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Comparison of Energy Usage for the Vacuum Separation of Acetic Acid/Acetic Anhydride Using an Internally Heat Integrated Distillation Column (HIDiC)

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Abstract: Energy savings for an internally heat-integrated distillation column (HIDiC) and a vapor recompression column for the vacuum separation of acetic acid-/acetic anhydride was theoretically analyzed and compared to the simulation of a reference column configuration of the Eastman Chemical Company using ASPEN Plus. In these simulations, the design and operating variables were defined and optimized to minimize total energy used. The effects of design variables such as quantity and location of the heat integration stages, reflux ratio, and rectifying section absolute pressure on energy consumption and product purity revealed that one HIDiC configuration had 62% energy savings over the reference column. The distillation column using vapor recompression was evaluated as a benchmark for comparing the HIDiC configurations and the reference column. The VRC column simulation predicted both increased product purity and an energy savings of 91% over the reference unit.

Keywords: Acetic acid, acetic anhydride, distillation, HIDiC

INTRODUCTION

Separation Technologies

Distillation operations, the most widely used separation processes, are major consumers of energy in both the chemical and refining industries

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(2). According to a past study, up to 4% of the total industrial energy requirements in the United States in 1988 was attributed to distillation processes (8). Recent estimates, which put distillation energy demands closer to 40% of the total energy requirement, indicate that obtaining the maximum energy efficiency is important (2). As a result, many research projects have focused on process optimization and overall system integration designs that use vapor recompression columns, VRCs. In VRC designs, the vapor is compressed in the top of the column and then condensed in the reboiler at the bottom of the column to provide heat for vapor generation (4). Although energy savings are observed in these alternative designs, energy saving efforts typically result in decreased product purity, which creates an economic tradeoff between energy and product recovery that must be balanced.

Recently, internally heat integrated distillation columns, HIDiC, were designed and studied as a way to achieve energy savings. It has been shown that not only has HIDiC offered saving potentials greater than conventional alternatives, HIDiC also has the ability to increase product recovery (7,10,11,17,19,20). Unlike the conventional distillation alternatives, i.e. VRCs, that have two sections located inside one column shell, the HIDiC configuration uses two separation columns, one for the rectifying section and one for the stripping section. The HIDiC configuration combines vapor recompression with diabatic operation to drive down energy requirements by thermally integrating the stages of the rectifying and stripping columns.

In a traditional distillation column, the temperature of the rectifying section is lower than that of the stripping section as a result of the purification between the lower boiling, more volatile component and the higher boiling, less volatile component in addition to the pressure drop down the length of the column. If the temperature of the rectifying section is higher than that of the stripping section, the heat from the rectifying section can be transferred to the stripping section and thus reduce energy losses. The HIDiC configuration, Fig. 1, increases the pressure of the rectifying column to a value high enough to create a temperature driving force between the rectifying column and the stripping column. In this configuration, the overhead vapor from the stripping section is compressed and fed to the bottom of the rectifying column and the liquid from the rectifying section is pressure-equalized using a throttling valve and fed back to the stripping column. The liquid leaving the top of the rectifying column is the light product and the bottom stream of the stripping column is the heavy product.

As indicated in Fig. 1, the two HIDiC columns are configured so that the energy of the high temperature rectifying column is used to heat the stripping column, resulting in continuous condensation of the vapor phase along the rectifying column and continuous evaporation along

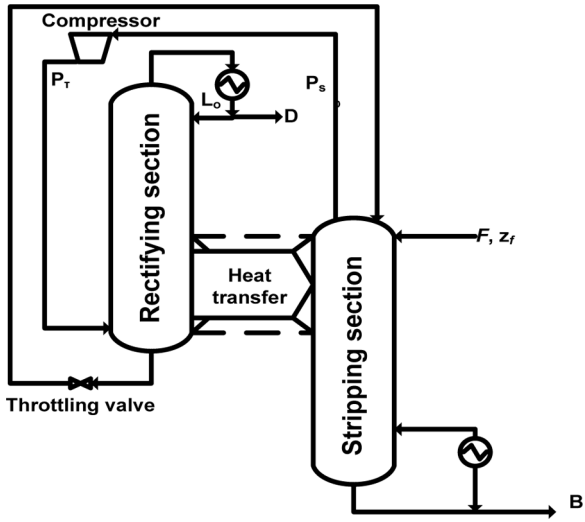

Figure 1. HIDiC configurations (6).

the stripping column. This concentrates the vapor and liquid flows at the center stages of the column, i.e. at the bottom stages of the rectifying column and at the top stages of the stripping column. The heat, which is transferred on each integrated stage through indirect contact of the high temperature vapor in the rectifying column and the lower temperature liquid in the stripping column, can be varied and has a direct effect on the reboiler duty. It has been reported previously that thermally integrating the two columns could reduce energy loss by 30% compared to VRCs (24).

Objectives

Although many simulations and bench scale experiments, as well as a full scale HIDiC pilot plant, have been investigated, no studies have been applied to separations at vacuum conditions where the pressures are limited by thermal degradation of the bottoms product. The objective of this present study was to simulate a HIDiC design capable of achieving component separation under vacuum conditions. For this simulation, the acetic acid/acetic anhydride split used at Eastman Chemical Company in Kingsport, TN was chosen because it meets the main three distillation criteria for HIDiC application:

1. the separation is achieved in a packed column;
2. the separation is an energy-intensive operation of a close-boiling mixture with a relative volatility close to unity; and

3. the separation is done for large-scale production (12,13,20,25). As it will be shown later on, this study indicates that it is feasible to achieve a 62% energy savings under vacuum conditions using a HIDiC configuration.

Additionally a vacuum system is of particular interest for two reasons. First, a pressure changing device (i.e. vacuum pump, steam jet, etc.) is already required to reach the vacuum pressures. While a HIDiC compressor or vacuum pump would be larger, it would not be a new unit operation, just a larger one. Second, often a column is run under vacuum to minimize the base heater temperature in order to use a lower utility or to minimize heat history of a thermally sensitive material. The upper portion of the column in this circumstance does not need to be under vacuum. Running this top portion of the column at a higher pressure would decrease the necessary diameter, offsetting some of the capital cost penalty of a HIDiC system.

DESIGN AND SIMULATION

Simulation Tool

ASPEN Plus was used to simulate and compare the separation performances of the Reference distillation column, the HIDiC and the VRC acetic acid/acetic anhydride splits. For this simulation, the Wilson-Hayden O'Connell model was used to obtain vapor-liquid equilibrium data. The two different HIDiC designs were simulated in ASPEN Plus as two thermally connected columns, where the rectifying stages were heat integrated with the equivalent number of stages in the stripping column. The stages in the stripping column without heat integration operate as normal column stages at specified pressures. The acetic acid/acetic anhydride separation simulations were conducted under vacuum pressures with an inert nitrogen atmosphere. The vacuum pressure allows the column to be operated at temperatures below the atmospheric boiling point of the products, which helps avoid thermal degradation of acetic anhydride. Since the temperature is exponentially dependent on pressure, the compressors used in the simulations were not operated above 24 psig so that the temperature never passed the atmospheric boiling point of acetic anhydride at 139°C.

Reference Distillation Case

Figure 2, shows a seventeen stage standard column, with seven stages in the rectifying section and ten stages in the stripping section. The liquid feed, which is a 50/50 wt% mixture of acetic acid/acetic anhydride, is

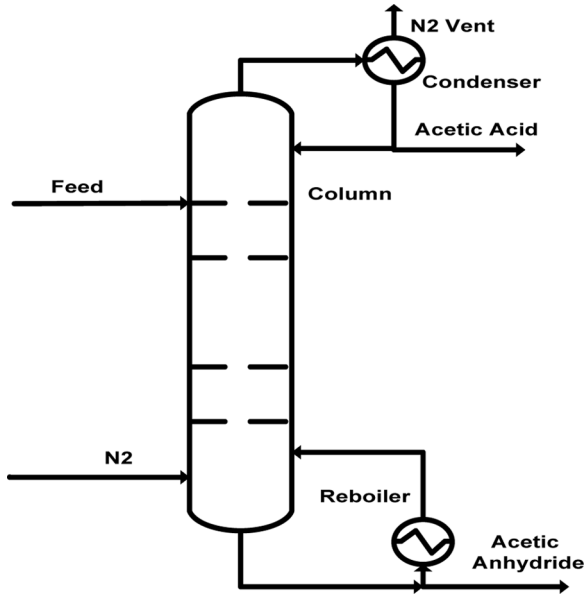

Figure 2. Schematic of reference distillation.

fed to the eighth stage of the column at a rate of 50,000 lb/hr. The reflux ratio of this column is 2.0, and the bottoms to feed ratio is 0.5. The top and bottom column pressures are 5.80 psia and 10.44 psia, respectively, with a pressure drop per equivalent stage of 0.27 psia. The top product in this column is 96.7 mol% acetic acid, and the bottom product is 91.0 mol% acetic anhydride. For the Reference Distillation case, the condenser duty is -1.66×10^7 BTU/hr and the reboiler duty is 1.77×10^7 BTU/hr.

Vapor Recompression Cases

The vapor recompression configuration (VRC), Fig. 3, is based on the operating conditions of the Reference Distillation case. The liquid feed of 50/50% mixture of acetic acid/acetic anhydride is fed to a preheater that increases the feed temperature to 90°C before it is fed to the eighth stage of the column. The reflux ratio for the VRC simulation was set at 1.4 with a boil-up rate of 254 lbmol/hr. The top and bottom pressures of the column were 5.8 psia and 10.4 psia respectively with a 0.25 psia pressure drop per stage. The product purities were 97% for the acetic acid out the top and 97.3% for the acetic anhydride out of the bottom. The

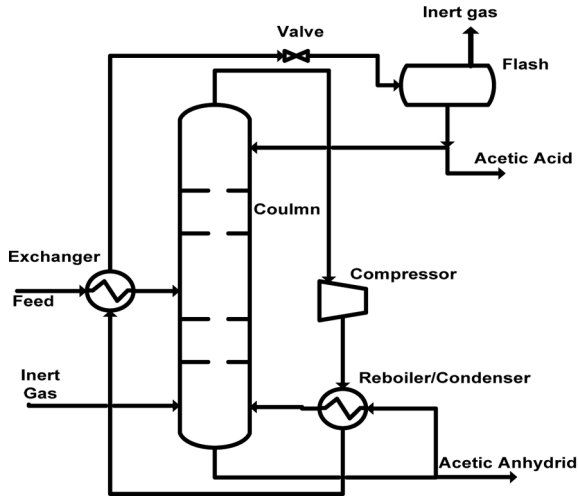

Figure 3. Schematic of Vapor Recompression Distillation.

VRC used the top product as the heat source for the reboiler by compressing the vapor flow to a pressure of 26 psia and a temperature of 138°C. The compressor required a duty of 1.57×10^6 BTU/hr and was the only required energy input for the system. The heated vapor is cooled using a heat exchanger on the feed stream, a throttle valve and a flash drum that takes the stream conditions to 5.8 psia and 30°C. The flowrate of the distillate is 24,700 lb/hr and the bottoms flowrate is 25,300 lb/hr.

HIDiC Cases

Both investigated configurations consist of a seven stage rectifying column and a ten stage stripping column, where five rectifying column stages are thermally integrated with five stripping column stages. In the HIDiC cases, the liquid from the bottom of the rectifying section is sent through a throttling valve and mixed with the 50,000 lb/hr, 50/50 wt% liquid feed, creating a high flow, partially vaporized feed that is sent to the top stage of the stripping column. The vapor from the stripping section is compressed before it is fed to the bottom stage of the rectifying column. Also in both HIDiC cases, the bottoms product rate is held constant at 284 lbmol/hr.

Higher Pressure HIDiC Case

In the Higher Pressure HIDiC case, Fig. 4, the pressures of the stripping column are consistent with the pressures of the stripping section of the

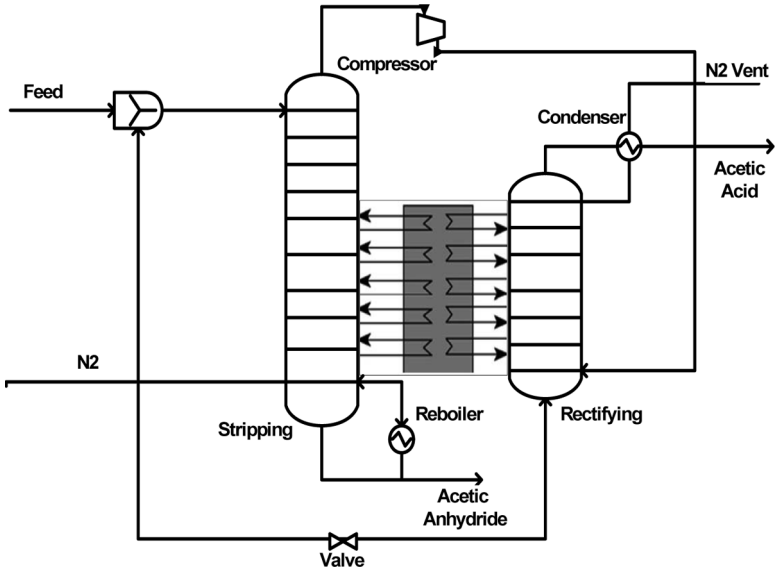

Figure 4. Schematic of the Higher Pressure HIDiC configuration.

Reference distillation column. The stripping column top pressure is 8.00 psia, and with a pressure drop per stage of 0.22 psia per stage, results in a bottom pressure of 10.42 psia. The pressures of the rectifying column were increased to 20.00 psia at the top and 22.51 psia at the bottom with a pressure drop per stage of 0.31 psia to allow for thermal integration between columns. In this HIDiC case, the heat is transferred from stages 2, 3, 4, 5, and 6 of the rectifying column to stages 5, 6, 7, 8, and 9 of the stripping column at a rate of 1.50×10^7 BTU/hr. At a reflux ratio of 0.4, the products from the rectifying and striping sections are 98.9% acetic acid and 94.7% acetic anhydride, respectively. This simulation resulted in a condenser duty of -9.32×10^6 BTU/hr, a reboiler duty of 2.17×10^5 BTU/hr, and a compressor duty of 1.02×10^7 BTU/hr. The total energy requirement for the Higher Pressure HIDiC case is 1.04×10^7 BTU/hr.

Lower Pressure HIDiC Case

For the Lower Pressure HIDiC case, Fig. 5, the pressures in the stripping column are 2.00 psia at the top and 4.42 psia at the bottom, which results from maintaining the 0.22 psia pressure drop used in the Higher Pressure HIDiC case. In the rectifying column, the reflux ratio is 0.4, the top and

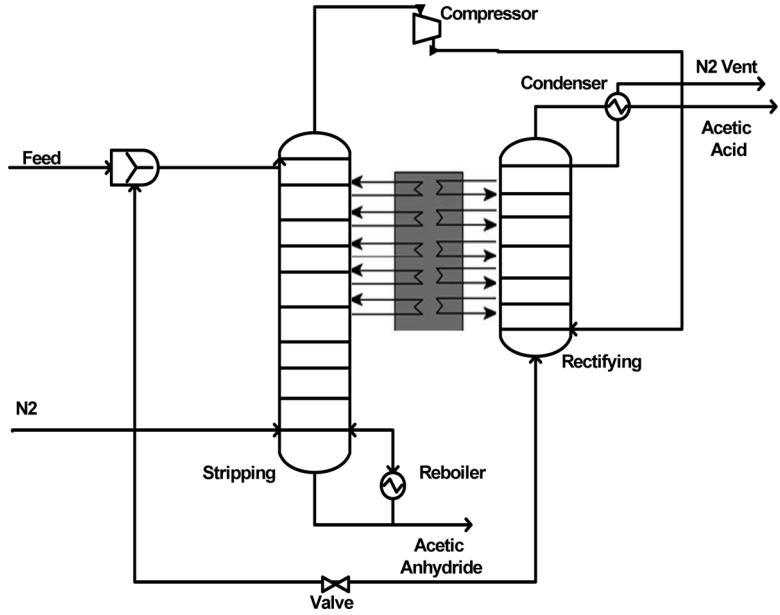

Figure 5. Schematic of the Lower Pressure HIDiC configuration.

bottom pressures are 10.00 psia and 12.52 psia, respectively, and the pressure drop per stage is again 0.31 psia. At these low pressures, the temperatures at the bottom stages of the stripping section are too high to allow proper thermal integration from the rectifying column. As a result, the heat is transferred from stages 2, 3, 4, 5, and 6 of the rectifying column to stages 2, 3, 4, 5, and 6 of the stripping column at a rate of 6.00×10^6 BTU/hr. These column parameters result in a top product that is 98.0% acetic acid, a bottom product that is 93.2% acetic anhydride, a condenser duty of -8.06×10^6 BTU/hr, a reboiler duty of 1.61×10^6 BTU/hr, and a compressor duty of 5.10×10^6 BTU/hr. The total energy requirement for the Lower Pressure HIDiC case is 8.62×10^6 BTU/hr.

RESULTS AND DISCUSSION

Comparison of the Reference, VRC and HIDiC Configurations

For the four cases described above, Table 1 summarizes the ASPEN input variables and Table 2 summarizes the results of each simulation. This comparison focused on the energy usage of each case in terms of

Table 1. Variables defined in the ASPEN simulations

Reference Distillation	
Feed Flowrate	50,000 lb/hr
Feed Composition	50/50 wt%
Feed Pressure	30 psia
Feed Temperature	50°C
Number of Stages	17
Feed Stage	8
Reflux Ratio	2.0
Bottoms/Feed Ratio	0.5
Air Leak Stage	18
Vent	1
Top Pressure	5.8 psia
Column ΔP	3.87 psia
Vapor Recompression Distillation	
Feed Flowrate	50,000 lb/hr
Feed Composition	50/50 wt%
Feed Pressure	30 psia
Feed Temperature (Preheater)	50°C
Feed Temperature (Column)	90°C
Number of Stages	17
Feed Stage	8
Reflux Ratio	1.4
Boil-up Ratio	0.5
Air Leak Stage	18
Vent	Flash Drum
Top Pressure	5.8 psia
Column ΔP	4.64 psia

	Stripping	Rectifying
Higher Pressure HIDiC		
Feed Flowrate	516,500 lb/hr	491,100 lb/hr
Feed Composition	82/18 wt%	87/13 wt%
Feed Pressure	8.0 psia	8.0 psia
Feed Temperature	101.3°C	101.7°C
Feed Vapor Frac	0.13	1.00
Number of Stages	10	7
Feed Stage	1	7
Bottom Product	284 mol/hr	N/A
Reflux Ratio	N/A	0.5

(*Continued*)

Table 1. Continued

	Stripping	Rectifying
Vent Stage	N/A	1
Air Leak Stage	11	N/A
Top Pressure	8 psia	20 psia
Column ΔP	2.2 psia	3.1 psia
Heat Integrated Stages	5, 6, 7, 8, & 9	2, 3, 4, 5, & 6
Heat Input per Stage	1.5E7 BTU/hr	− 1.5E7 BTU/hr
Lower Pressure HIDic		
Feed Flowrate	233,600 lb/hr	208,400 lb/hr
Feed Composition	69/31 wt%	76/24 wt%
Feed Pressure	2.0 psia	2.0 psia
Feed Temperature	67.2°C	68.3°C
Feed Vapor Frac	0.17	1.00
Number of Stages	10	7
Feed Stage	1	7
Bottom Product	284 mol/hr	N/A
Reflux Ratio	N/A	0.4
Vent Stage	N/A	1
Air Leak Stage	11	N/A
Top Pressure	2 psia	10 psia
Column ΔP	2.2 psia	3.1 psia
Heat Integrated Stages	2, 3, 4, 5, & 6	2, 3, 4, 5, & 6
Heat Input per Stage	3.0E6 BTU/hr	− 3.0E6 BTU/hr

reboiler duty, condenser duty, compressor duty, the purity of the distillate and the bottoms, and the vapor and liquid flow profiles. In addition, temperature, pressure, vapor fraction, and liquid fraction profiles were examined.

The energy summary in Fig. 6 compares the heat input per side stream, condenser duty, reboiler duty, compressor duty, and total required energy for each of the four cases. From Fig. 6, it is easily seen that the two HIDiC cases require much less total energy than the Reference case, with the Lower Pressure HIDiC having a 62.0% energy savings. The VRC simulation achieves the greatest energy savings of 91.1% with the only required energy input being the compressor. The required compressor energy for the VRC configuration is lower than the lower pressure HIDiC case because the vapor flowrate to the VRC compressor is only 30% of the vapor flowrate to the HIDiC compressor. The reduced vapor flowrate in the VRC only uses one third of the energy

Table 2. Simulation comparison

	Reference	VRC	HIDiC Higher P	HIDiC Lower P
Top Product Purity	0.9667	0.9705	0.9889	0.9797
Bottom Product Purity	0.9095	0.9732	0.9469	0.9317
Input per Stage (BTU/hr)	N/A	N/A	1.50E + 07	6.00E + 06
-Condenser Duty (BTU/hr)	1.66E7	N/A	9.32E6	8.06E6
Reboiler Duty (BTU/hr)	1.77E7	4.52E6	2.17E5	1.62E6
Compressor Duty (BTU/hr)	N/A	1.57E6	1.02E7	5.10E6
Total Energy (BTU/hr)	1.77E7	1.57E6	1.04E7	6.72E6
Energy Savings (%)	0.0%	91.1%	41.0%	62.0%

input of the HIDiC to achieve the required energy increase in the vapor stream to drive the reboiler. It is apparent from Fig. 6 that the majority of the total required energy for the HIDiC cases is actually energy needed for the compressor, not the reboiler. In fact, the reboiler has been nearly eliminated for the HIDiC cases and entirely eliminated in the VRC case.

Figure 7 shows that not only do the two HIDiC and VRC configurations result in significant energy savings; but also, they result in a higher distillate and bottoms purity than the Reference Distillation case. The greatest purity is observed for the Higher pressure HIDiC case with a distillate purity of 98.8% and bottoms purity of 95%. The VRC case resulted in distillate and bottom purities that were approximately equal

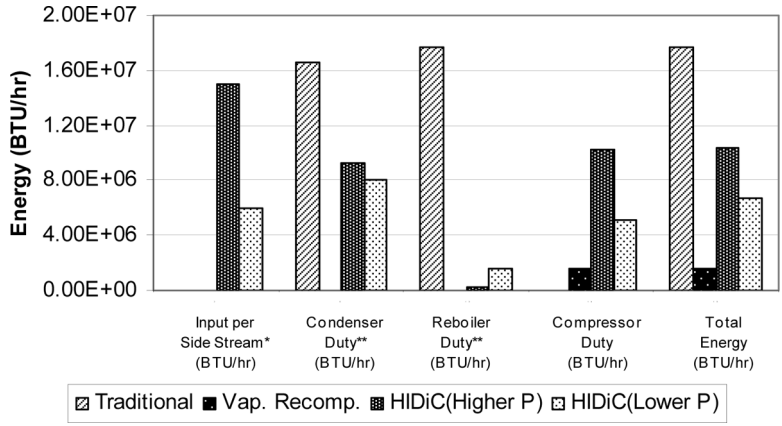

Figure 6. Energy comparison. *Note*: *Side stream energies for HIDiC only; ** VRC requires no reboiler nor condenser duty.

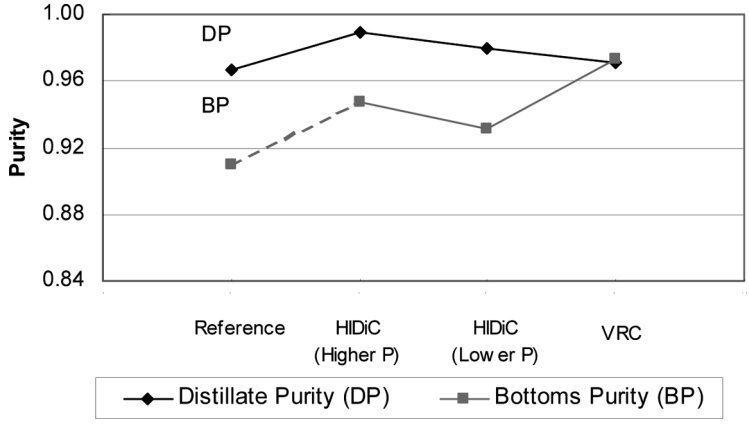

Figure 7. Purity comparison.

at 97%. The decreased purity observed for the Lower Pressure HIDiC case is most likely due to the lower column flowrates observed in Fig. 8. The Reference and VRC Distillations vapor and liquid flowrates remain relatively constant throughout the column, with the VRC paralleling the Reference column at slightly greater rates. The VRC vapor and liquid flowrates are greater than the Reference case due to an increase in

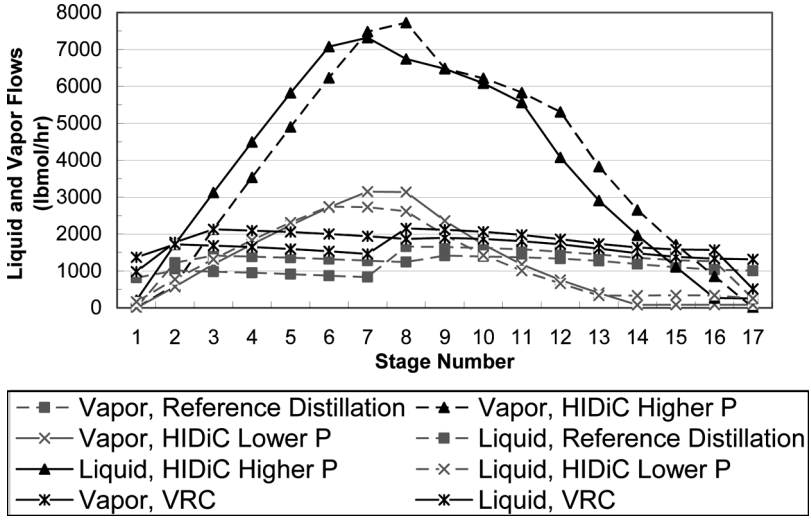

Figure 8. Vapor and liquid flow profiles.

the vaporization of the liquid at the bottom and the condensation of the vapor at the top. The HIDiC configurations have much greater flows in the middle stages, i.e. the bottom of the rectifying section and the top of the stripping section. This concentration of flows arises from the continuous condensation of the vapor in the rectifying column and the continuous evaporation of the liquid in the stripping column that results from the thermal integration of stages. The lower flowrates observed for the Lower Pressure HIDiC case require less heat input per side stream, which explains why in Fig. 8 the compressor duty the heat transfer per side stream are lower for the Lower Pressure HIDiC than for the Higher Pressure HIDiC. As a result, the Lower Pressure HIDiC configuration needs less heat transfer area to achieve the greater amount of energy savings.

The acetic acid vapor and liquid profiles are plotted versus stage number in Figs. 9 and 10, respectively. Interestingly enough, the fraction profiles for the two HIDiC cases differ dramatically. The Lower Pressure HIDiC vapor and liquid fraction flow profiles follow the same general trend as the Reference Distillation case, where the fraction of acetic acid decreases slowly until about stage twelve, after which point there is a sharp decrease in the amount of acetic acid present. For the Higher Pressure HIDiC, the sharp decrease in the acetic acid vapor and liquid fractions occur at stage nine with the bottoms purity achieved by stage fifteen. Although this suggests that perhaps the bottom two stages of the stripping column are not necessary to achieve the separation for the Higher Pressure HIDiC case, subsequent investigations revealed that

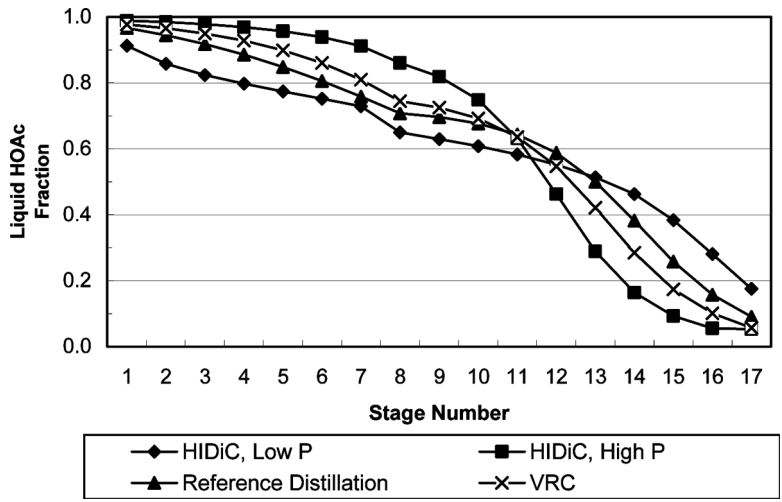

Figure 9. HOAc liquid mole fraction profile.

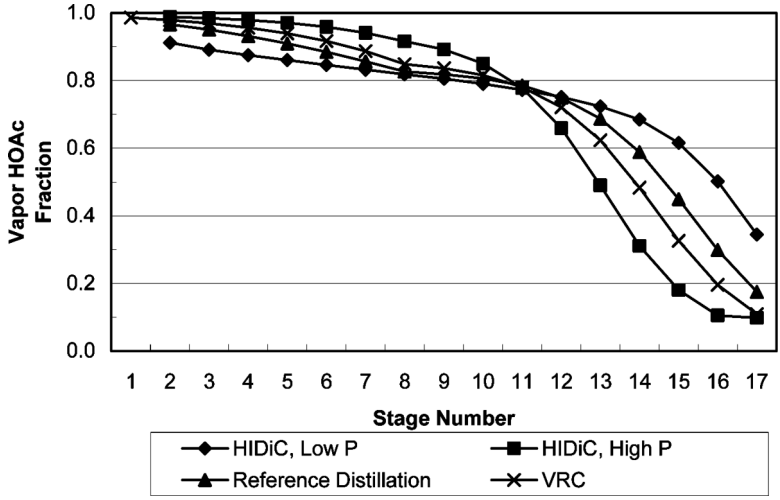

Figure 10. HOAc vapor mole fraction profile.

energy savings is maximized when there are seven stages in the rectifying column and ten stages in the stripping column. The VRC profiles followed the same trends as the Reference column, while maintaining a greater fraction in the rectifying section but decreasing in liquid and vapor fraction after stage twelve in the stripping section.

The pressure profile in Fig. 11 display that the HIDiC simulations operate at greater pressures in the rectifying section than the Reference and VRC cases. The stripping section of the higher pressure HIDiC case maintains the same pressure profile as the Reference case while the lower pressure HIDiC case is required to operate at lower pressures. Pressure profiles for the VRC case are approximately equal to that of the Reference column.

The temperature profiles in Fig. 12 follow the same trend as the pressure profile. The thermally integrated stripping column stages of the HIDiC cases are easily observed in Fig. 12 where the temperature profiles increase with a larger slope. Notice that the pressure and temperature profiles for the VRC and Reference Distillation cases are increasing with a constant slope down the column, the temperature and pressure profiles for the HIDiC cases are much higher in the rectifying column than the stripping column. The large temperature differences, which result from the large pressure differences, provide the driving force for heat integration between columns. The Reference and HIDiC columns top temperature are 30°C, which is set by the condenser N_2 vent conditions.

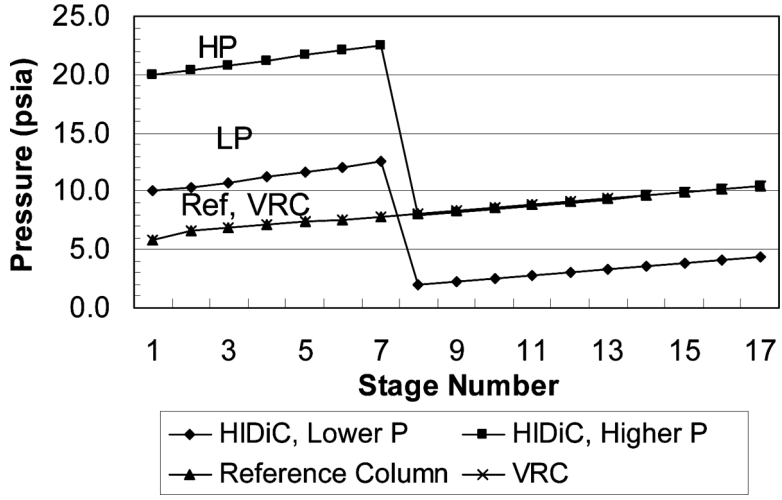

Figure 11. Pressure profiles.

VRC analysis

The analysis of the vapor recompression configuration was based on the actual operating conditions of the Eastman Reference column. The simulation was setup so that the condenser could be eliminated from the

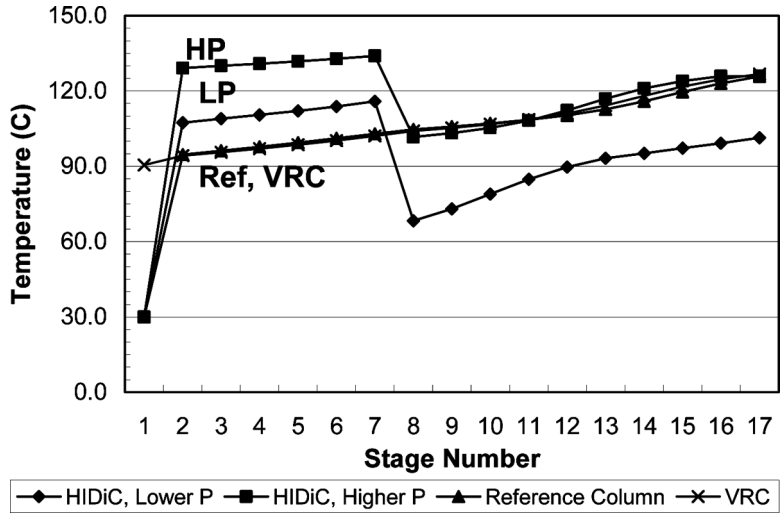

Figure 12. Temperature profiles.

configuration and the distillate vapor could be recompressed and utilized as a heat source for the reboiler, thus having the reboiler double as the condenser. The boil-up rate was adjusted to the maximum flowrate that the simulation could operate at without encountering heat exchanger crossover effects. The reflux ratio was adjusted to evaluate the effect of product purity. The result of the analysis is shown in Fig. 13 which is a plot of compressor duty and purity as a function of the reflux ratio. As seen in the plot the purity increases as the reflux ratio increases, but reaches a point of maximum purity of 99% for the distillate and bottoms by a reflux ratio of 3. A reflux ratio and boil-up rate of 1.4 and 508 lbmol/hr produced a similar column profile to the Reference column and were used in the analysis to compare the Reference case.

Since increasing the reflux rate effects the amount of distillate vapor produced an evaluation was made to compare the energy required in the condenser to the reflux ratio. The results of the energy analysis is shown in Fig. 13 as a function of the reflux ratio. The plot shows that the compressor duty and the reflux ratio are proportional and increase linearly at a rate of 619,000 BTU/hr per unit increase in reflux ratio. By operating at a reflux ratio of 1.4 the compressor only requires 1,578,000 BTU/hr.

Higher Pressure HIDiC Analysis

The first analysis of the Higher Pressure HIDiC configuration was done by examining the effect of the heat transfer per side stage on the reboiler

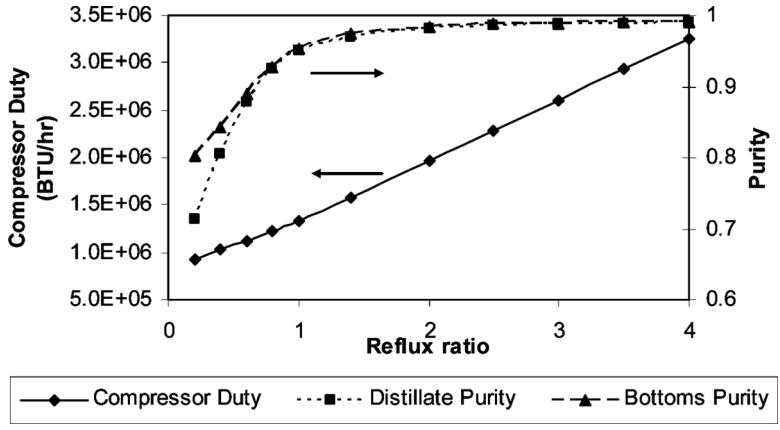

Figure 13. Compressor Duty and Purity as a function of reflux.

duty, condenser duty, and purity while holding the reflux ratio constant at 2.0. The results from this analysis are shown in Fig. 14, where the reboiler duty and condenser duty are plotted on the y-axes and the heat transfer per stage is on the x-axis. At a constant reflux ratio, the reboiler duty decreases linearly with increasing heat transfer per stage. The condenser duty also decreases with increasing heat transfer per stage, although it appears to reach a limiting value at higher heat transfer per stage. The decrease in the reboiler and condenser duties results from the thermal integration between stages.

Fig. 15 shows the effect of heat transfer per side stream on the purity. Both the bottoms and distillate purity increase with increasing heat transfer per stage, although the purity increases at a lower rate for higher values of high heat transfer per stage. It can be inferred from Fig. 15 that as a result of decreased separation performance at low heat transfer per stage values, larger column diameters are necessary for the HIDiC configurations than for the Reference Distillation columns in order to provide adequate heat transfer area.

The second analysis of the Higher Pressure HIDiC case consisted of holding the heat transfer per stage constant at 1.5×10^7 BTU/hr to examine the effect of reflux ratio on the reboiler duty, condenser duty, and purity. In this analysis, Fig. 16, both the reboiler duty and condenser duty decrease linearly with decreasing reflux ratio. This is because of the decreased flowrates through the reboiler and condenser resulting from the decreased reflux ratio. At a constant heat transfer per stage, decreasing the reflux ratio has no effect on the purity. The distillate and bottoms purities remain constant at 98.9% and 94.8%, respectively. The lack of effect on purity of changing the reflux ratio is attributed to the constant

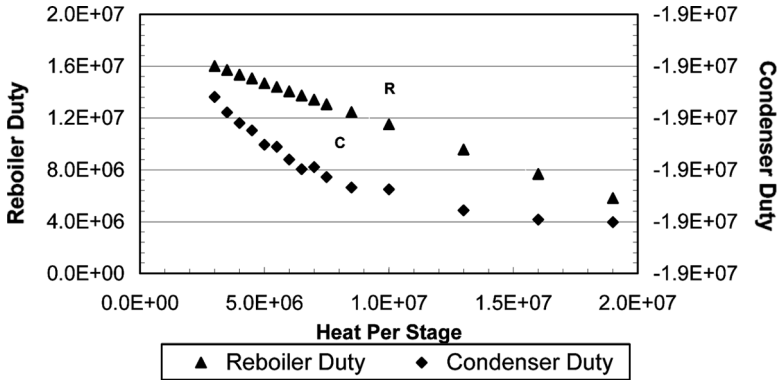

Figure 14. Reboiler and condenser duties (BTU/hr) as a function of heat transfer per stage (BTU/hr) at a reflux ratio of 2.0.

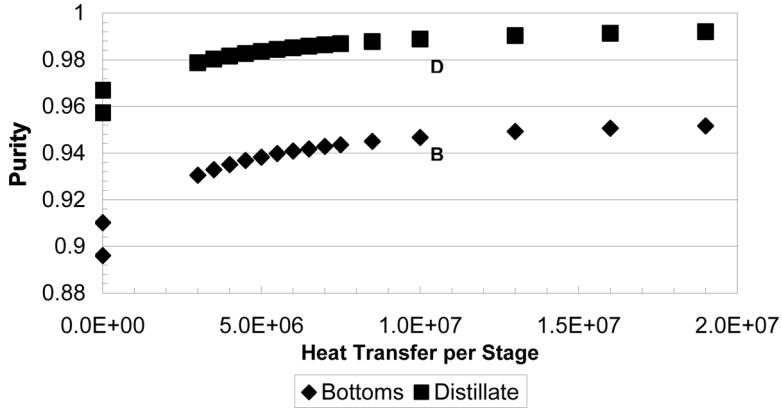

Figure 15. Purity as a function of heat transfer per stage (BTU/hr) at a reflux ratio of 2.0.

condensation and evaporation occurring along the columns, which ensures that the light component is continually evaporated in the stripping column and the heavy component is continually condensed in the rectifying column.

Lower Pressure HIDiC Analysis

The effect of pressure on the HIDiC configuration for the acetic acid/acetic anhydride split was analyzed by holding the reflux ratio constant

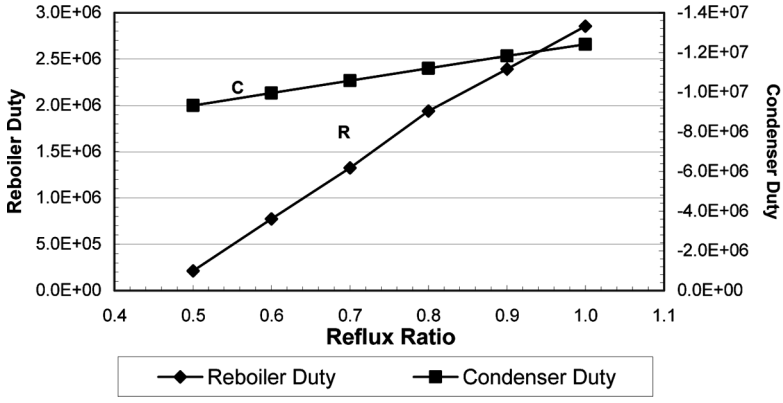

Figure 16. Reboiler and condenser duties (BTU/hr) as a function of reflux ratio at a constant heat transfer per stage (BTU/hr).

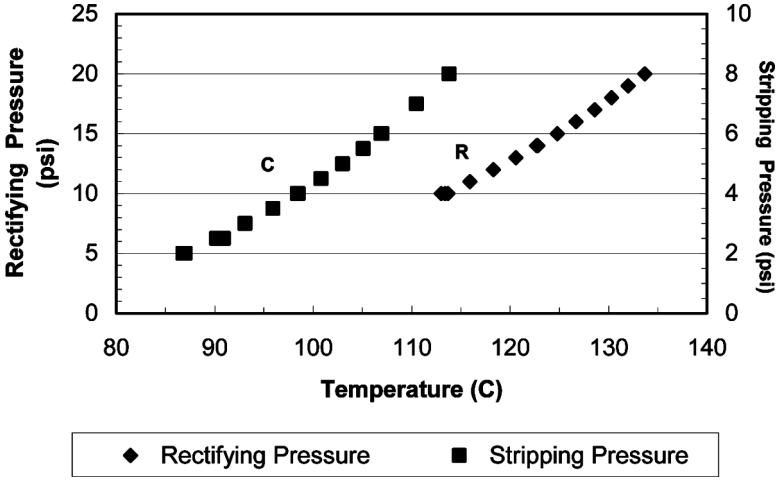

Figure 17. Effect of column pressure on the reboiler and condenser duty.

at 2.0×10^6 and the heat transfer per stage constant at 2.0 and 3.0×10^6 BTU/hr. The effect of changing the column pressure on the reboiler and condenser duty is shown in Fig. 17. Again, both the reboiler duty and the condenser duty decrease as the pressure decreases, but since the duties change only slightly with large changes in pressure; a question is raised about whether the decrease is a result of the pressure drop or the

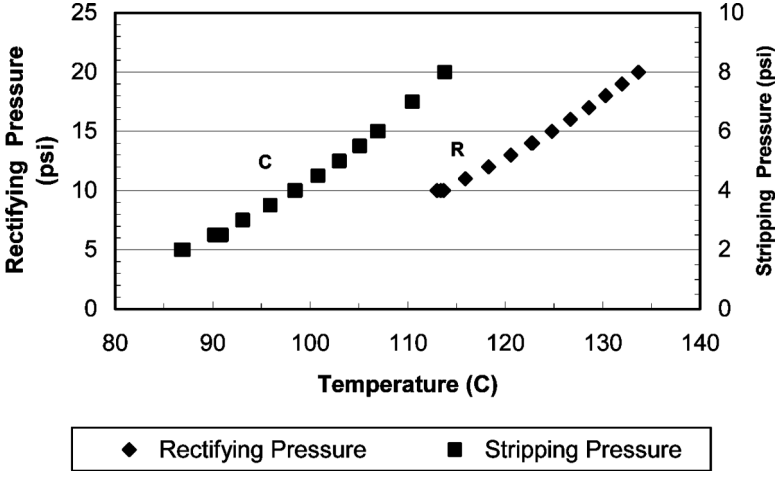

Figure 18. Effect of pressure on temperature.

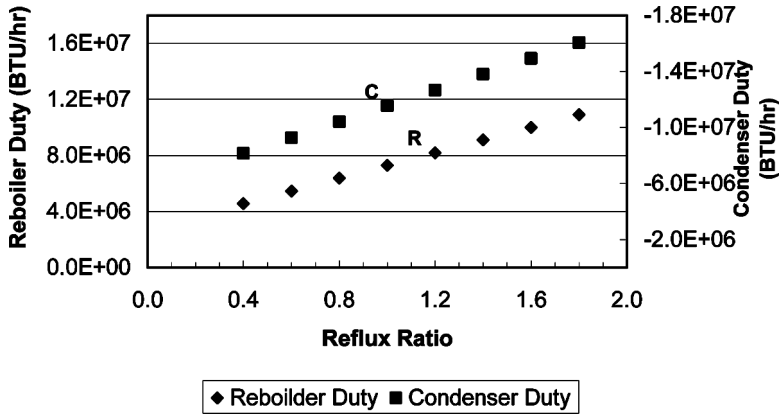

Figure 19. Effect of reflux ratio on the reboiler and condenser duties at a constant heat transfer per stage of 3.0×10^6 BTU/hr.

temperature drop. The effect of pressure on the temperature at the top of each column is shown in Fig. 18, which indicates that temperature changes dramatically enough with pressure that the decrease in reboiler and condenser duties could, in fact, be a result of the temperature drop.

The effect of the reflux ratio on the reboiler duty, the condenser duty, and the purity was investigated while holding the heat transfer per stage constant at 3.0×10^6 BTU/hr. The results from the energy analysis,

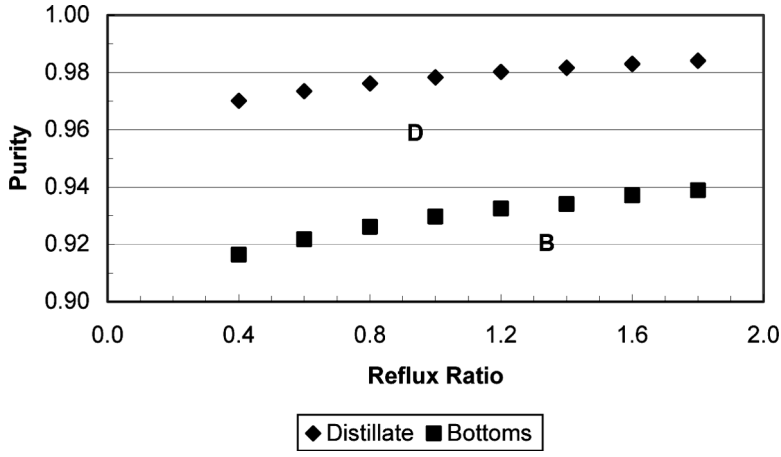

Figure 20. Effect of reflux ratio on the distillate and bottoms purities at a constant heat transfer per stage of 3.0×10^6 BTU/hr.

Fig. 19, indicate that both the reboiler duty and condenser duty decrease linearly with decreasing reflux ratio. This is because the flow through the column decreases as the reflux ratio is decreased. In Fig. 20, the bottoms and distillate purities are graphed versus the reflux ratio for the Lower Pressure HIDiC case. Unlike the Higher Pressure HIDiC case where the purities are constant with reflux ratio, the bottoms and distillate purities both decrease as the reflux ratio decreases. This observation explains why a reflux ratio of 0.4 in the Lower Pressure HIDiC case results in a lower purity than a reflux ratio of 0.5 in the Higher Pressure HIDiC case.

Ideal HIDiC Analysis

In order to make heat transfer between the rectifying and stripping columns possible, the temperature profile of the rectifying column must be above the temperature profile of the stripping column. The temperature profiles for the Higher Pressure HIDiC and Lower Pressure HIDiC cases are shown in Fig. 21. Although the rectifying column and stripping column profiles of the Lower Pressure HIDiC configuration are lower than the Higher Pressure HIDiC configuration because of the decreased column pressures, the temperature profiles for both columns follow the same trend because the stage pressure drops are the same in both cases. The larger temperature driving force ($T_R - T_S$) observed for the Lower Pressure HIDiC is

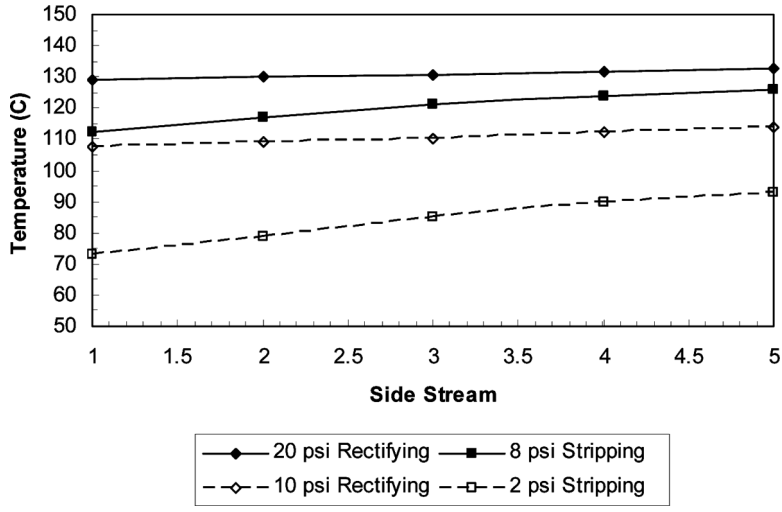

Figure 21. Temperature profiles for the rectifying column and stripping column of the Higher Pressure HIDiC and Lower Pressure HIDiC cases.

the result of the heat integration at the top stages of the stripping column, which are cooler than the bottom stages of the stripping column where the Higher Pressure HIDiC heat integration occurs. As previously stated, this change was made because in the Lower Pressure HIDiC case, the driving force resulting when the bottom stages of the stripping column were used was not large enough to allow heat integration.

ANALYSIS OF THE EFFECT OF DESIGN PARAMETERS ON THE LOWER PRESSURE HIDIC

For the optimized Lower Pressure HIDiC case, the effects of changing the heat input per stage and the number of stages in the stripping column and rectifying columns was investigated to determine if additional energy savings over 62% could be obtained at the specified column pressures, reflux ratio, bottoms rate, and heat integrated stages. When the heat input per stage was increased, the ASPEN simulation was unable to converge; when it was decreased, the energy savings and the purity of the distillate and the bottoms decreased dramatically. These results, shown in Fig. 22, indicate that for the Lower Pressure HIDiC configuration, lowering the heat input per stage has adverse effects on the mixture separation and energy savings.

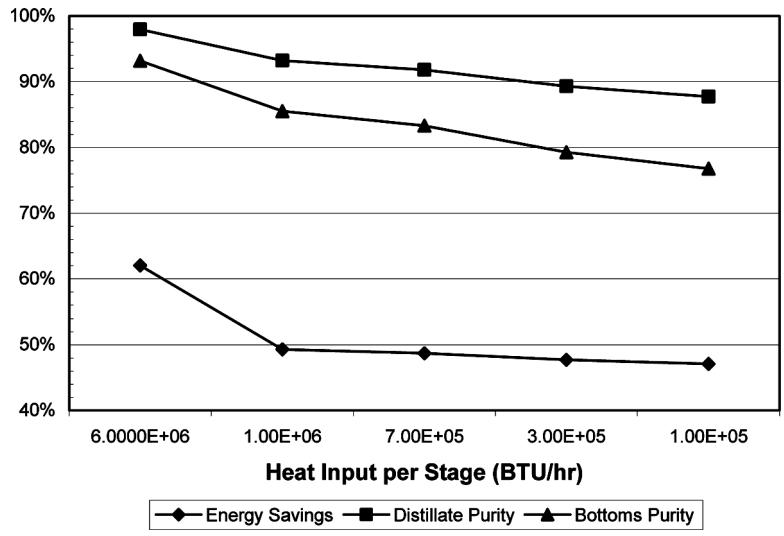

Figure 22. Effect of decreasing the heat input per stage after optimization for the Lower Pressure HIDiC configurations.

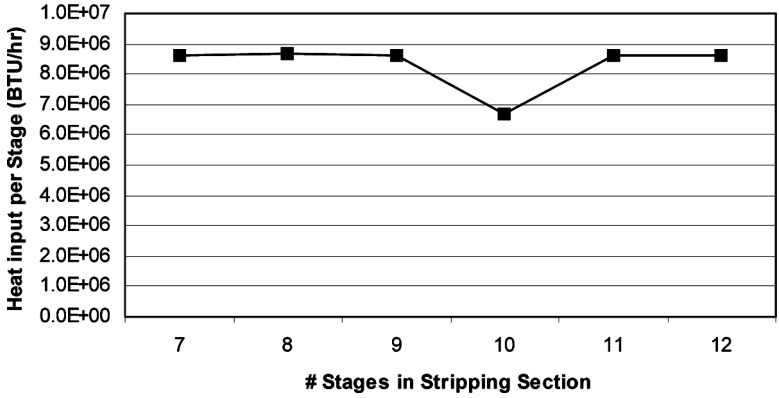

Figure 23. Effect of changing the number of stages in the stripping column on energy savings.

The effect of changing the number of stages in the stripping and rectifying columns was also investigated. Although increasing the number of stages in the two columns did slightly increase the purity, the increase was minimal. When the number of stages in the rectifying column is increased, there is no effect on the total energy savings because neither the reboiler nor compressor is associated with the rectifying column. But when the number of stages in the stripping column is increased or decreased, the energy savings decrease as shown in Fig. 23. This suggests that the optimal number of stages for the Lower Pressure HIDiC configuration is seven stages in the rectifying column and ten stages in the stripping column.

LIMITATIONS OF THE HIDIC CONFIGURATIONS

Heat Transfer Area

According to previous studies (2,23), a reasonable value for the overall heat transfer coefficient is $517\,\mathrm{kcal/m^2 \cdot hr \cdot K}$, but can be as high as $827\,\mathrm{kcal/m^2 \cdot hr \cdot K}$. Assuming the maximum overall heat transfer coefficient, the required heat transfer areas for each integrated stage can be found in Table 3. According to Gadalla *et al.*, 2006a, the heat panels designed by TU Delft can achieve the 88.6 m^2 of heat transfer area needed for the Lower Pressure HIDiC case. To achieve the required amount of heat transfer area, the rectifying and stripping column can be arranged as either concentric or multiconcentric columns with heat

Table 3. Maximum ($U = 516.75\,kcal/m^2{\cdot}hr{\cdot}K$) and minimum ($U = 826.79\,kcal/m^2{\cdot}hr{\cdot}K$) required heat transfer areas per stage

Heat Integrated Stage	A_{min} (m^2)	A_{max} (m^2)
Higher Pressure HIDiC		
1	270.76	433.21
2	348.51	557.60
3	463.51	741.61
4	579.94	927.89
5	657.63	1052.20
Lower Pressure HIDiC		
1	53.18	173.28
2	61.06	223.04
3	71.50	296.65
4	81.68	371.16
5	88.61	420.88

panels on the stripping side, the rectifying side, or both (21). According to this previous work (2), the heat transfer area of $658\,m^2$ needed to achieve the 1.5×10^7 BTU/hr of heat transfer for the Higher Pressure case cannot be attained. Therefore, HIDiC configurations that require heat transfer areas over $621\,m^2$ are not expected to be feasible.

Thermal Degradation

Columns used in acetic acid/acetic anhydride separations are operated at vacuum pressures because acetic anhydride will thermally degrade at its atmospheric boiling point, 139°C (1). The thermal degradation of acetic anhydride is the result of a kinetic reaction in which the degradation rate is expected to be exponentially dependent on temperature. Because the degradation rate is exponentially dependent on temperature, and the temperature is exponentially dependent on pressure (see Fig. 24), operating the column at vacuum pressures greatly reduces the degradation of acetic anhydride. The bottom stage of the rectifying column, which has the highest temperatures in the HIDiC configuration, is the area of concern with respect to thermal degradation. From the relationship observed in Fig. 24 for the seventh stage temperature as a function of pressure, seventh stage rectifying column pressures over 24.0 psia result in thermal degradation. Thermal degradation was also taken into account when designing the VRC simulation where the main concern was the high pressures and temperatures of the compressed vapor stream. To avoid thermal degradation of the acetic acid product the VRC compressor

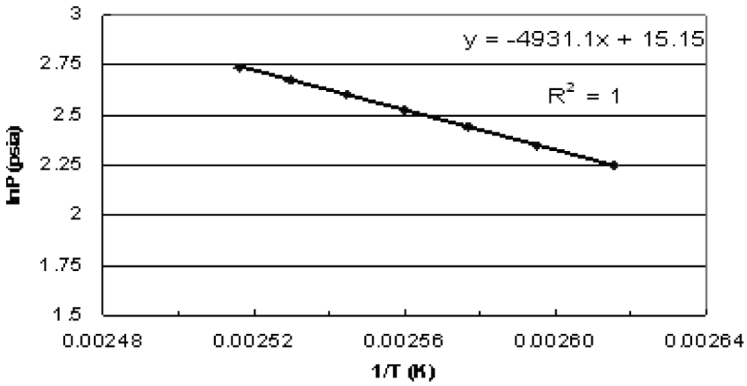

Figure 24. Temperature as a function of pressure on the bottom stage of the rectifying column.

ran at 24 psia with an output temperature of 135°C resulting in a slightly less than desirable heat exchanger design $\Delta T = 10^{\circ}C$ between fluid streams.

Design Specifications

According to Nakaiwa *et al.*, 2003, the three design variables that impose strict requirements to actual operation feasibility for the ideal HIDiC are the feed flowrate, the overhead product purity, and the bottoms product purity. These are the three variables that impose strict requirements because they control whether or not the HIDiC configuration that is more economical than the Reference distillation based on the cost of the feed and the revenue generated by the products. If the feed flowrate is high and the product purities are low, the energy savings are offset by operational costs. To offset the economic problems in the design of the two HIDiC configurations examined in this work, both the feed flowrate and the bottoms flowrate were set while manipulating the number and placement of integrated side streams, the amount of energy transferred, the pressures, and the reflux ratios to obtain cost effective purities.

SUMMARY AND CONCLUSIONS

It has been shown that the energy savings for this separation can reach 91.1% with VRC and 62% using HIDiC, with most of the energy

requirement arising for the work needed to compress the vapor stream rather than as reboiler duty. In addition, the distillate and bottoms product purities are increased for all three alternative cases, thus eliminating the economic tradeoff previously experienced in energy saving columns. The amount of heat transfer needed in the Lower Pressure HIDiC case is obtainable using heat panels when the rectifying and stripping columns are arranged either concentrically or multiconcentrically. The following concluding remarks can be made about the vacuum condition VRC and HIDiC design for separation of acetic acid and acetic anhydride:

VRC

1. The preheated feed stream reduced the amount of energy needed in the reboiler, which reduced the compressor duty to one tenth of the Reference distillations overall required energy. The distillate vapor flowrate to the compressor was one third that of the lower pressure HIDiC case causing the energy for the compressor to be much less than the HIDiC cases.
2. The vapor and liquid flows in the VRC column were greater that the Reference case, which increased the product purity, but the exiting distillate and bottoms rates were reduced.

HIDIC

1. The mixture from the bottom of the rectifying column is mixed with the feed before it is sent to the first stage of the stripping column. This is done to create a partially vaporized feed to the column, creating a thermal condition ($0 < q \leq 1$) that increases separation and energy recovery (Nakaiwa et al., 2003).
2. The HIDiC configurations increase the vapor and liquid flows throughout the column, which are both at a maximum in the stages at the bottom of the rectifying column and the top of the stripping column. Thus, the reflux ratio can be reduced due to the presence of constant internal reflux resulting from the condensation of vapor in the rectifying column and evaporation of liquid in the stripping column.
3. Having heat integration at the bottom stages of the stripping column decreases the column temperature differences, resulting in a lower reboiler duty. As the pressure decreases, the temperature difference decreases to the point where there it is no longer high enough to allow heat transfer from the rectifying section to the

stripping section. To overcome this problem, the upper stages of the stripping column are heat integrated in the Lower Pressure HIDiC case, which increases the temperature difference and decreases the required heat transfer per stage.

4. Energy requirements are reduced at lower pressures because the lower vapor flowrate resulting at decreased temperatures requires less work from the compressor.
5. The reboiler duty decreases linearly with decreasing reflux ratio and increasing heat transfer per stage. In addition, the bottoms and distillate purities also increase with increasing heat transfer per stage, but decrease with decreasing reflux ratio.
6. Manipulating the staging and decreasing the heat transfer per stage have adverse effects on an optimized HIDiC simulation.

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