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Increasing the Energy Efficiency of Extractive Distillation

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ABSTRACT

New flow sheets are developed for extractive distillation when it is used to break azeotropes. Detailed simulations of the model system, ethanol–water–ethylene glycol, are used to determine the energy use and column sizes. Replacing the total condenser used for the concentration column of the classical extractive distillation design with a partial condenser and thus connecting to the second column with a vapor instead of a liquid stream can result in reductions of both heating and cooling requirements of over 20%. An additional 9% reduction in heating and cooling requirements can be obtained with a new recycle design for extractive distillation.

Key Words: Energy; Extractive distillation.

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Extractive distillation is commonly used for the separation of chemical mixtures that form azeotropes or have low relative volatilities.[1] Unfortunately, extractive distillation is considered to be a major energy user. New flow sheets to reduce the energy use in extractive distillation to break azeotropes were developed and discussed in this article. Accurate simulations of the extractive distillation methods were done for the model system ethanol–water–ethylene glycol using Aspen Plus.[2]

CURRENT PROCESSES

The conventional three-column extractive distillation system for ethanol water separation with ethylene glycol as the solvent is shown in Fig. 1(a).[1,3–5] The first column concentrates the ethanol in the dilute feed and produces a pure water bottoms product. The second column adds solvent to make the azeotrope disappear allowing production of a pure ethanol distillate. The third column recovers solvent for recycle and produces a water distillate. Note that there are two water product streams: bottoms and effluent.

One defining feature of the standard process is that liquid product is taken from the total condenser of column 1 to be fed into column 2 [see Fig. 1(a)]. In a slight variation, a vapor distillate that is taken from a partial condenser of column 1 is fed into column 2[5,6] [see Fig. 1(b)]. While liquid transfer eases operational concerns, it increases cooling in the condenser of column 1 and increases energy requirements in the reboiler of column 2. Use of vapor transfer reduces energy requirements, but there is a trade-off in operability because intermediate storage is difficult and both columns are now linked. Also, if there is an increased vapor flow in column 2, a larger diameter will be required, raising capital cost. However, an advantage of introducing a vapor feed to the extractive distillation column is that it helps to “maintain a higher solvent concentration on the feed tray and the trays immediately below.”[5]

There is an optimum concentration of the distillate product from column 1 in both flow sheets. Approaching the azeotropic concentration in column 1 increases the energy input and the capital investment in column 1, but reduces the energy and equipment costs required for columns 2 and 3. On the other hand, approaching the feed concentration reduces the energy input and the capital investment in column 1, but increases the energy and equipment costs required for columns 2 and 3.

Multieffect[4,6,7] and heat-integrated[4,7] extractive distillation systems have also been developed. The multieffect systems essentially split column 1 in Fig. 1(a) or (b) into two parts at different pressures, and result in substantial

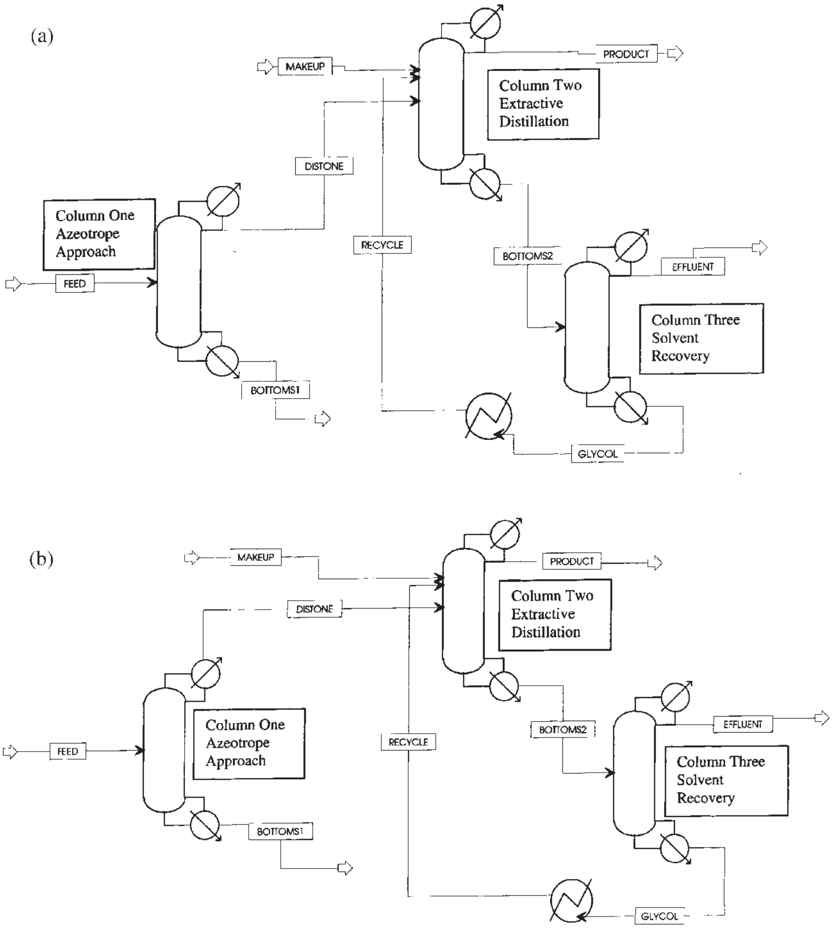


Figure 1. Conventional extractive distillation systems. (a) Liquid distillate feed to column 2.[3] (b) Vapor distillate to column 2.

energy savings. Comparisons[4,7] between multieffect and heat-integrated systems for ethanol–water separation showed that multieffect systems required less energy.

Another interesting extractive distillation process combines columns 2 and 3 in Fig. 1(a) and (b) into a single column with a vapor side withdrawal below the feed stage.[8] Pure ethanol is the distillate product. Pure ethylene glycol is the bottoms product. Approximately a 99 mol percentage water, 1 mol percentage ethylene glycol product is withdrawn as the side product.

NEW PROCESSES

In the standard and modified flow sheets [see Fig. 1(a) and (b)], the solvent is present in large quantities throughout column 2. However, based on the binary ethanol–water equilibrium data, separation of these components is easy at low ethanol concentrations. Thus, the solvent may be removed earlier in the flow sheet. Such a step corresponds with the heuristic, remove mass separating agents early.[9] Two new flow sheets [Fig. 2(a) and (b)] do this. Both flow sheets use a partial condenser on column 1 and a vapor transfer line to column 2.

The new flow sheets depart from the conventional partial condenser system in that the only water product is the bottoms from column 1. This requires recycle of the distillate from column 3 to column 1. The bottoms from column 2 and the distillate from column 3 can contain ethanol. The flow sheet in Fig. 2(a) uses a total condenser on column 3 and recycles a liquid to column 1. In Fig. 2(b), column 3 has a partial condenser with vapor recycle to column 1. The partial condenser reduces cooling in the condenser of column 3 and heating in the reboiler of column 1. To operate properly, the process in Fig. 2(b) must have $p_{\mathrm{column3}} > p_{\mathrm{column1}} > p_{\mathrm{column2}}$. This can be done conveniently by using a pump on the bottoms from column 2. The two recycle processes introduce two new key variables: the flow rate and purity of the recycle stream fed to column 1.

SIMULATIONS

The specifications for the feed, products, and solvent recycle stream are given in Table 1. A constant pressure of 1 atm was used. The RADFRAC distillation routine of Aspen Plus version 10.01[2] was used to model the processes. The nonrandom two-liquid (NRTL) thermodynamics package was used to describe the vapor-liquid equilibrium (VLE). In all cases, the operation of each column was defined by the reflux ratio and the ratio of distillate to feed rates D/F. This ratio can be related to the desired distillate mol fraction by external mass balances.

For the conventional systems [see Fig. 1(a) and (b)] modeling began with column 1. The distillate to feed ratio D/F proved to be the primary determinate of the energy consumption for these systems. First, $(L/D)_{\mathrm{min}}$ was found using a large number of stages, and $(L/D)_{\mathrm{actual}}$ was set to 1.15 ×

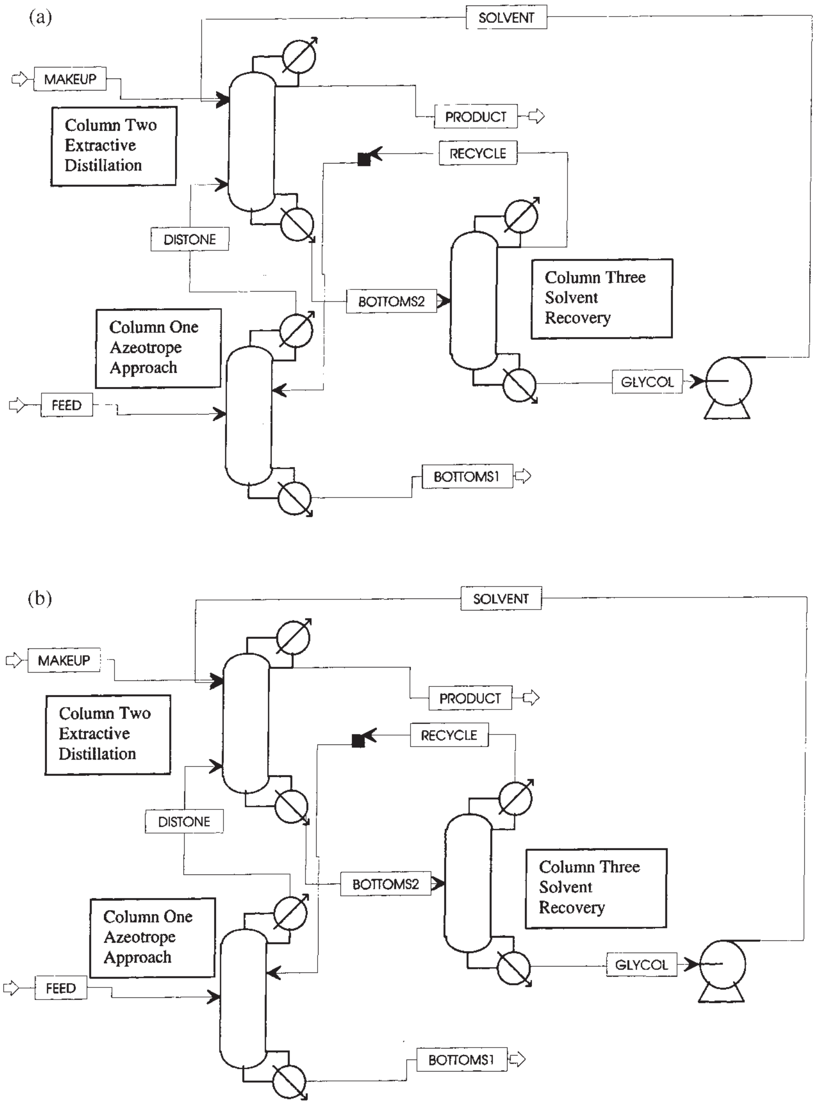


Figure 2. Proposed recycle extractive distillation systems. (a) Liquid recycle from column 3 to column 1. (b) Vapor recycle from column 3 to column 1.

Table 1. Specifications for streams.

Stream	Purity	Conditions	Flow rate
Feed	10.0 mole percentage ethanol	Saturated liquid	100.0 kmol/hr
Ethanol product	$>$99.7 mol percentage ethanol	Saturated liquid	—
Bottoms 1	$<$2.0 E−8 mol percentage ethanol	Saturated liquid	—
Solvent recycle in all cases	99.999 mole percentage ethylene glycol	78.2°C before fed to column 2	5.0 kmol/hr

$(L/D)_{min}$. Then, the optimum feed stage was determined by trial and error. The total number of stages was reduced until the column could just meet the bottoms purity requirements. The feed stage was adjusted to remain at the optimum. This procedure should result in operating conditions that are close to optimum for each D/F value.

Column 2 was modeled after column 1 was optimized. The initial solvent feed to column 2 was assumed to be pure ethylene glycol, and its temperature was set to 78.2°C based on the approximate tray temperature. Some experimentation showed that stage 5 below the condenser was always close to the optimum location for solvent addition. A solvent flow rate of 5 kmol/hr to the extractive distillation column (for the basis of 100 kmol/hr of feed to column 1) worked well in all flow sheets. Less solvent resulted in lower ethanol product purity. More solvent had little impact on purity and increased energy costs. An external reflux ratio in column 2 of approximately 1.2 was sufficient to remove the ethylene glycol from the distillate product. Column 2 was then optimized to meet the ethanol product specification and produce a bottoms product with enough ethanol that the specifications for the distillate of column 3 would be met.

Column 3 is a simple binary column that was operated at an external reflux ratio equal to 1.15 times the minimum reflux ratio and uses the optimum feed stage. Once column 3 was optimized, the solvent recycle stream was connected to column 2. A heat exchanger must be placed between columns 3 and 2, cooling the solvent to the predetermined temperature of 78.2°C. The solvent recycle stream must contain little water or the ethanol product purity from column 2 deteriorates significantly.

The modeling of the new systems [see Fig. 2(a) and (b)] was more challenging. Columns 1 and 2 are modeled simultaneously, with column 1 being optimized first. The flow rate and composition of the recycle stream to column 1 needs to be estimated to start the simulation. This recycle flow rate and composition proved to be the most important factors in the energy consumption for these flow sheets.

Column 2 is optimized in the same way as in the conventional flow sheet. Initially, pure solvent is fed to stage 5. The optimum feed location depends on the purity chosen for the recycle stream from column 3. Since column 3 essentially removes only ethylene glycol, the ethanol to water ratio in the recycle stream to column 1 and in the bottoms of column 2 are practically identical. It is possible to recycle pure water, which requires a higher feed stage in column 2. Modeling column 3 again presents relatively few challenges.

After all columns were individually optimized, the solvent recycle stream was connected first, and convergence was obtained. At this point, the distillate from column 3 should be very similar to the recycle stream being fed to column 1. Assuming they almost matched, the system usually converged when this recycle stream was connected. If they were different, obtaining convergence was difficult.

The effect of changes in design can often be estimated without doing complete economic calculations. Operating costs were assumed to depend linearly on the total system heating duty (sum of all reboilers) and the total system cooling duty (sum of all condensers plus the solvent recycle heat exchanger). Capital costs were assumed to be proportional to the volume of separation (height × area), which is appropriate for packed columns and approximate for tray columns. Since the height is proportional to N_j and area is proportional to $V_{\max,j}$ (assuming a constant approach to flooding), the volume factor is defined as

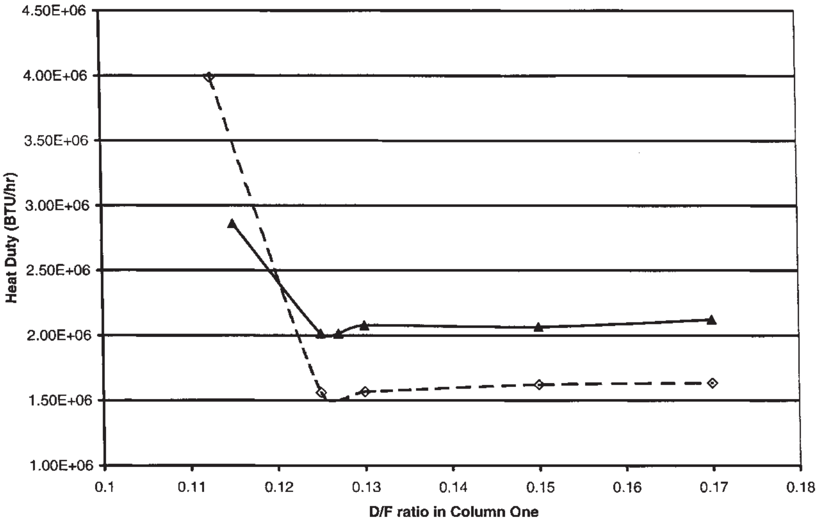
$$\text{Volume} - \text{factor} = \sum N_j * V_{\max,j}$$

where N_j is the number of theoretical stages in column j, and $V_{\max,j}$ is the maximum vapor flow rate within column j in kmol/hr.

RESULTS

Total system heating and cooling duties for the total condenser flow sheet [see Fig. 1(a)] and the partial condenser system [see Fig. 1(b)] are shown in Fig. 3(a) and (b). The ratio of D/F in column 1, which controls the approach to the azeotrope concentration, is a controlling variable in the simulations. At the optimum D/F (~0.125) and higher D/F values, the partial condenser system is significantly more energy efficient than the total condenser system for both heating and cooling duties. At D/F = 0.125, the reboiler difference of 453,000

(a)



(b)

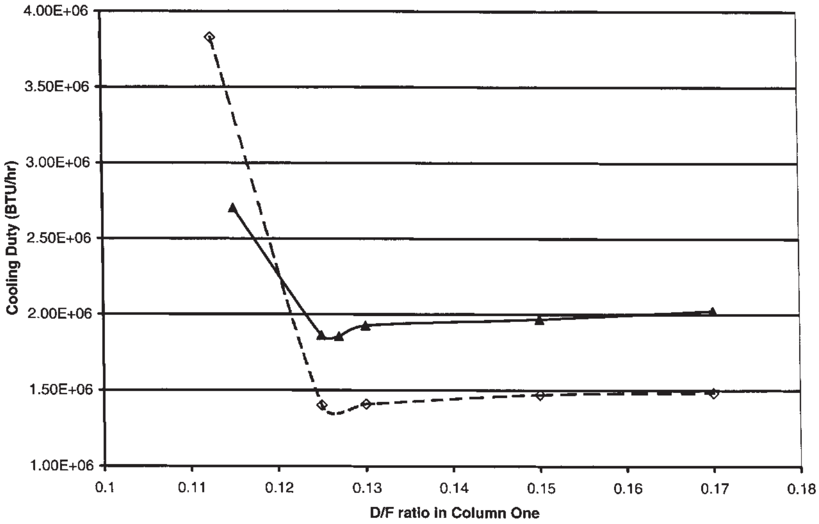


Figure 3. Results for conventional designs. (a) Sum of reboiler heating duties. (b) Sum of condenser cooling duties. (c) Volume factors. *Key:* ▲, total condenser; ◇, partial condenser.

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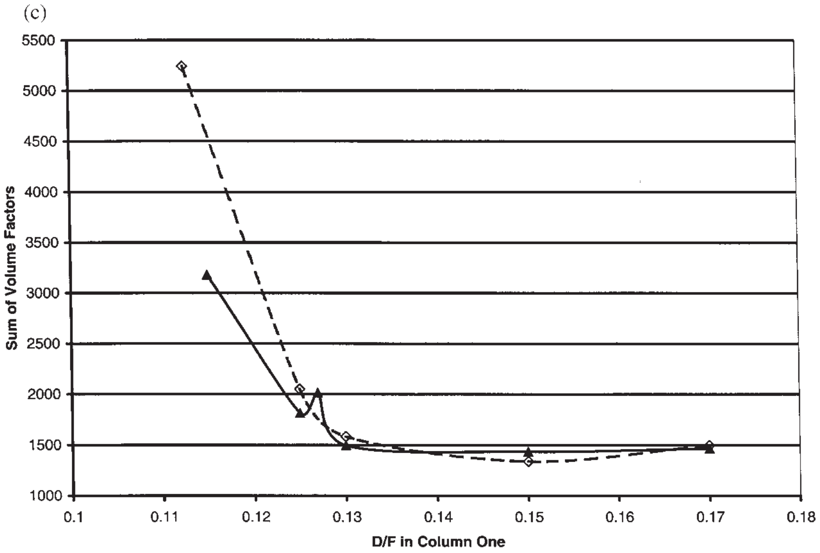

Figure 3. Continued.

BTU/hr represents a savings 22.5% for the partial condenser system compared to the total condenser system. The savings of −460,000 BTU/hr in the condensers represents a 24.7% advantage for the partial condenser system. Surprisingly, at the D/F that minimizes the volume factor (∼0.15), the partial condenser system requires a slightly lower volume factor than the total condenser system [see Fig. 3(c)]. Since the optimums for energy savings and separation volumes differ, a compromise value of D/F = 0.13 was used. The optimal designs at D/F = 0.13 for the total condenser and partial condenser systems are given in Tables 2 and 3, respectively. At D/F = 0.13, the partial condenser system saves 24.5% of the heating required in the reboiler and 27.0% of the cooling required in the condenser, while the volume factor increases by 5.8%.

The energy use in the new recycle process flow sheets [Fig. 2(a) and (b)] can be compared on a similar basis [Fig. 4(a) and (b)]. Missing data points in Fig. 4 occur when convergence could not be obtained. Initially, the recycle stream concentration was set at 15 mol percentage ethanol and the results for the liquid and vapor recycle were compared. At the optimum recycle rates (8.0 to 8.5 for vapor and 7.0 for liquid recycle) for the 15 mol percentage ethanol recycle, energy use in the vapor recycle system is considerably lower than with liquid recycle, and the liquid recycle system was inferior to the standard

Table 2. Design information for conventional design—total condenser optimum.

	Column 1	Column 2	Column 3	Heater	Total
Stages	23	40	20		
Feed stage	12	35	10		
Solvent feed stage		4			
Reboiler duty (BTU/hr)	1.18E+06	7.14E+05	1.89E+05		2.08E+06
Condenser duty (BTU/hr)	−1.03E+06	−6.66E+05	−1.34E+05	−9.85E+04	−1.93E+06
D/F	0.13	0.77	0.37		
Reflux (L/D)	1.10975	0.8	0.162725		
Boil-up ratio	0.349198	2.1446153	0.748366		
$V_{\max}$ (kmol/hr)	30.38	18.05	3.74		
Volume factor	699	722	75		1495

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Table 3. Design information for conventional design—partial condenser optimum.

	Column 1	Column 2	Column 3	Heater	Total
Stages	25	43	17		
Feed stage	12	39	9		
Solvent feed stage		4			
Reboiler duty (BTU/hr)	1.153E+06	2.269E+05	1.872E+05		1.567E+06
Condenser duty (BTU/hr)	−5.11E+05	−6.65E+05	−1.32E+05	−9.97E+04	−1.41E+06
D/F	0.13	0.77	0.37		
Reflux (L/D)	1.0465	0.798	0.1495		
Boil-up ratio	0.343	0.663	0.742		
V_{max} (kmol/hr)	29.81	18.02	3.71		
Volume factor	745	775	63		1584

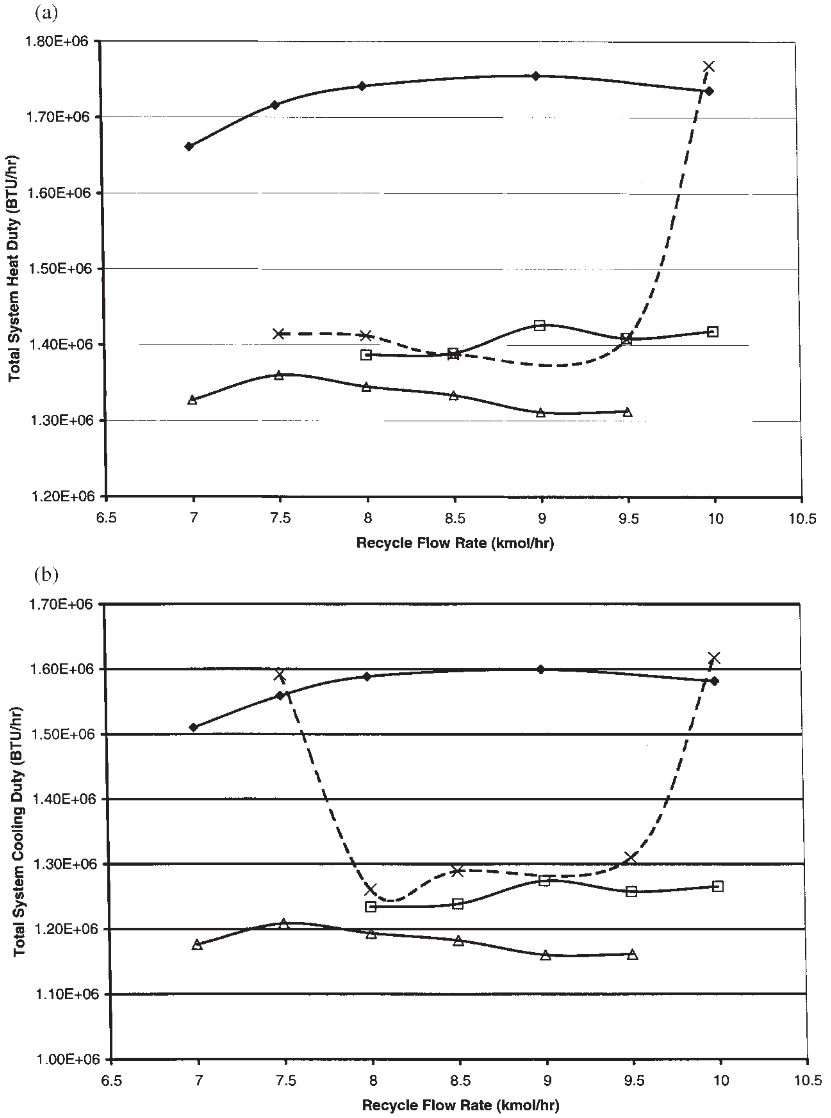

Figure 4. Results for proposed recycle designs. (a) Sum of reboiler heating duties. (b) Sum of condenser cooling duties. (c) Volume factors. *Key:* ◆, liquid recycle to column 1, 15% ethanol; □, vapor recycle to column 1, 5% ethanol; △, vapor recycle to column 1, 0% ethanol; and ×, vapor recycle to column 1, 15% ethanol.

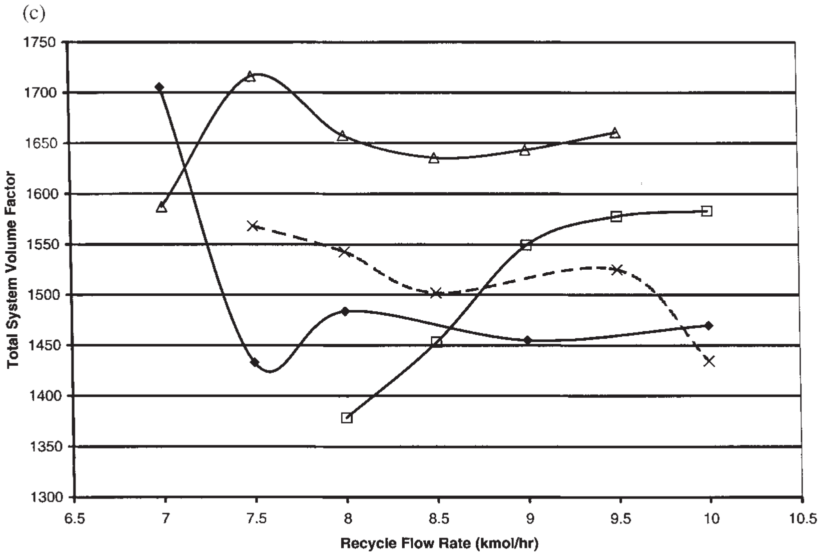

Figure 4. Continued.

system with a partial condenser [see Fig. 1(b)]. The liquid recycle system did have a somewhat lower volume factor than the vapor recycle system [see Fig. 4(c)]. Based on the significantly reduced energy usage of the vapor recycle system, the effect of recycle concentration was studied only for vapor recycle.

A pure water vapor recycle results in the most energy efficient system [see Fig. 4(a) and (b)] since the steam is input into the reboiler of column 1 where it serves as open steam heating. However, pure water vapor recycle has a significantly higher volume factor than the other vapor recycle systems [see Fig. 4(c)].

A vapor recycle system with 5 mol percentage ethanol appears to be a useful compromise. Since the volume factor for this recycle system is much more sensitive than the energy requirements, a recycle rate of 8.0, which minimizes the volume factor (1380), was chosen. The optimal design for this system is described in Table 4. At a recycle rate of 8.0, the heating requirements are 1,420,000 and the cooling requirements are 1,270,000 BTU/hr. The vapor recycle process can be compared to the conventional design with a partial condenser [see Fig. 1(b)] with the first column operating at D/F = 0.15, which minimizes the volume factor (volume factor = 1340, total reboiler duty = 1,620,000, and total condenser duty = −1,470,000 BTU/hr).

Table 4. Design information for proposed design—5% ethanol recycle, 8 kmol/hr recycle rate.

	Column 1	Column 2	Column 3	Heater	Total
Stages	16	35	16		
Feed stage	3	34	9		
Recycle/solvent feed stage	8	5			
Reboiler duty (BTU/hr)	8.14E+05	1.94E+05	4.11E+05		1.42E+06
Condenser duty (BTU/hr)	−2.95E+05	−8.50E+05	−2.08E+04	−9.97E+04	−1.27E+06
Distillate/feed (D/F)	0.167	0.435	0.615		
Reflux ratio (L/D)	0.416	1.297	0.067		
Boil-up ratio	0.234	0.345	1.628		
$V_{\max}$ (kmol/hr)	27.09	23.03	8.69		
Volume	433	806	139		1378

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When compared to this conventional partial condenser system, the vapor recycle system with 5 mol percentage ethanol operates at energy savings of 12.3% for heating and 13.6% for cooling. The volume factor for this vapor recycle system is 3.0% higher. The reduced sizes of the reboiler and condenser will help to compensate for the increased capital cost of the distillation column.

An alternative is to compare the vapor recycle system with 5 mol percentage ethanol to a partial condenser system with the first column operating at $D/F = 0.13$ (see Table 3). This D/F is very close to the minimum for energy use and the increase in the volume factor compared to $D/F = 0.15$ is modest (volume factor = 1580, total reboiler duty = 1,570,000, and total condenser duty = −1,410,000 BTU/hr). When compared to the conventional partial condenser system with $D/F = 0.13$ in column 1, the vapor recycle system operates at energy savings of 9.6% for heating and 9.9% for cooling. The volume factor for the vapor recycle system is 12.7% lower. For both comparisons the vapor recycle system is more economical.

DISCUSSION

The flow sheets in Fig. 2(a) and (b) have a solvent recycle to column 2 and a water/ethanol recycle to column 1. With two recycle streams and very nonlinear VLE, convergence was often difficult. The optimum recycle rate for the vapor recycle with 5 mol percentage ethanol may be less than 8.0 kmol/hr. Despite repeated attempts, we were unable to obtain convergence at a recycle rate of 7.5.

The new recycle processes [see Fig. 2(a) and (b)] were also designed with no condenser in column 1 and no reboiler for column 2. The liquid returning from column 3 would serve to create reflux for column 1, while the vapor leaving column 1 would serve as the source of boil-up for column 2. To maintain the energy requirements necessary for both column 1 and column 2, a high volume of recycled material was needed, which significantly increased energy use and volume factors in all three columns.

In some situations it may be possible to retrofit existing equipment to switch from liquid connections between columns 1 and 2 [see Fig. 1(a)] to a vapor connection [see Fig. 1(b)]. This will result in significant energy savings for both heating and cooling. However, since the optimum designs for the columns are somewhat different, retrofitting may work best when the plant is operating below design capacity. Retrofitting to operate in the fashion shown in Fig. 2(a) and (b) will be more difficult but, if possible, will result in additional energy savings.

Comparison of our results with the results obtained with a multieffect extractive distillation system is instructive. For Fig. 2(b) for the optimum design, the total energy use is 3244.5 kJ/kg of 99.7% ethanol product for a feed that is 10 mol percentage ethanol. Lynn and Hanson[6] found that for a 10 weight percentage (4.17 mol percentage) ethanol feed that the total energy use for a four-column, multieffect distillation was 2730 kJ/kg of ethanol product, which is significantly less. However, it would be easy to use a multieffect column instead of column 1 in Fig. 2(b). Lynn and Hanson[6] found that the total energy use for the multieffect system was 56.1% (for 6 weight percentage ethanol feed) of that for Fig. 1(b). Arbitrarily assuming the same percentage improvement when Fig. 2(b) is converted to a multieffect system, we can estimate its energy use with two effects for column 1 as approximately 1820 kJ/kg ethanol for the 10 mol percentage feed. Although detailed calculations are required for this configuration, the multieffect recycle design appears to be very competitive. If a higher ethylene glycol concentration can be tolerated in the water product stream than listed in Table 1, then the one column extractive distillation scheme[8] would also be of interest.

Although this study centered on using extractive distillation to break the azeotrope formed by ethanol and water, the results should be qualitatively correct for other azeotropic systems. Many of the specific details, such as the optimum recycle concentrations and flow rates, will depend on the system. A system in which the stripping process in column 2 is more difficult than that of ethanol and water is likely to be more energy efficient at higher recycle concentrations.

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