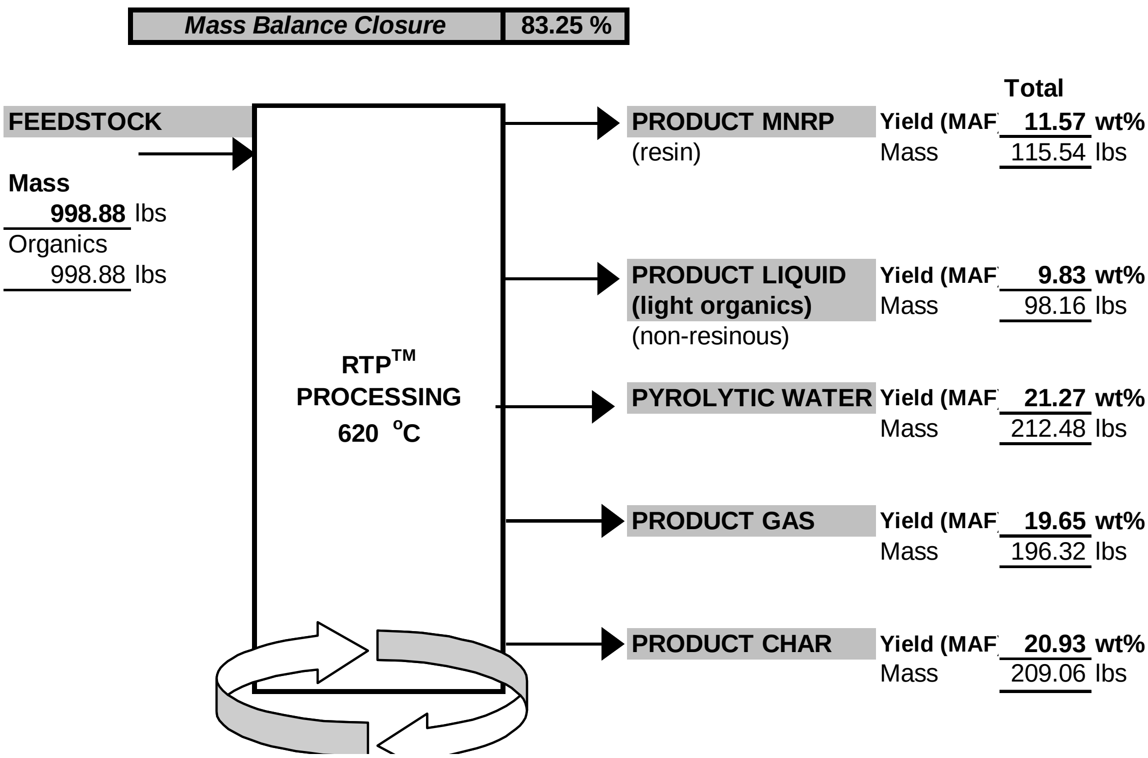
Run 87A
Feedstock ECN #287 Mill Residue (Planar Shavings)
Reactor Temperature 520°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Moisture Content | T208 om-94 | wt % | 5.75 |
| Ash Content | In-house | wt % | 0.30 |
| Carbon | D5291 | wt % | 51.05 |
| Hydrogen | D5291 | wt % | 6.54 |
| Nitrogen | D5291 | wt % | 0.26 |
| U.S Sieve # +8 | In-house | wt % | 0.8 |
| U.S Sieve # -8+14 | In-house | wt % | 0.0 |
| U.S Sieve # -14+18 | In-house | wt % | 3.5 |
| U.S Sieve # -18+20 | In-house | wt % | 11.1 |
| U.S Sieve # -20+35 | In-house | wt % | 58.0 |
| U.S Sieve # -35+70 | In-house | wt % | 22.8 |
| U.S Sieve # -70+100 | In-house | Wt % | 3.0 |
| U.S Sieve # -100+200 | In-house | Wt % | 0.9 |
| U.S Sieve # -200+325 | In-house | Wt % | 0.0 |
| U.S Sieve # -325 | In-house | Wt % | 0.1 |

| Composite Product Liquid | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | Wt % | 9.87 |
| Acetone/THF Insoluble (solids) | In-house | Wt % | 0.01 |
| Ash Content | In-house | Wt % | 0.062 |
| Carbon | D5291 | Wt % | 54.50 |
| Hydrogen | D5291 | Wt % | 7.26 |
| Nitrogen | D5291 | Wt % | 0.30 |
| Acid Number | In-house | mg/g | 56.42 |
| High Heating Value | D240-92 | MJ/kg | 21.65 |
| pH Value | In-house | | 2.16 |
| Bulk Density | D1298-85 | g/Ml | 1.10 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **0.14** |
| Carbon dioxide | In-house GC | vol % | **0.74** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **1.54** |
| Methane | In-house GC | vol % | **0.33** |
| Ethane | In-house GC | vol % | **0.02** |
| Ethylene | In-house GC | vol % | **0.06** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.00** |
| Propylene | In-house GC | vol % | **0.03** |
| I-Butane | In-house GC | vol % | **0.00** |
| N-Butane | In-house GC | vol % | **0.00** |
| 1-Butene | In-house GC | vol % | **0.00** |
| Isobutylene | In-house GC | vol % | **0.00** |
| T-2-Butene | In-house GC | vol % | **0.00** |
| C-2-Butene | In-house GC | vol % | **0.00** |
| 1,3-Butadiene | In-house GC | vol % | **0.00** |
| I-Pentane | In-house GC | vol % | **0.00** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **0.00** |
| 1-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-1-Butene | In-house GC | vol % | **0.00** |
| T-2-Pentene | In-house GC | vol % | **0.00** |
| C-2-Pentene | In-house GC | vol % | **0.00** |
| 2-ME-2-Butene | In-house GC | vol % | **0.00** |
| C6+ | In-house GC | vol % | **0.04** |
| Oxygen | In-house GC | vol % | **0.42** |
| Nitrogen | In-house GC | vol % | **95.76** |

## RTP-4 MATERIAL BALANCE FLOWSHEET (R269)



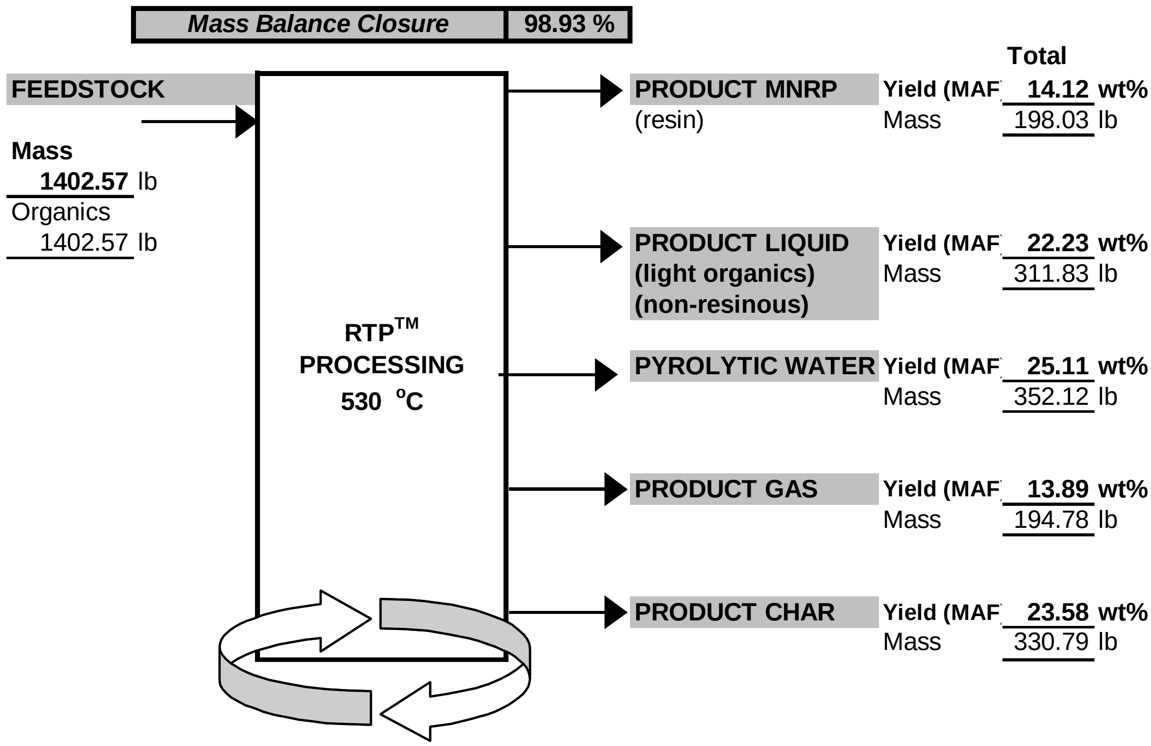
Run R269
Feedstock ECN #254 Aspen
Reactor Temperature 600°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.398 |
| Moisture Content | T208 om-94 | wt % | 4.8 |
| Ash Content | In-house | wt % | 4.67 |
| HHV | D240-92 | MJ/kg | 20.2 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 1.5 |
| U.S Sieve # -8+12 | In-house | wt % | 6.5 |
| U.S Sieve # -12+14 | In-house | wt % | 7.7 |
| U.S Sieve # -14+20 | In-house | wt % | 28.8 |
| U.S Sieve # -20+30 | In-house | wt % | 13.7 |
| U.S Sieve # -30+35 | In-house | wt % | 8.2 |
| U.S Sieve # -35+50 | In-house | wt % | 18.2 |
| U.S Sieve # -50 | In-house | wt % | 15.4 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 45.1 |
| Cellulose Content | T203 om-93 | wt % | 24.3 |
| Hemicellulose Content | T203 om-93 | wt % | 18.0 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 8.7 |
| Carbohydrates | T212 om-93 | wt % | 42.3 |
| **Mass Balance Analysis** | | | |
| S | D5291 | wt % | 0.08 |
| Na | Caleb Brett | ppm | 240 |
| Cl | Caleb Brett | ppm | < 1 |
| Phosphates | Caleb Brett | ppm | 319 |
| Ca | Caleb Brett | ppm | 17213 |
| $SiO_2$ | Caleb Brett | ppm | 56 |
| K | Caleb Brett | ppm | 3182 |
| Mg | Caleb Brett | ppm | 1013 |

| Product Liquid (Whole Oil) | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | wt % | 52.2 |
| Acid Content (dry) | In-house | wt % | 2.7 |
| Acetone/THF Insoluble (solids) | In-house | wt % | 9.12 |
| NRP (dry) | In-house | wt % | 42.5 |
| Ash Content | In-house | wt % | 7.42 |
| **Product MNRP-B(58)T** | | | |
| **Immediate Analysis** | | | |
| Weight | | G | 216.2 |
| Bulk Density | In-house | | 0.68 |
| Acid Content (dry) | In-house | wt % | 1.8 |
| Acid Number | In-house | mg/g | 38.0 |
| Ash Content | In-house | wt % | 0.09 |
| Acetone/THF Insoluble (solids) | In-house | wt % | 0.22 |
| Melting Point | In-house | °C | 56 |
| U.S Sieve # +8 | | wt % | 0.0 |
| U.S Sieve # -8+10 | | wt % | 1.2 |
| U.S Sieve # -10+100 | | wt % | 78.3 |
| U.S Sieve # -100+200 | | wt % | 20.0 |
| U.S Sieve # -200 | | wt % | 0.5 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **2.01** |
| Carbon dioxide | In-house GC | vol % | **12.85** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **5.78** |
| Methane | In-house GC | vol % | **2.06** |
| Ethane | In-house GC | vol % | **0.17** |
| Ethylene | In-house GC | vol % | **0.34** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.02** |
| Propylene | In-house GC | vol % | **0.16** |
| I-Butane | In-house GC | vol % | **trace** |
| N-Butane | In-house GC | vol % | **trace** |
| 1-Butene | In-house GC | vol % | **0.03** |
| Isobutylene | In-house GC | vol % | **0.01** |
| T-2-Butene | In-house GC | vol % | **0.01** |
| C-2-Butene | In-house GC | vol % | **0.01** |
| 1,3-Butadiene | In-house GC | vol % | **0.02** |
| I-Pentane | In-house GC | vol % | **trace** |
| N-Pentane | In-house GC | vol % | **0.00** |
| 3-ME-1-Butene | In-house GC | vol % | **trace** |
| 1-Pentene | In-house GC | vol % | **0.01** |
| 2-ME-1-Butene | In-house GC | vol % | **trace** |
| T-2-Pentene | In-house GC | vol % | **trace** |
| C-2-Pentene | In-house GC | vol % | **trace** |
| 2-ME-2-Butene | In-house GC | vol % | **trace** |
| C6+ | In-house GC | vol % | **0.07** |
| Oxygen | In-house GC | vol % | **7.03** |
| Nitrogen | In-house GC | vol % | **67.62** |

## RTP-4 MATERIAL BALANCE FLOWSHEET (R270)



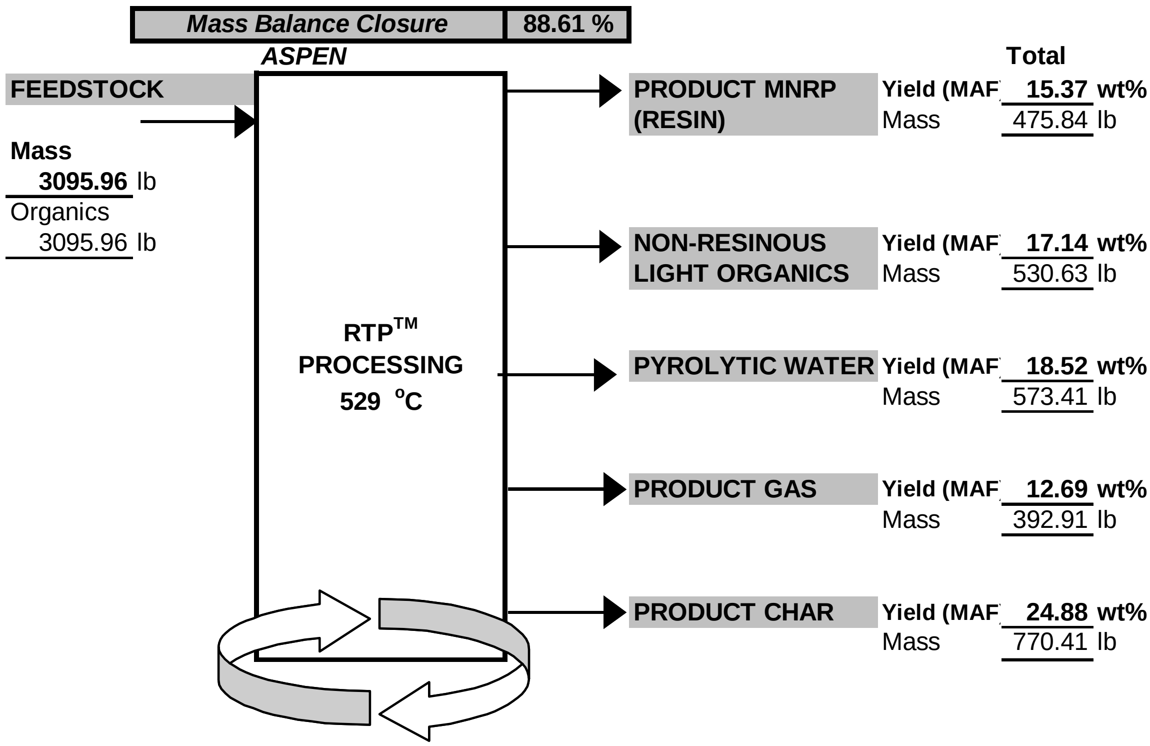
Run R270
Feedstock ECN #254 Aspen
Reactor Temperature 533°C

| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.398 |
| Moisture Content | T208 om-94 | wt % | 4.8 |
| Ash Content | In-house | wt % | 4.67 |
| HHV | D240-92 | MJ/kg | 20.2 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 1.5 |
| U.S Sieve # -8+12 | In-house | wt % | 6.5 |
| U.S Sieve # -12+14 | In-house | wt % | 7.7 |
| U.S Sieve # -14+20 | In-house | wt % | 28.8 |
| U.S Sieve # -20+30 | In-house | wt % | 13.7 |
| U.S Sieve # -30+35 | In-house | wt % | 8.2 |
| U.S Sieve # -35+50 | In-house | wt % | 18.2 |
| U.S Sieve # -50 | In-house | wt % | 15.4 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 45.1 |
| Cellulose Content | T203 om-93 | wt % | 24.3 |
| Hemicellulose Content | T203 om-93 | wt % | 18.0 |
| Wood Extractives (Resins) | T204 om-88 | wt % | 8.7 |
| Carbohydrates | T212 om-93 | wt % | 42.3 |
| **Mass Balance Analysis** | | | |
| S | D5291 | wt % | 0.08 |
| Na | Caleb Brett | ppm | 240 |
| Cl | Caleb Brett | ppm | < 1 |
| Phosphates | Caleb Brett | ppm | 319 |
| Ca | Caleb Brett | ppm | 17213 |
| $SiO_2$ | Caleb Brett | ppm | 56 |
| K | Caleb Brett | ppm | 3182 |
| Mg | Caleb Brett | ppm | 1013 |

| Product Liquid (Whole Oil) | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Water Content | E 203-96 | wt % | 37.4 |
| Acid Content (dry) | In-house | wt % | |
| Acetone/THF Insoluble (solids) | In-house | wt % | 3.1 |
| NRP (dry) | In-house | wt % | 40.2 |
| Ash Content | In-house | wt % | 0.4 |
| **Product MNRP-B(58)T** | | | |
| **Immediate Analysis** | | | |
| Weight | | G | 1127.4 |
| Bulk Density | In-house | | 0.69 |
| Acid Content (dry) | In-house | wt % | 1.8 |
| Acid Number | In-house | mg/g | 40.0 |
| Ash Content | In-house | wt % | 0.29 |
| Acetone/THF Insoluble (solids) | In-house | wt % | 0.22 |
| Melting Point | In-house | °C | 56 |
| U.S Sieve # +8 | | wt % | 0.0 |
| U.S Sieve # -8+10 | | wt % | 0.5 |
| U.S Sieve # -10+100 | | wt % | 85.2 |
| U.S Sieve # -100+200 | | wt % | 13.8 |
| U.S Sieve # -200 | | wt % | 0.5 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **1.78** |
| Carbon dioxide | In-house GC | vol % | **30.48** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **26.75** |
| Methane | In-house GC | vol % | **6.30** |
| Ethane | In-house GC | vol % | **0.71** |
| Ethylene | In-house GC | vol % | **1.43** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.16** |
| Propylene | In-house GC | vol % | **0.73** |
| I-Butane | In-house GC | vol % | **0.01** |
| N-Butane | In-house GC | vol % | **0.02** |
| 1-Butene | In-house GC | vol % | **0.16** |
| Isobutylene | In-house GC | vol % | **0.05** |
| T-2-Butene | In-house GC | vol % | **0.04** |
| C-2-Butene | In-house GC | vol % | **0.03** |
| 1,3-Butadiene | In-house GC | vol % | **0.09** |
| I-Pentane | In-house GC | vol % | **trace** |
| N-Pentane | In-house GC | vol % | **0.01** |
| 3-ME-1-Butene | In-house GC | vol % | **0.02** |
| 1-Pentene | In-house GC | vol % | **0.05** |
| 2-ME-1-Butene | In-house GC | vol % | **0.01** |
| T-2-Pentene | In-house GC | vol % | **0.02** |
| C-2-Pentene | In-house GC | vol % | **0.01** |
| 2-ME-2-Butene | In-house GC | vol % | **0.01** |
| C6+ | In-house GC | vol % | **0.21** |
| Oxygen | In-house GC | vol % | **0.84** |
| Nitrogen | In-house GC | vol % | **29.14** |

**RTP-4 MATERIAL BALANCE FLOWSHEET (R271)**



Run R271
Feedstock ECN #257Aspen
Reactor Temperature 529°C

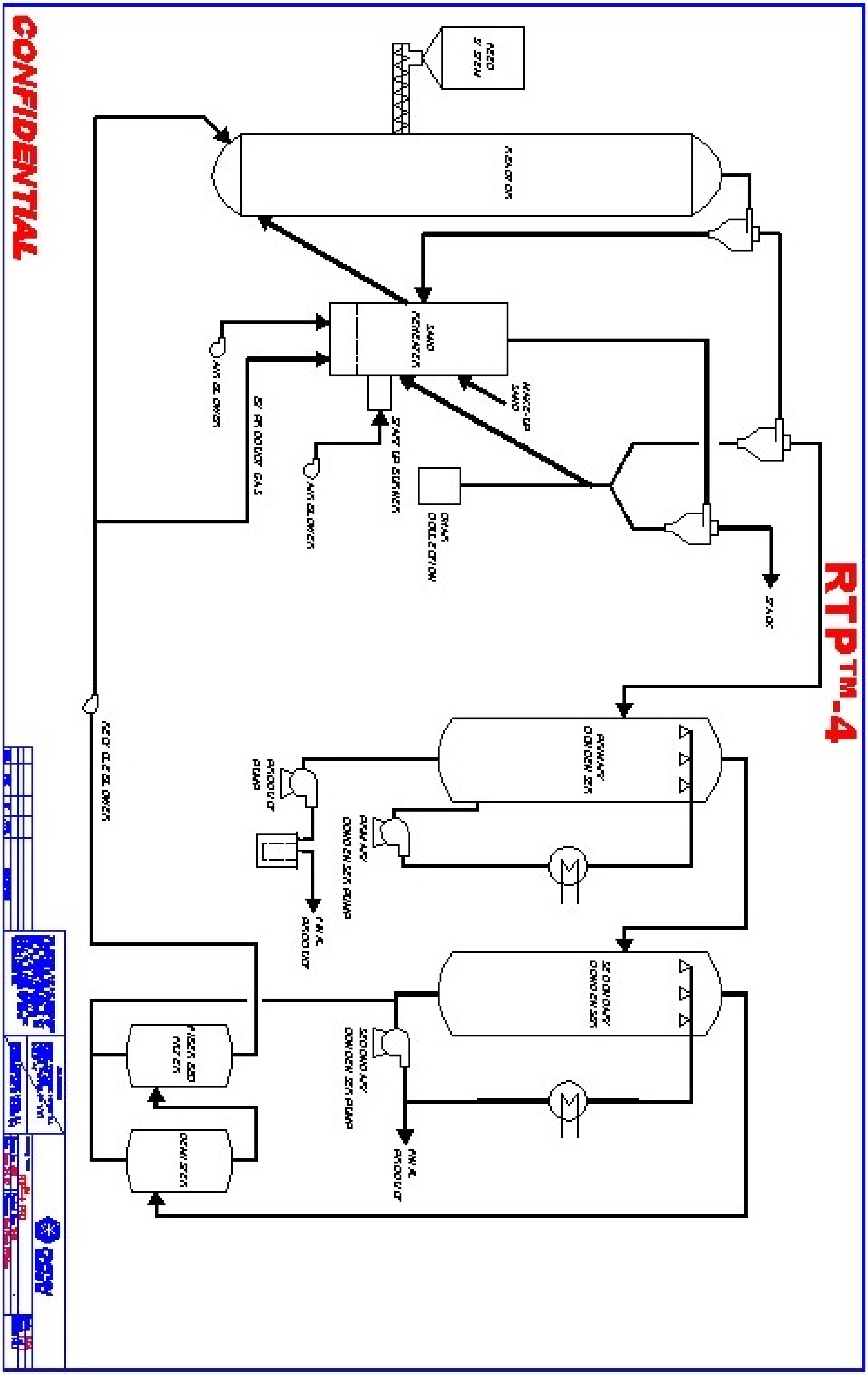
| Reactor Feed (as fed) | Method ASTM | Units | Results |
|---|---|---|---|
| **Immediate Analysis** | | | |
| Bulk Density @ 25°C | In-house | g/mL | 0.339 |
| Moisture Content | T208 om-94 | wt % | 2.9 |
| Ash Content | In-house | wt % | 3.61 |
| HHV | D240-92 | MJ/kg | 20.9 |
| U.S Sieve # +1/4 | In-house | wt % | 0.0 |
| U.S Sieve # -1/4+8 | In-house | wt % | 13.5 |
| U.S Sieve # -8+12 | In-house | wt % | 11.1 |
| U.S Sieve # -12+14 | In-house | wt % | 7.6 |
| U.S Sieve # -14+20 | In-house | wt % | 17.2 |
| U.S Sieve # -20+30 | In-house | wt % | 11.8 |
| U.S Sieve # -30+35 | In-house | wt % | 8.8 |
| U.S Sieve # -35+50 | In-house | wt % | 14.7 |
| U.S Sieve # -50 | In-house | wt % | 15.2 |
| **Basic Analysis** | | | |
| Lignin (Klason) Content | T222 om-88 | wt % | 43.7 |
| Cellulose Content | T203 om-93 | wt % | 20.8 |
| Hemicellulose Content | T203 om-93 | wt % | 18.4 |
| Wood Extractives (acetone | T204 om-88 | wt % | 3.6 |
| Wax (by chloroform) | T204 om-88 | wt % | 6.7 |
| Carbohydrates | T212 om-93 | wt % | 39.2 |
| **Mass Balance Analysis** | | | |
| C | D5291 | wt % | 49.27 |
| H | D5291 | wt % | 6.00 |
| N | D5291 | wt % | 0.22 |
| S | D5291 | wt % | 0.09 |
| Na | GalbraithLab | ppm | 0.051 |
| Cl | GalbraithLab | ppm | 39 |
| Phosphates | GalbraithLab | ppm | <0.03 |
| Ca | GalbraithLab | ppm | 1.15 |
| $SiO_2$ | GalbraithLab | ppm | 0.35 |
| K | GalbraithLab | ppm | 0.26 |
| Mg | GalbraithLab | ppm | 0.077 |

| Product Gas | Method ASTM | Units | Results |
|---|---|---|---|
| **Mass Balance Analysis (6 working days)** | | | **Average** |
| Hydrogen | In-house GC | vol % | **1.98** |
| Carbon dioxide | In-house GC | vol % | **33.56** |
| Hydrogen Sulfide | In-house GC | vol % | **0.00** |
| (M) Carbonyl Sulfide | In-house GC | vol % | **0.00** |
| Carbon monoxide | In-house GC | vol % | **33.41** |
| Methane | In-house GC | vol % | **7.78** |
| Ethane | In-house GC | vol % | **0.93** |
| Ethylene | In-house GC | vol % | **1.90** |
| Acetylene | In-house GC | vol % | **0.00** |
| Propane | In-house GC | vol % | **0.20** |
| Propylene | In-house GC | vol % | **0.88** |
| I-Butane | In-house GC | vol % | **0.01** |
| N-Butane | In-house GC | vol % | **0.02** |
| 1-Butene | In-house GC | vol % | **0.17** |
| Isobutylene | In-house GC | vol % | **0.06** |
| T-2-Butene | In-house GC | vol % | **0.05** |
| C-2-Butene | In-house GC | vol % | **0.03** |
| 1,3-Butadiene | In-house GC | vol % | **0.10** |
| I-Pentane | In-house GC | vol % | **trace** |
| N-Pentane | In-house GC | vol % | **0.01** |
| 3-ME-1-Butene | In-house GC | vol % | **0.02** |
| 1-Pentene | In-house GC | vol % | **0.04** |
| 2-ME-1-Butene | In-house GC | vol % | **0.01** |
| T-2-Pentene | In-house GC | vol % | **0.02** |
| C-2-Pentene | In-house GC | vol % | **0.01** |
| 2-ME-2-Butene | In-house GC | vol % | **0.01** |
| C6+ | In-house GC | vol % | **0.26** |
| Oxygen | In-house GC | vol % | **1.53** |
| Nitrogen | In-house GC | vol % | **17.55** |

| Product Liquid (Whole Oil) | | | |
|---|---|---|---|
| **Immediate Analysis** | | | **(calculated)** |
| Density @ 25°C | D1298 | $g/cm^3$ | 1.09 |
| pH | In-house | | 3.40 |
| Water Content | E 203-96 | wt % | 65.52 |
| Acid Content (dry) | In-house | wt % | 18.85 |
| Acetone/THF Insoluble (solids) | In-house | wt % | 1.07 |
| Ash Content on Solid | In-house | wt % | 2.88 |
| Ash Content | In-house | wt % | 0.34 |
| Wax Content | In-house | wt % | 1.65 |

# APPENDIX A4

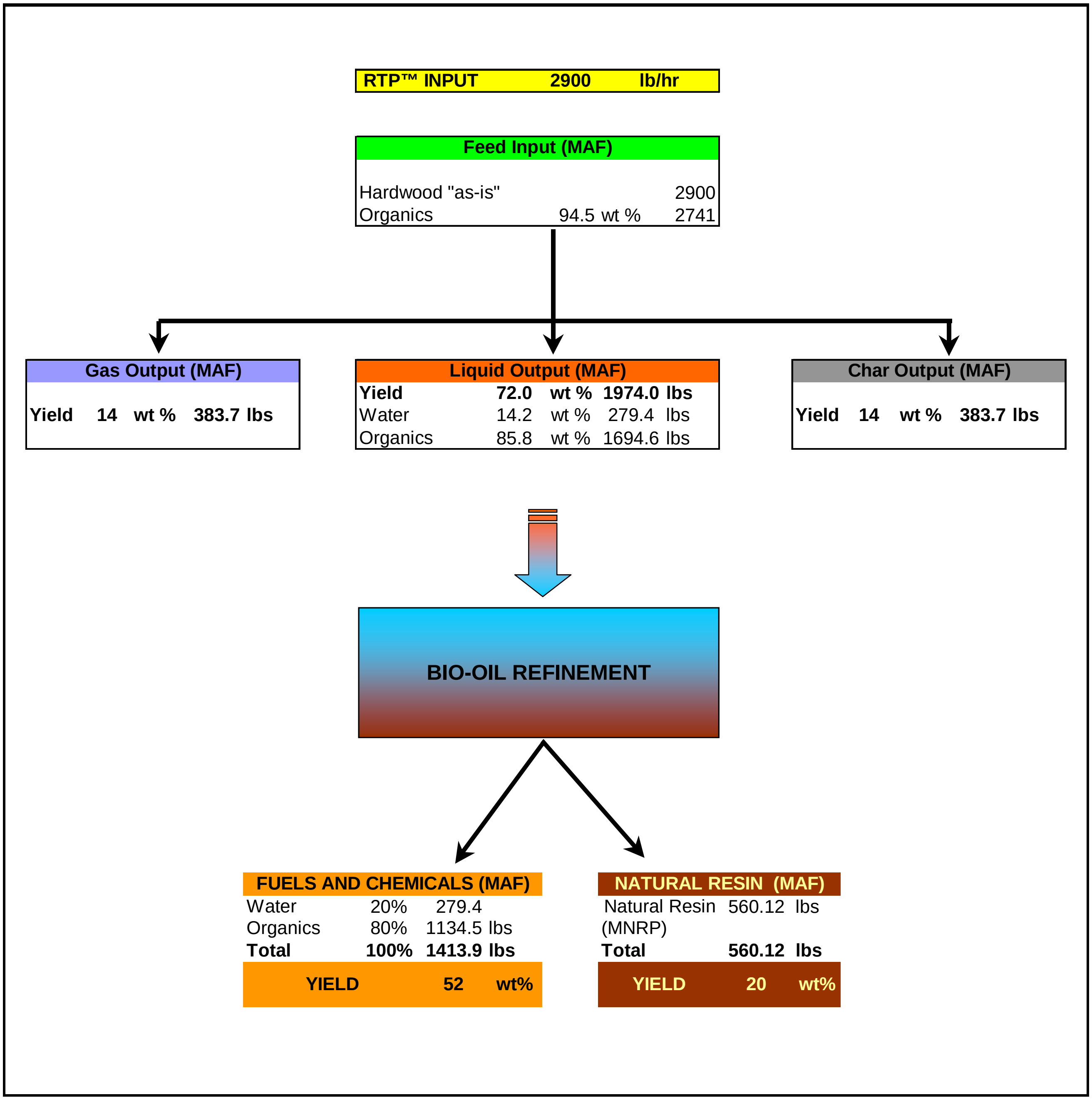
**Pilot Scale Unit (RTP-4) Configuration – Process Flow Diagram**


CONFIDENTIAL
RTP™-4

# APPENDIX A5

## Large-Scale Production of Whitewood Residue-Derived MNRP in Rhinelander

**MASS FLOW DIAGRAM FOR THE PRODUCTION OF NATURAL RESIN FROM A COMMERCIAL RTP™**



# MNRP - 63

Shipment : **001**
Date : **03/05/02**
Purchase Order :
Amount: **3,027 LBS**

| Item | Bag Identification Numbers | Weight (lbs) |
|---|---|---|
| 1 | RH- 001- 2002-05-03 -01 | 1001 |
| 2 | RH- 001- 2002-05-03 -02 | 1008 |
| 3 | RH- 001- 2002-05-03 -03 | 1018 |

Totals 40 # of Bags 3,027 Pounds

| Analysis | MNRP (63) | MNRP (63) + | MNRP (63) | MNRP (63) | MNRP (63) + | MNRP (63) + | MNRP (63) | Distillate | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Bag 1 | Bag 2 | Bag 3 | Bag 4 | Bag 5 | Bag 6 | Bag 7 | light (Pail 4) | *dark (Pail 1) | **light (Pail 1) |
| **Ash Content (wt%)** | 0.126 | 0.0573 | 0.101 | 0.164 | 0.126 | 0.0969 | 0.155 | 0.107 | 0.255 | 0.159 |
| **Solid Content (wt%)** | 1.31 | 1.23 | 1.05 | 1.35 | 0.813 | 1.45 | 1.30 | 0.241 | 0.056 | 0.131 |
| **Melting Point (°C)** | 77.0 | 66.3 | 61.0 | 63.3 | 64.0 | 60.8 | 63.0 | n/a | n/a | n/a |
| **Acid Number (mg/g)** | 19.51 | 22.72 | 26.07 | 25.41 | 23.07 | 24.32 | 26.97 | 122.2 | 80.1 | 121.2 |
| **Moisture (wt%)** | | | | | | | | 17 Brix | | 17 Brix |
| **Bulk Density (g/cm3)** | 0.7363 | 0.7729 | 0.7456 | 0.7372 | 0.7343 | 0.7914 | 0.6982 | 1.0399 | 1.0190 | 1.0563 |
| **pH** | | | | | | | | 1.60 | 1.77 | 1.60 |

* Dark (Pail #1) – Dark fraction left after separation.
** Light (Pail #1) – Light fraction left after separation.

# APPENDIX A6

## Char Activation

# Activated Char

For Char Derived from Planar Shavings and Forest Residue

**Background Information**

Activated char is a carbonaceous adsorbent with high internal porosity, produced from the heat treatment or activation of raw materials such as wood, coal, peat, coconut shells etc. It is used extensively in the food and chemical industry for applications including; decolorization of sugars and sweeteners, odor control, water purification and gas cleanup. Steam activation is one of the common processes used for the manufacture of activated carbon. Raw materials are subjected to various pre-treatment (acid washing, pelletization), processing temperatures (700 to 1100C), steam loadings (.5 to 1) and residence times (.5 to 1 hr) to achieve tailor made materials suitable for specific commercial applications. Adsorptive and physical characteristics play an important role in activated carbon performance and may include; surface area, pore size, iodine number, hardness, pH, bulk density and molasses number. This work examines the potential use of by-product char from bench-scale RTP processing of planar shavings and forest residue feedstocks as activated carbon

**Small Scale Activation Unit**

Ensyn designed, constructed and tested a small-scale activation unit to evaluate the potential use of RTP by-product char from planar shavings and forest residue feedstock's as activated carbon. The small-scale activation unit allows Ensyn to evaluate yield and adsorption for 20 g samples of char under varying conditions of temperature, steam to carbon ratio and residence time.

The small-scale activator is illustrated in Figure 5. The reactor is approximately 20 mm in diameter and is constructed of stainless steel, set inside an electrical resistance furnace. Three thermocouples mounted inside the reactor were used to determine the average processing temperature. Steam was produced in a custom made generator and fed into the reactor for a predetermined amount of time. A weigh scale was used to measure the amount of water entering the system. The exhaust from the activation unit was vented to atmosphere.

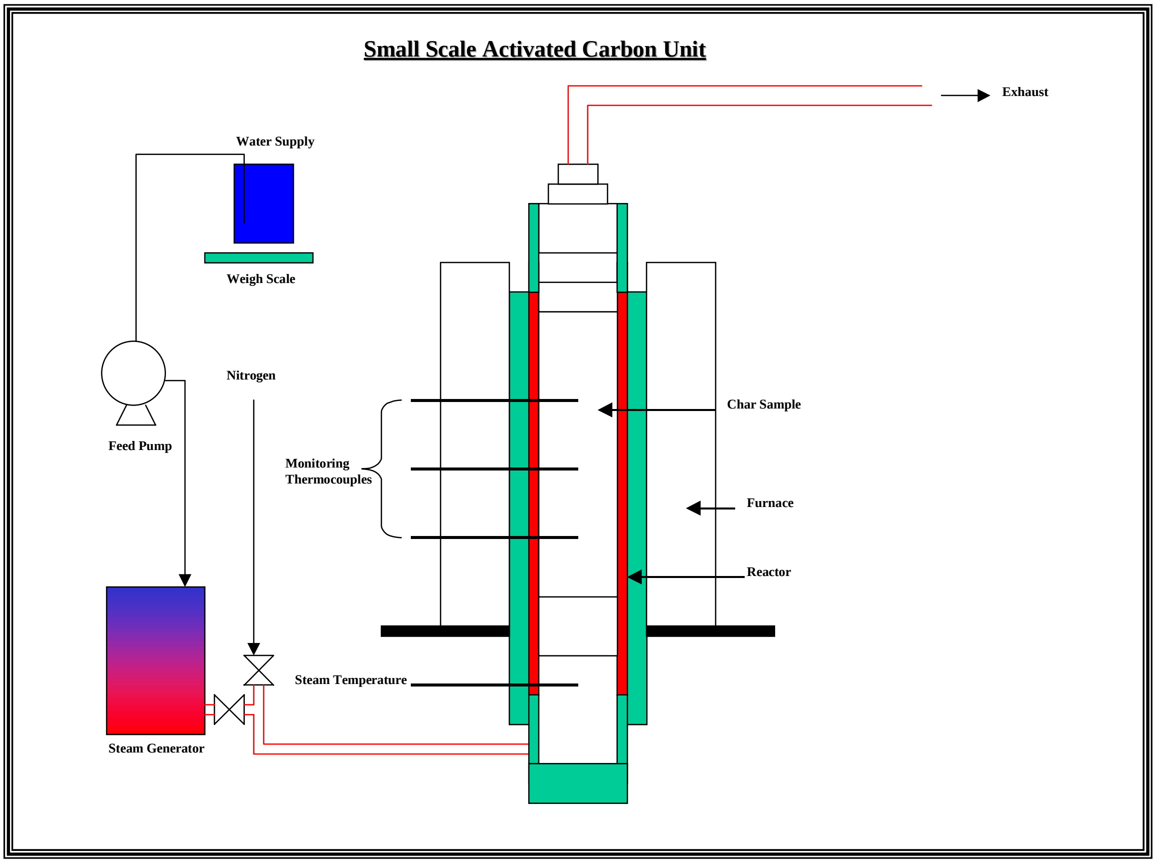



**Figure 5 - Small Activated Carbon Unit**

## Experimental Procedures

For each experiment, the reactor tube was properly cleaned and assembled, with an appropriate amount of char sample loaded. The furnace was turned on with nitrogen purge through the reactor. Once the desired reaction temperature was reached (700°C to 1000°C), steam was turned on and flow rates adjusted to maintain the desired steam to carbon ratio. Temperature, water flow rates and pressure readings were recorded every five minutes for the duration of the run (30min to 120 min). At the end of each test, the electric heaters and steam were switched off and nitrogen purged through the reactor for cool down (approx. 2 hrs). The activated carbon sample was removed and weighed to obtain yield and analyzed for adsorption (iodine number).

<u>Activation</u>

Nineteen activation runs were performed using the small-scale activation unit. The initial twelve runs were shake down and commissioning experiments using hardwood char produced from a commercial RTP plant (commercial hardwood char has been successfully activated to meet commercial activated carbon specifications). The remaining runs were performed on by-product char, produced from small scale RTP processing of planar shavings and forest residue feedstocks. The by-product char samples originally produced from RTP, contained large concentrations of sand and had to be floated to remove sand prior to activation. Char sample characteristics and activation results are summarized in the tables below.

**Table 11 - "As Is" Char (Before Activation)**

| Sample ID | Yield (g) | Ash Content (wt%) | Bulk Density (g/mL) |
|---|---|---|---|
| **R84a** (ECN 308) Forest Residue | 81.3672 | 6.42 | 0.1986 |
| **R85a** (ECN 287) Mill Residue | 122.6877 | 5.94 | 0.1759 |
| **R86a** (ECN 308) Forest Residue | 177.6026 | 6.57 | 0.2118 |
| **R87a** (ECN 287) Mill Residue | 187.7374 | 10.04 | 0.1731 |
| **Hardwood Char** (ECN 222) | ~ 500 | 8.85 | 0.2276 |

**Table 12 - Char (Activated)**

| Sample ID | Run Date | Yield (g) | Yield (%) | Iodine Number (mg/g) |
|---|---|---|---|---|
| MACU – 1<br>ECN 222 Hardwood | May 7, 2003 | 12.0 | 48.0 | 379.19 |
| MACU – 2<br>ECN 222 Hardwood | May 8, 2003 | 16.5 | 34.2 | 288 |
| MACU – 3<br>ECN 222 Hardwood | May 9, 2003 | 7.67 | 30.7 | 282.55 |
| MACU – 4<br>ECN 222 Hardwood | May 12, 2003 | 1.41 | 5.6 | Not enough sample. |
| MACU – 5<br>ECN 222 Hardwood | May 13, 2003 | 5.5 | 22.0 | 302.24 |
| MACU – 6<br>ECN 222 Hardwood | May 15, 2003 | 11.5 | 45.3 | 247.28 |
| MACU – 7<br>ECN 222 Hardwood | May 20, 2003 | 9.0 | 60.0 | 473.44 |
| MACU – 8<br>ECN 222 Hardwood | May 22, 2003 | - | - | - |
| MACU – 9<br>ECN 222 Hardwood | May 23, 2003 | 2.0 | 19.0 | 286.12 |
| MACU – 10<br>ECN 222 Hardwood | May 26, 2003 | 4.3 | 30.3 | Top – 702.17<br>Middle – 788.99<br>Bottom – Not enough sample |
| MACU – 11<br>ECN 222 Hardwood | May 27, 2003 | 6.8 | 45.9 | Top (Fine) – 677.73<br>Top (Coarse) – 557.62<br>2nd Stage (Fine) – 648.57<br>Middle (Med.) – 653.56 |
| MACU – 12<br>ECN 222 Hardwood | May 28, 2003 | 4.6 | 30.4 | Top (Fine) – 653.86<br>Bottom (Coarse) – 531.39 |
| MACU – 13<br>ECN 287 (R85+R87) | June 3, 2003 | 3.46 | 24.0 | 352.57 |
| MACU – 14<br>ECN 287 (R85+R87) | June 4, 2003 | 2.71 | 18.4 | 665.17 |
| MACU – 15<br>ECN 287 (R85+R87) | June 9, 2003 | 3.42 | 22.3 | 896.37 |
| MACU – 16<br>ECN 308 (R84+R86) | June 10, 2003 | 4.12 | 27.2 | 714.95 |
| MACU – 17<br>ECN 308 (R84+R86) | June 16, 2003 | 3.01 | 20.1 | 930.42 |
| MACU – 18<br>ECN 308 (R84+R86) | June 18, 2003 | 2.87 | 19.1 | 895.46 |
| MACU – 19<br>ECN 308 (R84+R86) | June 19, 2003 | 2.38 | 15.9 | 946.97 |

## Conclusion

A small-scale activation unit was successfully designed, constructed and tested. By-product char produced from the RTP processing of planar shavings and forest residues was successfully activated, achieving iodine numbers of approximately 900 and yields of 22 wt%. Further optimization of yield and iodine number should be performed at a slightly larger scale.

# APPENDIX B

Product Characterizations at NREL

**Analysis of Biomass Feedstocks and NR Products.**

The work performed by the National Renewable Energy Laboratory (NREL) for this project has four different components.

Analysis of Natural Resin (NR) samples produced by Ensyn.
Analysis of the effects of temperature on composition of bark pyrolysis products.
Analysis of the effects of temperature on composition of mill and forest residue pyrolysis products.
Identifying tools for process monitoring of the biomass feedstocks.

**Analysis of NR samples produced by Ensyn**

This was a critical task since it involved identification of the preferred NR materials that were carried into resin development work. The scientific literature clearly documents the chemical features that will impact the formation of a crosslinked phenolic resin network. These features are the ratio of reactive phenolic sites to formaldehyde reactive sites, the number of reactive sites on the average phenolic molecules, and the number of monofunctional (only one reactive site) phenolic molecules. The chemical features dictate the polymerization reaction and the characteristics of the fully crosslinked network.

These crosslinking reactions can be modeled based on simple statistical approaches. One of the first approaches for predicting gelation or the gel point of complex mixtures was developed by Flory and Stockmayer (see equation 1).

$$X_n = \frac{f(1 - \rho + 1/r) + 2\rho}{f(1 - \rho + 1/r - 2p) + 2\rho} \qquad \text{Equation 1}$$

where

$X_n$ = number average degree of polymerization
f = average functionality of the system or the sum of all phenolic sites divided by the number of reactive molecules.
$\rho$ = ratio of nonbranching phenolic reactive sites to the total number of phenolic reactive sites
r = ratio of phenol reactive sites to formaldehyde reactive sites
p = extent of reaction which is related to reaction time

Equation 1 can be used to predict increases in the degree of polymerization (or molecular weight) as the extent of reaction (or reaction time) increases. In an industrial environment this equation is expressed in terms of reaction time and solution viscosity. It predicts the well-known induction period with little increase in molecular weight or viscosity until a critical point is reached where the degree of polymerization or molecular weight raises rapidly. It also shows how changing the ratio of reactive groups, the number of reactive sites on a single phenolic molecule, and the concentration of nonbranching phenolic molecules can affect the polymerization reactions that lead to gelation.

Controlling the ratio of the reactants (in this case the ratio of reactive phenolic sites to reactive formaldehyde sites) in a commercial resin is relatively simple and requires controlling the weight of the two reactants. In a complex mixture like NR, it is very difficult to directly measure the number of reactive sites on the phenolic rings so controlling the ratio of the reactants is more difficult. The number of reactive sites can be estimated by measuring the number of aromatic rings with a phenolic hydroxyl and the number of methoxyl or alky substituents on the aromatic rings.

The number of reactive sites on a single phenolic molecule, i.e., the “functionality” of that molecule, can also have a dramatic impact on the gel point. Increasing the functionality from phenol (f=3) to bisphenol A (f=4) to trisphenol (f=6) decreases the reaction time by about 15%. If the PF resins are made with phenolics that have higher functionalities, e.g., NR, these changes in functionality will reduce the cooking time needed to reach the gel point. Equation 1 suggests that adding 33 mol% of difunctional molecules, such as a methoxylated phenolic, to a standard PF resin formulation will increase the extent of reaction or the time to reach the gel point by about 5-10%.

Finally, equation 1 considers the impact of adding non-branching phenolics, which include phenolics with two or even one reactive site, to the reaction mixture. Phenolics with two reactive sites will extend the molecular weight between the branch points but will not contribute to a crosslinked network. Monofunctional molecules will terminate the growing chain end, and are particularly important because they will create a chain end or “defect” in the resin network. Although monofunctional molecules have a disastrous impact on the formation of high molecular weight linear polymers, they have much less effect on crosslinked resins. Although adding nonbranching molecules increases the extent of reaction or time required to reach the gel point, the increase is only 2%-3% for every 10 mol% of monofunctional molecules added to the reaction.

Based on these fundamental considerations a series of NR samples produced by Ensyn were analyzed to determine their functionality.

Table 1. Samples of natural Resin (NR) supplied by Ensyn.

| Species | Sample Name | Pyrolysis Temperature | NR Secondary Process |
|---|---|---|---|
| Aspen | ASP-500 | 500 | Standard |
| Aspen | ASP-550 | 550 | Standard |
| Aspen | ASP-550b | 550 | Standard |
| Aspen | ASP-600 | 600 | Standard |
| Aspen | ASP-550-LA | 550 | Low Acid |
| Aspen | ASP550-LMW | 550 | Low Molecular Weight |
| Spruce, Pine, Fir (SPF) | SPF-550 | 550 | Standard |
| Spruce, Pine, Fir (SPF) | SPF-600 | 600 | Standard |
| Southern Yellow Pine (SYP) | SYP-500 | 500 | Standard |
| Southern Yellow Pine (SYP) | SYP-550 | 550 | Standard |
| Southern Yellow Pine (SYP) | SYP-550b | 550 | Standard |
| Southern Yellow Pine (SYP) | SYP-600 | 600 | Standard |
| Southern Yellow Pine (SYP) | SYP-550-LA | 550 | Low Acid |

Table 2. Functionality of phenolic fragments measured with GC-MS.

| Sample Name | Pyrolysis Temperature | From GC-MS Analysis (area %) | | | | | |
|---|---|---|---|---|---|---|---|
| | | f>3 | f=2 | f=1 | f-0 | Methoxyl Content (% total phenolics) | Carbohydrates |
| ASP-500 | 500 | 2.0 | 4.3 | 18.3 | 7.4 | 21.3 | 10.4 |
| ASP-550 | 550 | 12.1 | 12.6 | 14.5 | 6.8 | 12.4 | 13.6 |
| ASP-550b | 550 | 12.3 | 7.8 | 16.6 | 7.3 | 14.4 | 7.0 |
| ASP-600 | 600 | 22.1 | 17.7 | 6.3 | 7.0 | 3.4 | 7.9 |
| ASP-550-LA | 550 | 10.9 | 9.4 | 16.7 | 7.1 | 16.0 | 1.8 |
| ASP550-LMW | 550 | 12.1 | 10.0 | 16.5 | 5.7 | 14.5 | 1.5 |
| SPF-550 | 550 | 16.8 | 2.9 | 4.6 | 2.9 | 4.6 | 33.5 |
| SPF-600 | 600 | 20.9 | 1.8 | 0 | 1.5 | 0 | 38.0 |
| SYP-500 | 500 | 15.8 | 0 | 8.1 | 1.2 | 6.2 | 50.4 |
| SYP-550 | 550 | 9.1 | 0 | 6.2 | 1.0 | 4.5 | 57.3 |
| SYP-550b | 550 | 4.5 | 0.7 | 8.4 | 1.5 | 7.1 | 35.8 |
| SYP-600 | 600 | 16.7 | 1.9 | 0 | 1.2 | 0 | 55.3 |
| SYP-550-LA | 550 | 25.4 | 3.5 | 18.0 | 1.9 | 13.9 | 25.9 |

The samples are listed in Table 1. They include three bark sources, Aspen, Southern Pine and Mixed Spruce, Pine, Fir, and three pyrolysis processing temperatures. There is a secondary process used to convert the raw pyrolysis oil from a liquid to a solid. This secondary processing can be conducted in several ways including routes that produce a low acid NR and routes that produce a low molecular weight NR.

Table 3. Functionality of phenolic fragments measured with NMR.

| Sample Name | Pyrolysis Temperature | | | | |
|---|---|---|---|---|---|
| | | Number of free sites per phenolic | Waxes | Methoxyl Content (% total phenolics) | Carbohydrates |
| ASP-500 | 500 | 2.5 | Medium | High | Medium |
| ASP-550 | 550 | 2.7 | Medium | High | Medium |
| ASP-550b | 550 | 2.7 | Medium | High | Medium |
| ASP-600 | 600 | 2.6 | Medium | High | Medium |
| ASP-550-LA | 550 | 2.7 | High | High | Low |
| ASP550-LMW | 550 | 2.7 | High | High | Low |
| SPF-550 | 550 | 2.4 | High | - | High |
| SPF-600 | 600 | 2.0 | High | Low | High |
| SYP-500 | 500 | 2.4 | Medium | Low | High |

| | | | | | |
|---|---|---|---|---|---|
| SYP-550 | 550 | 2.5 | Low | Low | High |
| SYP-550b | 550 | 2.3 | Low | Low | High |
| SYP-600 | 600 | 2.3 | Low | Low | High |
| SYP-550-LA | 550 | 2.4 | Medium | Low | High |

All of the samples supplied by Ensyn were subjected to chemical analyses with Gas Chromatography-Mass Spectroscopy (GC-MS), Nuclear Magnetic Spectroscopy (NMR) and Gel Permeation Chromatography (GPC). In combination these tools provide information of the number of reactive sites on the average phenolic ring and the number of non-branching phenolic rings, e.g., difunctional and monofunctional molecules. These results are summarized in Table 2. These trends are shown in Figure 1. There is a clear trend towards increasing functionality with increasing pyrolysis temperature. This trend is beneficial in terms of the chemical composition of the oils. But at the same time there is a decrease in the over yield of the NR with increasing temperature, which will increase the cost of the NR. The softwood barks have a relatively high concentration of trifunctional and difunctional phenolics, which is expected given their moderate levels of tannins and polyphenolic extractives.

The molecular weight and molecular weight distribution will also have a significant impact on the performance of the NRs. Specifically, increasing the molecular weight or molecular weight distribution will increase the number of individual phenolic molecules that have high functionality, which will decrease the gel-time. These effects are particularly important for the highest molecular weight components of the distribution. Thus the molecular weight and molecular weight distribution of the NRs were measured with GPC. The results of these tests are shown in Table 3. Both the number average molecular weight and the weight average molecular weight are very similar for all of the NR samples. The higher molecular weight tail (Z-average molecular weight) might show more of a trend of decreasing molecular weight with increasing temperature, but these results suggest that the molecular weight distribution of the processed NR is relatively insensitive to the original pyrolysis temperature.

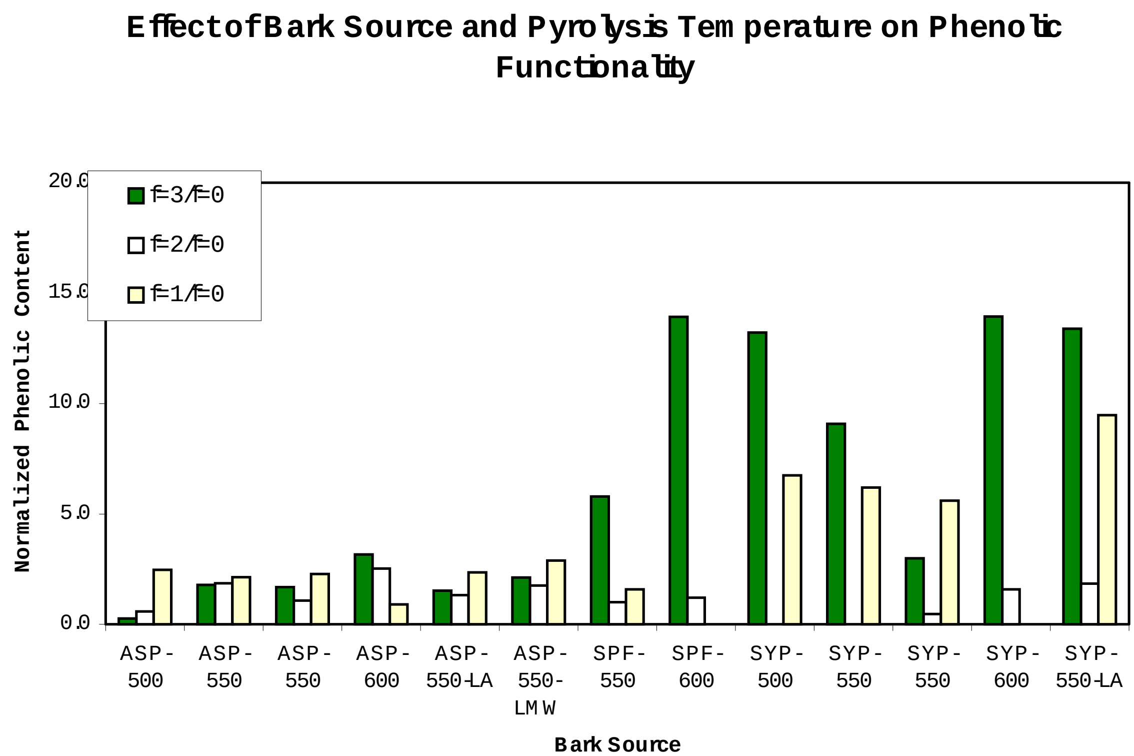



Figure 1. Relative functionality of NR samples prepared from bark.

Table 4. Molecular weight of NR.

| Sample Name | Pyrolysis Temperature | Number Ave MW | Weight Ave MW | Particulates |
|---|---|---|---|---|
| ASP-500 | 500 | 360 | 790 | Fine solids remaining (char) |
| ASP-550 | 550 | 330 | 610 | Fine solids remaining (char) |
| ASP-550b | 550 | 330 | 610 | Completely dissolved |
| ASP-600 | 600 | 320 | 580 | Completely dissolved |
| ASP-550-LA | 550 | 340 | 640 | Completely dissolved |
| ASP550-LMW | 550 | 330 | 620 | Completely dissolved |
| SPF-550 | 550 | 340 | 560 | Fine solids remaining (char) |
| SPF-550 (rep) | 550 | 340 | 550 | Fine solids remaining (char) |
| SPF-550 (rep) | 550 | 340 | 560 | Fine solids remaining (char) |
| SPF-600 | 600 | 330 | 510 | Fine solids remaining (char) |
| SYP-500 | 500 | 350 | 540 | Completely dissolved |
| SYP-550 | 550 | 360 | 540 | Completely dissolved |
| SYP-550b | 550 | 380 | 580 | Fine solids remaining (char) |
| SYP-600 | 600 | 330 | 490 | Completely dissolved |
| SYP-550-LA | 550 | 350 | 520 | Fine solids remaining (char) |

**Analysis of the effects of temperature on composition of bark pyrolysis products.**

To better understand the effects of pyrolysis temperature on the chemical composition of bark pyrolysis products we conducted a series of tests at different temperatures. The three bark sources, Aspen, Southern Pine and Mixed Spruce, Pine Fir, were all subjected to pyrolysis at a series of different temperatures and the pyrolysis products were monitored with molecular beam mass spectrometry (MBMS). Examples of the py-MBMS results are shown in Figure 2. The fragmentation patterns are very complex but specific peaks can be assigned to individual chemical components, specifically the phenolic fragments that are the based on the NRs.

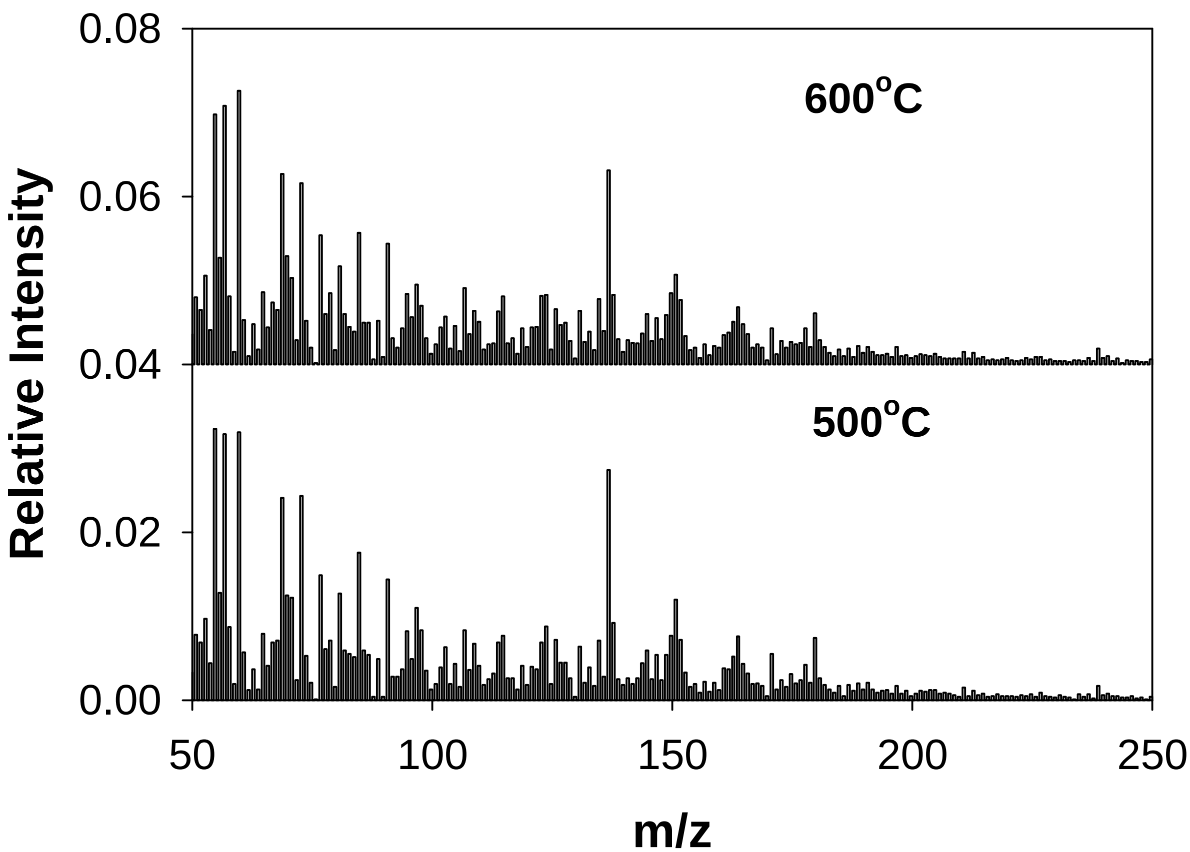



Figure2. py-MBMS fragmentation spectra from the pyrolysis of Aspen bark at different temperatures

The effects of the initial pyrolysis temperature on the composition of the pyrolysis oils can be modeled with Projection to Latent Structures (PLS) modeling. In this case the PLS modeling allows one to draw correlations between the pyrolysis temperature and the fragmentation pattern of the pyrolysis vapors. This correlation is shown in Figure 3. There is a strong correlation (r=0.96) between the experimental temperature and the temperature indicated from the fragmentation patterns. The specific chemical fragments that are driving this correlation are shown in Figure 4. This figure shows that masses 94, 110 and 124 are increasing and masses 137, 154 and 194 are decreasing as the initial pyrolysis temperature increases. These results are consistent with an increase in the relative concentration of phenol, catechol and guaiacol and a loss in the relative concentration of lignin dimers as the temperature increases. These changes in the suite of phenolics are consistent across all three bark sources.

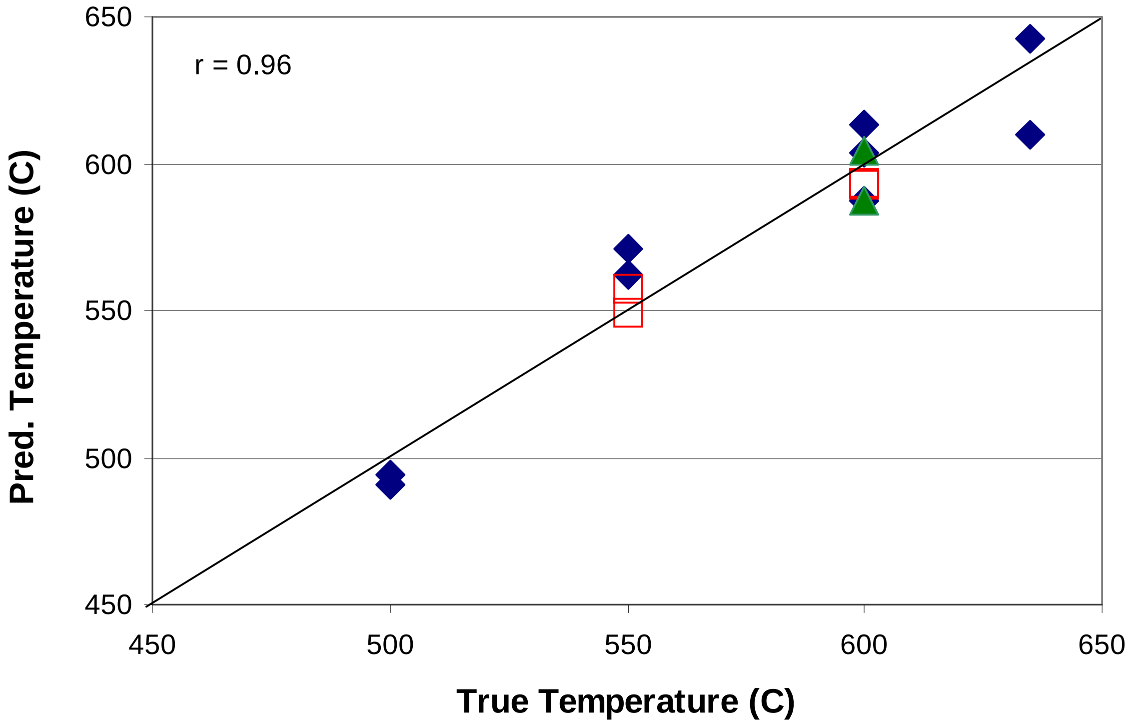



Figure 3. PLS model of NRs produced at different temperatures showing that the effects of temperature are similar for all three bark sources

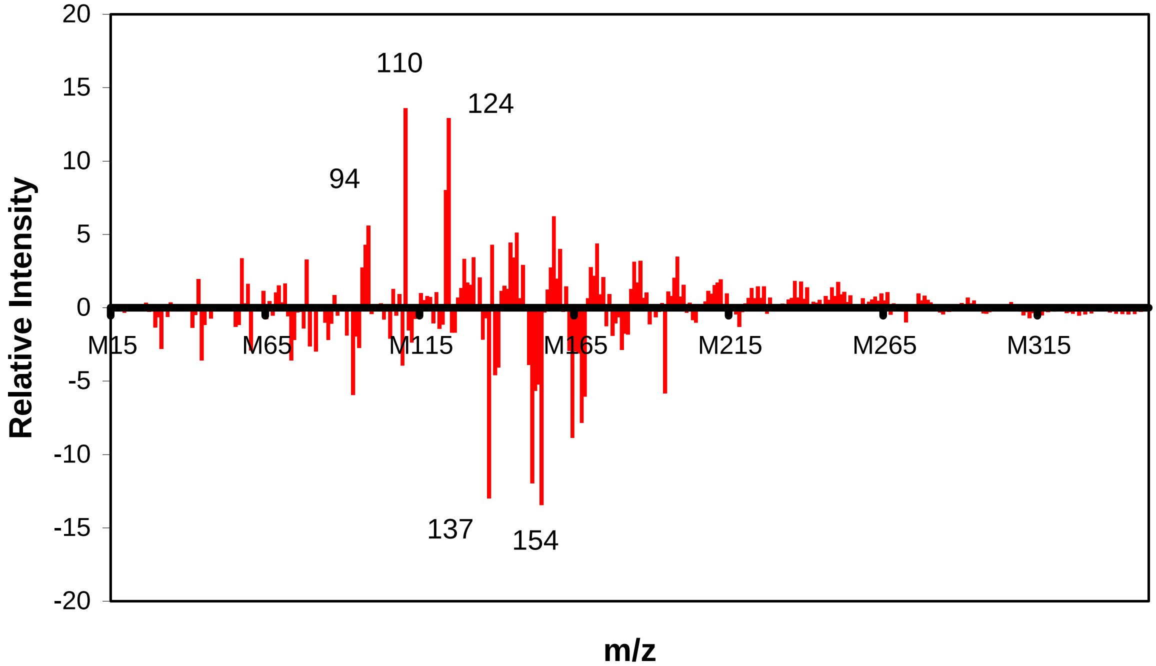



Figure 4. Regression coefficients for the PLS model of NRs produced at different temperatures showing the chemical changes as a function of NR production temperature.

While the effects of temperature on the composition of phenolics is clear, as expected there are still significant differences in the composition of the pyrolysis oils for the different bark feedstocks. These difference are highlighted in Figure 5. The differences between the different sources of bark are much greater than the effects of temperature.

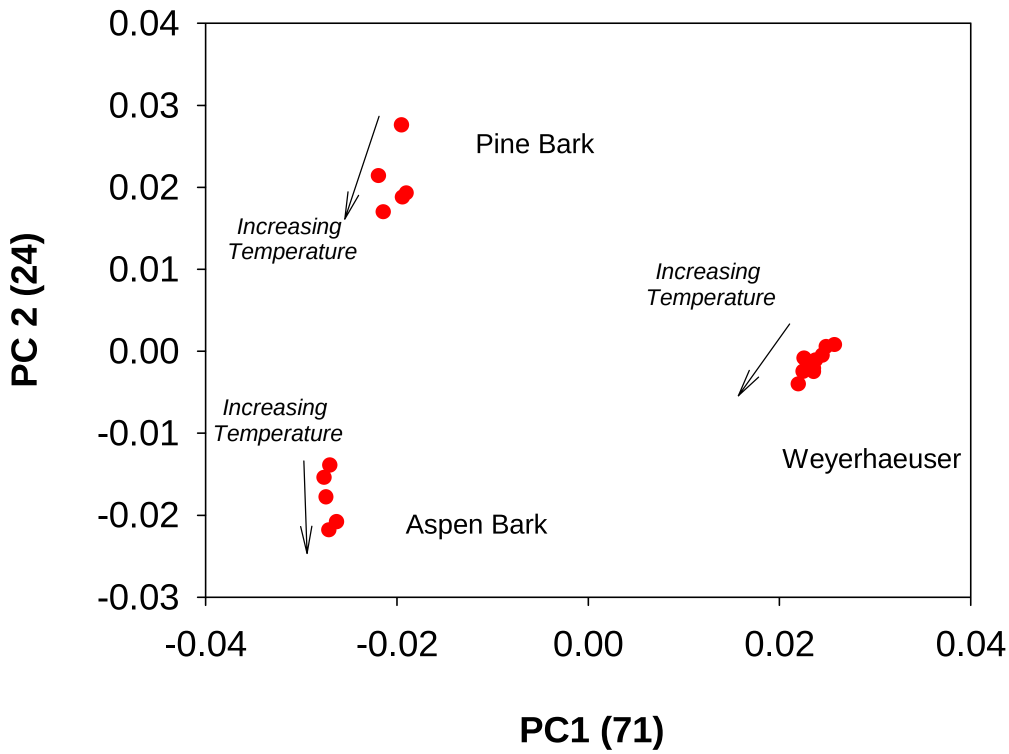



Figure 5. Results of PCA model of the different bark and wood feedstocks highlighting the significant chemical differences between the feedstocks.

**Analysis of the effects of temperature on composition of mill and forest residue pyrolysis products.**

Two analytical tools, py-MBMS and NIR spectroscopy, were used to study the Weyerhaeuser "forest residue" and "planar shavings" samples.

As expected the analysis with py-MBMS showed that there were significant differences in the chemical composition of the pyrolysis vapors based on both the source of the biomass feedstock and the pyrolysis temperature. These py-MBMS experiments were run at temperatures between 500 and 600$^{o}$C and measured the chemical composition of the vapors produced when the biomass was pyrolyzed at these different temperatures.

The effects of the biomass feedstock and pyrolysis temperature are shown in Figure 6 and 7. Figure 6 shows the results of PCA of py-MBMS analysis of different biomass feedstock, e.g., Aspen bark, Pine bark, "forest residue" and "planar shavings". The first PCA is based on chemical species with a nominal mass (mass to charge ratio) between 50 and 310 amu. These results show that forest residue and planar shavings are very different than the bark samples and similar to one another. When compared to the bark samples the forest residue and planar shavings appear similar, but when the bark samples are removed from the analysis there are subtle differences between the forest residue and planar shavings. Differences between the forest residue and planar shavings are highlighted when the PCA is run so that it only focused on the phenolic fragments (Figure 7). Based on this analysis there was clear separation between the different feedstocks. This result suggests that the yield and quality of the phenolic fraction that makes up the NR will be sensitive to variations in the source of the biomass feedstock, and that the two feedstocks are **not** interchangeable. The differences between the results shown in Figure 6 and Figure 7 indicate that the main differences between the two feedstocks are in their carbohydrate and extractive fractions.

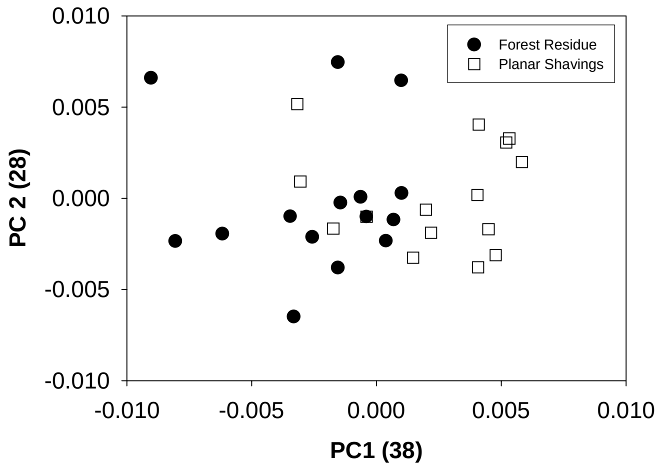



Figure 6. Results of PCA showing the overall chemical similarity of mill and forest residues.

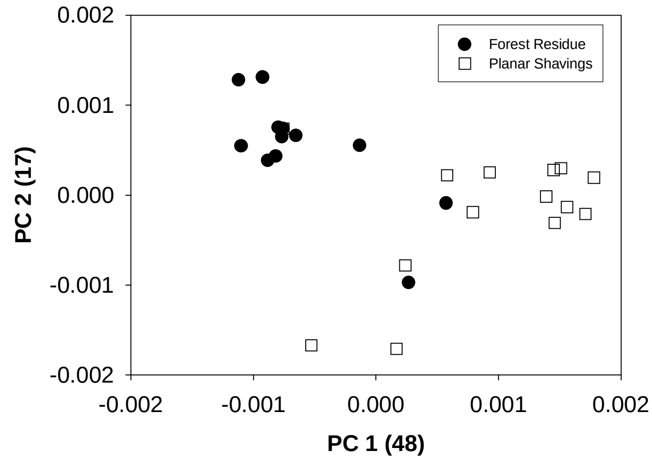



Figure 7. Results of PCA showing the differences in the phenolic component of mill and forest residues.

To better understand the effects of different biomass feedstocks the results of the py-MBMS analysis on the forest residue and planar shavings were combined with a prior study on wood bark samples supplied early in the project by LP. The results of this analysis are shown in Figure 8. There are dramatic differences between the two bark samples and the Weyerhaeuser samples, highlighting the importance of feedstock source.

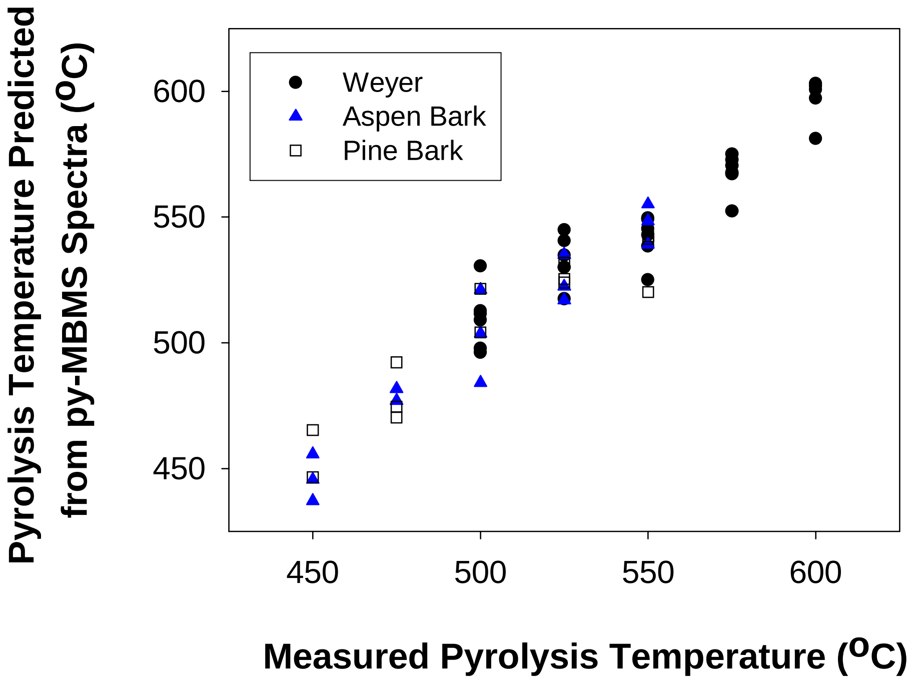



Figure 8. PLS model showing the effects of temperature on the composition of bark and residue feedstocks.

There are clear differences in the chemical composition of the vapors produced at different temperature as seen in Figure 8. These results show that over a 150°C temperature range all four sets of samples, e.g., Aspen bark, Pine bark, forest residue and planar shavings, exhibit similar changes in their chemical composition. These changes are related to a loss of carbohydrate, a corresponding increase in phenolics and cracking of the lignin-based phenolics to lower molecular weight compounds.

The results from the py-MBMS analysis clearly show that composition of the feedstock will have a significant impact on the chemical composition of the NR. Based on the importance of knowing the composition of the feedstock we conducted a brief series of experiments to demonstrate that NIR could be used to measure the composition of mixtures of forest residue and planar shavings. While several spectroscopic techniques could be used to measure the composition of these types of mixtures NIR is particularly attractive since it can be used as an online process control system.

**Identifying tools for process monitoring of the biomass feedstocks**

The results of this NIR screening study are shown in Figures 9 and 10. Figure 9 shows results of a PCA of the spectra gathered from the samples “as received” and samples ground to the same particle size. As expected, due to differences in specular scattering samples with the different particle size are clearly differentiated, and the actual composition of the mixture is less important. Nevertheless, both sets of samples are used to construct high quality PLS models of the type that would be used to measure the composition of mixtures flowing into a process (Figure 10.) The results shown in Figure 10 show that the composition of a mixture of “forest residue” and “planar shavings" can be measured with NIR. With this small sample size the standard deviation of the measurement is 5%, but it is likely that this standard deviation would be reduced with a more robust sample set.

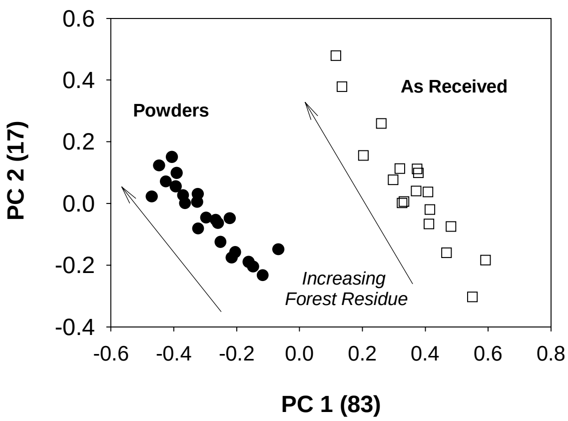



Figure 9. Results of PCA of NIR spectra showing differences between the forest residues and planar shavings.

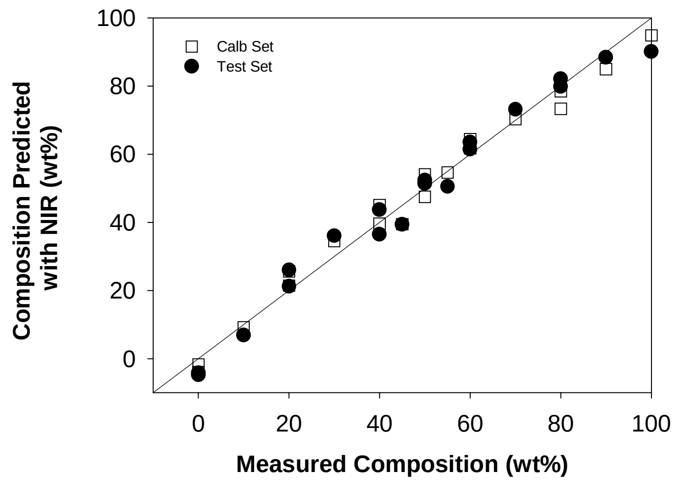



Figure 10. Results of PLS model showing the accuracy of NIR for measuring the composition of forest and mill residues in a feedstock.

# APPENDIX C

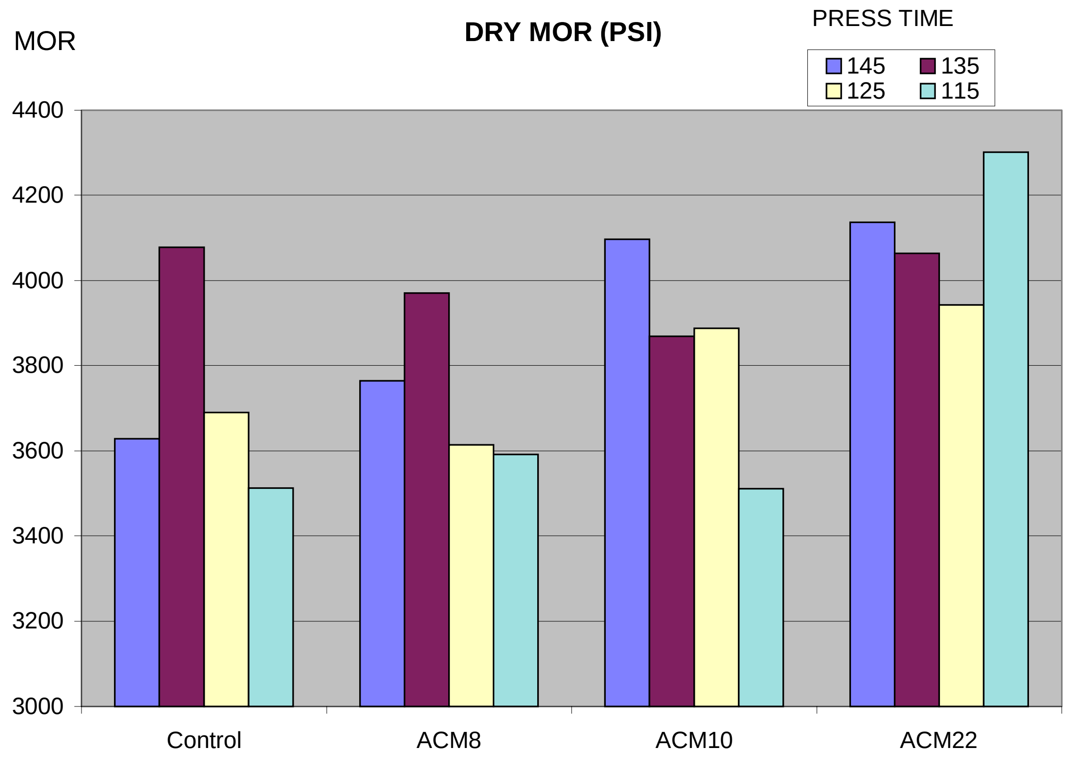
Properties Report for Bark Derived Resin by L-P

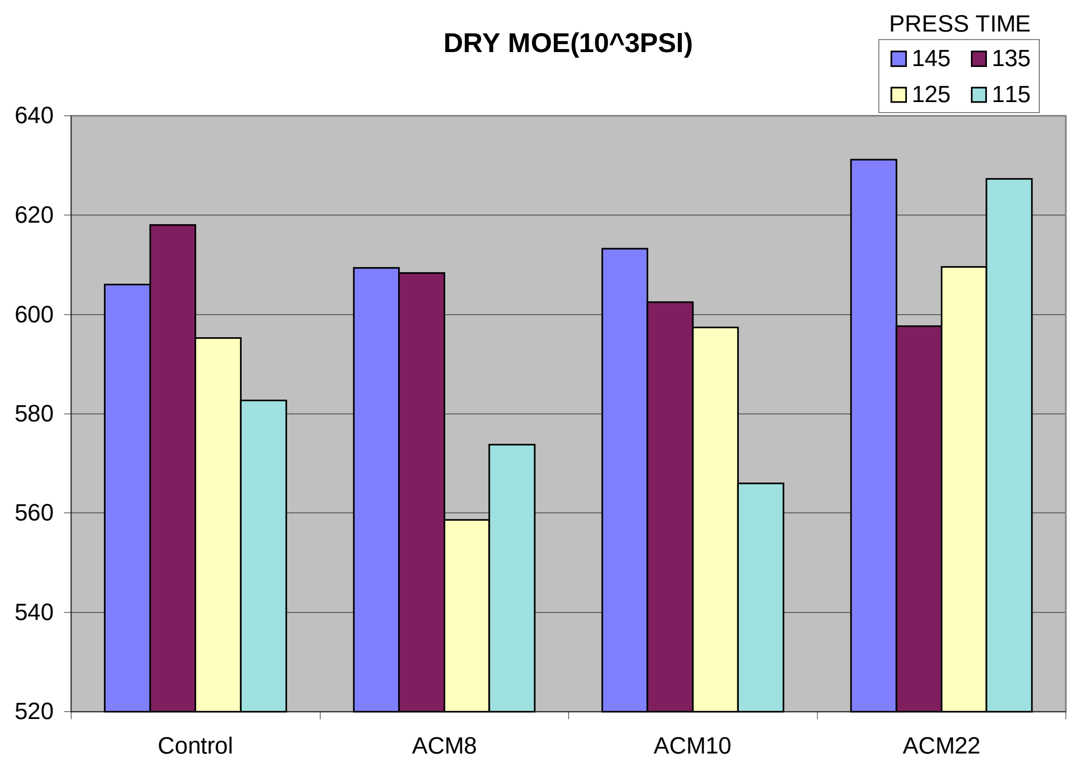
## Properties Report for Bark Derived Resin

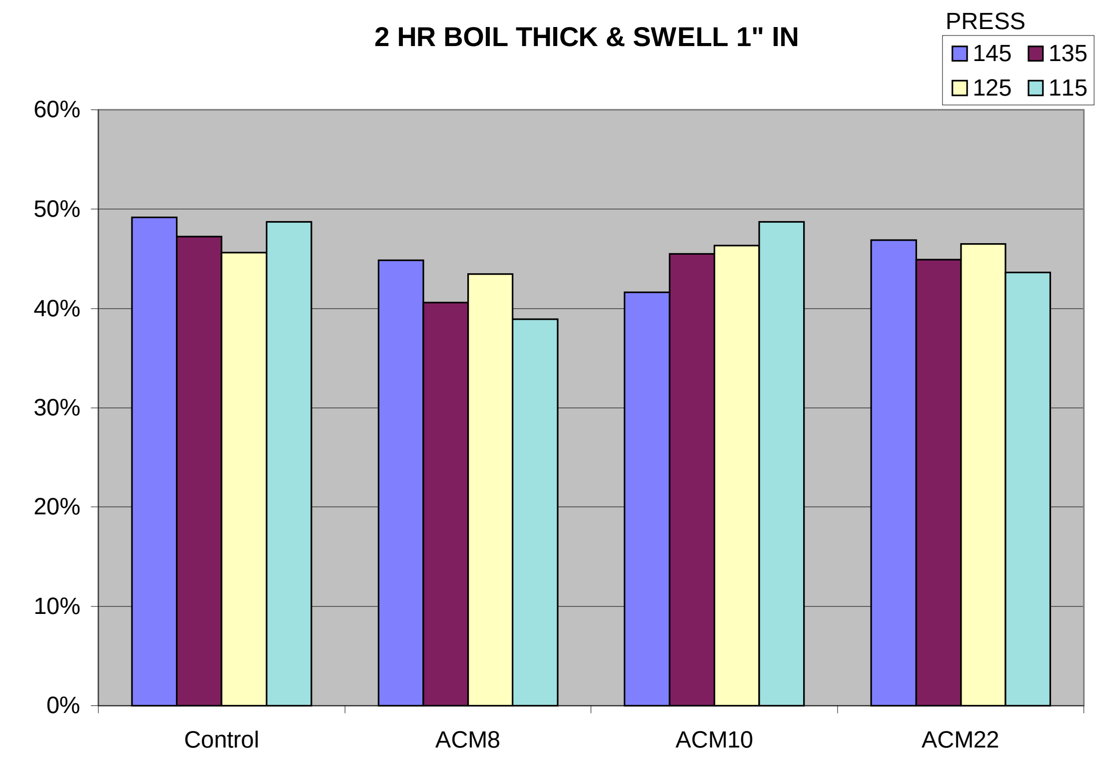
**Control -** 3% LPF (Borden OS745)
**ACM08 -** 3% Bark resin (ACM 2508) 50% subst. SPF based resin
**ACM10 -** 3% Bark resin (ACM 2510) 40% subst. SPF based resin
**ACM22 -** 3% Bark resin (ACM 2522) 40% subst. Aspen based resin

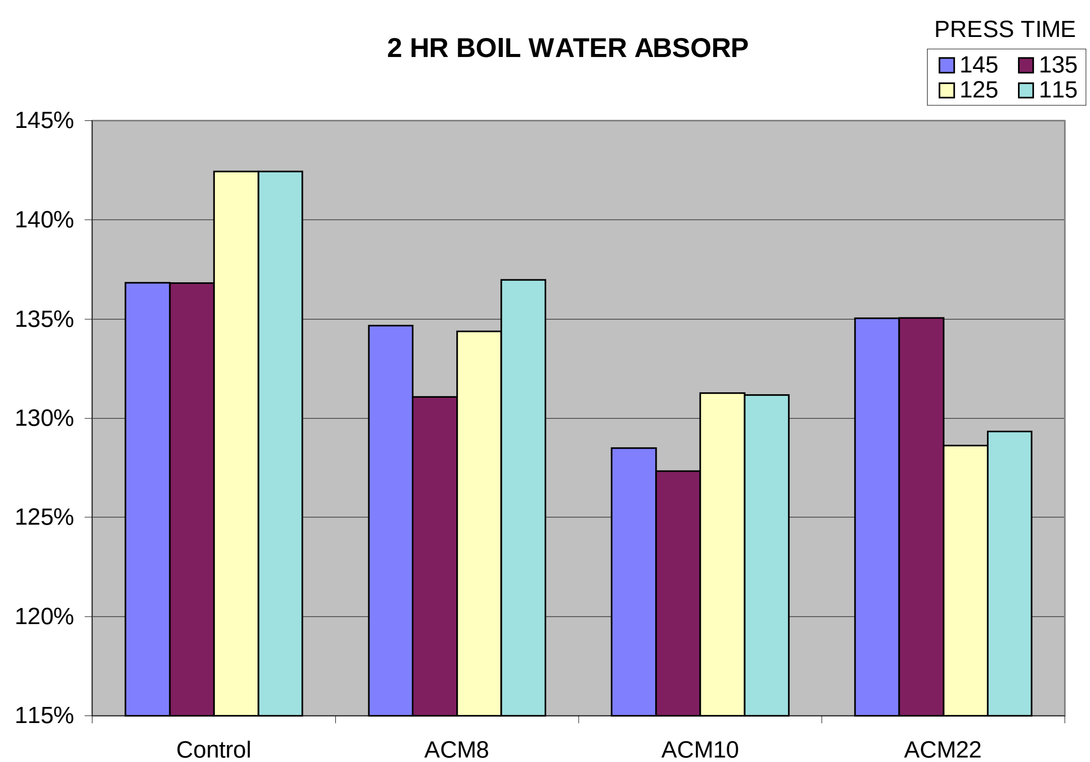
Panels utilized 1% wax (EW58-S) throughout panel and 2% MDI (R1840) in the core
Board orientation was 25:50:25 (random:random:random)
Target Thickness = 0.437   Target Density = 38 lb/ft^3

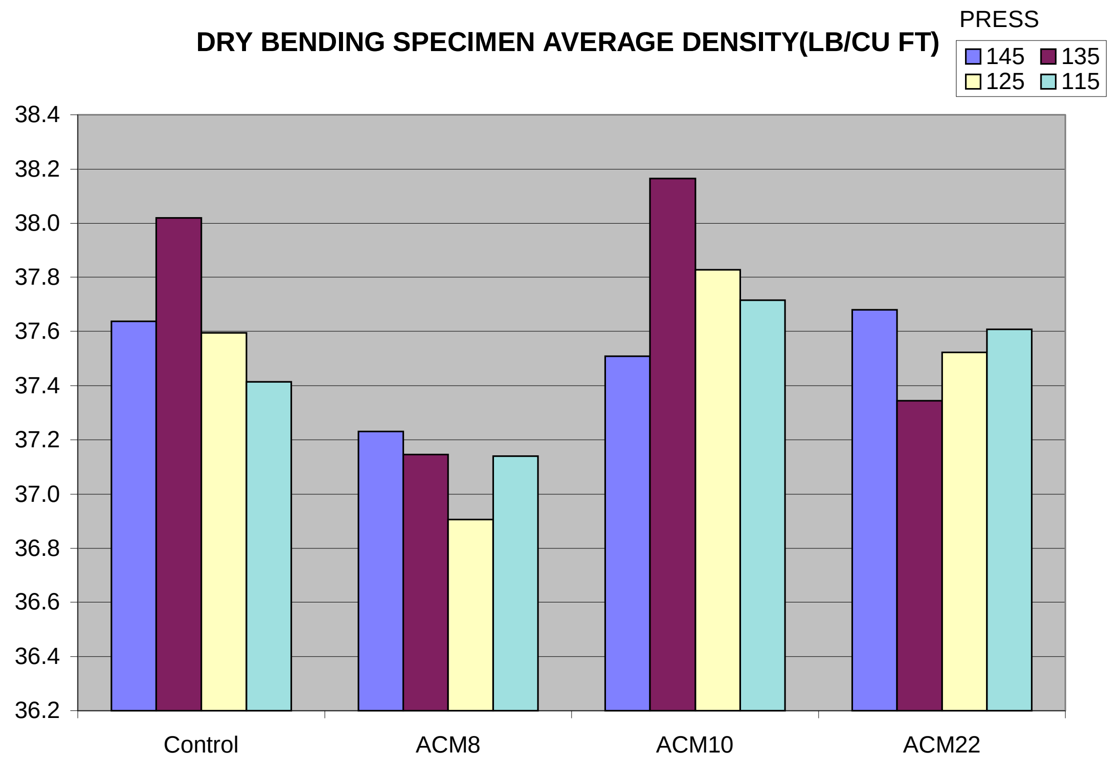
| | | Control | | ACM8 | | ACM10 | | ACM22 | |
|---|---|---|---|---|---|---|---|---|---|
| **Properties** | # | Average | Std Dev | Average | Std Dev | Average | Std Dev | Average | Std Dev |
| Dry MOR (psi) | 145 | 3628 | 508.6 | 3764 | 638.7 | 4097 | 741.1 | 4137 | 821.2 |
| | 135 | 4078 | 716.3 | 3970 | 475.0 | 3869 | 882.4 | 4064 | 883.3 |
| | 125 | 3690 | 520.6 | 3614 | 644.1 | 3888 | 629.3 | 3943 | 680.8 |
| | 115 | 3512 | 518.9 | 3591 | 677.4 | 3511 | 255.5 | 4301 | 274.2 |
| Dry MOE (10^3 psi) | 145 | 606.0 | 44.7 | 609.4 | 87.3 | 613.2 | 71.0 | 631.2 | 81.9 |
| | 135 | 618.0 | 66.8 | 608.3 | 74.4 | 602.5 | 53.8 | 597.6 | 87.7 |
| | 125 | 595.3 | 61.6 | 558.7 | 78.1 | 597.4 | 59.9 | 609.6 | 74.2 |
| | 115 | 582.7 | 73.2 | 573.8 | 50.5 | 566.0 | 21.0 | 627.3 | 35.6 |
| Dry MM (lb-in/ft) | 145 | 1501 | 229 | 1597 | 273.8 | 1692 | 308.6 | 1696 | 345.1 |
| | 135 | 1712 | 354.5 | 1700 | 176.1 | 1598 | 406.3 | 1679 | 362.8 |
| | 125 | 1560 | 273.8 | 1558 | 295.1 | 1613 | 248.8 | 1622 | 301.4 |
| | 115 | 1475 | 224.4 | 1569 | 307.7 | 1462 | 80.4 | 1783 | 116.5 |
| Dry EI (lb-in$^2$/ft) | 145 | 57014 | 5837 | 59523 | 8537 | 57551 | 6615 | 58706 | 9153 |
| | 135 | 59247 | 9304 | 60321 | 6030 | 56320 | 7371 | 56038 | 7498 |
| | 125 | 57825 | 9251 | 55910 | 8897 | 56471 | 5087 | 56660 | 6324 |
| | 115 | 55987 | 6313 | 58597 | 6589 | 53928 | 3291 | 59266 | 4167 |
| 2 Hour Boil MOR (psi) | 145 | 1829 | 319.4 | 1881 | 257.7 | 1938 | 380.2 | 2058 | 424.4 |
| | 135 | 1973 | 215.7 | 1767 | 222.7 | 1968 | 347.0 | 1947 | 283.4 |
| | 125 | 1973 | 367.4 | 1913 | 276.0 | 1924 | 214.0 | 2086 | 336.4 |
| | 115 | 1801 | 245.6 | 1829 | 361.8 | 1986 | 275.2 | 2027 | 259.2 |
| 2 Hour Boil MM (lb-in/ft) | 145 | 757.6 | 119.4 | 792.1 | 108.1 | 790.5 | 140.7 | 848.4 | 185.6 |
| | 135 | 825.4 | 77.54 | 756.3 | 94.18 | 799.1 | 151.8 | 802.5 | 114.0 |
| | 125 | 825.5 | 169.3 | 814 | 128.10 | 794.5 | 85.3 | 858.9 | 143.6 |
| | 115 | 746.8 | 91.21 | 800.6 | 171.2 | 816.1 | 120.80 | 850.2 | 103.1 |
| 24 hr Water Absorp (%) | 145 | | | 21.21% | 1.81% | 19.43% | 1.49% | 22.14% | 2.24% |
| | 135 | | | 19.19% | 1.84% | 19.99% | 0.38% | 21.04% | 1.27% |
| | 125 | | | 19.45% | 0.63% | 22.48% | 1.45% | 20.66% | 1.94% |
| | 115 | | | 19.89% | 2.49% | 25.92% | 6.49% | 22.55% | 5.30% |
| 24 hr Thick Swell (%) 1" in | 145 | | | 9.91% | 1.09% | 10.06% | 0.37% | 10.27% | 0.72% |
| | 135 | | | 9.87% | 0.60% | 10.74% | 0.20% | 10.15% | 1.06% |
| | 125 | | | 9.76% | 0.34% | 10.55% | 0.60% | 9.99% | 0.71% |
| | 115 | | | 9.64% | 0.76% | 11.65% | 1.41% | 9.52% | 1.35% |
| 24 hr Thick Swell (%) Edge | 145 | | | 21.14% | 1.10% | 20.28% | 1.41% | 22.10% | 1.29% |
| | 135 | | | 19.90% | 1.24% | 21.19% | 1.17% | 21.77% | 0.68% |
| | 125 | | | 21.25% | 0.98% | 22.09% | 0.50% | 20.91% | 1.52% |
| | 115 | | | 19.69% | 0.94% | 25.08% | 3.06% | 20.38% | 1.52% |
| 24 hr Differential Swell (%) | 145 | | | 11.23% | 1.86% | 10.22% | 1.44% | 11.83% | 1.45% |
| | 135 | | | 10.03% | 1.26% | 10.45% | 1.26% | 11.62% | 0.91% |
| | 125 | | | 11.49% | 1.28% | 11.54% | 0.76% | 10.93% | 0.90% |
| | 115 | | | 10.05% | 1.04% | 13.43% | 1.80% | 10.86% | 0.49% |
| 2 hr Boil Water Absorp (%) | 145 | 136.83% | 4.90% | 134.66% | 4.30% | 128.49% | 5.69% | 135.03% | 4.92% |
| | 135 | 136.81% | 4.38% | 131.07% | 8.37% | 127.34% | 2.69% | 135.05% | 5.82% |
| | 125 | 142.44% | 7.64% | 134.38% | 6.07% | 131.26% | 4.87% | 128.62% | 4.72% |
| | 115 | 142.43% | 3.09% | 136.98% | 4.77% | 131.16% | 6.26% | 129.33% | 6.08% |
| 2 hr Boil Thick Swell (%) 1" in | 145 | 49.16% | 2.08% | 44.83% | 1.46% | 41.62% | 2.16% | 46.89% | 1.02% |
| | 135 | 47.24% | 2.12% | 40.58% | 2.88% | 45.50% | 2.22% | 44.91% | 3.29% |
| | 125 | 45.61% | 4.42% | 43.45% | 1.39% | 46.35% | 2.06% | 46.51% | 2.07% |
| | 115 | 48.72% | 4.02% | 38.94% | 2.17% | 48.69% | 1.83% | 43.62% | 4.07% |
| 2 hr Boil Thick Swell (%) Edge | 145 | 47.78% | 2.54% | 46.42% | 1.93% | 44.00% | 2.83% | 50.03% | 4.60% |
| | 135 | 45.02% | 2.62% | 41.84% | 2.86% | 45.86% | 3.64% | 48.00% | 1.52% |
| | 125 | 46.96% | 5.20% | 43.31% | 2.64% | 47.18% | 2.84% | 45.93% | 2.03% |
| | 115 | 49.72% | 5.65% | 41.30% | 1.99% | 48.44% | 3.71% | 46.79% | 2.87% |
| **Density (lb/ft$^3$)** | 145 | 37.6 | 1.6 | 37.2 | 1.1 | 37.5 | 0.8 | 37.7 | 1.5 |
| | 135 | 38.0 | 1.2 | 37.1 | 1.8 | 38.2 | 1.8 | 37.3 | 1.2 |
| | 125 | 37.6 | 1.4 | 36.9 | 1.5 | 37.8 | 0.8 | 37.5 | 1.0 |
| | 115 | 37.4 | 1.5 | 37.1 | 1.9 | 37.7 | 1.2 | 37.6 | 0.7 |


MOR
DRY MOR (PSI)
PRESS TIME
145
135
125
115
4400
4200
4000
3800
3600
3400
3200
3000
Control
ACM8
ACM10
ACM22


DRY MOE(10^3PSI)
PRESS TIME
145
135
125
115
640
620
600
580
560
540
520
Control
ACM8
ACM10
ACM22


2 HR BOIL THICK & SWELL 1" IN
PRESS
145
135
125
115
60%
50%
40%
30%
20%
10%
0%
Control
ACM8
ACM10
ACM22


2 HR BOIL WATER ABSORP
PRESS TIME
145
135
125
115
145%
140%
135%
130%
125%
120%
115%
Control
ACM8
ACM10
ACM22


DRY BENDING SPECIMEN AVERAGE DENSITY(LB/CU FT)
PRESS
145
135
125
115
38.4
38.2
38.0
37.8
37.6
37.4
37.2
37.0
36.8
36.6
36.4
36.2
Control
ACM8
ACM10
ACM22

# APPENDIX D

L-P Summary Report on Bark Derived Resin Formulations Reported by Dynea and GP

| Specimen (145) | | MOR | MOE | Density (lb/ft3) |
|---|---|---|---|---|
| Dynea-1 | BD1 | 2962 | 494 | 35.67 |
| | BD2 | 3423 | 540 | 35.40 |
| | BD3 | 3263 | 560 | 38.23 |
| | BD4 | 4408 | 698 | 38.48 |
| Dynea-2 | BD1 | 4560 | 698 | 39.28 |
| | BD2 | 3645 | 563 | 35.20 |
| | BD3 | 3447 | 523 | 36.43 |
| | BD4 | 2633 | 525 | 34.59 |
| Dynea-3 | BD1 | 3577 | 605 | 38.01 |
| | BD2 | 3446 | 554 | 35.04 |
| | BD3 | 3577 | 563 | 38.79 |
| | BD4 | 3609 | 547 | 35.61 |
| GP-1 | BD1 | 4217 | 720 | 38.93 |
| | BD2 | 3405 | 603 | 37.11 |
| | BD3 | 4502 | 672 | 39.03 |
| | BD4 | 3592 | 648 | 38.10 |
| GP-2 | BD1 | 3942 | 633 | 36.62 |
| | BD2 | 4586 | 662 | 39.74 |
| | BD3 | 3904 | 628 | 38.25 |
| | BD4 | 4172 | 645 | 39.31 |
| GP-3 | BD1 | 3464 | 564 | 36.59 |
| | BD2 | 3787 | 639 | 37.10 |
| | BD3 | 4788 | 726 | 40.48 |
| | BD4 | 3629 | 557 | 36.22 |
| C1 | BD1 | 4061 | 637 | 36.67 |
| | BD2 | 3217 | 551 | 38.81 |
| | BD3 | 3347 | 607 | 37.45 |
| | BD4 | 3619 | 594 | 36.22 |
| C2 | BD1 | 4349 | 695 | 40.21 |
| | BD2 | 4018 | 613 | 36.74 |
| | BD3 | 4179 | 648 | 41.10 |
| | BD4 | 3736 | 576 | 35.87 |
| C3 | BD1 | 3128 | 603 | 36.53 |
| | BD2 | 3953 | 643 | 37.68 |
| | BD3 | 3265 | 557 | 36.59 |
| | BD4 | 2661 | 547 | 37.77 |

| Specimen (135) | | MOR | MOE | Density (lb/ft3) |
|---|---|---|---|---|
| A1 | BD1 | 4116 | 637 | 39.24 |
| | BD2 | 3949 | 612 | 36.02 |
| | BD3 | 3090 | 542 | 38.25 |
| | BD4 | 2974 | 540 | 35.76 |
| A2 | BD1 | 3162 | 529 | 35.18 |
| | BD2 | 3306 | 547 | 36.79 |
| | BD3 | 3094 | 516 | 38.79 |
| | BD4 | 2548 | 526 | 35.80 |
| GP-A1 | BD1 | 4930 | 648 | 39.08 |
| | BD2 | 3712 | 589 | 38.12 |
| | BD3 | 4779 | 632 | 38.65 |
| | BD4 | 2372 | 451 | 33.34 |
| GP-A2 | BD1 | 3782 | 568 | 37.56 |
| | BD2 | 3365 | 589 | 37.63 |
| | BD3 | 3503 | 558 | 37.17 |
| | BD4 | 4161 | 599 | 38.31 |
| C | BD1 | 4885 | 687 | 39.03 |
| | BD2 | 4777 | 662 | 40.06 |
| | BD3 | 4038 | 576 | 37.40 |
| | BD4 | 2963 | 583 | 38.10 |
| C | BD1 | 4057 | 602 | 38.50 |
| | BD2 | 4029 | 658 | 36.94 |
| | BD3 | 3175 | 494 | 36.24 |
| | BD4 | 4703 | 683 | 37.44 |

| Specimen (125) | | MOR | MOE | Density (lb/ft3) |
|---|---|---|---|---|
| Dynea-B1 | BD1 | 4255 | 687 | 36.85 |
| | BD2 | 4292 | 624 | 36.85 |
| | BD3 | 3095 | 541 | 37.79 |
| | BD4 | 3028 | 538 | 35.35 |
| Dynea-B2 | BD1 | 4254 | 726 | 38.08 |
| | BD2 | 3224 | 577 | 36.79 |
| | BD3 | 3363 | 571 | 36.85 |
| | BD4 | 3426 | 561 | 34.22 |
| GP-B1 | BD1 | 3521 | 580 | 37.00 |
| | BD2 | 3861 | 644 | 36.83 |
| | BD3 | 3813 | 552 | 37.94 |
| | BD4 | 3954 | 594 | 36.93 |
| GP-B2 | BD1 | 3137 | 552 | 38.57 |
| | BD2 | 2404 | 452 | 35.52 |
| | BD3 | 3899 | 601 | 38.70 |
| | BD4 | 3482 | 591 | 36.81 |
| C-B1 | BD1 | 3251 | 560 | 38.39 |
| | BD2 | 4543 | 660 | 38.22 |
| | BD3 | 3320 | 510 | 35.86 |
| | BD4 | 3583 | 606 | 36.73 |
| C-B2 | BD1 | 4423 | 654 | 40.07 |
| | BD2 | 3705 | 660 | 38.19 |
| | BD3 | 3182 | 517 | 36.32 |
| | BD4 | 3510 | 595 | 36.96 |

| Specimen (115) | | MOR | MOE | Density (lb/ft3) |
|---|---|---|---|---|
| Dynea-C1 | BD1 | 3986 | 665 | 38.10 |
| | BD2 | 2976 | 536 | 36.84 |
| | BD3 | 2998 | 498 | 38.06 |
| | BD4 | 3880 | 662 | 37.11 |
| Dynea-C2 | BD1 | 2701 | 449 | 35.27 |
| | BD2 | 3318 | 601 | 36.05 |
| | BD3 | 3027 | 495 | 36.67 |
| | BD4 | 3719 | 583 | 37.47 |
| GP-C1 | BD1 | 3848 | 672 | 39.63 |
| | BD2 | 4070 | 629 | 36.85 |
| | BD3 | 4125 | 606 | 38.20 |
| | BD4 | 3871 | 533 | 35.91 |
| GP-C2 | BD1 | 3674 | 591 | 37.42 |
| | BD2 | 4013 | 656 | 39.00 |
| | BD3 | 3520 | 558 | 37.48 |
| | BD4 | 4511 | 693 | 37.52 |
| C-C1 | BD1 | 3930 | 625 | 39.15 |
| | BD2 | 4127 | 593 | 39.17 |
| | BD3 | 2957 | 525 | 36.05 |
| | BD4 | 3988 | 697 | 36.60 |
| C-C2 | BD1 | 3215 | 550 | 37.59 |
| | BD2 | 2905 | 496 | 35.40 |
| | BD3 | 3076 | 515 | 36.44 |
| | BD4 | 3900 | 661 | 38.90 |

**Properties Report for Bark Derived Resin**

Control - 3% LPF (Borden OS745)
Dynea - 3% Bark resin (Neste 2063-22)
GP - 3% Bark resin (GPRI RPP073G40)

All Panels utilized 1% wax (EW58-S) throughout panel and 2% MDI (R1840) in the core
Board orientation was 25:50:25 (random:random:random)
Target Thickness = 0.437 Target Density = 38 lb/ft^3

| | | Control | | DYNEA | | GP | |
|---|---|---|---|---|---|---|---|
| **Properties** | # | Average | Std Dev | Average | Std Dev | Average | Std Dev |
| **Dry MOR (psi)** | 145 | 3628 | 508.6 | 3546 | 529.0 | 3999 | 456.9 |
| | 135 | 4078 | 716.3 | 3280 | 515.4 | 3826 | 818.2 |
| | 125 | 3690 | 520.6 | 3617 | 553.3 | 3509 | 524.6 |
| | 115 | 3512 | 518.9 | 3325 | 479.1 | 3954 | 302.4 |
| **Dry MOE (10^3 psi)** | 145 | 606.0 | 44.7 | 572.6 | 64.5 | 641.5 | 51.7 |
| | 135 | 618.0 | 66.8 | 556.0 | 43.9 | 579.2 | 59.9 |
| | 125 | 595.3 | 61.6 | 603.1 | 69.9 | 570.6 | 56.3 |
| | 115 | 582.7 | 73.2 | 561.2 | 79.8 | 617.3 | 56.0 |
| **Dry MM (lb-in/ft)** | 145 | 1501 | 229 | 1503 | 237.1 | 1651 | 215.6 |
| | 135 | 1712 | 354.5 | 1393 | 221.2 | 1599 | 343.9 |
| | 125 | 1560 | 273.8 | 1546 | 249.9 | 1467 | 231.5 |
| | 115 | 1475 | 224.4 | 1433 | 216.4 | 1653 | 128.7 |
| **Dry EI ($lb\text{-}in^2/ft$)** | 145 | 57014 | 5837 | 55843 | 7035 | 60141 | 5967 |
| | 135 | 59247 | 9304 | 54424 | 4785 | 55389 | 6706 |
| | 125 | 57825 | 9251 | 59539 | 7195 | 54519 | 6181 |
| | 115 | 55987 | 6313 | 56128 | 8350 | 59069 | 6198 |
| **2 Hour Boil MOR (psi)** | 145 | 1829 | 319.4 | 1646 | 239.1 | 1849 | 303.5% |
| | 135 | 1973 | 215.7 | 1706 | 311.5 | 1919 | 321.9% |
| | 125 | 1973 | 367.4 | 1741 | 214.3 | 2020 | 324.4% |
| | 115 | 1801 | 245.6 | 1682 | 289.1 | 1714 | 328.8% |
| **MOR Retention (%)** | 145 | 50.4% | | 46.4% | | 46.2% | |
| | 135 | 53.5% | | 47.2% | | 54.7% | |
| | 125 | 53.5% | | 48.1% | | 57.6% | |
| | 115 | 51.3% | | 50.6% | | 43.3% | |
| **2 Hour Boil MM (lb-in/ft)** | 145 | 757.6 | 119.4 | 693.9 | 97.07 | 796.4 | 130.9 |
| | 135 | 825.4 | 77.54 | 724.7 | 120.50 | 795.3 | 131.2 |
| | 125 | 825.5 | 169.3 | 736.4 | 87.86 | 831.4 | 117.3 |
| | 115 | 746.8 | 91.21 | 723.2 | 117.30 | 703.8 | 133.7 |

## Properties Report for Bark Derived Resin

Control - 3% LPF (Borden OS745)
Dynea - 3% Bark resin (Neste 2063-22)
GP - 3% Bark resin (GPRI RPP073G40)

All Panels utilized 1% wax (EW58-S) throughout panel and 2% MDI (R1840) in the core
Board orientation was 25:50:25 (random:random:random)
Target Thickness = 0.437 Target Density = 38 lb/ft^3

| | | Control | | DYNEA | | GP | |
|---|---|---|---|---|---|---|---|
| **Properties** | # | Average | Std Dev | Average | Std Dev | Average | Std Dev |
| **24 hr Water Absorp (%)** | 145 | 27.96% | 2.48% | 27.05% | 2.61% | 26.45% | 4.41% |
| | 135 | 27.55% | 15.82% | 25.95% | 2.14% | 23.39% | 2.65% |
| | 125 | 31.11% | 5.93% | 25.49% | 3.15% | 24.60% | 2.12% |
| | 115 | 31.16% | 1.78% | 23.74% | 2.43% | 27.18% | 1.54% |
| **24 hr Thick Swell (%) 1" in** | 145 | 11.94% | 0.74% | 11.61% | 1.50% | 11.43% | 0.98% |
| | 135 | 11.61% | 2.64% | 10.74% | 0.61% | 11.14% | 0.64% |
| | 125 | 13.11% | 1.58% | 10.43% | 0.87% | 12.14% | 1.51% |
| | 115 | 12.77% | 0.57% | 10.23% | 1.15% | 12.52% | 1.52% |
| **24 hr Thick Swell (%) Edge** | 145 | 22.84% | 0.97% | 22.37% | 1.21% | 21.90% | 1.53% |
| | 135 | 22.18% | 3.56% | 21.24% | 0.80% | 22.62% | 1.69% |
| | 125 | 23.86% | 2.25% | 20.10% | 1.31% | 23.06% | 1.48% |
| | 115 | 24.08% | 2.07% | 20.64% | 0.50% | 23.39% | 1.14% |
| **24 hr Differential Swell (%)** | 145 | 10.90% | 1.35% | 10.77% | 1.06% | 10.47% | 1.15% |
| | 135 | 10.57% | 2.17% | 10.50% | 0.97% | 11.47% | 1.77% |
| | 125 | 10.75% | 3.72% | 9.67% | 1.27% | 10.92% | 1.05% |
| | 115 | 11.31% | 1.56% | 10.42% | 1.49% | 10.87% | 2.48% |
| **2 hr Boil Water Absorp (%)** | 145 | 136.8% | 4.90% | 156.6% | 6.66% | 150.1% | 3.51% |
| | 135 | 136.8% | 4.38% | 152.3% | 5.38% | 149.8% | 1.48% |
| | 125 | 142.4% | 7.64% | 156.6% | 10.97% | 153.2% | 3.47% |
| | 115 | 142.4% | 3.09% | 150.2% | 3.81% | 158.3% | 7.96% |
| **2 hr Boil Thick Swell (%) 1" in** | 145 | 49.16% | 2.08% | 46.39% | 2.31% | 44.07% | 2.56% |
| | 135 | 47.24% | 2.12% | 46.35% | 2.42% | 47.76% | 2.46% |
| | 125 | 45.61% | 4.42% | 45.45% | 4.57% | 48.16% | 1.34% |
| | 115 | 48.72% | 4.02% | 48.23% | 4.76% | 49.44% | 0.83% |
| **2 hr Boil Thick Swell (%) Edge** | 145 | 47.78% | 2.54% | 46.21% | 3.66% | 45.40% | 3.57% |
| | 135 | 45.02% | 2.62% | 44.75% | 4.44% | 47.65% | 2.94% |
| | 125 | 46.96% | 5.20% | 46.95% | 4.52% | 48.61% | 2.04% |
| | 115 | 49.72% | 5.65% | 48.63% | 2.90% | 49.25% | 4.58% |
| **Density ($lb/ft^3$)** | 145 | 37.6 | 1.6 | 36.7 | 1.7 | 38.1 | 1.4 |
| | 135 | 38.0 | 1.2 | 37.0 | 1.6 | 37.5 | 1.8 |
| | 125 | 37.6 | 1.4 | 36.6 | 1.3 | 37.3 | 1.1 |
| | 115 | 37.4 | 1.5 | 36.9 | 1.0 | 37.8 | 1.2 |

| | | Control | Dynea | GP |
|---|---|---|---|---|
| Properties | # | Average | Average | Average |
| Density (lb/ft3) | 145 | 37.6 | 36.7 | 38.1 |
| | 135 | 38.0 | 37.0 | 37.5 |
| | 125 | 37.6 | 36.6 | 37.3 |
| | 115 | 37.4 | 36.9 | 37.8 |
| Dry MOR (psi) | 145 | 3628 | 3546 | 3999 |
| | 135 | 4078 | 3280 | 3826 |
| | 125 | 3690 | 3617 | 3509 |
| | 115 | 3512 | 3325 | 3954 |
| Dry MOE (10^3 psi) | 145 | 606.0 | 572.6 | 641.5 |
| | 135 | 618.0 | 556.0 | 579.2 |
| | 125 | 595.3 | 603.1 | 570.6 |
| | 115 | 582.7 | 561.2 | 617.3 |
| 2 hr Boil Thick Swell (%) 1" in | 145 | 49.16% | 46.39% | 44.07% |
| | 135 | 47.24% | 46.35% | 47.76% |
| | 125 | 45.61% | 45.45% | 48.16% |
| | 115 | 48.72% | 48.23% | 49.44% |
| 2 hr Boil Water Absorp (%) | 145 | 136.83% | 156.58% | 150.12% |
| | 135 | 136.81% | 152.31% | 149.79% |
| | 125 | 142.44% | 156.63% | 153.16% |
| | 115 | 142.43% | 150.24% | 158.28% |
| 2 hr Boil MOR Retention (%) | 145 | 50.4% | 46.4% | 46.2% |
| | 135 | 53.5% | 47.2% | 54.7% |
| | 125 | 53.5% | 48.1% | 57.6% |
| | 115 | 51.3% | 50.6% | 43.3% |
| 24 hr Water Absorp (%) | 145 | 27.96% | 27.05% | 26.45% |
| | 135 | 27.55% | 25.95% | 23.39% |
| | 125 | 31.11% | 25.49% | 24.60% |
| | 115 | 31.16% | 23.74% | 27.18% |
| 24 hr Thick Swell (%) 1" in | 145 | 11.94% | 11.61% | 11.43% |
| | 135 | 11.61% | 10.74% | 11.14% |
| | 125 | 13.11% | 10.43% | 12.14% |
| | 115 | 12.77% | 10.23% | 12.52% |
| 24 hr Thick Swell (%) Edge | 145 | 22.84% | 22.37% | 21.90% |
| | 135 | 22.18% | 21.24% | 22.62% |
| | 125 | 23.86% | 20.10% | 23.06% |
| | 115 | 24.08% | 20.64% | 23.39% |
| 24 hr Differential Swell (%) | 145 | 10.90% | 10.77% | 10.47% |
| | 135 | 10.57% | 10.50% | 11.47% |
| | 125 | 10.75% | 9.67% | 10.92% |
| | 115 | 11.31% | 10.42% | 10.87% |

# APPENDIX E

Pictures

**Surge Bin**

**Bulk Bag Unloader**

**Bulk Bag Unloader (wide angle)**

**OSB Containing MNRP (darker) and OSB Containing Standard PF (lighter)**

**Finished Product OSB Panels**

# APPENDIX F

Comparative Tests of OSB Panels Containing MNRP Produced at Tembec

July 10th, 2003

# Report of trial

**Project:**

## Third Trial of « Green Resin » AEG-02

**Introduction:**

Ensyn's mission is to develop industrial applications for its core technology, Rapid Thermal Processing, and to then implement these applications in industry. In fact, they developed some "green" additive from biomass, which can be added to phenolic resin to improve performance of OSB. Objectives of this project were to certify OSB surface resin AEG-02 (with TECO), which contains "MNRP" as the green additive. During the second trial (ran in February 2003), the proportion of fines in the boards had occurred some properties problems, and also the low density of panel produced. Two trials were already run, and the present trial is to obtain more stability in the density of boards, to be able to get TECO standard. The trial was run on June 26th, 2003, in Panneaux Tembec plant location, St-Georges-de-Champlain, Quebec.

The comparison of properties were done on a wide range of pressing time (145-200 sec.), because the pressing time seems to have no affect on the boards properties in this mill location. This decision was based on a previous investigation from the mill. This document reports the results obtained for internal bond (IB), bending properties (MOE & MOR), thickness swell (TS) and elasticity index (EI) of OSB produced. The density of boards was constant at 39.3 ± 0.6 lbs/ft$^3$.

**Pressing conditions used:**

- Board thickness : 7/16 inch
- Moisture content of wood :
    - Face : (6.4 ± 0.4) %
    - Core : (3.3 ± 0.5) %
- Face/Core ratio : 61 % / 39 %
- Resin Content (solid basis) :
    - Face : 4.0 % + ~ 2.5 % wax
    - Core : 2.65 % + 2.2 % wax
- Pressing Temperature : 220 °C
- Board pressure : 505 psi
- Line speed : 342 mm/sec
- Pressing time : 145-200 sec. including : - 51 sec to reach the thickness
    - 37 sec of degas

**Results and discussion**

Figure 1 shows the internal bond properties (IB) of boards before, during and after the run trial. As it is shown, there is a significant difference between ecological resin (AEG-02) and normal production (SL-624), because of standard deviation areas. The AEG-02 resin seems to influence the quality of the internal bond of the OSB by using the pressing conditions described above. However, we can also observe a difference between SL-624 before and after the trial. This could be explained by a variation in pressing parameters, the line speed, the forming variation, etc. All boards seems to pass the internal standard of 40 psi

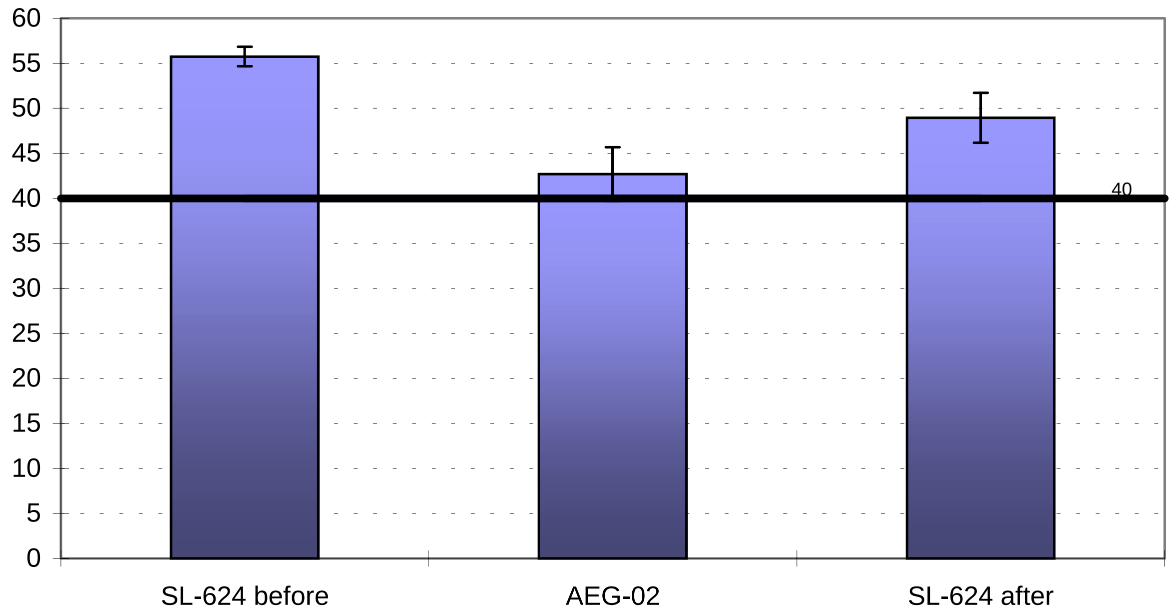



Figure 1, Internal bond of OSB before, during and after the trial.

Figure 2 represents the thickness swell property of OSB pressed before, during and after the test. It is not possible to find a statistical significant difference between the properties of boards tested during and after the trial. However, the thickness swell property seems to be statistically different before the trial. The tendency of thickness swell of boards during the trial seems to be higher than two others, but the standard deviation is also higher, because fewer samples were tested during the trial then before and after, increasing this deviation area. All the boards tested meet the 25 % thickness swell criteria of the plant.

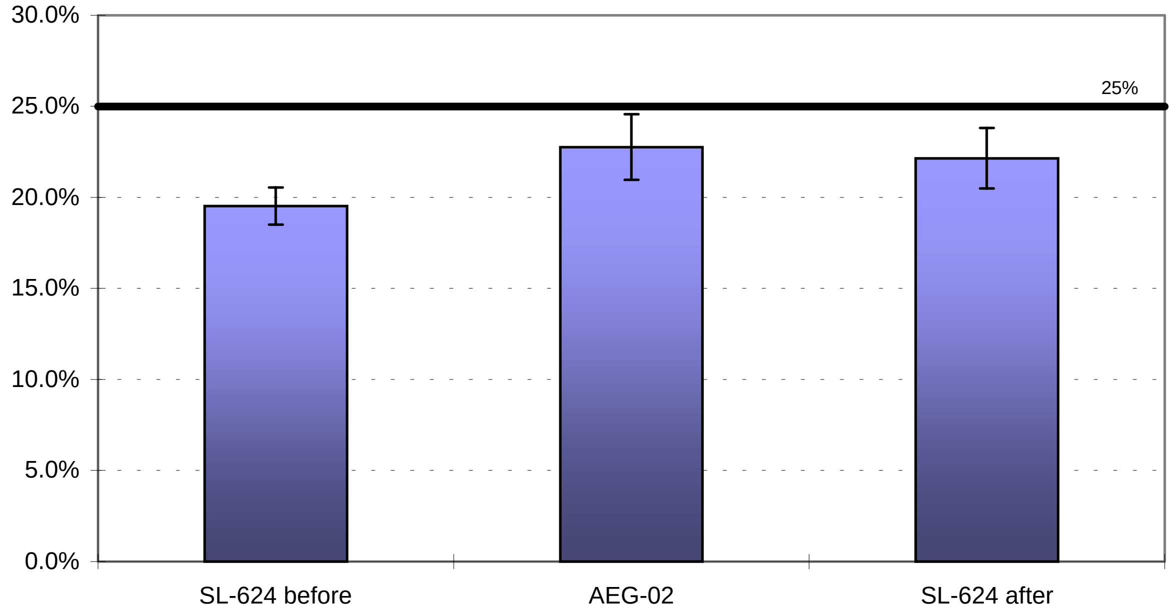



Figure 2, Thickness swell of OSB before, during and after AEG-02

Figure 3 and 4 shows the bending property (MOE – MOR) of OSB prior, during and after the test run. The // indicate that the sample length is parallel to the face strand direction. This value is always higher than perpendicular (T) ones because it combines the strength of the glue with the strength of wood strand itself (two layer of parallel layers oriented in the length direction). Natural strength of wood came from lignin, wood structure, cell direction, etc.

No significant difference in these properties seems to be shown in MOE and MOR // because of the superposition of standard deviations with a degree of confidence (α) of 5 %. For the perpendicular properties, it seems that MOE and MOR T before the trial is different from during and after the trial. The same trend is shown for TS and IB, meaning that the boards made before the trail get better properties. This situation could be explained by different press operator, the line speed, the quality of wood used (fresh wood vs. old wood), the sharpness of waferizer edges, the variation in amount of resin used, etc. It could also be a combination of all those factors. Boards made before the trial seems also to pass the standard of quality for all the bending properties, where it is not the case for the AEG-02 and SL-624 boards made after the trial. The MOE T during the trial does not meet the requirement of 280000 psi, and the one after the trial does not pass it either. However, those plant requirement are designed for boards which got the “hot stacking” treatment of 72 h. With this information, less attention could be put to meet those requirements. With the hot stack treatment, properties of boards in general will be improved. This treatment consists in the storage of tight package of boards for 72 h right after their exit of the press. The heat of boards will complete the cure of the adhesive and help to get strong adhesive linkage.

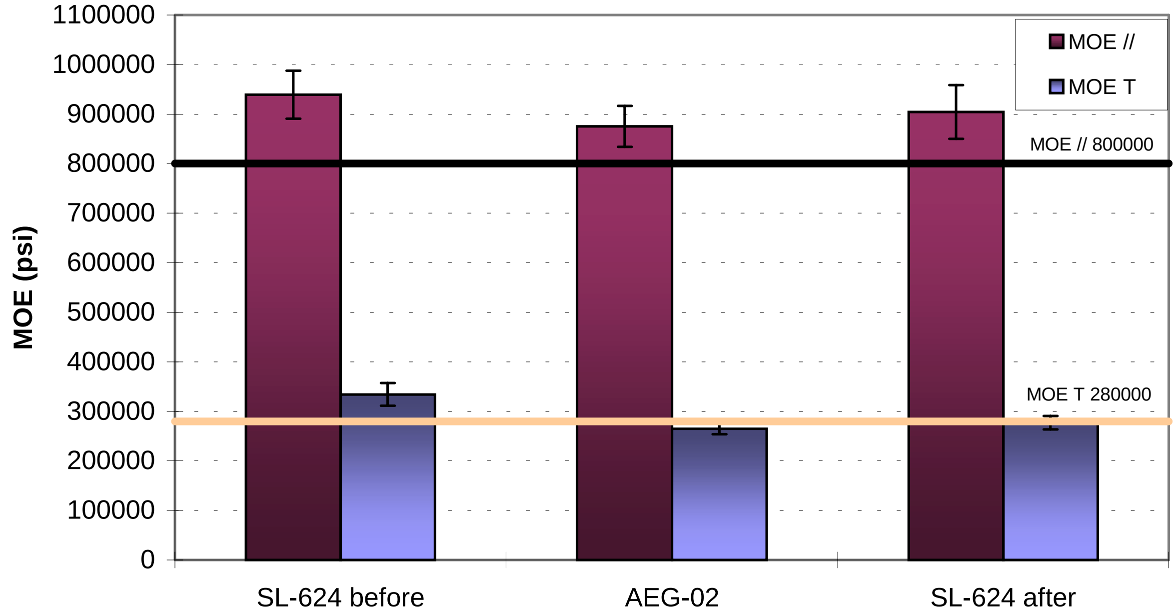



Figure 3, MOE property of OSB before, during and after the trial

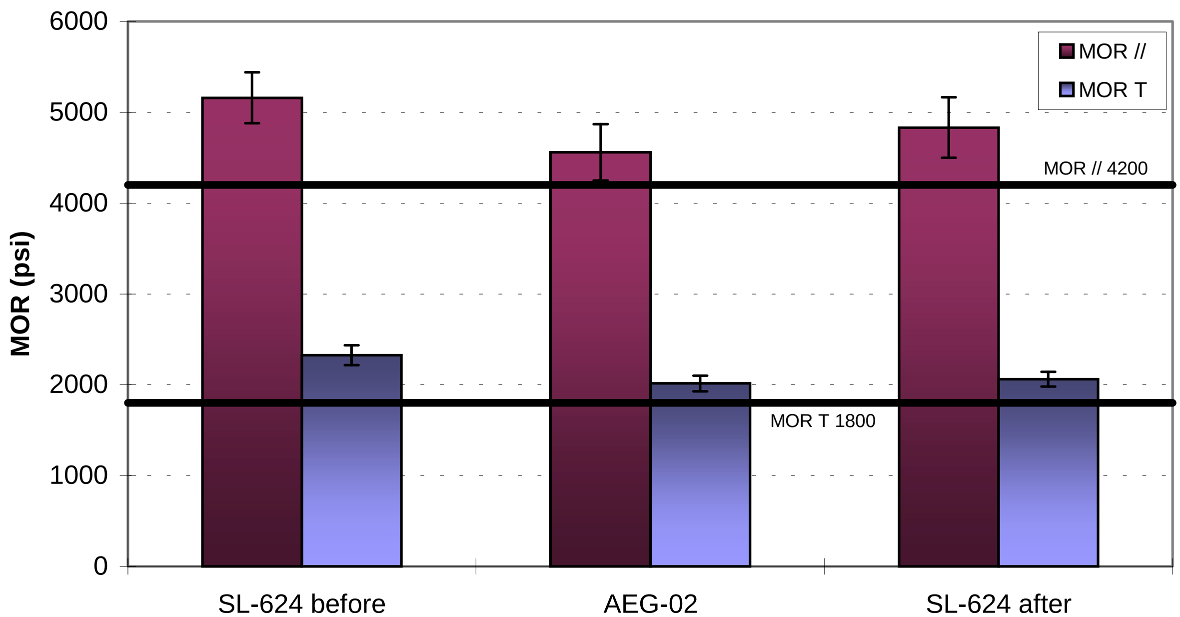



Figure 4, MOR property of OSB before, during and after the trial

Figure 5 shows the Elasticity Index (EI) of boards. This property is measured by the amount of deflection under a charge for a 8' x 4' board. A hot stacking process also improves this property. The EI is used in the industry as an indicator of the board quality for a construction purpose. As it is shown in Figure 5, there is no significant difference between quality of EI for boards pressed before, during or after the trial. The measurement of EI was done once the board was pressed, without any hot stack treatment. All the boards passed the requirement.

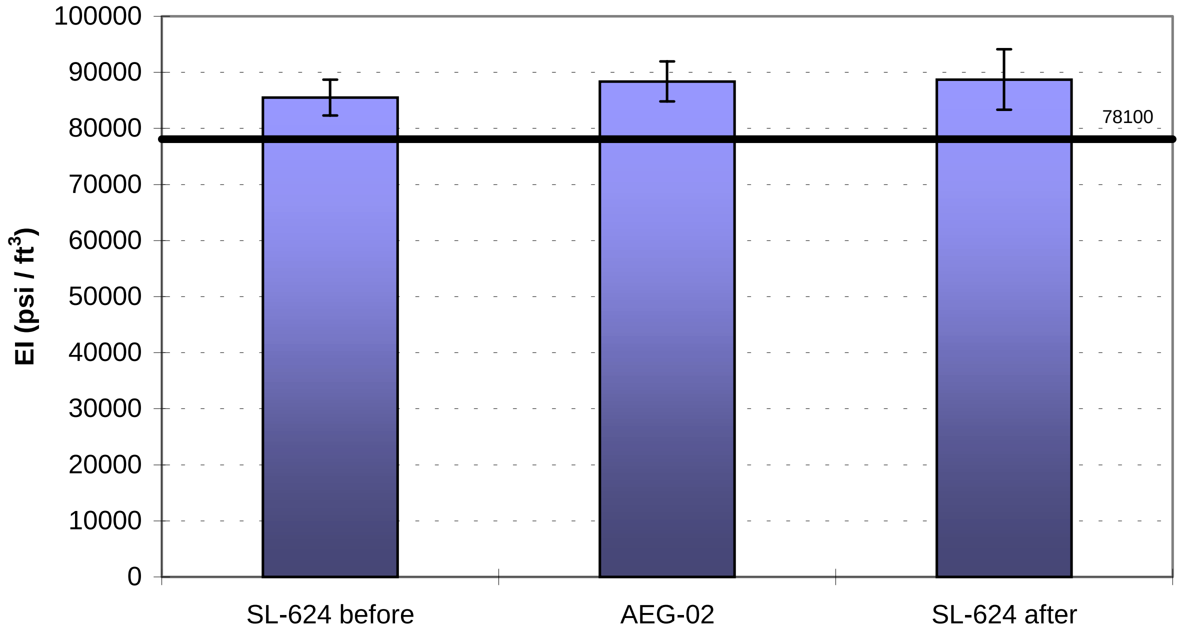



Figure 5, EI property of OSB before, during and after the trial

Conclusion

Results shown in this study indicate that there is a significant difference between AEG-02 and regular boards in term of IB and thickness swell properties. However, further statistical study should be done on more samples during the trial, to improve the standard deviation. As compared to the previous trial (second trial), the standard deviation of properties in general seems to be lower for this trial. It could also be interesting to see if the same trend is observed on a trial in a different plant. Some problem with the forming head had been highlighted during that trial, while last trial, there was a problem with the quality of wafers (amount of fines). All those process variation can affect the results. It would be interesting to see if the same trend of properties is observed in a different OBS plant. The following table gives exact results obtained prior, during, and after the trial of AEG-02, for properties analyzed. The highlighted results shown the results that have not passed the requirement.

Table 1, Properties of OSB with regular and test resin (AEG-02)

| Resin used | IB (psi) | Thickness swell (%) | MOE ( x $10^5$ psi) | | MOR (x$10^3$ psi) | | EI (x$10^4$ psi/ft$^3$) |
|---|---|---|---|---|---|---|---|
| | | | // | T | // | T | |
| SL-624 before | 56 ± 1 | 20 ± 1 | 9.4 ± 0.5 | 3.3 ± 0.2 | 5.2 ± 0.3 | 2.3 ± 0.1 | 8.6 ± 0.3 |
| AEG-02 trial | 43 ± 3 | 23 ± 2 | 8.8 ± 0.4 | 2.7 ± 0.1 | 4.6 ± 0.3 | 2.01 ± 0.09 | 8.8 ± 0.4 |
| SL-624 after | 49 ± 3 | 22 ± 2 | 9.0 ± 0.5 | 2.8 ± 0.1 | 4.8 ± 0.3 | 2.06 ± 0.08 | 8.9 ± 0.5 |

Melany Gagnon, M.Sc. Chemist

# APPENDIX G

OSB Tests at Forintek

Forintek Canada Corp.
Western Division
2665 East Mall
Vancouver, BC
V6T 1W5

**Confidential**

# Evaluation of Two Ensyn Resins as OSB Adhesives

by

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Prepared for

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July 2003

Contract No. 4139

| | | |
|---|---|---|
| Martin Feng | Kevin Groves | Jean Cook |
| Project Leader | Reviewed | Department Manager |

## Objective

To investigate the bonding quality of two Ensyn resins in comparison with a Dynea control PF resin at a loading level of **3.0%** resin solids add-on rate.

## Background

Ensyn Technologies Inc. produces bio-oils from wood residues and bark materials via a biomass fast pyrolysis process. A significant fraction of the pyrolysis products can be used as a major ingredient in the formulation of NR resins for the manufacture of oriented strand board and plywood.

This contract work is to evaluate two NR resin samples (of different formulations, ID: 2104-67 and 2104-69) against a commercial phenol-formaldehyde liquid face resin (from Dynea, ID: 2104-70) as a control resin in the manufacture of OSB panels.

## Experimental Procedure

1. Dried 16 kg aspen strands to about 2% MC for each of three blends.
2. Warmed liquid resin to 25ºC.
3. Conducted one blend each with the following parameters :

| | |
|---|---|
| Strands: | 15 kgs dry aspen / blend. **Actual loading depends on MC**. |
| EW-58S wax: | 260 g / blend |
| Liquid resin: | A) **790** g of 2104-70 resin (57.0% solids, 172 cp, pH10.5) |
| (no dilution) | B) **893** g of 2104-67 resin (50.38% solids, 145 cp, pH 10.7) |
| | C) **898** g of 2104-69 resin (50.10% solids, 165 cp, pH 10.7) |

4. Checked resinated furnish MC.
5. Pressed four panels from each blend using the following parameters:

| | |
|---|---|
| Mat: | 28” x 28” x 7/16”. 3610 g / mat |
| Density: | 40 lb/f³ |
| Press temp.: | 204ºC or 400ºF |
| Press time: | 225, 240, 255 and 270 seconds (conventional OSB press strategy). |

For the 1st blend (control resin 2104-70), label the panels as:

**4139-1-1A**
**4139-1-1B**
**4139-1-1C**
**4139-1-1D**

For the 2nd blend (Ensyn resin 2104-67), label the panels as:

**4139-1-2A**
**4139-1-2B**
**4139-1-2C**
**4139-1-2D**

For the 3rd blend (Ensyn resin 2104-69), label the panels as:

**4139-1-3A**
**4139-1-3B**
**4139-1-3C**
**4139-1-3D**

5. Test the panels for IB (5 samples / panel), MOR & MOE (2 samples / panel), 2-hour boil (1 sample / panel) and 24 hour water soak (1 sample / panel).

## Test Results

All the test results are summarized in the following sections.

Section I – Furnish Moisture Content before and after Blending
Section II – Board Density and Internal Bond Strength
Section III – Modulus of Rupture and Modulus of Elasticity
Section IV – Thickness Swell and Water Absorption after 24 Hour Water Soak
Section V – Centre Point Loading Flexure Test after 2 Hour Water Boil

# Section I
## Furnish Moisture Content before and after Blending

**Initial Furnish**

| | Blend 1 | Blend 2 | Blend 3 |
|---|---|---|---|
| beaker | 226.2 | 226.2 | 226.2 |
| wet wt | 261.93 | 260.1 | 269.87 |
| dry wt | 261.16 | 259.52 | 268.9 |
| wet wt | 35.73 | 33.9 | 43.67 |
| dry wt | 34.96 | 33.32 | 42.7 |
| %MC | 2.2% | 1.7% | 2.3% |

**After Blending**

| | Blend 1 | Blend 2 | Blend 2 | Blend 3 |
|---|---|---|---|---|
| beaker | 226.2 | 226.2 | 226.2 | 226.2 |
| wet wt | 267.4 | 280.6 | 261.36 | 272.45 |
| dry wt | 265.66 | 277.78 | 259.49 | 270.22 |
| wet wt | 41.2 | 54.4 | 35.16 | 46.25 |
| dry wt | 39.46 | 51.58 | 33.29 | 44.02 |
| %MC | 4.4% | 5.5% | 5.6% | 5.1% |

# Section II
## Board Density and Internal Bond Strength

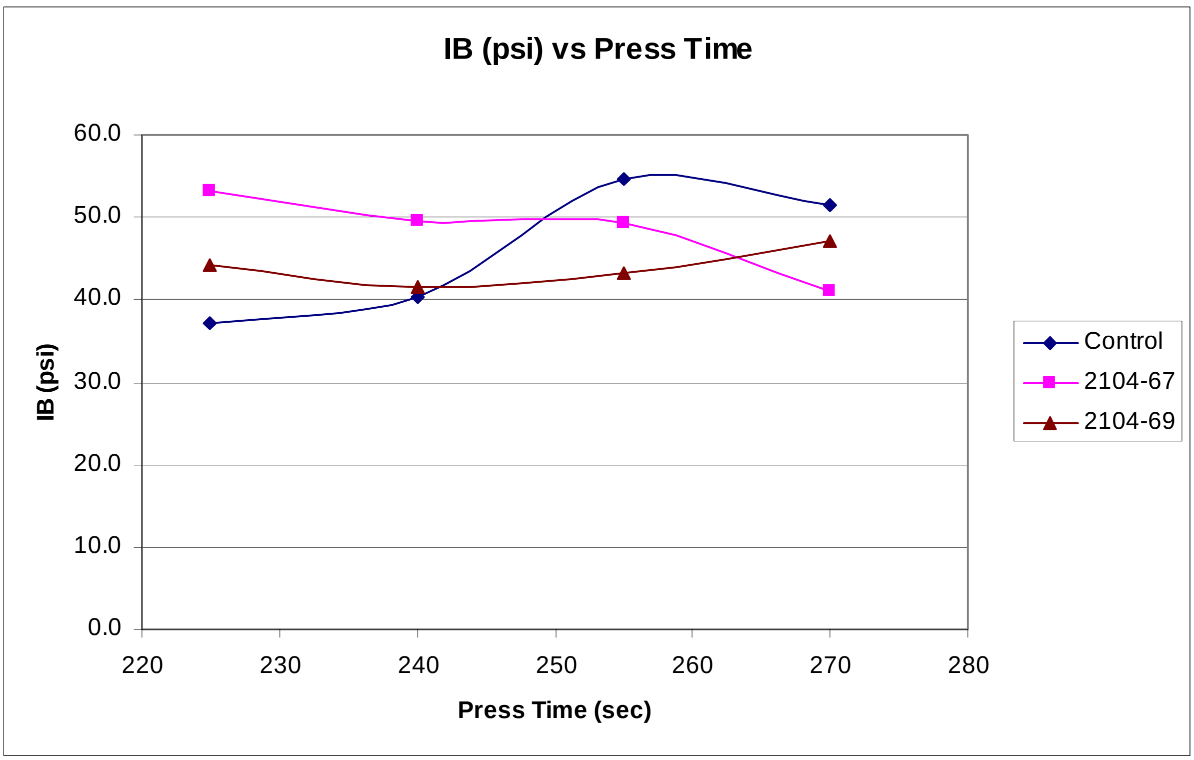
## DATA INPUT SHEET FOR INTERNAL BOND MEASUREMENT

**Project: 4139-1** **Date of entry: May 8, 2003**

**NOTE:** In the panel no. description the first digit (ie 1A-1) 1 is 225 sec board press time, 2 is 240 sec, 3 is 255 sec, 4 is 270 sec.
2104-70 is the control. 2104-67 and 2104-69 are Ensyn resins
All panels are made with 3% resin solids add-on rate and 1% wax solids - 5 IB samples per panel. * excluded values

| Form Row No. | | Panel No. | Position in Panel | Dimensions and Weight Length (mm) | Width (mm) | Thickness (mm) | Weight (g) | Internal Bond (lbs) | Internal Bond PSI | Density (lbs/cu.ft.) | Density (g/cu.cm.) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Control | 1A-1 | | 50.76 | 51.02 | 11.64 | 17.01 | 98.81 | 24.62 | 35.19 | 0.564 |
| 2 | Control | 1A-2 | | 50.63 | 51.02 | 11.71 | 19.54 | 163.90 | 40.94 | 40.29 | 0.646 |
| 3 | Control | 1A-3 | | 50.61 | 51.19 | 11.77 | 21.05 | 160.50 | 39.97 | 43.06 | 0.690 |
| 4 | Control | 1A-4 | | 50.70 | 51.43 | 11.74 | 21.73 | 137.50 | 34.02 | 44.27 | 0.710 |
| 5 | Control | 1A-5 | | 50.57 | 51.28 | 11.71 | 21.14 | 184.90 | 46.00 | 43.42 | 0.696 |
| | | Average | | 50.65 | 51.19 | 11.71 | 20.09 | 149.12 | 37.1 | 41.25 | 0.661 |
| | | St. Dev. | | 0.08 | 0.18 | 0.05 | 1.90 | 32.77 | 8.2 | 3.70 | 0.059 |
| 1 | Control | 1B-1 | | 50.45 | 51.27 | 11.78 | 19.71 | 164.10 | 40.93 | 40.35 | 0.647 |
| 2 | Control | 1B-2 | | 50.60 | 51.20 | 11.66 | 18.53 | 141.40 | 35.21 | 38.26 | 0.613 |
| 3 | Control | 1B-3 | | 50.71 | 51.16 | 11.54 | 19.23 | 26.28 | * 6.50 | 40.06 | 0.642 |
| 4 | Control | 1B-4 | | 50.61 | 51.06 | 11.69 | 19.14 | 154.20 | 38.50 | 39.52 | 0.633 |
| 5 | Control | 1B-5 | | 50.54 | 51.18 | 11.70 | 18.84 | 187.10 | 46.67 | 38.83 | 0.622 |
| | | Average | | 50.58 | 51.17 | 11.67 | 19.09 | 134.62 | 40.3 | 39.40 | 0.631 |
| | | St. Dev. | | 0.10 | 0.08 | 0.09 | 0.44 | 62.83 | 4.8 | 0.86 | 0.014 |
| 1 | Control | 1C-1 | | 50.76 | 51.16 | 11.74 | 22.16 | 202.10 | 50.21 | 45.34 | 0.727 |
| 2 | Control | 1C-2 | | 50.61 | 51.15 | 11.71 | 21.35 | 234.00 | 58.32 | 43.93 | 0.704 |
| 3 | Control | 1C-3 | | 50.56 | 51.08 | 11.65 | 23.23 | 267.90 | 66.92 | 48.16 | 0.772 |
| 4 | Control | 1C-4 | | 50.59 | 51.06 | 11.44 | 22.13 | 129.10 | 32.24 | 46.71 | 0.749 |
| 5 | Control | 1C-5 | | 50.49 | 51.12 | 11.49 | 21.93 | 262.20 | 65.54 | 46.12 | 0.739 |
| | | Average | | 50.60 | 51.11 | 11.61 | 22.16 | 219.06 | 54.6 | 46.05 | 0.738 |
| | | St. Dev. | | 0.10 | 0.04 | 0.13 | 0.68 | 56.68 | 14.2 | 1.57 | 0.025 |
| 1 | Control | 1D-1 | | 50.47 | 51.34 | 11.72 | 21.89 | 330.50 | 82.29 | 44.96 | 0.720 |
| 2 | Control | 1D-2 | | 50.59 | 51.06 | 11.72 | 20.41 | 61.92 | 15.47 | 42.05 | 0.674 |
| 3 | Control | 1D-3 | | 50.45 | 51.03 | 11.64 | 21.54 | 254.90 | 63.88 | 44.83 | 0.718 |
| 4 | Control | 1D-4 | | 50.72 | 51.19 | 11.67 | 20.67 | 125.70 | 31.23 | 42.55 | 0.682 |
| 5 | Control | 1D-5 | | 50.65 | 51.10 | 11.57 | 18.69 | 256.90 | 64.04 | 38.93 | 0.624 |
| | | Average | | 50.58 | 51.14 | 11.66 | 20.64 | 205.98 | 51.4 | 42.66 | 0.684 |
| | | St. Dev. | | 0.12 | 0.13 | 0.06 | 1.25 | 109.19 | 27.2 | 2.47 | 0.040 |
| 1 | 2104-67 | 2A-1 | | 50.49 | 51.14 | 10.98 | 19.32 | 182.10 | 45.50 | 42.50 | 0.681 |
| 2 | 2104-67 | 2A-2 | | 50.47 | 51.15 | 10.99 | 18.96 | 202.70 | 50.66 | 41.68 | 0.668 |
| 3 | 2104-67 | 2A-3 | | 50.89 | 51.07 | 10.99 | 19.71 | 260.60 | 64.69 | 43.04 | 0.690 |
| 4 | 2104-67 | 2A-4 | | 50.53 | 51.35 | 11.04 | 20.42 | 206.20 | 51.27 | 44.46 | 0.713 |
| 5 | 2104-67 | 2A-5 | | 50.72 | 51.03 | 11.03 | 20.71 | 215.20 | 53.64 | 45.25 | 0.725 |
| | | Average | | 50.62 | 51.15 | 11.01 | 19.82 | 213.36 | 53.2 | 43.39 | 0.695 |
| | | St. Dev. | | 0.18 | 0.12 | 0.03 | 0.73 | 29.06 | 7.1 | 1.45 | 0.023 |
| 1 | 2104-67 | 2B-1 | | 50.43 | 51.30 | 11.17 | 18.53 | 178.10 | 44.41 | 39.99 | 0.641 |
| 2 | 2104-67 | 2B-2 | | 50.77 | 51.28 | 11.24 | 23.29 | 273.20 | 67.70 | 49.64 | 0.796 |
| 3 | 2104-67 | 2B-3 | | 50.74 | 51.30 | 11.20 | 20.38 | 135.80 | 33.66 | 43.60 | 0.699 |
| 4 | 2104-67 | 2B-4 | | 50.59 | 51.16 | 11.11 | 20.07 | 238.80 | 59.53 | 43.53 | 0.698 |
| 5 | 2104-67 | 2B-5 | | 50.66 | 51.18 | 11.15 | 18.90 | 169.40 | 42.15 | 40.78 | 0.653 |
| | | Average | | 50.64 | 51.24 | 11.17 | 20.23 | 199.06 | 49.5 | 43.51 | 0.697 |
| | | St. Dev. | | 0.14 | 0.07 | 0.05 | 1.88 | 55.67 | 13.8 | 3.79 | 0.061 |
| 1 | 2104-67 | 2C-1 | | 50.45 | 51.29 | 11.10 | 19.02 | 182.90 | 45.60 | 41.30 | 0.662 |
| 2 | 2104-67 | 2C-2 | | 50.51 | 51.26 | 11.04 | 18.54 | 205.20 | 51.13 | 40.46 | 0.648 |
| 3 | 2104-67 | 2C-3 | | 50.54 | 51.27 | 10.99 | 18.62 | 211.80 | 52.73 | 40.78 | 0.654 |
| 4 | 2104-67 | 2C-4 | | 50.56 | 51.24 | 10.77 | 20.70 | 150.20 | 37.40 | 46.27 | 0.742 |
| 5 | 2104-67 | 2C-5 | | 50.58 | 51.10 | 10.89 | 17.98 | 238.10 | 59.43 | 39.84 | 0.639 |
| | | Average | | 50.53 | 51.23 | 10.96 | 18.97 | 197.64 | 49.3 | 41.73 | 0.669 |
| | | St. Dev. | | 0.05 | 0.08 | 0.13 | 1.03 | 33.02 | 8.3 | 2.59 | 0.042 |
| 1 | 2104-67 | 2D-1 | | 50.68 | 51.08 | 11.01 | 22.98 | 211.30 | 52.66 | 50.29 | 0.806 |
| 2 | 2104-67 | 2D-2 | | 50.56 | 51.29 | 11.13 | 18.69 | 155.30 | 38.64 | 40.39 | 0.647 |
| 3 | 2104-67 | 2D-3 | | 50.65 | 51.13 | 11.16 | 18.12 | 116.00 | 28.90 | 39.10 | 0.627 |
| 4 | 2104-67 | 2D-4 | | 50.50 | 51.20 | 11.27 | 18.31 | 16.81 | * 4.20 | 39.19 | 0.628 |
| 5 | 2104-67 | 2D-5 | | 50.59 | 51.38 | 11.15 | 18.54 | 177.30 | 44.01 | 39.90 | 0.639 |
| | | Average | | 50.60 | 51.22 | 11.14 | 19.33 | 135.34 | 41.1 | 41.77 | 0.669 |
| | | St. Dev. | | 0.07 | 0.12 | 0.09 | 2.05 | 74.75 | 10.0 | 4.79 | 0.077 |
| 1 | 2104-69 | 3A-1 | | 50.64 | 51.20 | 11.38 | 19.38 | 133.60 | 33.24 | 40.97 | 0.657 |
| 2 | 2104-69 | 3A-2 | | 50.57 | 51.29 | 11.25 | 21.18 | 154.00 | 38.31 | 45.27 | 0.726 |
| 3 | 2104-69 | 3A-3 | | 50.53 | 51.07 | 11.14 | 19.61 | 191.90 | 47.98 | 42.55 | 0.682 |
| 4 | 2104-69 | 3A-4 | | 50.52 | 51.06 | 11.21 | 23.08 | 204.80 | 51.22 | 49.78 | 0.798 |
| 5 | 2104-69 | 3A-5 | | 50.60 | 51.17 | 11.23 | 19.96 | 200.10 | 49.86 | 42.82 | 0.686 |
| | | Average | | 50.57 | 51.16 | 11.24 | 20.64 | 176.88 | 44.1 | 44.28 | 0.710 |
| | | St. Dev. | | 0.05 | 0.10 | 0.09 | 1.53 | 31.39 | 7.9 | 3.44 | 0.055 |
| 1 | 2104-69 | 3B-1 | | 50.42 | 51.17 | 11.33 | 19.44 | 82.64 | 20.67 | 41.48 | 0.665 |
| 2 | 2104-69 | 3B-2 | | 50.56 | 51.19 | 11.18 | 20.99 | 177.50 | 44.25 | 45.24 | 0.725 |
| 3 | 2104-69 | 3B-3 | | 50.44 | 51.05 | 11.12 | 21.46 | 222.30 | 55.70 | 46.75 | 0.749 |
| 4 | 2104-69 | 3B-4 | | 50.49 | 51.09 | 11.15 | 18.65 | 173.10 | 43.29 | 40.44 | 0.648 |
| 5 | 2104-69 | 3B-5 | | 50.60 | 51.23 | 11.15 | 19.84 | 176.80 | 44.00 | 42.81 | 0.686 |
| | | Average | | 50.50 | 51.15 | 11.19 | 20.08 | 166.47 | 41.6 | 43.35 | 0.695 |
| | | St. Dev. | | 0.08 | 0.07 | 0.08 | 1.15 | 51.03 | 12.8 | 2.62 | 0.042 |
| 1 | 2104-69 | 3C-1 | | 50.54 | 51.18 | 11.28 | 19.17 | 140.60 | 35.07 | 40.98 | 0.657 |
| 2 | 2104-69 | 3C-2 | | 50.74 | 51.35 | 11.23 | 20.94 | 203.30 | 50.34 | 44.64 | 0.715 |
| 3 | 2104-69 | 3C-3 | | 50.82 | 51.15 | 11.24 | 19.71 | 150.10 | 37.25 | 42.08 | 0.674 |
| 4 | 2104-69 | 3C-4 | | 50.77 | 51.18 | 11.10 | 22.44 | 208.00 | 51.64 | 48.53 | 0.778 |
| 5 | 2104-69 | 3C-5 | | 50.70 | 51.18 | 11.01 | 17.88 | 167.90 | 41.75 | 39.04 | 0.626 |
| | | Average | | 50.71 | 51.21 | 11.17 | 20.03 | 173.98 | 43.2 | 43.05 | 0.690 |
| | | St. Dev. | | 0.11 | 0.08 | 0.11 | 1.74 | 30.57 | 7.5 | 3.67 | 0.059 |
| 1 | 2104-69 | 3D-1 | | 50.66 | 51.38 | 11.25 | 17.73 | 188.80 | 46.80 | 37.76 | 0.605 |
| 2 | 2104-69 | 3D-2 | | 50.89 | 51.24 | 11.11 | 18.58 | 180.50 | 44.66 | 40.00 | 0.641 |
| 3 | 2104-69 | 3D-3 | | 50.76 | 51.31 | 11.13 | 20.64 | 211.20 | 52.32 | 44.41 | 0.712 |
| 4 | 2104-69 | 3D-4 | | 50.65 | 51.16 | 11.00 | 19.63 | 174.70 | 43.50 | 42.95 | 0.688 |
| 5 | 2104-69 | 3D-5 | | 50.80 | 51.34 | 11.03 | 18.69 | 197.20 | 48.78 | 40.52 | 0.649 |
| | | Average | | 50.75 | 51.29 | 11.10 | 19.05 | 190.48 | 47.2 | 41.13 | 0.659 |
| | | St. Dev. | | 0.10 | 0.09 | 0.10 | 1.11 | 14.37 | 3.5 | 2.60 | 0.042 |

| | Internal Bond (psi) | | |
|---|---|---|---|
| Press Time | Control | 2104-67 | 2104-69 |
| 225 | 37.1 | 53.2 | 44.1 |
| 240 | 40.3 | 49.5 | 41.6 |
| 255 | 54.6 | 49.3 | 43.2 |
| 270 | 51.4 | 41.1 | 47.2 |



# Section III
## Modulus of Rupture and Modulus of Elasticity

# CENTRE POINT LOADING FLEXURE TEST

**Liquid Control Resin and Two Ensyn Resins**

**Project: 4139-1** **Date of entry:** 08-May-03

Test Span: 10.5" or 266.7mm Date Tested: 08-May-03

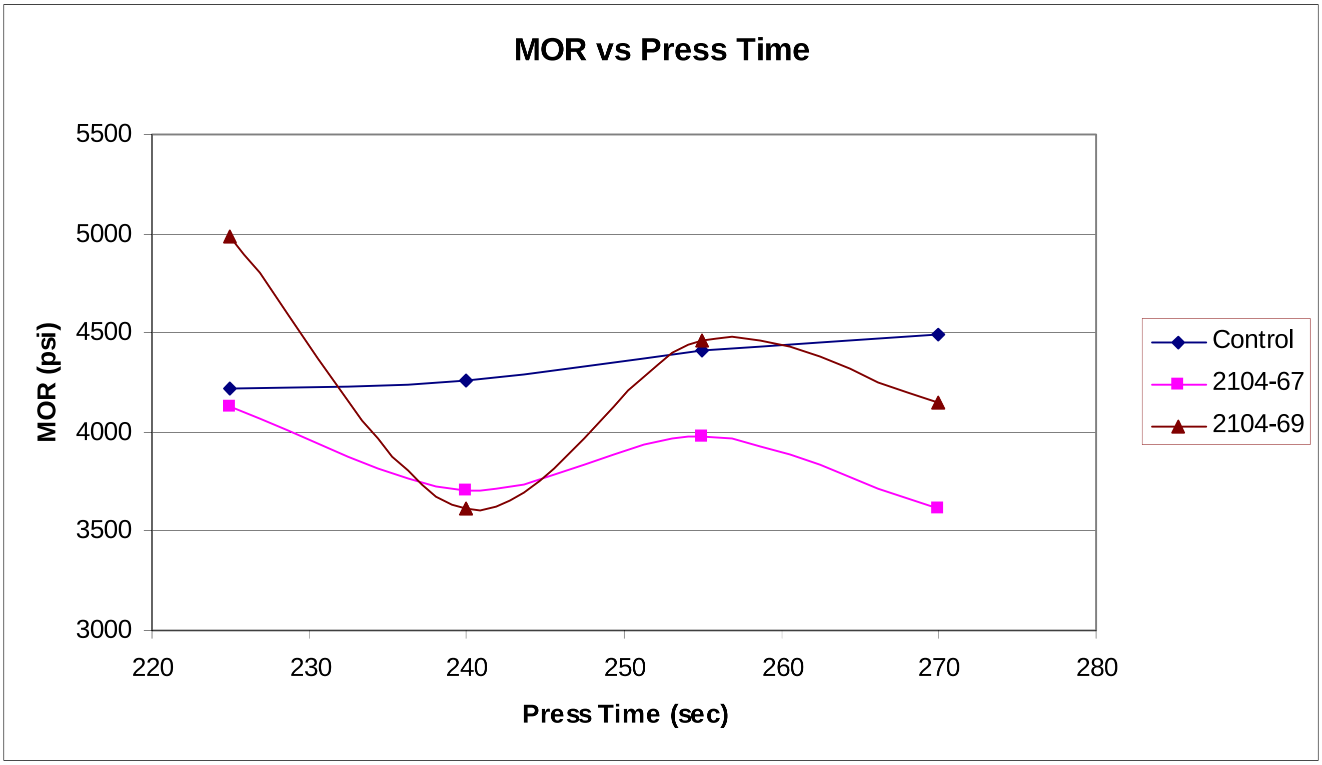
Specimen Size: 3.0" x 7/16" x 12 1/2" Test Speed: 5.5 mm/min

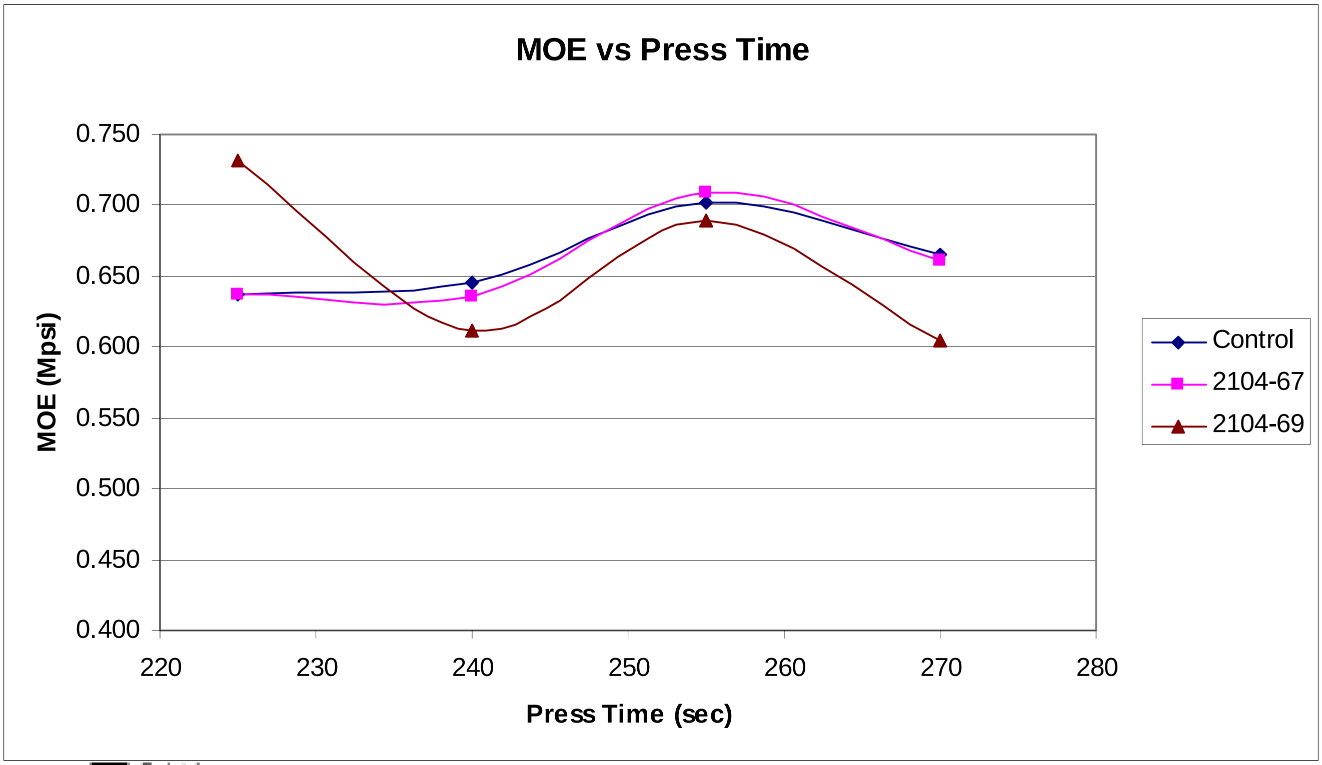
**NOTE:** In the panel no. description the first digit (ie 1A-1) 1 is 225 sec board press time, 2 is 240 sec, 3 is 255 sec, 4 is 270 sec. 2104-70 is the control. 2104-67 and 2104-69 are Ensyn resins.
All panels are made with 3 % resin solids add-on rate and 1% wax solids - 2 flexure samples per panel.

| | | Code # | Width (mm) | Depth (mm) | Max. Load (kg) | MOR (MPa) | MOE (GPa) | MOR (psi) | MOE (Mpsi) | M.C. % | S.G. Product | Density (lb/cu.ft) | Length (mm) | Weight (gms) | Dry wt. (grams) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Control | 4139-1-1A-1 | 76.68 | 11.81 | 94.71 | 34.72 | 4.91 | 5036 | 0.712 | 1.7 | 0.621 | 39.4 | 319 | 182.3 | 179.3 |
| 2 | Control | 4139-1-1A-2 | 76.73 | 11.69 | 62.60 | 23.41 | 3.88 | 3395 | 0.563 | 1.7 | 0.622 | 39.5 | 319 | 181.0 | 178.0 |
| | | Average | 76.71 | 11.75 | 78.66 | 29.07 | 4.40 | 4216 | 0.637 | 1.7 | 0.621 | 39.4 | 319 | 181.7 | 178.6 |
| | | St. Dev. | 0.04 | 0.08 | 22.71 | 8.00 | 0.73 | 1160 | 0.106 | 0.0 | 0.001 | 0.1 | 0 | 0.9 | 0.9 |
| 3 | Control | 4139-1-1B-1 | 76.72 | 11.74 | 80.72 | 29.93 | 4.36 | 4341 | 0.632 | 1.4 | 0.648 | 40.9 | 319 | 188.6 | 186.1 |
| 4 | Control | 4139-1-1B-2 | 76.63 | 11.58 | 75.49 | 28.80 | 4.55 | 4178 | 0.660 | 1.4 | 0.661 | 41.8 | 319 | 189.9 | 187.2 |
| | | Average | 76.68 | 11.66 | 78.11 | 29.37 | 4.45 | 4260 | 0.646 | 1.4 | 0.654 | 41.4 | 319 | 189.2 | 186.6 |
| | | St. Dev. | 0.06 | 0.11 | 3.70 | 0.80 | 0.14 | 115 | 0.020 | 0.0 | 0.010 | 0.6 | 0 | 0.9 | 0.8 |
| 5 | Control | 4139-1-1C-1 | 76.76 | 11.60 | 62.76 | 23.82 | 4.84 | 3455 | 0.702 | 1.3 | 0.651 | 41.1 | 319 | 187.3 | 184.8 |
| 6 | Control | 4139-1-1C-2 | 76.65 | 11.72 | 99.49 | 37.05 | 4.84 | 5374 | 0.703 | 1.2 | 0.639 | 40.4 | 319 | 185.4 | 183.2 |
| | | Average | 76.71 | 11.66 | 81.13 | 30.44 | 4.84 | 4415 | 0.702 | 1.3 | 0.645 | 40.7 | 319 | 186.4 | 184.0 |
| | | St. Dev. | 0.08 | 0.08 | 25.97 | 9.36 | 0.00 | 1357 | 0.001 | 0.1 | 0.008 | 0.5 | 0 | 1.3 | 1.2 |
| 7 | Control | 4139-1-1D-1 | 76.72 | 11.49 | 84.19 | 32.59 | 4.29 | 4727 | 0.622 | 1.4 | 0.646 | 40.8 | 319 | 184.2 | 181.6 |
| 8 | Control | 4139-1-1D-2 | 76.90 | 11.71 | 78.82 | 29.30 | 4.88 | 4251 | 0.709 | 1.3 | 0.634 | 40.1 | 319 | 184.7 | 182.3 |
| | | Average | 76.81 | 11.60 | 81.51 | 30.95 | 4.59 | 4489 | 0.665 | 1.4 | 0.640 | 40.5 | 319 | 184.4 | 181.9 |
| | | St. Dev. | 0.13 | 0.16 | 3.80 | 2.33 | 0.42 | 337 | 0.061 | 0.1 | 0.008 | 0.5 | 0 | 0.4 | 0.5 |
| 9 | 2104-67 | 4139-1-2A-1 | 76.75 | 11.07 | 64.21 | 26.77 | 4.52 | 3882 | 0.656 | 1.4 | 0.655 | 41.4 | 319 | 180.0 | 177.6 |
| 10 | 2104-67 | 4139-1-2A-2 | 76.82 | 10.90 | 70.09 | 30.11 | 4.26 | 4367 | 0.618 | 1.3 | 0.582 | 36.8 | 319 | 157.5 | 155.4 |
| | | Average | 76.79 | 10.99 | 67.15 | 28.44 | 4.39 | 4125 | 0.637 | 1.3 | 0.619 | 39.1 | 319 | 168.7 | 166.5 |
| | | St. Dev. | 0.05 | 0.12 | 4.16 | 2.36 | 0.18 | 343 | 0.027 | 0.0 | 0.052 | 3.3 | 0 | 16.0 | 15.7 |
| 11 | 2104-67 | 4139-1-2B-1 | 76.76 | 11.11 | 58.20 | 24.08 | 4.19 | 3493 | 0.607 | 1.2 | 0.654 | 41.3 | 319 | 180.1 | 178.0 |
| 12 | 2104-67 | 4139-1-2B-2 | 76.77 | 11.06 | 64.67 | 27.00 | 4.58 | 3916 | 0.664 | 1.2 | 0.644 | 40.7 | 319 | 176.5 | 174.5 |
| | | Average | 76.77 | 11.09 | 61.44 | 25.54 | 4.38 | 3705 | 0.636 | 1.2 | 0.649 | 41.0 | 319 | 178.3 | 176.2 |
| | | St. Dev. | 0.01 | 0.04 | 4.57 | 2.06 | 0.28 | 299 | 0.040 | 0.0 | 0.007 | 0.5 | 0 | 2.5 | 2.5 |
| 13 | 2104-67 | 4139-1-2C-1 | 76.90 | 10.86 | 77.66 | 33.57 | 5.50 | 4869 | 0.798 | 1.1 | 0.664 | 41.9 | 319 | 179.0 | 176.9 |
| 14 | 2104-67 | 4139-1-2C-2 | 76.82 | 10.89 | 49.48 | 21.29 | 4.27 | 3089 | 0.619 | 1.2 | 0.649 | 41.0 | 319 | 175.2 | 173.1 |
| | | Average | 76.86 | 10.88 | 63.57 | 27.43 | 4.89 | 3979 | 0.709 | 1.2 | 0.656 | 41.4 | 319 | 177.1 | 175.0 |
| | | St. Dev. | 0.06 | 0.02 | 19.93 | 8.68 | 0.87 | 1259 | 0.127 | 0.1 | 0.011 | 0.7 | 0 | 2.6 | 2.7 |
| 15 | 2104-67 | 4139-1-2D-1 | 76.69 | 11.33 | 52.91 | 21.07 | 3.80 | 3056 | 0.551 | 1.3 | 0.631 | 39.9 | 319 | 177.1 | 174.8 |
| 16 | 2104-67 | 4139-1-2D-2 | 76.64 | 10.99 | 67.89 | 28.75 | 5.32 | 4171 | 0.772 | 1.2 | 0.697 | 43.9 | 319 | 189.3 | 187.2 |
| | | Average | 76.67 | 11.16 | 60.40 | 24.91 | 4.56 | 3614 | 0.661 | 1.2 | 0.664 | 41.9 | 319 | 183.2 | 181.0 |
| | | St. Dev. | 0.04 | 0.24 | 10.59 | 5.43 | 1.08 | 788 | 0.156 | 0.1 | 0.046 | 2.9 | 0 | 8.6 | 8.7 |
| 17 | 2104-69 | 4139-1-3A-1 | 76.81 | 11.25 | 79.95 | 32.24 | 4.97 | 4677 | 0.721 | 1.2 | 0.658 | 41.5 | 319 | 183.6 | 181.3 |
| 18 | 2104-69 | 4139-1-3A-2 | 76.81 | 11.13 | 88.70 | 36.55 | 5.12 | 5301 | 0.742 | 1.3 | 0.670 | 42.3 | 319 | 185.1 | 182.8 |
| | | Average | 76.81 | 11.19 | 84.33 | 34.40 | 5.04 | 4989 | 0.732 | 1.3 | 0.664 | 41.9 | 319 | 184.4 | 182.1 |
| | | St. Dev. | 0.00 | 0.08 | 6.19 | 3.05 | 0.11 | 441 | 0.015 | 0.0 | 0.009 | 0.6 | 0 | 1.1 | 1.1 |
| 19 | 2104-69 | 4139-1-3B-1 | 76.81 | 11.24 | 64.08 | 25.89 | 4.22 | 3755 | 0.613 | 1.2 | 0.650 | 41.0 | 319 | 181.2 | 179.0 |
| 20 | 2104-69 | 4139-1-3B-2 | 76.80 | 11.04 | 57.07 | 23.90 | 4.20 | 3467 | 0.610 | 1.2 | 0.655 | 41.3 | 319 | 179.2 | 177.1 |
| | | Average | 76.81 | 11.14 | 60.58 | 24.90 | 4.21 | 3611 | 0.611 | 1.2 | 0.652 | 41.2 | 319 | 180.2 | 178.1 |
| | | St. Dev. | 0.01 | 0.14 | 4.96 | 1.41 | 0.02 | 204 | 0.002 | 0.0 | 0.003 | 0.2 | 0 | 1.4 | 1.4 |
| 21 | 2104-69 | 4139-1-3C-1 | 76.80 | 11.31 | 79.17 | 31.59 | 4.80 | 4583 | 0.696 | 1.2 | 0.662 | 41.8 | 319 | 185.6 | 183.4 |
| 22 | 2104-69 | 4139-1-3C-2 | 76.83 | 11.19 | 73.34 | 29.89 | 4.70 | 4335 | 0.682 | 1.1 | 0.678 | 42.7 | 319 | 187.9 | 185.8 |
| | | Average | 76.82 | 11.25 | 76.26 | 30.74 | 4.75 | 4459 | 0.689 | 1.2 | 0.670 | 42.3 | 319 | 186.8 | 184.6 |
| | | St. Dev. | 0.02 | 0.08 | 4.12 | 1.20 | 0.07 | 175 | 0.010 | 0.0 | 0.011 | 0.7 | 0 | 1.6 | 1.7 |
| 23 | 2104-69 | 4139-1-3D-1 | 76.65 | 11.22 | 68.72 | 27.92 | 4.22 | 4050 | 0.612 | 1.3 | 0.664 | 41.9 | 319 | 184.5 | 182.1 |
| 24 | 2104-69 | 4139-1-3D-2 | 76.93 | 10.95 | 68.83 | 29.25 | 4.13 | 4243 | 0.599 | 1.2 | 0.644 | 40.7 | 319 | 175.2 | 173.1 |
| | | Average | 76.79 | 11.09 | 68.78 | 28.59 | 4.17 | 4147 | 0.605 | 1.3 | 0.654 | 41.3 | 319 | 179.9 | 177.6 |
| | | St. Dev. | 0.20 | 0.19 | 0.08 | 0.94 | 0.06 | 136 | 0.009 | 0.1 | 0.014 | 0.9 | 0 | 6.6 | 6.4 |

| Press Time | MOR Control Control | 2104-67 | 2104-69 |
|---|---|---|---|
| 225 | 4216 | 4125 | 4989 |
| 240 | 4260 | 3705 | 3611 |
| 255 | 4415 | 3979 | 4459 |
| 270 | 4489 | 3614 | 4147 |

| Press Time | MOE Control Control | 2104-67 | 2104-69 |
|---|---|---|---|
| 225 | 0.637 | 0.637 | 0.732 |
| 240 | 0.646 | 0.636 | 0.611 |
| 255 | 0.702 | 0.709 | 0.689 |
| 270 | 0.665 | 0.661 | 0.605 |



# Section IV
## Thickness Swell and Water Absorption after 24 Hour Water Soak

## 24 HR Soak test - 6" x6" Samples - Forintek Canada Corp.

**Project: 4139-1 Date of entry: May 8 and 9**

**NOTE:** In the panel no. description the first digit (ie 1A-1) 1 is 225 sec board press time, 2 is 240 sec, 3 is 255 sec, 4 is 270 sec.
Control is 2104-70 resin, 2104-67 and 2104-69 are Ensyn resins.
All panels are made with 3% resin solids add-on rate and 1% wax solids- 2 water soak sample per panel

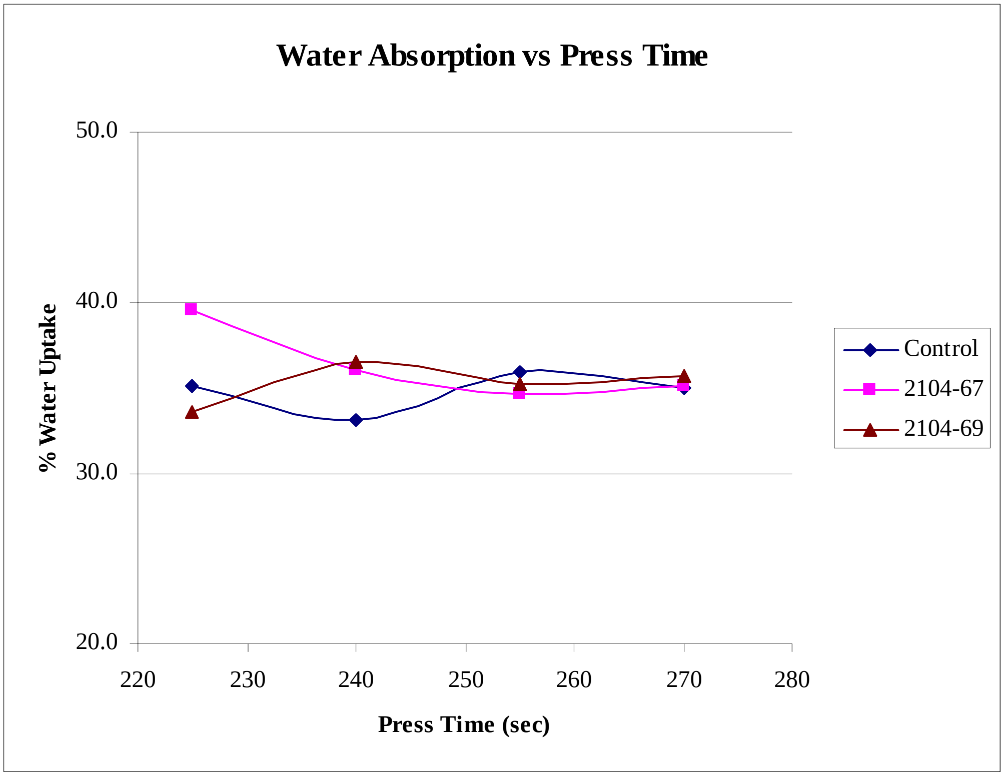
| | Sample No. | Initial Wt. (gm) | Wet Wt. (gm) | Width (mm) | Length (mm) | Initial Dim (mm) | Initial Dim (mm) | Initial Dim (mm) | Initial Dim (mm) | Center Initial Dim (mm) | Average Initial Dim (mm) | Wet Dim (mm) | Wet Dim (mm) | Wet Dim (mm) | Wet Dim (mm) | Center Final Dim (mm) | Average Wet Dim (mm) | Density (lb/cu.ft) | Water Absorb (%) | Th Swell Outside (%) | Th Swell Inside (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | 1A 1 | 185.17 | 251.57 | 153.05 | 152.72 | 11.79 | 11.86 | 11.83 | 11.80 | 11.73 | 11.82 | 13.791 | 14.016 | 13.529 | 13.522 | 14.903 | 13.715 | 41.8 | 35.9 | 16.0 | 27.1 |
| | 1A 2 | 197.13 | 264.91 | 153.09 | 152.79 | 11.82 | 11.80 | 11.80 | 11.87 | 11.79 | 11.82 | 14.951 | 13.712 | 13.734 | 14.299 | 14.205 | 14.174 | 44.5 | 34.4 | 19.9 | 20.5 |
| | Average | 191.15 | 258.24 | 153.07 | 152.76 | 11.81 | 11.83 | 11.81 | 11.84 | 11.76 | 11.82 | 14.37 | 13.86 | 13.63 | 13.91 | 14.55 | 13.94 | 43.1 | 35.1 | 18.0 | 23.8 |
| | St. Dev. | 8.46 | 9.43 | 0.03 | 0.05 | 0.02 | 0.05 | 0.03 | 0.05 | 0.04 | 0.00 | 0.82 | 0.21 | 0.14 | 0.55 | 0.49 | 0.32 | 1.9 | 1.0 | 2.7 | 4.6 |
| Control | 1B 1 | 183.49 | 246.36 | 153.19 | 152.79 | 11.83 | 11.80 | 11.90 | 11.83 | 11.75 | 11.84 | 14.118 | 13.737 | 13.805 | 13.855 | 13.767 | 13.879 | 41.3 | 34.3 | 17.2 | 17.2 |
| | 1B 2 | 188.13 | 248.11 | 153.20 | 153.18 | 11.72 | 11.75 | 11.79 | 11.79 | 11.76 | 11.76 | 13.517 | 13.444 | 13.541 | 14.006 | 13.566 | 13.627 | 42.5 | 31.9 | 15.9 | 15.4 |
| | Average | 185.81 | 247.24 | 153.20 | 152.99 | 11.77 | 11.78 | 11.85 | 11.81 | 11.76 | 11.80 | 13.82 | 13.59 | 13.67 | 13.93 | 13.67 | 13.75 | 41.9 | 33.1 | 16.5 | 16.3 |
| | St. Dev. | 3.28 | 1.24 | 0.01 | 0.28 | 0.08 | 0.04 | 0.08 | 0.03 | 0.01 | 0.06 | 0.42 | 0.21 | 0.19 | 0.11 | 0.14 | 0.18 | 0.9 | 1.7 | 0.9 | 1.3 |
| Control | 1C 1 | 175.41 | 240.75 | 153.06 | 152.84 | 11.63 | 11.69 | 11.81 | 11.73 | 11.69 | 11.72 | 13.168 | 14.294 | 13.553 | 13.215 | 12.970 | 13.558 | 39.9 | 37.2 | 15.7 | 10.9 |
| | 1C 2 | 171.84 | 231.20 | 153.04 | 152.82 | 11.63 | 11.76 | 11.76 | 11.79 | 11.72 | 11.74 | 13.481 | 13.620 | 13.795 | 13.772 | 13.520 | 13.667 | 39.0 | 34.5 | 16.5 | 15.4 |
| | Average | 173.63 | 235.98 | 153.05 | 152.83 | 11.63 | 11.73 | 11.78 | 11.76 | 11.71 | 11.73 | 13.32 | 13.96 | 13.67 | 13.49 | 13.25 | 13.61 | 39.5 | 35.9 | 16.1 | 13.2 |
| | St. Dev. | 2.52 | 6.75 | 0.01 | 0.01 | 0.00 | 0.05 | 0.03 | 0.04 | 0.02 | 0.02 | 0.22 | 0.48 | 0.17 | 0.39 | 0.39 | 0.08 | 0.6 | 1.9 | 0.5 | 3.1 |
| Control | 1D 1 | 180.02 | 243.81 | 153.15 | 152.52 | 11.58 | 11.64 | 11.76 | 11.68 | 11.64 | 11.66 | 13.226 | 13.404 | 13.839 | 13.497 | 13.475 | 13.492 | 41.2 | 35.4 | 15.7 | 15.8 |
| | 1D 2 | 185.63 | 249.83 | 153.10 | 152.85 | 11.46 | 11.52 | 11.62 | 11.59 | 11.56 | 11.55 | 12.973 | 13.276 | 13.459 | 13.245 | 13.315 | 13.238 | 42.8 | 34.6 | 14.6 | 15.2 |
| | Average | 182.83 | 246.82 | 153.13 | 152.69 | 11.52 | 11.58 | 11.69 | 11.63 | 11.60 | 11.61 | 13.10 | 13.34 | 13.65 | 13.37 | 13.40 | 13.36 | 42.0 | 35.0 | 15.2 | 15.5 |
| | St. Dev. | 3.97 | 4.26 | 0.04 | 0.23 | 0.08 | 0.08 | 0.10 | 0.06 | 0.05 | 0.08 | 0.18 | 0.09 | 0.27 | 0.18 | 0.11 | 0.18 | 1.2 | 0.6 | 0.7 | 0.5 |
| 2104-67 | 2A 1 | 153.00 | 218.00 | 153.07 | 152.63 | 10.95 | 10.89 | 10.93 | 10.98 | 10.89 | 10.94 | 12.544 | 12.473 | 12.718 | 12.492 | 12.380 | 12.557 | 37.4 | 42.5 | 14.8 | 13.7 |
| | 2A 2 | 154.63 | 211.45 | 153.02 | 152.81 | 10.78 | 10.69 | 10.78 | 10.76 | 10.76 | 10.75 | 12.233 | 11.928 | 12.462 | 12.094 | 12.297 | 12.179 | 38.4 | 36.7 | 13.3 | 14.3 |
| | Average | 153.82 | 214.73 | 153.05 | 152.72 | 10.87 | 10.79 | 10.85 | 10.87 | 10.83 | 10.84 | 12.39 | 12.20 | 12.59 | 12.29 | 12.34 | 12.37 | 37.9 | 39.6 | 14.0 | 14.0 |
| | St. Dev. | 1.15 | 4.63 | 0.04 | 0.13 | 0.12 | 0.14 | 0.11 | 0.15 | 0.09 | 0.13 | 0.22 | 0.39 | 0.18 | 0.28 | 0.06 | 0.27 | 0.7 | 4.1 | 1.1 | 0.4 |
| 2104-67 | 2B 1 | 168.29 | 226.79 | 153.06 | 152.72 | 11.12 | 11.17 | 11.28 | 11.26 | 11.17 | 11.21 | 12.621 | 13.057 | 13.487 | 12.489 | 12.785 | 12.914 | 40.1 | 34.8 | 15.2 | 14.5 |
| | 2B 2 | 164.29 | 225.53 | 152.99 | 152.91 | 11.11 | 11.08 | 11.13 | 11.17 | 11.07 | 11.12 | 13.115 | 13.189 | 13.341 | 13.002 | 12.926 | 13.162 | 39.4 | 37.3 | 18.3 | 16.8 |
| | Average | 166.29 | 226.16 | 153.03 | 152.82 | 11.11 | 11.12 | 11.21 | 11.21 | 11.12 | 11.17 | 12.87 | 13.12 | 13.41 | 12.75 | 12.86 | 13.04 | 39.7 | 36.0 | 16.8 | 15.7 |
| | St. Dev. | 2.83 | 0.89 | 0.05 | 0.13 | 0.00 | 0.06 | 0.11 | 0.06 | 0.07 | 0.06 | 0.35 | 0.09 | 0.10 | 0.36 | 0.10 | 0.18 | 0.5 | 1.8 | 2.2 | 1.6 |
| 2104-67 | 2C 1 | 178.93 | 241.86 | 153.13 | 152.75 | 11.04 | 11.15 | 11.22 | 11.09 | 11.14 | 11.12 | 12.913 | 13.071 | 13.521 | 12.845 | 13.038 | 13.088 | 42.9 | 35.2 | 17.7 | 17.1 |
| | 2C 2 | 173.13 | 232.02 | 153.00 | 153.41 | 11.17 | 11.22 | 11.19 | 11.19 | 11.21 | 11.19 | 12.819 | 12.774 | 12.986 | 13.094 | 13.139 | 12.918 | 41.1 | 34.0 | 15.4 | 17.2 |
| | Average | 176.03 | 236.94 | 153.07 | 153.08 | 11.10 | 11.18 | 11.21 | 11.14 | 11.17 | 11.16 | 12.87 | 12.92 | 13.25 | 12.97 | 13.09 | 13.00 | 42.0 | 34.6 | 16.5 | 17.1 |
| | St. Dev. | 4.10 | 6.96 | 0.09 | 0.47 | 0.09 | 0.05 | 0.02 | 0.07 | 0.05 | 0.05 | 0.07 | 0.21 | 0.38 | 0.18 | 0.07 | 0.12 | 1.3 | 0.8 | 1.6 | 0.1 |
| 2104-67 | 2D 1 | 169.62 | 228.84 | 153.16 | 152.61 | 11.22 | 11.34 | 11.39 | 11.26 | 11.27 | 11.30 | 12.723 | 13.479 | 13.187 | 12.761 | 13.165 | 13.038 | 40.1 | 34.9 | 15.4 | 16.8 |
| | 2D 2 | 164.80 | 222.97 | 153.07 | 152.69 | 11.30 | 11.35 | 11.39 | 11.33 | 11.36 | 11.34 | 13.055 | 12.736 | 13.324 | 12.719 | 12.853 | 12.959 | 38.8 | 35.3 | 14.2 | 13.1 |
| | Average | 167.21 | 225.91 | 153.12 | 152.65 | 11.26 | 11.35 | 11.39 | 11.29 | 11.31 | 11.32 | 12.89 | 13.11 | 13.26 | 12.74 | 13.01 | 13.00 | 39.4 | 35.1 | 14.8 | 15.0 |
| | St. Dev. | 3.41 | 4.15 | 0.06 | 0.06 | 0.06 | 0.01 | 0.00 | 0.05 | 0.07 | 0.03 | 0.23 | 0.53 | 0.10 | 0.03 | 0.22 | 0.06 | 0.9 | 0.3 | 0.8 | 2.6 |
| 2104-69 | 3A 1 | 173.04 | 235.64 | 153.12 | 152.77 | 11.29 | 11.30 | 11.41 | 11.28 | 11.31 | 11.32 | 12.828 | 12.800 | 13.233 | 13.029 | 12.843 | 12.973 | 40.8 | 36.2 | 14.6 | 13.6 |
| | 3A 2 | 177.43 | 232.64 | 153.05 | 152.89 | 11.26 | 11.26 | 11.39 | 11.32 | 11.30 | 11.31 | 12.845 | 12.805 | 13.201 | 13.124 | 13.059 | 12.994 | 41.8 | 31.1 | 14.9 | 15.5 |
| | Average | 175.24 | 234.14 | 153.09 | 152.83 | 11.28 | 11.28 | 11.40 | 11.30 | 11.30 | 11.31 | 12.84 | 12.80 | 13.22 | 13.08 | 12.95 | 12.98 | 41.3 | 33.6 | 14.8 | 14.6 |
| | St. Dev. | 3.10 | 2.12 | 0.05 | 0.08 | 0.02 | 0.03 | 0.01 | 0.03 | 0.00 | 0.01 | 0.01 | 0.00 | 0.02 | 0.07 | 0.15 | 0.02 | 0.7 | 3.6 | 0.2 | 1.4 |
| 2104-69 | 3B 1 | 159.06 | 217.37 | 153.02 | 152.85 | 11.25 | 11.26 | 11.26 | 11.27 | 11.32 | 11.26 | 12.738 | 13.391 | 13.321 | 12.877 | 13.274 | 13.082 | 37.7 | 36.7 | 16.2 | 17.3 |
| | 3B 2 | 164.26 | 223.94 | 153.07 | 152.66 | 11.14 | 11.34 | 11.31 | 11.29 | 11.30 | 11.27 | 12.638 | 13.030 | 13.474 | 13.059 | 13.083 | 13.050 | 38.9 | 36.3 | 15.8 | 15.8 |
| | Average | 161.66 | 220.66 | 153.05 | 152.76 | 11.20 | 11.30 | 11.28 | 11.28 | 11.31 | 11.26 | 12.69 | 13.21 | 13.40 | 12.97 | 13.18 | 13.07 | 38.3 | 36.5 | 16.0 | 16.5 |
| | St. Dev. | 3.68 | 4.65 | 0.04 | 0.13 | 0.08 | 0.05 | 0.03 | 0.01 | 0.01 | 0.00 | 0.07 | 0.26 | 0.11 | 0.13 | 0.14 | 0.02 | 0.9 | 0.2 | 0.2 | 1.0 |
| 2104-69 | 3C 1 | 171.78 | 232.10 | 153.03 | 152.71 | 11.35 | 11.36 | 11.43 | 11.41 | 11.37 | 11.39 | 12.830 | 12.733 | 12.783 | 12.861 | 12.902 | 12.802 | 40.3 | 35.1 | 12.4 | 13.5 |
| | 3C 2 | 175.25 | 237.26 | 153.05 | 152.64 | 11.28 | 11.37 | 11.42 | 11.36 | 11.32 | 11.36 | 13.036 | 13.306 | 13.752 | 13.636 | 13.441 | 13.433 | 41.2 | 35.4 | 18.3 | 18.7 |
| | Average | 173.52 | 234.68 | 153.04 | 152.68 | 11.31 | 11.37 | 11.43 | 11.38 | 11.34 | 11.37 | 12.93 | 13.02 | 13.27 | 13.25 | 13.17 | 13.12 | 40.7 | 35.2 | 15.3 | 16.1 |
| | St. Dev. | 2.45 | 3.65 | 0.01 | 0.05 | 0.05 | 0.01 | 0.01 | 0.04 | 0.03 | 0.02 | 0.15 | 0.41 | 0.69 | 0.55 | 0.38 | 0.45 | 0.7 | 0.2 | 4.1 | 3.7 |
| 2104-69 | 3D 1 | 175.57 | 238.55 | 152.99 | 152.79 | 11.18 | 11.24 | 11.42 | 11.20 | 11.22 | 11.26 | 13.661 | 13.701 | 13.731 | 13.868 | 14.094 | 13.740 | 41.6 | 35.9 | 22.1 | 25.6 |
| | 3D 2 | 175.85 | 238.19 | 152.95 | 152.99 | 11.24 | 11.26 | 11.24 | 11.27 | 11.25 | 11.25 | 13.244 | 13.143 | 13.468 | 13.413 | 13.495 | 13.317 | 41.7 | 35.5 | 18.4 | 20.0 |
| | Average | 175.71 | 238.37 | 152.97 | 152.89 | 11.21 | 11.25 | 11.33 | 11.24 | 11.23 | 11.25 | 13.45 | 13.42 | 13.60 | 13.64 | 13.79 | 13.53 | 41.6 | 35.7 | 20.2 | 22.8 |
| | St. Dev. | 0.20 | 0.25 | 0.03 | 0.14 | 0.05 | 0.01 | 0.13 | 0.05 | 0.02 | 0.00 | 0.29 | 0.39 | 0.19 | 0.32 | 0.42 | 0.30 | 0.0 | 0.3 | 2.6 | 4.0 |

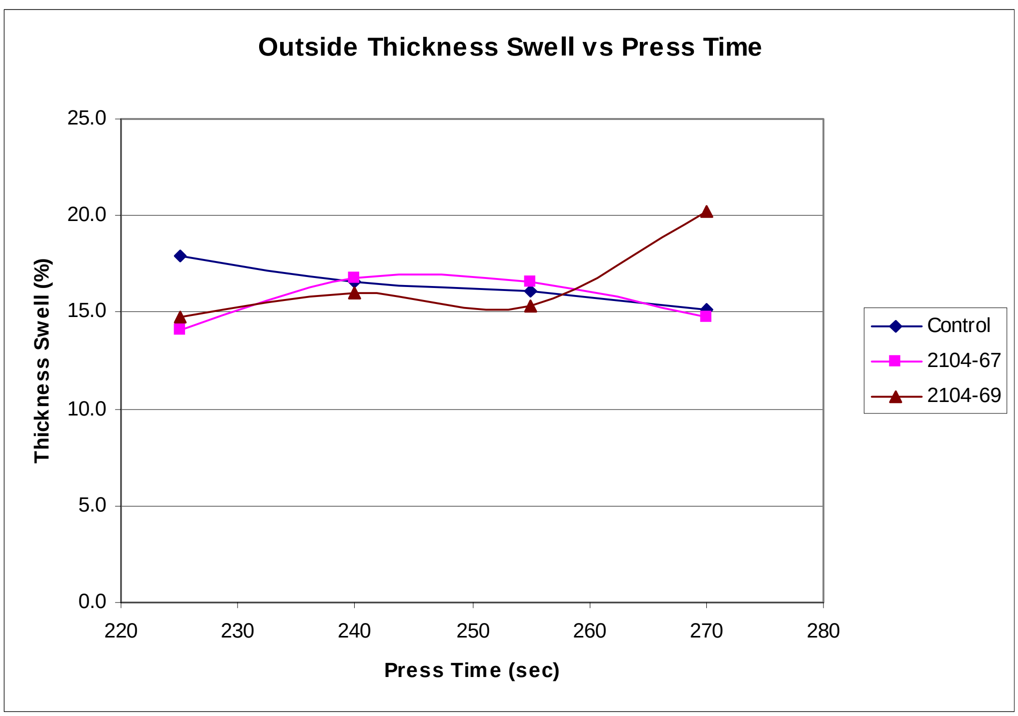
Water Absorption

| Press Time | Control | 2104-67 | 2104-69 |
|---|---|---|---|
| 225 | 35.1 | 39.6 | 33.6 |
| 240 | 33.1 | 36.0 | 36.5 |
| 255 | 35.9 | 34.6 | 35.2 |
| 270 | 35.0 | 35.1 | 35.7 |

Thickness Swell

| Press Time | Control | 2104-67 | 2104-69 |
|---|---|---|---|
| 225 | 18.0 | 14.0 | 14.8 |
| 240 | 16.5 | 16.8 | 16.0 |
| 255 | 16.1 | 16.5 | 15.3 |
| 270 | 15.2 | 14.8 | 20.2 |



# Section V
## Centre Point Loading Flexure Test after 2 Hour Water Boil

# CENTRE POINT LOADING FLEXURE TEST AFTER 2 HR BOIL

**Liquid Control Resin and Two Ensyn Resins**

**Project:** **4139-1** **Date of entry:** May 8,2003

Test Span: 10.5" or 266.7mm Date Tested: May 8,2003

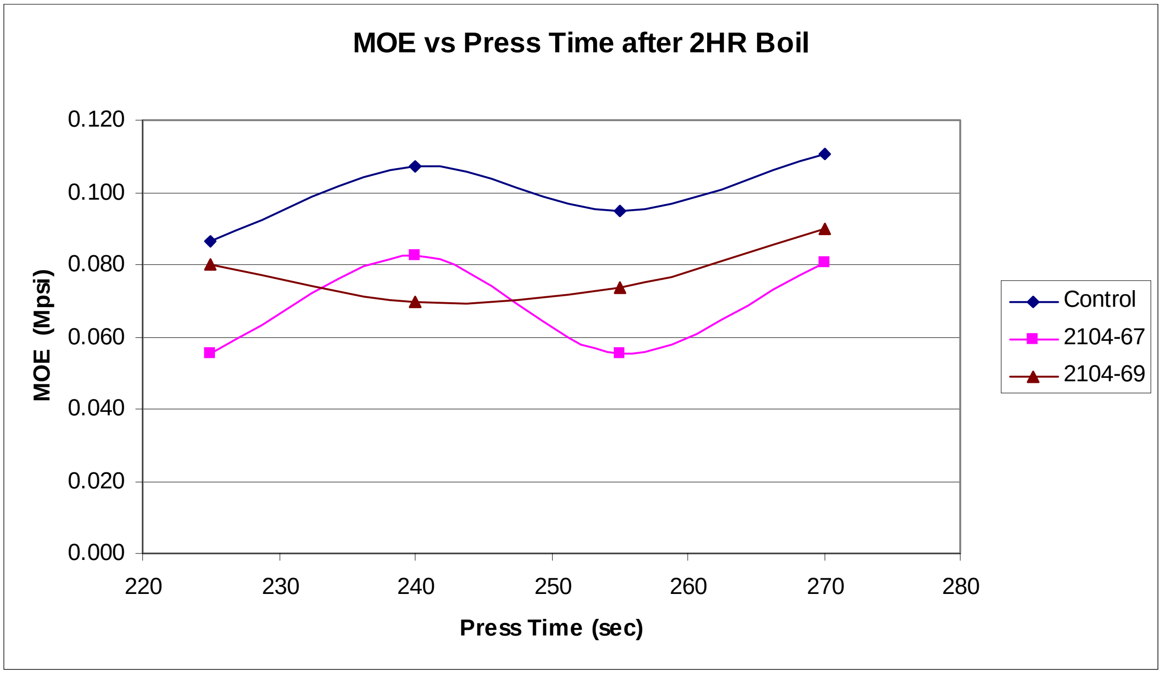
Specimen Size: 3.0" x 7/16" x 12 1/2" Test Speed: 10.5 mm/min

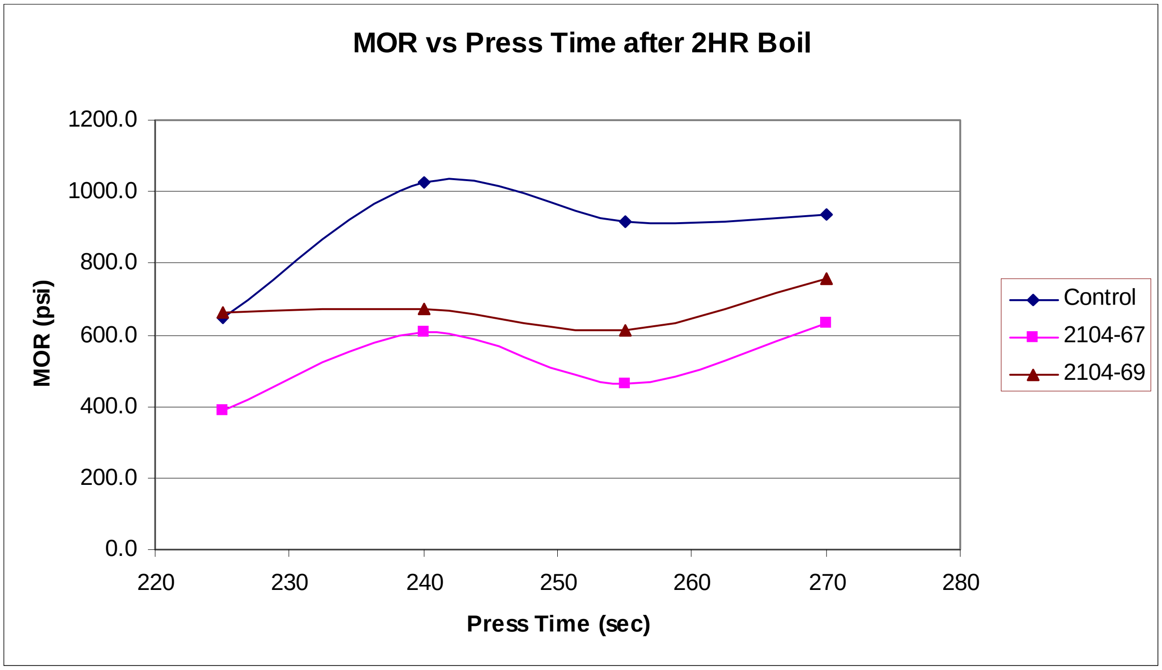
**NOTE:** In the panel No. description the first digit (ie 1A-1) 1 is 225 sec board press time, 2 is 240 sec, 3 is 255 sec, 4 is 270 sec.
Control is 2104-70 resin, 2104-67 and 2104-69 are Ensyn resins
All panels are made with 3 % resin solids add on rate and1% wax solids- 1 flexure samples per panel

| | | Code # | Before Soak Width (mm) | Before Soak Depth (mm) | After Soak Width (mm) | After Soak Depth (mm) | Max. Load (kg) | MOR (MPa) | MOE (GPa) | MOR (psi) | MOE (Mpsi) | M.C. after Soak % | S.G. Product | Density (lb/cu.ft) | Length (mm) | Before Soak Weight (gms) | After Soak Weight (gms) | Dry wt. (grams) | Thick. Swell % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Control | 4139-1-1A-1 | 76.74 | 11.47 | 77.00 | 18.80 | 30.9 | 4.451 | 0.597 | 645.7 | 0.087 | 163.8 | 0.624 | 40.0 | 319 | 180.10 | 462.19 | 175.23 | 63.9 |
| 2 | Control | 4139-1-1B-1 | 76.78 | 11.48 | 77.14 | 17.52 | 42.63 | 7.058 | 0.739 | 1024.0 | 0.107 | 138.9 | 0.702 | 41.0 | 319 | 184.74 | 471.36 | 197.29 | 52.6 |
| 3 | Control | 4139-1-1C-1 | 76.90 | 11.43 | 76.99 | 19.33 | 46.44 | 6.329 | 0.653 | 918.0 | 0.095 | 170.9 | 0.646 | 44.7 | 319 | 200.76 | 490.74 | 181.14 | 69.1 |
| 4 | Control | 4139-1-1D-1 | 76.59 | 11.34 | 76.90 | 17.84 | 40.24 | 6.446 | 0.763 | 935.0 | 0.111 | 154.4 | 0.249 | 42.8 | 319 | 190.28 | 475.11 | 186.73 | 57.3 |
| 5 | 2104-67 | 4139-1-2A-1 | 76.66 | 10.98 | 77.32 | 19.26 | 19.65 | 2.686 | 0.382 | 389.6 | 0.056 | 174.8 | 0.635 | 40.7 | 319 | 175.30 | 468.57 | 170.54 | 75.4 |
| 6 | 2104-67 | 4139-1-2B-1 | 76.72 | 10.98 | 77.27 | 19.24 | 30.44 | 4.172 | 0.568 | 605.2 | 0.082 | 167.5 | 0.672 | 42.8 | 319 | 184.43 | 482.88 | 180.51 | 75.2 |
| 7 | 2104-67 | 4139-1-2C-1 | 76.76 | 11.07 | 77.12 | 19.60 | 24.24 | 3.208 | 0.380 | 465.3 | 0.055 | 168.8 | 0.659 | 42.1 | 319 | 182.99 | 480.42 | 178.70 | 77.1 |
| 8 | 2104-67 | 4139-1-2D-1 | 76.83 | 10.92 | 77.30 | 18.76 | 30.28 | 4.364 | 0.555 | 633.0 | 0.081 | 167.6 | 0.662 | 42.1 | 319 | 180.81 | 473.97 | 177.09 | 71.8 |
| 9 | 2104-69 | 4139-1-3A-1 | 76.73 | 11.03 | 77.15 | 18.10 | 29.42 | 4.563 | 0.550 | 661.9 | 0.080 | 166.9 | 0.659 | 42.1 | 319 | 182.09 | 475.06 | 177.98 | 64.1 |
| 10 | 2104-69 | 4139-1-3B-1 | 76.75 | 11.05 | 77.05 | 19.48 | 34.66 | 4.647 | 0.481 | 674.1 | 0.070 | 172.2 | 0.654 | 41.7 | 319 | 180.86 | 481.27 | 176.83 | 76.3 |
| 11 | 2104-69 | 4139-1-3C-1 | 76.76 | 10.89 | 77.12 | 18.81 | 29.48 | 4.236 | 0.509 | 614.4 | 0.074 | 162.9 | 0.660 | 42.1 | 319 | 179.92 | 462.90 | 176.05 | 72.7 |
| 12 | 2104-69 | 4139-1-3D-1 | 76.74 | 10.95 | 77.17 | 17.54 | 31.54 | 5.208 | 0.621 | 755.5 | 0.090 | 168.9 | 0.647 | 41.3 | 319 | 177.41 | 466.66 | 173.52 | 60.2 |

| | MOR | | |
|---|---|---|---|
| Press Time | Control | 2104-67 | 2104-69 |
| 225 | 645.7 | 389.6 | 661.9 |
| 240 | 1024.0 | 605.2 | 674.1 |
| 255 | 918.0 | 465.3 | 614.4 |
| 270 | 935.0 | 633.0 | 755.5 |

| | MOE | | |
|---|---|---|---|
| Press Time | Control | 2104-67 | 2104-69 |
| 225 | 0.087 | 0.056 | 0.080 |
| 240 | 0.107 | 0.082 | 0.070 |
| 255 | 0.095 | 0.055 | 0.074 |
| 270 | 0.111 | 0.081 | 0.090 |



# APPENDIX H

Odor Tests

Weyerhaeuser

Naomi High
WTC 1F3
Weyerhaeuser Company
Tacoma, WA 98477
(253) 924-6412
(253) 924-6324 FAX

August 21, 2002

Ensyn Group, Inc.
Mr. Terry Bentley, (V.P. Development)
380 Hunt Club Road
Suite 201
Ottawa, Ontario
Canada
K1V 1C1
FAX: 613-248-2260

Post-it™ brand fax transmittal memo 7671 | # of pages ▸ 8

| | |
|---|---|
| To Mr. Bentley | From Naomi High |
| Co. Ensyn | Co. Weyerhaeuser |
| Dept. Development | Phone # 253 924 6412 |
| Fax # 613 248 2260 | Fax # 253 924 6324 |

Dear Mr. Bentley,

In response to a previous agreement between Weyerhaeuser and Ensyn, a brief study was recently conducted in an effort to access differences in odor between conventional OSB and that made with a surface resin that was partially comprised of MNRP. The protocol used for this study was that which was previously disclosed to Weyerhaeuser from Ensyn in an e-mail message (please see attachment). The following paragraph provides some additional information regarding the actual study. The raw data gathered is also attached to this memo.

On Tuesday, August 20, 2002, control samples and samples from ARC (Alberta Research Council) were laid out on a table in a room. The control samples were 28-inch squares of 7/16" Edson board with a PF resin surface and a pMDI core. The ARC samples were 28-inch squares of 7/16", with a MNRP based surface and a pMDI core. Both sets of boards were made with the same strands. The control samples were numbered 1 through 5. The samples from ARC were shipped to us numbered 41-45. Four subjects, three male and 1 female, were brought into the room at different times. The subjects do not work for wood products and have no experience in this area. The subjects had the most unpleasant perceptions of the control samples. The total score of samples 5, 41, 44, and 45, was an eleven. The total score of samples 4, 42, and 43, was a twelve. Samples 2 and 3, had a total score of 6. Sample 1 scored a 9.

Please let me know if I can be of further assistance.

Sincerely,

Naomi High
Weyerhaeuser Company

cc. Jack G. Winterowd

Please Mark Box with a X based on your perception of the sample

| Sample # | | 1-unpleasant aroma | 2-slightly unpleasant aroma | 3-no unusual aroma | 4-slightly pleasant odor | 5-pleasant odor |
|---|---|---|---|---|---|---|
| 1 | | ✓ | | | | |
| 2 | | ✓ | | | | |
| 3 | | ✓ | | | | |
| 4 | | | ✓ | | | |
| 5 | | | ✓ | | | |
| 41 | | | ✓ | | | |
| 42 | | | ✓ | | | |
| 43 | | | ✓ | | | |
| 44 | | | ✓ | | | |
| 45 | | | ✓ | | | |

Thank You!

Please Mark Box with a X based on your perception of the sample

| Sample # | | 1-unpleasant aroma | 2-slightly unpleasant aroma | 3-no unusual aroma | 4-slightly pleasant odor | 5-pleasant odor |
|---|---|---|---|---|---|---|
| 1 | | | | X | | |
| 2 | | | X | | | |
| 3 | | | | X | | |
| 4 | | | | | X | |
| 5 | | | | X | | |
| 41 | | | | X | | |
| 42 | | | | X | | |
| 43 | | | | X | | |
| 44 | | | | X | | |
| 45 | | | | X | | |

Thank You!

Please Mark Box with a X based on your perception of the sample

| Sample # | | 1-unpleasant aroma | 2-slightly unpleasant aroma | 3-no unusual aroma | 4-slightly pleasant odor | 5-pleasant odor |
|---|---|---|---|---|---|---|
| 1 | | X | | | | |
| 2 | | X | | | | |
| 3 | | X | | | | |
| 4 | | | | | X | |
| 5 | | | X | | | |
| 41 | | | X | | | |
| 42 | | | | | X | |
| 43 | | | | | X | |
| 44 | | | X | | | |
| 45 | | | | X | | |

Thank You!

| Sample # | | 1-unpleasant aroma | 2-slightly unpleasant aroma | 3-no unusual aroma | 4-slightly pleasant odor | 5-pleasant odor |
|---|---|---|---|---|---|---|
| 1 | | | | | X | |
| 2 | | | X | | | |
| 3 | | X | | | | |
| 4 | | | X | | | |
| 5 | | | | X | | |
| 41 | | | | | X | |
| 42 | | | | X | | |
| 43 | | | | X | | |
| 44 | | | | | X - but 43 smells | |
| 45 | | | | X - very slight | | |

Thank You!

# Memorandum

Date: November 15, 2002

To: Terry Bentley

From: Garth Gorsky

RE: Odour Test of Ensyn OSB Panels

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Two boards (a control board and an Ensyn board identified only as 1w2 and 5w2) were cut into squares two inches by two inches and randomly labelled with numbers from one to ten. I was later informed that 1w2 was the control board and 5w2 was the Ensyn board.

On October 22, 2002 I conducted a test panel of 7 individuals in a government office. Some were clerical staff, some management and some professionals but none were experts in wood technology or marketing. The samples were laid out on a table and each individual was asked to smell the samples and then rate them according to the attached table.

**Results**

In total there were 70 individual ratings (7 people times 10 samples) and 54 of the 70 ratings fell in the “No Unusual Aroma” s category). Three of the individuals found “No Unusual Aroma” in any of the samples. Five ratings were “Slightly Unpleasant” and 11 ratings were “Slightly Pleasant. Three of the five “Unpleasant” ratings were from the Ensyn board and two were from the control. On the “Slightly Pleasant Odour” rating, five were from the Ensyn board and six were from the control.

In all cases where individuals had ratings other than “No Unusual Aroma”, they always rated at least one Ensyn sample and at least one control sample as either pleasant or unpleasant. In other words no one found only one of the boards to be pleasant or unpleasant.

Using a scoring system like that used by Weyerhaeuser resulted in a total score for the Ensyn board of 107 and the control board 109 signifying no significant difference in the aroma of the two boards. In this scoring, “Slightly Unpleasant Aroma” ratings were given 2 points, “Slightly Pleasant Odour” were given 4 points and the middle rating was given 3 points.

Attached are copies of the score sheets.

Garth Gorsky

# Summary of Ratings for Each Sample

| Sample No. | Total Points | 1- Unpleasant Aroma | 2- Slightly unpleasant aroma | 3- No unusual aroma | 4- Slightly pleasant odor | 5- pleasant odor |
|---|---|---|---|---|---|---|
| 1 E | 20 | Nil | 1 | 6 | Nil | Nil |
| 2 E | 20 | Nil | 1 | 6 | Nil | Nil |
| 3 C | 23 | Nil | Nil | 5 | 2 | Nil |
| 4 C | 21 | Nil | 1 | 5 | 1 | Nil |
| 5 C | 23 | Nil | Nil | 5 | 2 | Nil |
| 6 E | 24 | Nil | Nil | 4 | 3 | Nil |
| 7 E | 21 | Nil | 1 | 5 | 1 | Nil |
| 8 E | 22 | Nil | Nil | 6 | 1 | Nil |
| 9 C | 22 | Nil | Nil | 6 | 1 | Nil |
| 10 C | 20 | Nil | 1 | 6 | Nil | Nil |

E = Ensyn Resin 107 points C = Control Resin 109