

*Solvent fractionation of switchgrass optimisation, mixed feedstock
experiments and new lignin isolation methods*

REPORT OF KNOXVILLE

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1. SYMBOLS AND ABBREVIATIONS

1.1. Abbreviations

AQ	aqueous
CF	clean fractionation
P_{eq}	equilibrium pressure
P_{offset}	offset pressure
VI	virtual instrument
SG	switchgrass
TTEP	true thermodynamic equilibrium pressure

1.2. Chemicals

MIBK	methyl isobutyl ketone
DI H ₂ O	deionized water
EtOH	ethanol 190 proof
NaCl	sodium chloride
Et ₂ O	diethyl ether

1.3. Tradenames

Excel	Microsoft Corporation
LabVIEW	National Instruments Corp.

2. ABSTRACT

Converting lignocellulosic biomass into biofuels is an option for improving energy independence, increasing national security and reducing greenhouse emissions. The burning of fossil fuels like coal and oil release CO₂ which is a major cause of global warming. Biofuels are produced from plants that grow today and thus do not increase CO₂ to the atmosphere. The use of switchgrass as feedstock is supported by the fact that it is a non-food biomass source, it is a versatile and adaptable plant that grows in poor soils and land conditions that don't support food crops. To isolate the required cellulose from switchgrass, the solvent fractionation process, a organosolv process, has been investigated. The organosolv biomass fractionation process (Clean Fractionation) separates the lignocellulosic raw material into cellulose, hemicellulose and lignin. The biomass is treated with a ternary mixture of methyl isobutyl ketone, ethanol, water and an acid promoter. The process is rapid, simple, and gives good separation of the individual lignocellulosic components of different feedstocks. Overall, the process is a simple model of initial biorefinery pretreatment steps, demonstrating the conversion of a complex raw material into three simpler renewable process streams, each of which can be used for the production of fuels and chemicals.

A new a proprietary method of lignin and hemicelluloses recovery from black liquor obtained from the solvent fractionation process has been investigated. This method offers a fast separation of layers and thus rapid access to the lignin component and aqueous phases. This method is simpler, has fewer processing steps, is easier to handle, and more amenable to the measurement of the amount of solvent used in each step.

Key words: biofuels, solvent fractionation, switchgrass, salting out method, organosolv

3. INTRODUCTION

The solvent fractionation process is an organosolv process (biomass treated with organic solvents). The delignification process is carried out using organic solvents and a mineral acid as catalyst. The organosolv process breaks the internal bonds between lignin and hemicellulose and separates the lignin and hemicellulose fractions. (Panday 2009) Advantages of the solvent fractionation process in comparison to more conventional pulping processes are (Bozell, O'Lenick et al. 2009):

1. The presence of organic solvents reduces the viscosity of the pretreatment medium, improving penetration into the biomass and facilitating a more efficient removal of lignin.
2. The presence of solvents can retard the redeposition of lignin onto the other biomass components after separation is complete. pH control and alkali washing of the cellulose have been used to address redeposition.
3. Organosolv cellulose is easier to purify. This is important in the paper industry as a solution for environmental issues associated with pulp bleaching, but also to the chemical industry, as it frequently requires starting materials of high purity.
4. The cellulose shows improved bleachability and viscosity retention when compared to cellulose prepared using conventional processes such as kraft or soda.
5. The improved properties of organosolv cellulose have been exploited in the production of viscose and carboxymethylcellulose.

The solvent fractionation also called clean fractionation or organosolv process and is the ternary solvent mixture remains a single phase throughout the entire pulping process.

The biomass source is treated with a ternary mixture of MIBK (methyl isobutyl ketone, ethanol and water in the presence of an acid promoter. Optimized conditions developed at UT use a 16/34/50 mass ratio of MIBK/EtOH/H₂O for a period of 56 minutes at 140°C in the presence of 0.05 H₂SO₄. The lignin and hemicelluloses are dissolved by the solvent system and are continuously washed by the reactor in flowthrough mode. After separation, the reactor is drained leaving behind cellulose as a solid which is fiberized and washed with water. The black liquor contains the lignin

and hemicellulose and is then processed using a new proprietary method to cause phase separation to give the organic phase containing the lignin and an aqueous phase containing the hemicelluloses. The Clean Fractionation process can be used to process a wide range of biomass feedstocks. (Bozell, O'Lenick et al 2009)

4. MATERIALS AND METHODS

The feedstock used during this project is all non-food biomass sources. All feedstocks were dried to a moisture content of less than 10%.

4.1. Feedstock.

- Switchgrass (*Panicum virgatum*) a versatile and adaptable summer perennial grass native to North America that grows in poor soils and land conditions that don't support food crops. It's a non-food biomass source.
- White poplar (*Populus alba*) pulp grade chips. Dimensions of 4 by 2 cm and a thickness of 0.5 cm

4.2. Separations.

Figure shows the 4 phases of the CF process. The vacuum fragment, the filling phase, the heat-up phase and the flow fragment.

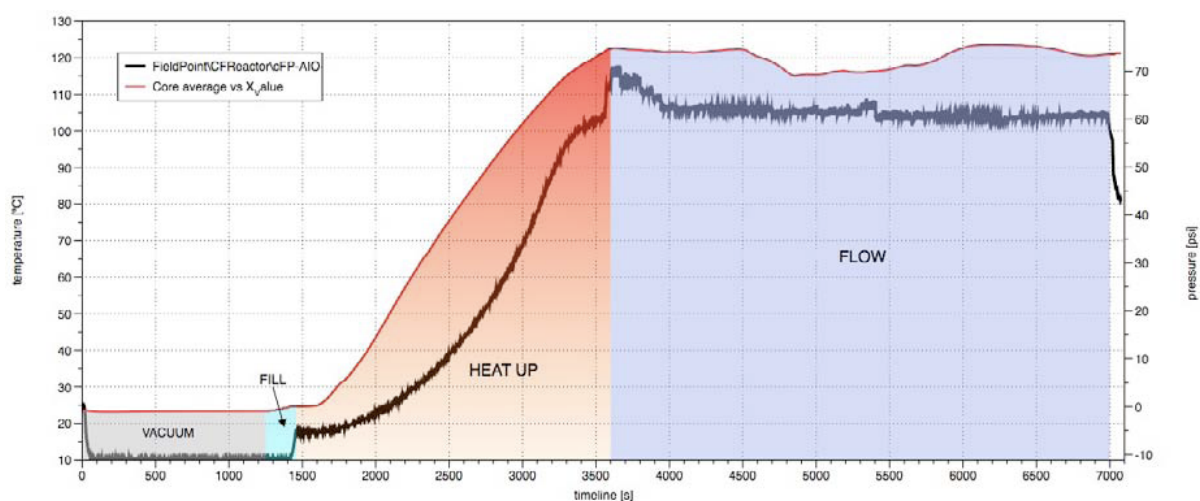


Figure 1 shows the 4 phases of the CF process (Buchinger, 2009)

A perforated Teflon basket was charged with the feedstock and put into the reactor. The run can be split into four phases. In the vacuum fragment, a vacuum pump pulls a vacuum on the entire system, causing the feedstock to degas and improve the penetration of the feedstock with solvent, in the filling phase. After 20 minutes the valve of the feed tank is opened and the vacuum fills the reactor to the outlet of the reactor. The height of filling is controlled by the amount of solvent introduced into a vacuum trap (100ml) installed at the reactor outlet. After the filling height is reached, the valve to the feed tank is closed and the heating phase is initiated. The heater bands around the reactor are controlled by electrical controllers, which are set to the operation temperature. The heating process takes approximately 40 minutes to come to a core temperature of 140°C. The solvent pump is switched on as soon as three of the heater bands have reached operation temperature, and the fourth heater band is 5°C below operation temperature. The pressure that can be reached by the heating process itself is called the equilibrium pressure (P_{eq}). The flow phase is initiated as soon as:

$$P_{eq} + P_{offset} = \text{Valve opening}$$

P_{offset} is chosen by the operator and entered into the computer. It has a control and stabilizing function. This extra pressure is generated by the solvent pump.

The Badger valve is controlled by LabVIEW and programmed to open as soon as operation pressure is reached. The offset was set to 0.015 psi (before 10PSI). The stroke rate of the pump was set to 12 s^{-1} . The stroke rate is not changed during the run. As soon as the flow starts the count-down timer on the LabVIEW virtual instrument, set to run for 56 minutes, is activated. During this flow phase, a flow measurement is taken at the reactor outlet every 5 minutes and should be at 3.5 l/run(56 minutes of flowthrough). After the flow phase the heaters were turned off and the remaining black liquor is drained into the collection pot.

Figure 2 shows the correlation between temperature and P_{eq} . It also shows the three operation temperature and the reached pressures.

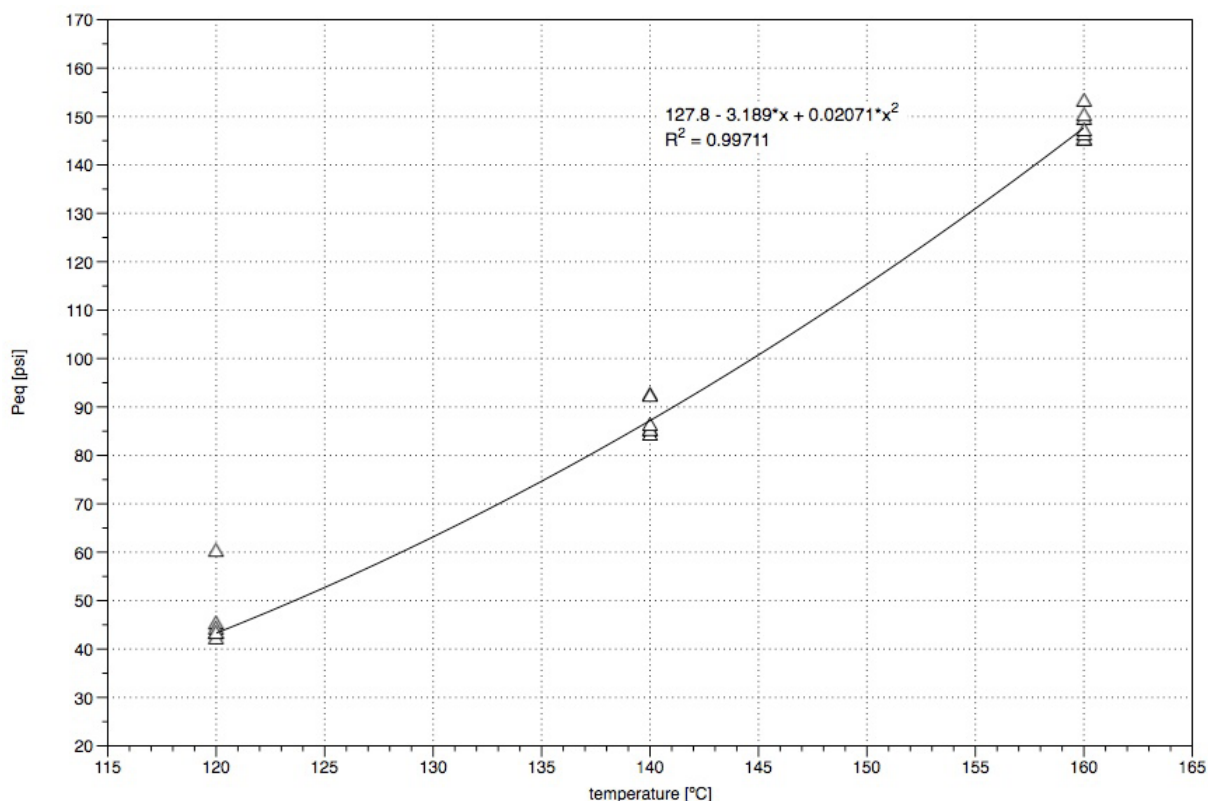


Figure 2: Shows the correlation between temperature and P_{eq} (Buchinger, 2009)

After the solvent fractionation process is complete there are two fractions left. The cellulose in the reactor and the black liquor collected in the collection pot.

4.3. Cellulose

To ease the process of fiberizing the cellulose 3 liters of deionized water are added to the cellulose 24 hours prior to blending it. The cellulose is then fiberized with a mixer for a period of 5 minutes to help release the solvent from the biomass and to reduce the particle size of the cellulose for easier downstream processing.

The fiberized cellulose is then placed into a Buchner funnel that is under vacuum and is washed with deionized water for 1 hour. After the washing the cellulose is covered with a latex membrane causing the vacuum to press and dry the cellulose for 1 hour. The dry cellulose is then weighed and stored in a freezer for further processing.

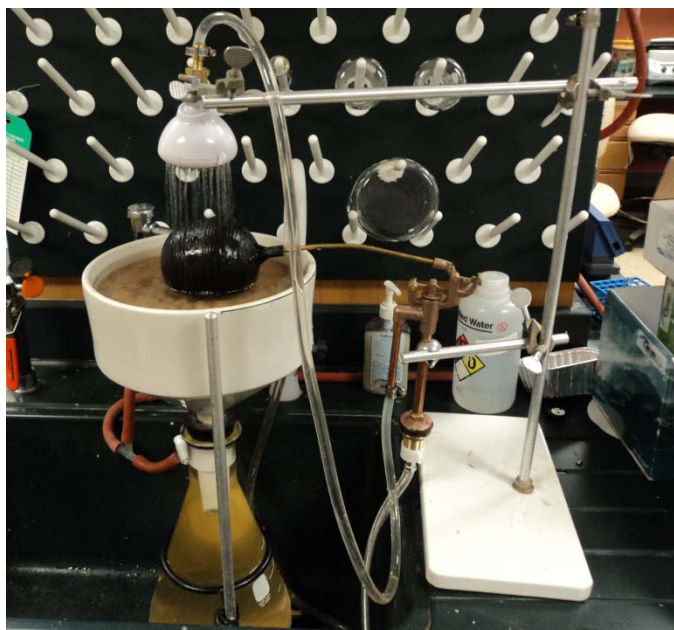


Figure 3 shows the washing of the fiberized cellulose (Maraun,2010)

4.4. Black Liquor

The black liquor was separated using the a new separation method. This new separation method was introduced by Prof. J. Bozell of the Centre for Renewable Carbon at University of Tennessee, Knoxville, USA.

4.4.1. New lignin isolation method

This new method is used to separate the black liquor into the organic and the aqueous phase. This new separation method is still in the phase of patenting and thus is not discussed in this paper.

4.4.2. Organic Phase

For lignin recovery, the organic layer is stripped via rotary evaporation at 45°C to give a dry gummy clay like material. This material is treated with 100ml diethyl ether (Et_2O) and allowed to sit overnight. Following the overnight treatment, the Et_2O is filtered with a Buchner funnel and filter paper. A second treatment with 100ml Et_2O is performed followed by a second filtration. The filtrate is hooked to a vacuum and is allowed to dry overnight in the round bottomed flask. The next day, a dry, brown solid powdered lignin is recovered.

4.4.3. Aqueous phase lignin

The aqueous phase still contains lignin and thus is collected and stripped of ethanol and MIBK via rotary evaporation at 50°C. The AQ is then filtered through a filter paper in a medium sized Büchner funnel. The lignin is then dried by vacuum for 12 hours and weighed.

4.5. Hemicellulose

After the separation a 500ml sample of aqueous was stored in the freezer for further analysis. HPLC will give a qualitative result of the composition of the hemicelluloses in the aqueous phase (in progress). Ash and acid soluble lignin will be found in this phase (Fengel and Wegener 2003).

5. MIXED FEEDSTOCK CAMPAIGN

The idea for the mixed feedstock campaign was introduced by Prof. J.Bozell.

Mixed Feedstock Campaign

Run temperature 140°C

Acid concentration in solvent: 0.05 M H₂SO₄

Run#	SG	Poplar	SG / Poplar
	g	g	In % of Basket
# 76	243	50	90/10
# 77	202	125	75/25
# 78	135	250	50/50
# 79	67	375	25/75
# 80	27	450	10/90

Replicates

# 81	243	50	90/10
# 82	202	125	75/25
# 83	135	250	50/50
# 84	67	375	25/75
# 85	27	450	10/90

To fill a reactor with feedstock it requires:

270 Gram of Switchgrass OR
500 Gram of woodchips

Feedstock: Switchgrass / Poplar

1%= 2.7 Gram of Switchgrass
1%= 5 Gram of Poplar chips

Figure 4: Run matrix for mixed feedstock campaign (Maraun, 2010)

The main aim for the mixed feedstock campaign was, to collect data on the quality of lignin, hemicelluloses and cellulose that the mixed feedstock would provide, and to determine how the fractionation process would have to be adjusted to get similar results as those of single feedstock.

The new proprietary method was used to separate the black liquor from the mixed feedstock. The cellulose was fiberized, washed and frozen for further analysis. This step is still in progress.

6. Reactor

Figure 5 shows the reactor schematic used at UT. The reactor used at the University of Tennessee is a flow-through design. The reactor is constructed from Hastelloy C276. This metal is resistant to many aggressive chemicals along with the sulfuric acid used in this study. The reactor is heated by 4 electric heater bands. The reactor is loaded with biomass contained in a perforated Teflon basket. The reactor is then filled with solvent provided by a solvent feed tank. The solvent and biomass is then heated to operation temperature by heater bands controlled via controllers in the electric enclosure. When the selected temperature is reached, the reactor is then operated in a flow-through mode, solvent is pumped through the bottom of the reactor for about 1 hour and the extract is collected in a collection tank. A small scale heat exchanger is used to preheat solvent entering the bottom of the reactor. Pressure is maintained using a pressure control valve controlled by LabVIEW. LabVIEW is a virtual programming environment designed to interface with measurement and control instruments.

After completion the remaining solvent in the reactor is drained in the collection tank.

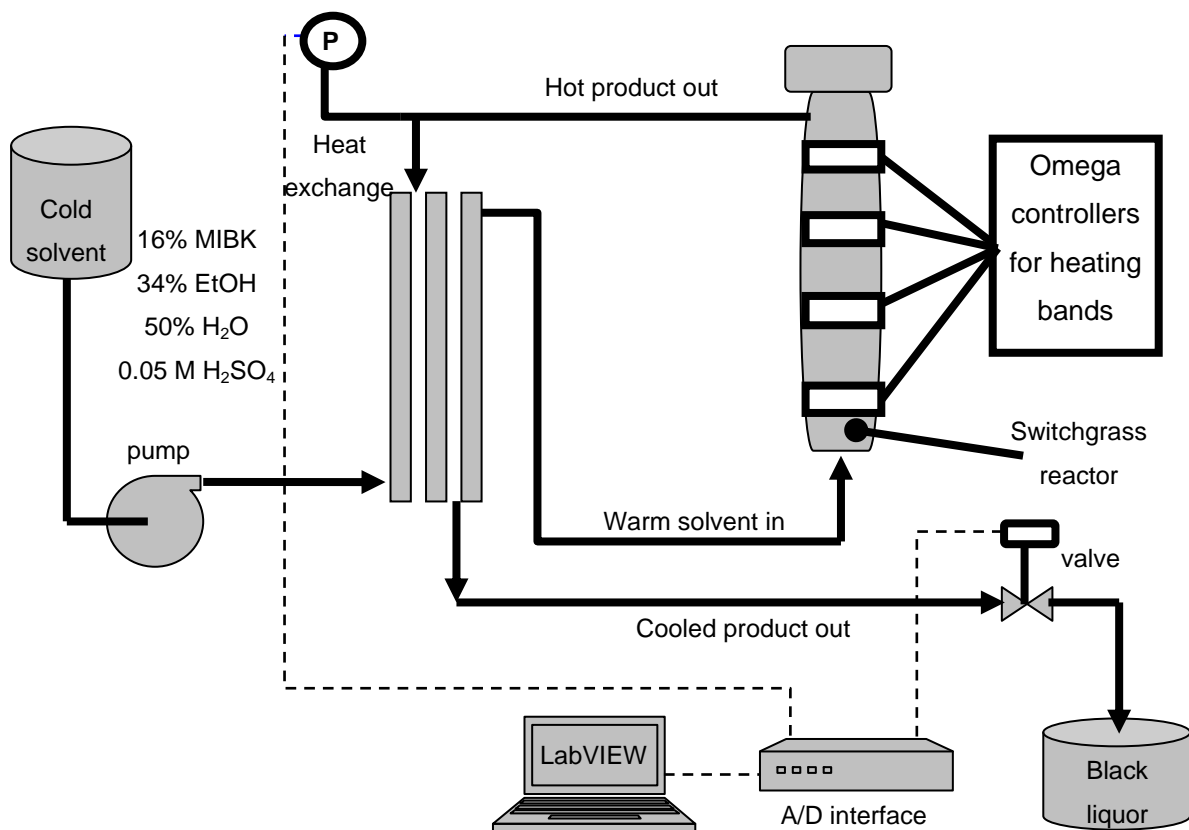
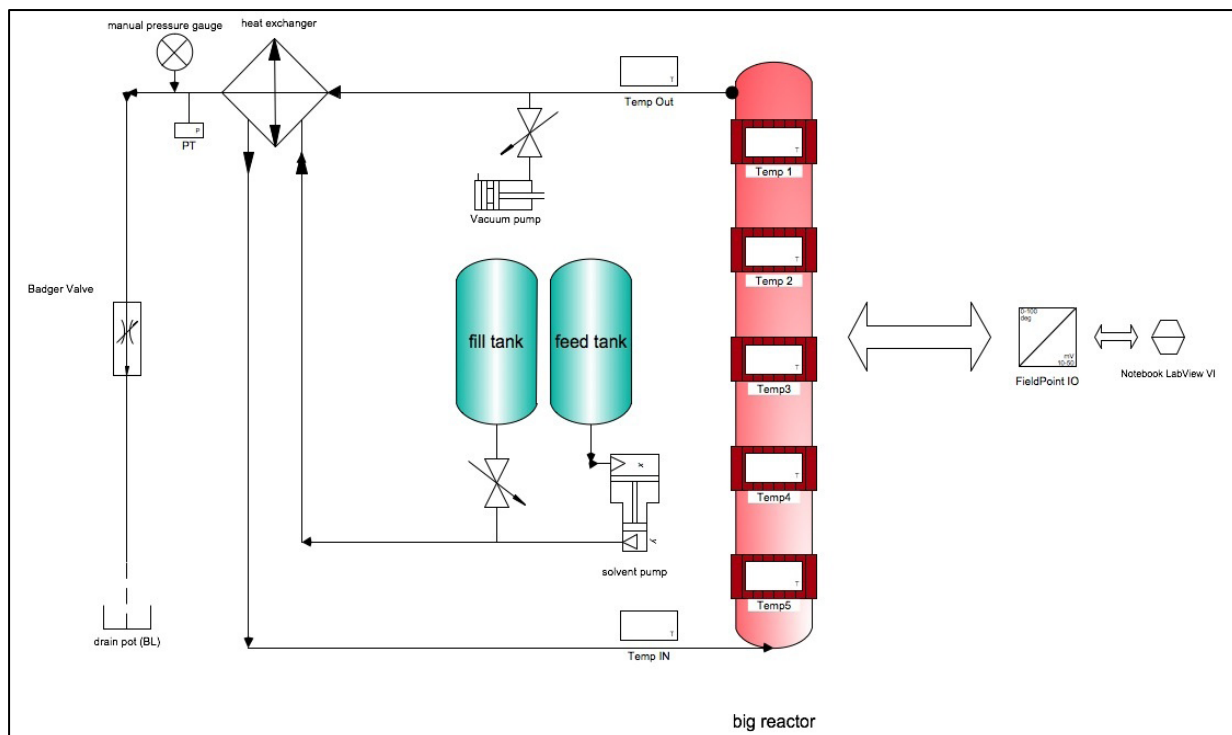


Figure 5: Reactor schematic used at UT (CJ O’Lenick, 2009)

6.1. Materials

Figure 7 shows the big reactor schematic used at UT. The reactor used for this experiment is a small version of the one shown but has the same build-up.



aggressive chemicals. Hasteloy consist of Ni and Mo and is a trade name of the Company Haynes International Ltd. (Buchinger, 2009).

Prior to the solvent fractionation, a vacuum is drawn on the biomass by a vacuum pump (GAST DOA-P707-AA PUMP). Then the reactor is filled with solvent by opening a valve. After the reactor is filled the 4 heater bands (Watlow) heat the system to a set temperature. When operation temperature is reached the solvent pump (Williams CR P500V225 CR TC.) starts to push solvent through the reactor. The flow through of solvent is then controlled by a badger valve type (766 BLRA Model 4). This model operates pneumatically. The badger valve itself is controlled by an ITT Conoflow Model GT4108EB, which converts a input signal of 4-20mA DC to a proportional 3-27 PSIG output signal (ITT 2009). The signal is connected to the Field Point Module and controlled by LabVIEW. (Buchinger, 2009). An approximate of 3.5 L is used for filling the reactor and another 3 L of solvent are used for pumping through the system.(small reactor). All the preparation steps are documented on a reactor logsheet.

At the start of this project, the small reactor had not been used for a period of more than two months. The latest run's had been problematic and it was decided to overhaul the reactor. The solvent pump had been losing its prime with the result that flow stopped and quick action had to be taken to restore flow. The cause for the loss of prime in the solvent pump was not yet identified and thus no specific action could be taken. An early test run ended abruptly with the opening of an emergency release valve which again left more unexplained questions.

6.2. Reactor repair and enhancements

Figure 9 shows the different repairs and enhancements made to the reactor during this project.

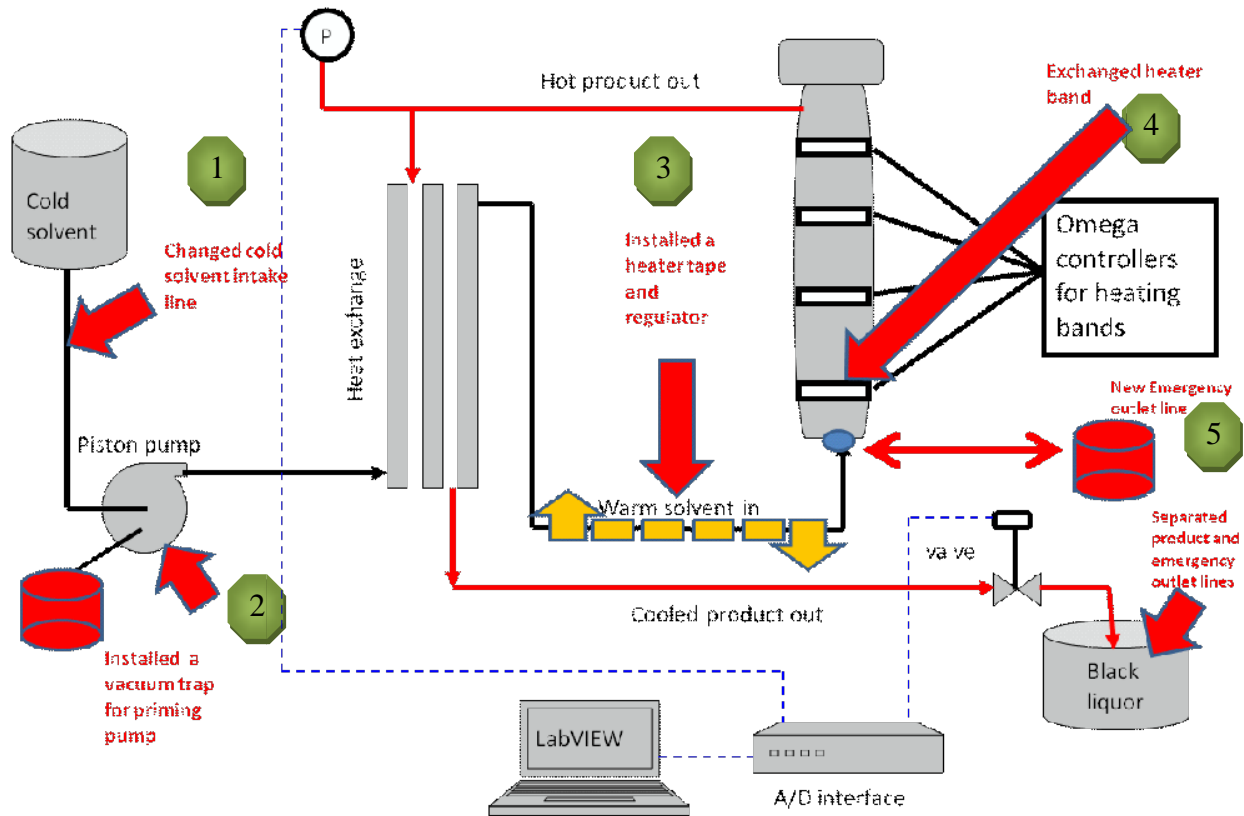


Figure 8: Shows repairs and enhancements made during the project (Maraun, 2010)

1. Solvent intake lines. Diagnosis: Leaks at multiple junctions and a connector pair caused air to leak into the inlet lines impeding the suction process performed by the Williams solvent pump thus causing trouble during operation or even breakdown. Action taken: Dismantled the inlet lines, removed the connector pair (Swage lock – standard fittings) and retightened all junctions that were indispensable.
2. Installation of a flow trap for better control: This would be a quicker, easier way to restore flow in the case of flow loss during operation and would also help when preparing the reactor for a run. Furthermore, visible proof of liquid flowing is an advantage for the operator. The flow trap was connected to both the vacuum pump and the bleeder valve on the Williams solvent pump.

3. Rheostat controlled heater tape. This elastic heater tape was installed by wrapping it around the solvent inlet line at the bottom of the reactor. Its purpose was to support and help the operator to keep and control operation temperature in the reactor. This had a positive impact on maintaining the temperature.
4. Heater band: During operation heater band No.4 at the bottom of the reactor burned out and had to be replaced.
5. New emergency release line: During a test run an unexpected emergency release occurred. Before, the emergency line, led into the same collection pot in front of the reactor as the extracted black liquor. Since flow measurements are taken by the operator repeatedly it was considered a hazard and the new emergency outlet line connected to a emergency valve was laid to lead to the back of the reactor into a new collection pot.

6.3. Reactor optimization using LabVIEW

Figure 10, shows the virtual control panel LabVIEW used for reactor operation. LabVIEW is a virtual programming environment designed to interface with measurement and control instruments.

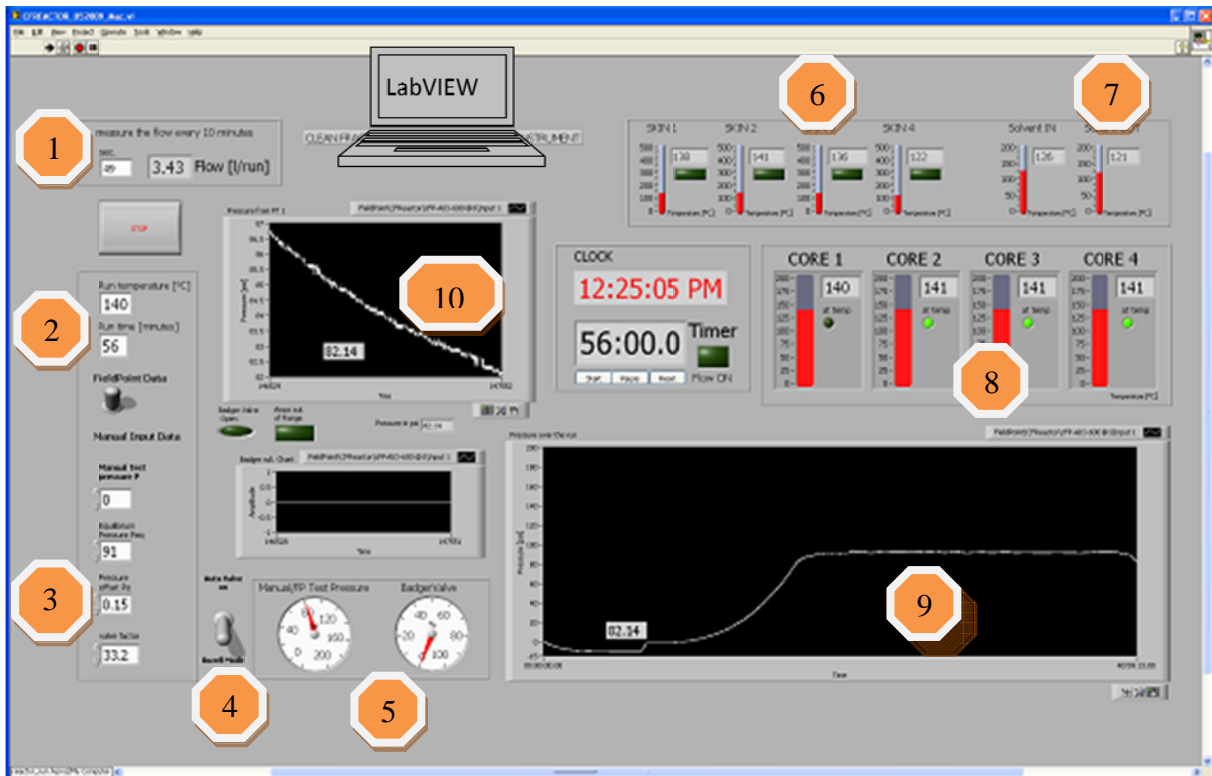


Figure 9: Virtual control panell (LabVIEW) (Maraun, 2010)

1. Flow measurement. During the actual run flow measurements are made every 5 or 10 minutes. A 50 ml cup is held under the outlet line, the time it takes to fill up is recorded and entered into the given textbox. The computer then calculates the flow in [l/run].
2. Run temperature and duration in minutes. This does not regulate the temperature of the reactor. The information is entered and stored with the operation data collected by LabVIEW.
3. Badger valve control. These 4 parameters are used to control the badger valve, which can run in 2 different modes, the “Bozell” mode or the “Auto valve” mode (4).
4. Pressure gages for both reactor and badger valve.

5. Reading of the thermocouples attached to the outside skin of the reactor beneath the insulation.
6. Solvent IN and Solvent OUT thermocouples giving information about the solvent temperature flowing in and out of the reactor.
7. Thermocouples reading the core temperatures inside of the reactor.
8. Pressure diagram. This diagram gives a very detailed picture of happenings inside of the reactor.
9. Pressure – Time Diagramm

6.3.1. Parameters

Figure 11 shows the adjustable parameters for reactor operation can be divided into computer controllable and external adjustable parameters.

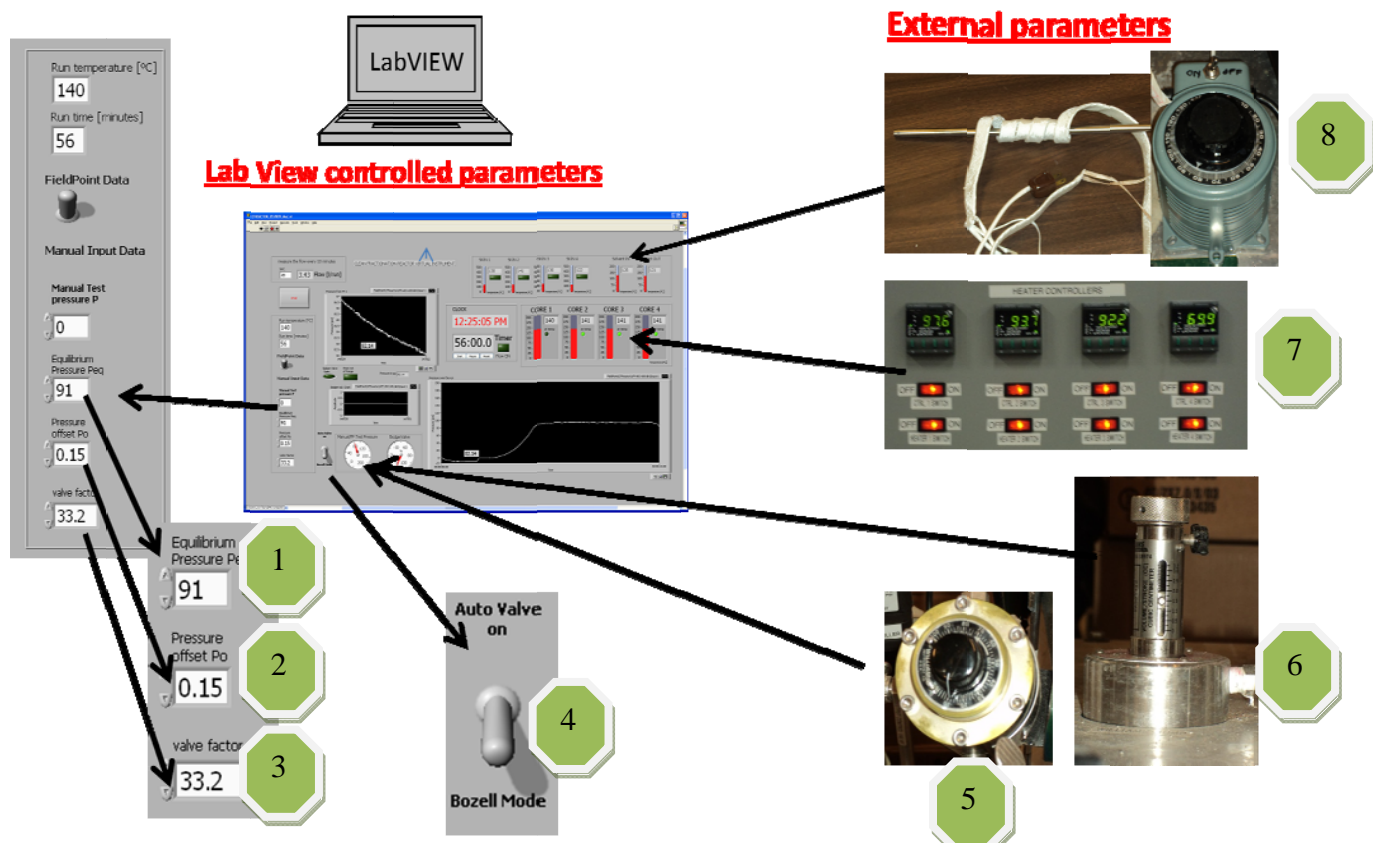


Figure 10: Controllable or adjustable parameters of the reactor (Maraun, 2010)

6.3.1.1. Computer controlled parameters:

1. Equilibrium Pressure. This pressure is chosen by the operator (Thus not the real equilibrium pressure at the given temperature. The word “equilibrium” is used as an artifact from earlier nomenclature. The true thermodynamic equilibrium pressure is simply the pressure of the reactor at a fixed, static temperature in the closed reactor system without flow). Tests showed that the actual pressure during the run should not be less than 6 psi higher than the thermodynamic equilibrium pressure at the set temperature. This helped stabilize the pressure fluctuations in the reactor.

Temperatures used at UT and their true thermodynamic equilibrium pressure plus the additional 6 PSI gives the chosen run Pressure. (see Figure 1)

- 120°C – 45 PSI TTEP plus the additional 6 PSI = 51 PSI
 - 140°C – 85 PSI TTEP plus the additional 6 PSI = 91 PSI
 - 160°C – 145 PSI TTEP plus the additional 6 PSI = 151 PSI
2. Pressure offset. This Pressure is added to the above chosen equilibrium pressure and triggers the badger valve. As soon as the pressure rises above the run Pressure the badger valve starts to open. The offset Pressure was lowered from 10 PSI to as little as 0.15 PSI, this had an immediate effect on the pressure fluctuations and stability throughout the run.
 3. Badger valve opening percent. The badger valve has an opening range between 0 and 100 %. The badger valve opening percent is the crucial part in achieving the optimal operation conditions. The opening percentage has to be corrected to the first decimal if not to cause excess or low pressure. The aim was to achieve the right amount of flow. This means to release (using the Badger valve) and at the same time feed (using the Williams pump) the right amount of solvent through the reactor.

4. "Auto valve" or "Bozell" mode switch. Before the process would be started in Bozell mode (manual mode) and then after some time would be switched into auto valve mode. The computer would then take pressure readings of the reactor and calculate the opening percentage of the badger valve for every reading taken. During the project it was decided not to use "auto valve" mode any more since there was no possibility to make adjustments during operation. The "auto valve" mode re-programming is currently in progress.

6.3.1.2. External adjustable parameters and their reconfiguration

These are parameters that are adjusted manually by the operator at different stages.

5. The Stroke Rate control. This switch controls the stroke rate of the Williams piston pump. Before, the pump would be started with a stroke rate of 25 strokes then reduced to 10 strokes. This was changed to 12 strokes throughout the run.
6. Stroke length. The length of the stroke of the Williams pump can be changed to shorter or longer. Tests with different stroke lengths were done, but no changes decided.
7. Temperature controllers. These were used to power and regulate the heater bands on the reactor. On the lower heater band we added an additional 2.5°C to every chosen run temperature. This proved to help keep the temperatures more stable. We encountered less temperature fluctuations.
8. Rheostat. This was used to control the temperature of the newly installed heater tape. The heater tape proved to be effective in keeping temperatures stable.

7. RESULTS

7.1. Reactor repair, enhancements and optimization

The optimization and enhancements made, improved and simplified manual operation of the reactor and had a significant impact on the operation sequence of the reactor. These changes led to a more stable run with less pressure fluctuations make it easier to reproduce.

7.1.1. Process Parameter reconfiguration

Table 1 shows the process parameter reconfiguration that were tested and decided during this project.

Parameters	Prior	Optimized
1, heater bands	120/140/160 °C	heater 4 inc.by 2.5°C
2. Pump stroke rate (pre set)	25/10	12
3. Pump stroke length	pre set to 1.5	no change
4.VI Equilibrium pressure	85 PSI at 140°C	optimized to 91 PSI
5. Pressure offset	10 PSI Bozell then Auto valve	Bozell mode
6.Valve factor (%)	starting value25 at 140 °C	33.2 at 140 °C
7.Heater tape	not available	150 °C

Table 1 shows the process parameter reconfiguration (Maraun. H. 2010)

Changes to the process parameters were tested and implemented to further improve the solvent fractionation process and to reduce operation changes that influence the performance of the process.

1. The heater band 4 was set 2.5°C higher than the rest of the heater bands. This helped stabilize the core temperature of the reactor, it was observed that both temperature fluctuations and their occurrence were in a lesser extent.
2. The solvent pump stroke rate was fixed to 12 stokes.

3. There were no changes observed when adjusting the stroke length of the Williams pump. Thus no changes were made.
4. The VI equilibrium pressure was set to 91 PSI ((140°C – 85 PSI TTEP plus the additional 6 PSI = 91 PSI)) after it was observed that a lower pressure had an impact on the Badger valve behavior causing pressure fluctuations.
5. Pressure offset: The pressure build-up of 10 PSI added to the true thermodynamic equilibrium pressure is built up by the solvent pump. This value was reduced to 0.15 PSI. Due to this reduction the pressure, to activate the Badger valve, is reached with every pump stroke. Before this it would take several strokes. The badger valve is now constantly working, making the run go smooth, with very low fluctuation of about 0.2 PSI.
6. The valve factor is essential for the amount of flow and for pressure balance and stability during operation.
7. The installed heater tape helped stabilize the temperature in the reactor.

7.1.2. Mixed feedstock campaign

These results summarize the analysis performed on the lignin.

7.1.3. Lignin

Table 1 shows the distribution of both SG and Poplar from RUN# 76 - # 80. The distribution was calculated out of the knowledge that 270g of SG and 500g of Poplar will fill the perforated Teflon basket to the top.

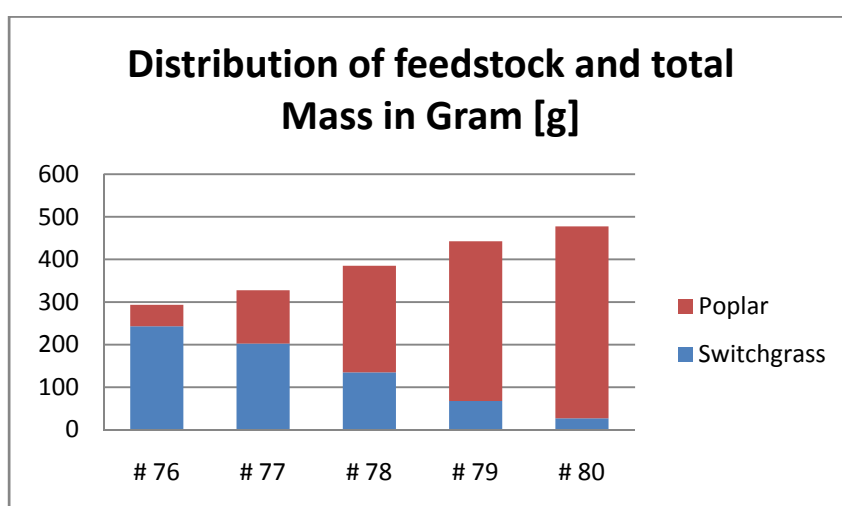


Table 2 shows the total mass of feedstock at equal volume. (Maraun, 2010)

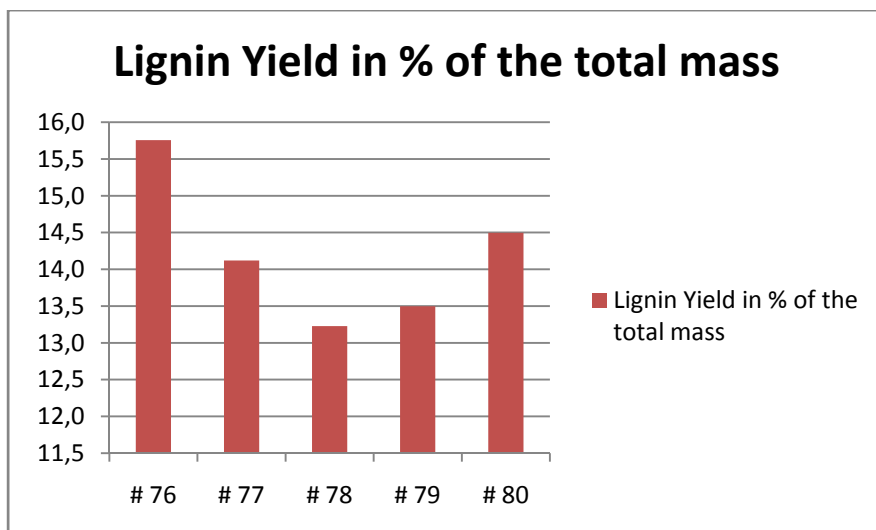


Table 3 shows the amount of Lignin recovered from the pulp in percent (Maraun, 2010)

Table 2 shows the amount of lignin recovered from RUN #76 – #80. The lignin yield of switchgrass found in literature is 17.2% (Mosier, Qin). Run #76, which had a 90% fraction of switchgrass came to lignin yield of 15, 8%to the figure found in the literature.

It turned out that the higher the poplar fraction the more incomplete the break down. A number of unreacted poplar wood chips were observed. There a few alternatives that should be tested to complete the organosolv process in this case. Higher operation temperatures as well as higher acid concentration are possible solutions.

Further experiment should be carried out to figure out how time affects the yield.

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