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OPTIMIZATION OF A FLUIDIZED BED REACTOR FOR THE PRODUCTION OF ACETONE FROM ISOPROPYL ALCOHOL

By:

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A thesis submitted to the faculty of The University of Mississippi in partial fulfillment of the requirements of the Sally McDonnell Barksdale Honors College

Oxford

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Abstract:

For the optimization of the isopropyl alcohol to acetone process, Unit 1100, the Net Present Value (NPV) was used as the objective function to observe the economic impact of each of the optimization factors being tested. During the optimization process, it is not only important to optimize the process's profitability but also to ensure that the process operates efficiently and meets product specifications. The goal of this optimization was to alter variables from the base case calculations of Unit 1100 to maximize the NPV of the unit. AVEVA ProII software was used to simulate the process and make changes to the desired optimization variables. The results of each simulation were then used to calculate the NPV after each change to the process. A new design was made to maximize the process's NPV. This report details the optimization strategies used to maximize the acetone production process's NPV and several other key considerations

Table of Contents

Abstract	1
Table of Contents	2
Executive Summary	3
Design	4
Optimization	5
High Temperature Catalyst	5
Low Temperature Catalyst	9
Final Optimization	13
Optimized Design	16
Process Safety	18
Impact Factors	18
Global	18
Cultural/Environmental/Social	18
Economic	19
Fluidized Bed Reactor Optimization	19
Conclusion	22
References	23
Appendix	24

Executive Summary:

The OM petrochemical facility is designing a pharmaceutical-grade acetone process with a required purity of 99.9 wt%. The acetone is made using the endothermic dehydrogenation of isopropyl alcohol (IPA).

$$(CH_3)_2 CHOH \xrightarrow{1} (CH_3)_2 CO + H_2$$

IPA acetone hydrogen

This process uses a catalyst-filled plug flow reactor (R-1101) to facilitate the reaction of IPA to acetone and hydrogen gas. For this process, two catalyst options are given: a high-temperature catalyst and a low-temperature catalyst. The high temperature catalyst has an operating temperature range of 240-700°C and the low-temperature catalyst has an operating temperature range of 170-250°C. For both of these catalysts, a base case model is made to determine the favorable option to optimize further.

After performing optimization simulation on Microsoft Excel and a chemical production simulation software, AVEVA ProII, it was found that the higher temperature catalyst would be the better option to optimize. This decision was made by testing similar optimization steps on both the low and high-temperature base case processes and finding that the high-temperature catalyst has the highest net present value (NPV) at the end of the first cycle of \$128.7 million dollars. After choosing the best catalyst option, the number of tubes in the reactor, the length of the reactor, the temperature of the heating loop, and heat integration were tested. The final NPV of the optimized process was \$140.5 million. It is recommended for the company to move forward with the project

and to begin drafting the P&ID, move to the next class of process design cost estimation, and continue looking for future areas of optimization.

Design:

For the production of pharmaceutical-grade acetone, there are three major sections in the processing facility: reactor preparation and reactor, separation preparation, and separation. For the reactor preparation and reactor, this section includes a heat exchanger and a pump to directly adjust the temperature and pressure before the IPA enters the reactor. Once in the reactor, the IPA reacts to form acetone and hydrogen gas. The next major section is the separation preparation where the mixture enters two heat exchangers in series which is then fed to a flash drum. Once out of the flash drum, the vapor flows into an absorber where the vapor IPA and acetone are absorbed by the water. The liquid phase from the flash drum meets this new liquid stream of IPA, acetone, and water and is sent to the separation section. In the final major section, two distillation towers are used to separate the mixture. The first distillation tower separates the acetone from the mixture and the second distillation tower separates the water from the IPA. The unreacted IPA is recycled. The PFD for the acetone production can be seen in Figure 1 below.



Figure 1: Process Flow Diagram for the Production of Acetone

Optimization:

High Temperature Catalyst:

The completed base case for the high temperature catalyst was \$122.2 million. The first step in the optimization process was to improve the pressure feeding into the reactor. The base case operating pressure for the reactor feed pump (P-1101) was 2.3 bar. The company then tested 5 different pressures to find the optimal value that gives the highest resulting NPV. After the optimization cycles, it was found that the optimum pressure was 2.4 bar. The increase in NPV is due to the relationship between gas-phase reaction kinetics and increasing pressure. As the pressure increases in a system the volume decreases, which ensures better mixing. The effects of the operating pressure of P-1101 on the NPV can be seen in Figure 2 below. The color coding for data points is as follows: blue data points are tested values, black data points are the base case

values, and red data points are the optimized values. The same convention is used in all of the other figures as well.



Figure 2: High-Temperature Catalyst P-1101 Outlet Pressure

The next area of optimization in the reactor preparation is to change the outlet temperature of E-1101. This heat exchanger uses high-pressure steam as a heating medium and directly changes the inlet temperature in the reactor, R-1101. At higher temperatures the reaction rate increases which increases the single-pass conversion. This increases the NPV of the process as the temperature increases. With the optimized temperature being 250°C increasing the NPV to \$123.4 million. Temperatures above 250°C were not tested because they violate the basic principles of thermodynamics. The high-pressure steam that is being used for the utility stream operates at 254°C. This means that it is not feasible for this utility stream to heat the process stream to a temperature above its own operating temperature. The effects of the inlet reactor temperature can be seen in Figure 3 below.



Figure 3: High-Temperature Catalyst E-1101 Outlet Temperature

The stream exiting the reactor has four major components that need to be separated: Hydrogen, Acetone, IPA, and Water. To prepare this stream for separation two heat exchangers are used in series to a flash drum, where the vapor mixture goes to an absorber T-1101. Adding more trays to the absorber results in better separation. Starting with the base case number of 10 trays, more trays were added until there was a decrease in the NPV. With the optimized number of trays being 25, this increases the NPV to \$123.6 million, as seen in Figure 4.



Figure 4: High-Temperature Catalyst T-1101 Number of Trays

The next area of optimization in T-1101 is the water flow rate into the tower. A higher flow rate of water results in better absorption but adding too much water increases the duty in the two distillation towers later used in the process. Starting with the base case flow rate of 41 kmol/hr the NPV increases until it approaches the optimized value of 200 kmol/hr. This optimized flow rate increases the NPV to \$128.7 million, as seen in Figure 5.



Figure 5: High-Temperature Catalyst Water Flow Rate T-1101

The last area of optimization for the high temperature catalyst was the separation section. In this area, the two distillation tower trays and feed trays are changed to lower the initial duty values. After each optimization, the distillation towers were resized, and the feed tray was changed. For T-1102 the optimized number of trays and the optimized feed tray location were already used so the NPV remained unchanged at \$128.7 million. In T-1103 another tray was added increasing the number of trays from 9 trays to 10 trays. However, this change was minor, only increasing the NPV by around \$40,000. The final NPV for the high temperature catalyst is \$128.7 million after the six optimized changes. The effects of each of the optimization variables can be seen in Figure 6 below.



Figure 6: High-Temperature Catalyst Waterfall Diagram

Low Temperature Catalyst:

When optimizing the low-temperature catalyst process, the company started with the same area of optimization as with the high temperature catalyst. Due to the gas-phase reaction, an increase in pressure has a positive correlation with the increase of the kinetics in a reaction. Starting with a base case of 2.3 bar, the optimized operating pressure was found to be 2.7 bar. The optimum pressure had a final NPV of \$118 million; an increase of just over \$2 million, as seen in Figure 7.



Figure 7: Low-Temperature Catalyst P-1101 Outlet Pressure

The next stage of optimization taken was to optimize the temperature leaving the heat exchanger (E-1101) heating the reactor feed. Due to the relationship between temperature and reaction kinetics, the company tested higher temperatures. The company assumed a temperature difference in the reactor of 15°C. That decision was made based on the idea that as the temperature difference decreases, the kinetics in the reaction increase. Moving forward with the process, it would be beneficial to investigate and optimize the temperature difference to determine the optimum exiting temperature for the heat exchanger. With an optimum temperature found to be 235°C, the optimized NPV was \$119.8 million. This was another almost \$2 million increase from the base case value of 230°C, as seen in Figure 8.



Figure 8: Low-Temperature Catalyst E-1101 Outlet Temperature

Following the base reactor optimization, the next step was to optimize the absorber tower (T-1101). The absorber column's purpose was to remove any vapor IPA and acetone from the exiting hydrogen stream and return it to the process. An increase of trays increases the efficiency of the tower. Knowing this, the company tested different numbers of trays to find the maximum NPV for the process. The optimized tray number was found to be 20, doubling the base case value of 10. With the increase of trays, the new NPV was found to be almost \$119.93 million, increasing the value from the base \$119.86 million, as shown in Figure 9.



Figure 9: Low-Temperature Catalyst T-1101 Number of Trays

After improving the tray number for T-1101, the company then optimized the flow rate of water into the tower. One of the largest problems in the process was the amount of acetone and IPA lost into the hydrogen vapor stream. An increase in the flow rate allows for more efficient absorption of the acetone and IPA. Doing this allowed for more improved uses of raw materials and helped decrease the cost spent on the raw materials into the process. Shown in Figure 10, the optimized flow rate of 150 kmol/hr increased the NPV of the system to \$122 million.



Figure 10: Low-Temperature Catalyst Water Flow Rate T-1101

The next two areas of optimization were the number of feed trays in distillation columns T-1102 and T-1103. An increase in trays increases the separation of acetone from the IPA and water stream in T-1102 and the IPA recycle stream from the wastewater stream in T-1103. The changes in tray number had a minimal effect on the NPV for both towers. The change for T-1102 had a \$600,000 increase on the NPV and the change for T-1103 had a \$319,000 increase on the NPV. In total, the changes to both towers had an almost \$1 million increase, giving a final NPV for the optimized process of \$123 million. The effects of each of the optimization variables can be seen in Figure 11 below.



Figure 11: Low-Temperature Catalyst Waterfall Diagram

Final Optimization:

Upon completion of the initial optimization cycles for both the high and low temperature catalyst options, the company chose to further optimize the high temperature catalyst option. This decision was based on the NPV values found from the initial optimization cycles. The high temperature catalyst yielded an NPV that was \$5.5 million more than the low temperature catalyst. After selecting the catalyst for further optimization, the company investigated several areas of optimization that were not changed in the first cycle.

The first area of optimization investigated was the number of tubes in the reactor. In the base case, the plug flow reactor, R-1101, contained 450 tubes. When optimized, the company chose a reactor with 350 tubes; the new reactor design increased the project's NPV by \$3.8 million, as shown in Figure 12.



Figure 12: Optimization of Reactor Tube Number

After optimizing the number of tubes in the reactor, the company began investigating the optimum length of the reactor. In the base case, the length of the reactor was 6.096 meters. From the ProII simulations, the NPV of the project continually increased as the reactor length was decreased. Reactor lengths were tested from 7 meters down to 1 centimeter. Although the simulations showed that very short reactor lengths were possible, the company decided to test the shorter reactor lengths using numeric integration. These tests showed that reactor lengths below 2 meters were not actually possible. At lengths less than 2 meters, the simulations and numeric integration calculations were widely varied for the reaction conversion. Because of these variations, the company selected a reactor length of 2 meters as the optimum. This change yielded an NPV increase of \$5.8 million bringing the NPV to \$138.3 million, as shown in Figure 13.



Figure 13: Optimization of Reactor Length

The company also optimized the temperature in the NC-17 heating loop. In the base case, the heater, H-1101, was operating at a temperature of 360°C. The company found an optimum operating temperature of 410°C would increase the NPV by \$600,000 yielding an NPV of \$138.9 million, as shown in Figure 14.



Figure 14: Optimization of Temperature in H-1101

In the separation section, the company further optimized the acetone recovery of the process. During the final optimization cycle, another heat exchanger and a flash drum, E-1109 and V-1105 respectively, were added to the process on the vapor stream exiting the reflux drum of the acetone column. These changes were made because there was a significant amount of acetone remaining in the vapor phase after exiting the tower in the base case. This caused some of the product to be lost to the hydrogen fuel gas stream. Adding the heat exchanger and flash drum allows for this acetone to be condensed and recovered in the acetone product stream. These changes increased the NPV by \$2.2 million to a new value of \$141.2 million.

Finally, the company performed heat integration calculations using the minimum utilities minimum number of exchangers (MUMNE) approach for the reactor section of the process. These calculations yielded a redesigned heat exchanger network around the reactor. In the new design, the reactor effluent is used as the hot utility in E1101 to heat the reactor feed. Also, a new heat exchanger, E-1110, was added after E-1101 to heat the reactor feed to the desired temperature. These changes increased the NPV by \$300,000 to \$141.5 million.

During the optimization process, there was some miscommunication on the allowable error in product purity. Therefore, the purity specification was changed from 99.88 wt% acetone to exactly 99.90 wt% acetone. This adjustment in purity decreased the NPV by \$1 million to a final NPV of \$140.5 million.

Optimized Design:

Heat integration was used in the reactor preparation section and the separation preparation. E-1102 was removed from the optimized process and replaced with E-1110. This new heat exchanger uses the high-temperature reactor effluent to heat the IPA going into the reactor.

In the distillate section of T-1102, a new heat exchanger and flash drum were added to the vapor stream of V-1103. This maximizes the amount of acetone recovered in the separation section of this process. Both of these changes can be seen in Figure 15 below.



Figure 15: Optimized Process Flow Diagram for the Production of Acetone

Process Safety:

For the final optimized design, there are two areas of concern for safety. The first area is the reactor effluent, the temperature in this steam is 410°C. With a temperature this high, proper insulation is required around this section. The next area for safety concerns is the heating loop. This stream contains NC-17 operating at a pressure of 30.5 bar and 450°C. With pressure and temperature values this high, the proper insulation and material of construction are required. Proper training and PPE are required for all sections of this process.

Impact Factors:

Global:

The company's process could be impacted negatively if the global market for pharmaceuticalgrade acetone decreases. If a new, more effective solvent is discovered it could hinder the company from getting a profitable price on the acetone product. If that were to be the case, it could be beneficial to look into reducing the purity specifications and producing low-grade acetone or seeing if the plant can be converted to the production of a different product.

Cultural/Environmental/Social:

As the world moves towards a more sustainable, environmentally conscious mindset, it is imperative that the company operates this plant in ways that will not only be safe to the environment but the people in the surrounding area as well. Ensuring that any wastewater and plant emissions are operating below EPA guidelines ensures that the plant has the lowest possible impact on the people living near the plant and that the air and water quality are clean and safe. Further, each state has independent environmental guidelines and safety procedures, and it would be imperative to operate in accordance to those regulations presented during construction.

Economic:

Economically, there are many factors that could impact the construction of the plant. Seen recently, the Covid-19 pandemic impacted production lines and caused shipping shortages. If something like that were to happen again, there could be difficulty getting materials of construction for the different pieces of operating equipment and would thus delay plant operation. This would further push back the NPV of the plant, in turn losing the company money. Barring another global pandemic, an increase of raw material prices is something that the company should be wary of. If raw material prices increase, the process would not be as profitable.

Fluidized Bed Reactor Optimization:

In the previous optimization cycles of the IPA to acetone process, a plug flow reactor was used to carry out the necessary reaction. After these cycles were completed, a fluidized bed reactor was investigated in order to explore other possible means of conducting this reaction.

Fluidized bed reactors are commonly used in industry. These reactors offer improved heat transfer, the ability to better fluidize solids, and the ability to handle a wide range of particle sizes. However, these reactors are also more difficult to design and operate.

During the optimization of the fluidized bed reactor, there were several design parameters that had to be met: the inlet feed pressure had to range from 0.75 to 5 bar, the inlet feed temperature had to range from 300 to 750°C, the length to diameter (L/D) ratio of the reactor had to range from 2 to 10, and the ratio of the superficial gas velocity to the minimum fluidizing velocity had to range from 3 to 10. Also, in a fluidized bed reactor, some of the feed gas bypasses the catalyst

due to bubbling in the reactor. This effect means that the single pass conversion of the reactor can never exceed 90%. While staying within the design parameters, the objective of this optimization was to maximize the selectivity and conversion in the reactor.

With these parameters in mind, the new fluidized bed reactor was simulated in AVEVA ProII using the same reaction data and kinetics as in the previous optimization cycles. ProII does not have the ability to simulate a true fluidized bed reactor, so a plug flow reactor was used in this optimization. In order to abide by the design parameters of the fluidized bed reactor, a 10% bypass of the reactor feed was also included in the simulation. When optimizing the reactor, there were four variables considered: inlet pressure, inlet temperature, reactor volume, and L/D ratio. During each simulation of the reactor, the ProII calculator function was used to observe changes in selectivity and conversion while monitoring the inlet and outlet velocity ratios to ensure that they remained within the acceptable range.

To begin the optimization process, a functioning base case for the fluidized bed reactor had to be established. In the base case, the inlet pressure was 0.75 bar, the inlet temperature was 300°C, the reactor length was 8 meters, and the reactor diameter was 2 meters. These conditions yielded a selectivity of 0.07 and a conversion of 6.7%. Once the base case was established, optimization of the inlet temperature and pressure were conducted.

To optimize the inlet temperature and pressure, the temperature was manually varied using 50°C intervals from 300 to 750°C while having ProII conduct case studies varying the pressure from 0.75 to 5 bar with 0.125 bar intervals. By manually varying the temperature, the optimum reactor temperature and pressure were evaluated simultaneously using the results of the ProII case studies. These case studies showed that optimum selectivity and conversion were achieved at a

temperature of 600°C and a pressure of 4.75 bar. These feed conditions yielded a selectivity of 8.76 and a conversion of 89.75%.

After optimizing the temperature and pressure, the reactor volume was varied to observe its effects while maintaining the existing L/D ratio of 4. The volume was varied by changing the reactor length in 1 meter intervals while adjusting the diameter accordingly. Lengths from 6.1 meters to 15 meters were tested. It was found that the optimum selectivity and conversion in the reactor were still achieved at the base case reactor volume, 25.13 m³.

Finally, the reactor L/D ratio was varied from 2 to 10 while maintaining a constant reactor volume. The optimum selectivity and conversion were still found at the base case value of an L/D of 4.

After optimizing all of these variables, the optimum fluidized bed reactor selected operated at a temperature of 600°C, a pressure of 4.75 bar, a volume of 25.13 m³, and an L/D ratio of 4. This reactor yielded a selectivity of 8.76 and a conversion of 89.75%.

During the optimization process, there were two factors other than maximizing selectivity and conversion that had to be considered. First, the inlet and outlet velocity ratios were required to be from 3 to 10. Any possible configuration which yielded values for these ratios which were out of range were excluded as viable options. Additionally, there were significant issues with the reactor pressure drop while simulating this reactor. With lower inlet pressures, the simulation was failing to run because the reactor pressure drop was causing the reactor to begin operating in a vacuum. Also, when the L/D ratio was increased past 5, a similar issue was occurring even at the higher pressure of 4.75 bar being used. This effect was occurring because increasing the reactor of the

reactor increases the pressure drop as well. When increasing the L/D ratio, the length of the reactor must increase, or the diameter of the reactor must decrease. Either of these changes increases the reactor pressure drop and causes the reactor to begin operating in a vacuum. Because of this effect, all possible configurations experiencing these pressure drop issues were excluded as viable options.

Despite the issues surrounding the reactor pressure drop, a highly effective optimum design was still found. The reactor selected has a high selectivity of 8.76 and a conversion of 89.75% which is nearly equal to the theoretical maximum of 90%.

Conclusion:

After optimizing both the high and low temperature catalyst options, the high temperature catalyst was selected for further optimization. Upon completion of the final optimization cycle, several key changes were made to the process. The heat exchanger network in the reactor section was redesigned, the separations section was improved in order to increase acetone recovery, and the reactor size was changed to maximize the project's NPV. The final NPV after completing all three optimization cycles was \$140.5 million. Moving forward, a P&ID of the process must be developed which would include instruments currently missing from the design such as controllers, sensors, and valves. Once the P&ID is completed, the next class of cost estimation can be conducted which will provide a more accurate NPV value for the process. Also, the company will continue to pursue other possible areas of optimization for the process.

References:

Turton, Richard, et al. Analysis, Synthesis and Design of Chemical Processes. 5th ed., Prentice Hall, 2018.

Production of Acetone Case Study Document- Version 6

Appendix:

Stream No.	1	2	3	4	5	6	7	8
Temperature (°C)	25	28.90857907	28.96973839	250	410	45	20	20
Pressure (bar)	1	1	2.4	2.12	2.054294471	1.914294759	1.774294759	1.774294759
Vapor Mole Fraction	0	0	0	1	1	0.52379843	0.415996676	1
Total Mass Flow (MT/h)	4.5	4.9	4.7	4.9	4.9	4.9	4.9	0.6
Total Molar Flow Rate (kmol/h)	97.27539455	106.603032	106.603032	106.603032	171.7712911	171.7705911	171.7705911	73.28964914
Component Flowrate (kmol/hr)								
Hydrogen	0	5.21832E-11	5.21832E-11	5.21832E-11	65.16825905	65.16720315	65.16720315	64.77275058
Acetone	0	2.356703727	2.356703727	2.356703727	67.52496278	67.52312265	67.52312265	7.690476916
IPA (Isopropyl Alcohol)	65.17451435	68.15429502	68.15429502	68.15429502	2.986035971	2.985933536	2.985933536	0.082217612
Water	32.1008802	36.0920333	36.0920333	36.0920333	36.0920333	36.09433173	36.09433173	0.744204036
NC-17	0	0	0	0	0	0	0	0

Appendix A: Case Study Optimization Final Excel Stream Table

Stream No.	9	10	11	12	13	14	15	16
Temperature (°C)	20	25	37.49586911	26.02289799	30.48426476	36.26694555	36.28449003	98.38986669
Pressure (bar)	1.774294759	2	1.73	1.6	1.73	1.2	1.5	1.4
Vapor Mole Fraction	0	0	0	1	8.47E-05	1	0	0
Total Mass Flow (MT/h)	4.3	3.6	4.0	0.2	8.3	0.0	3.7	4.5
Total Molar Flow Rate (kmol/h)	98.48094192	200	207.461054	65.82859513	305.9419959	1.291559794	64.7932596	239.8571766
Component Flowrate (kmol/hr)								
Hydrogen	0.39445238	0	0.486912888	64.28583769	0.881365268	0.767080874	0.114284399	5.21735E-11
Acetone	59.83264584	0	7.528867733	0.161609183	67.36151357	0.523441429	64.48039022	2.357681918
IPA (Isopropyl Alcohol)	2.903715931	0	0.082217612	1.59413E-16	2.985933543	4.81431E-07	0.000133229	2.985799832
Water	35.35012777	200	199.3630558	1.381148262	234.7131835	0.00103701	0.198451754	234.5136948
NC-17	0	0	0	0	0	0	0	0

Stream No.	17	18	19	20	21	22	23	24
Temperature (°C)	26.42584281	98.39245618	72.89040475	109.3256068	45	410	410.0354657	449.92994
Pressure (bar)	1.2	1.5	1.2	1.4	1.26	30	30.5	30.25
Vapor Mole Fraction	1	0	0	0	0	0	0	0
Total Mass Flow (MT/h)	0.2	4.5	0.4	4.2	4.2	36.1	36.1	36.1
Total Molar Flow Rate (kmol/h)	67.12015492	239.8571766	9.327709647	230.5294669	230.5294669	150	150	150
Component Flowrate (kmol/hr)								
Hydrogen	65.05291856	5.21735E-11	5.21735E-11	7.23455E-21	7.23455E-21	0	0	0
Acetone	0.685050611	2.357681918	2.356837054	0.000844864	0.000844864	0	0	0
IPA (Isopropyl Alcohol)	4.81431E-07	2.985799832	2.979828232	0.0059716	0.0059716	0	0	0
Water	1.382185272	234.5136948	3.991044361	230.5226504	230.5226504	0	0	0
NC-17	0	0	0	0	0	150	150	150

Appendix B: Case Study Optimization Heat Exchanger Duties

Exchanger ID	E-1101	E-1102	E-1103	E-1104	E-1105	E-1106	E-1107	E-1108
In	hps	cw	rw	cw	lps	cw	lps	cw
Out	bfw	cw ret	rw ret	cw ret	bfw	cw ret	bfw	cw ret
Temp (°C)	234.00	350.00	45.00	50.00	92.00	108.80	108.80	108.80
Pressure (Barg)	1.30	0.91	0.77	0.63	0.63	0.50	0.50	0.40
Duty (GJ/h)	4.17	1.09	0.94	16.58	20.01	1.70	1.86	1.12
Price (\$/GJ) IN	5.66	0.38	4.77	0.38	4.54	0.38	4.54	0.38
Price (\$/GJ) OUT	1.52	0.38	4.77	0.38	1.52	0.38	1.52	0.38



Appendix C: Case Study Optimization Cash Flow Diagram

Appendix D: Case Study Optimization Final ProII Simulation