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HEAT INTEGRATED ETHANOL DEHYDRATION FLOWSHEETS

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ABSTRACT

A theoretical evaluation of heat-integrated heterogeneous-azeotropic ethanol-water distillation flowsheets is presented. Simulations of two column flowsheets using several different hydrocarbon entrainers reveal a region of potential heat integration and substantial reduction in operating energy. In this paper, methods for comparing hydrocarbon entrainers are shown.

Two aspects of entrainers are related to operating and capital costs. The binary azeotropic composition of the entrainer-ethanol mixture is related to the energy requirements of the flowsheet. A temperature difference in the azeotropic column is related to the size of the column and overall process staging requirements. Although the hydrophobicity of an entrainer is essential for specification of staging in the dehydration column, no substantial increase in operating energy results from an entrainer that has a higher water content. Likewise, liquid-liquid equilibria between several entrainer-ethanol-water mixtures have no substantial effect on either staging or operation. Rather, increasing the alcohol content of the entrainer-ethanol azeotrope limits its recovery in the dehydration column, and increases the recycle and reflux streams. These effects both contribute to increasing the separation energy requirements and reducing the region of potential heat integration.

A cost comparison with a multieffect extractive distillation flowsheet reveals that the costs are comparable; however, the extractive distillation flowsheet is more cost effective as operating costs increase.

INTRODUCTION

In azeotropic distillation, a separating agent, usually called the entrainer, is added to modify the activities of the compounds being separated; that is, to increase the relative volatility between the two components being separated and thereby make it easier or even possible to separate a binary mixture that has close-boiling points or forms a binary azeotrope. To be able to affect the activities of other components, a large amount (or high concentration) of the entrainer should be maintained in the liquid phase. Thus, the boiling point of the entrainer should be close, but not too close, to those of the other components. The entrainer may form an azeotrope with the component to be taken overhead. Frequently, the azeotrope is heterogeneous; Treybal (1) recommends this. Upon condensation it will split into two different phases, one on each side of the distillation boundaries (see Doherty et al.(2)). The classic paper by Benedict and Rubin (3) provides an overview of the subject.

Figure 1 is a typical flowsheet with two distillation columns and one decanter for purifying ethanol from ethanol-water mixtures. Since water is more polar than ethanol, it will be more strongly repelled from a nonpolar liquid than ethanol. Thus, the presence of a nonpolar hydrocarbon will significantly increase the water activity and thus enhance its volatility. This dehydration of ethanol by enhancing the water volatility permits a pure ethanol product to be withdrawn from the bottom of the column. The overhead vapor of the dehydrator condenses into two liquid phases. The aqueous phase is recycled to the concentrator and the organic phase is returned to the dehydrator as reflux. Typical operating specifications for this system were reported by Prokopakis and Seider (4). Black et al. (5) proposed pentane as an alternative entrainer to benzene for this system.

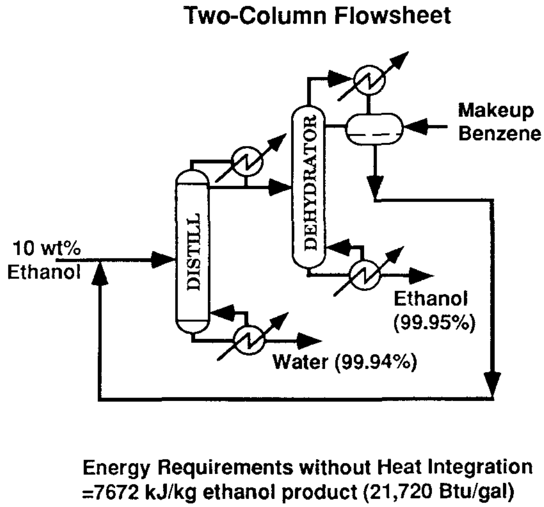

FIGURE 1. Typical two column flowsheet for dehydration of ethanol.

In order to reduce the energy requirement associated with the process flowsheet of Figure 1, a heat integrated flowsheet shown in Figure 2 was investigated. The column pressures are specified such that the condenser load of the concentrator can be used to supply heat to the dehydrator reboiler. The heat from the product streams is recovered by using it to preheat the feed to the concentrator. Thus, the heat input to the concentrator reboiler, QRB1, constitutes all of the required energy input to the system.

Figure 3 shows binary azeotropic data for several potential hydrocarbon entrainers and ethanol where the mole fraction entrainer in the azeotrope is plotted against the temperature difference between the azeotrope's boiling temperature and ethanol's normal boiling point. Considering only those parameters, it can be expected that a hydrocarbon which has high entrainer concentration in its entrainer-ethanol azeotrope

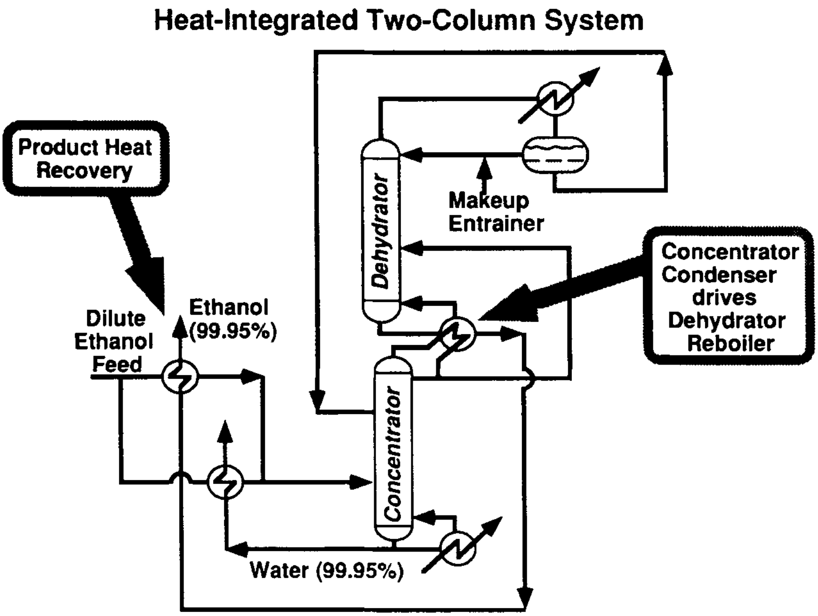

FIGURE 2. Heat integrated two column flowsheet for ethanol dehydration.

(proportional to reflux or energy requirement) and high temperature difference (proportional to staging) might be a better candidate. The simulation with a potential entrainer from the middle of this hydrocarbon family of potential entrainers will provide a clearer picture of the costs associated with this family.

Rion and Van Brunt (6) made a comparison of the energy and staging requirements between n-pentane and benzene entrainers. They concluded that the heat integrated flowsheets require less equipment than extractive distillation and less energy than conventional two-column azeotropic distillation flowsheets. The objective of this work is to extend their research to include the heat integrated flowsheets using 2-methyl-pentane

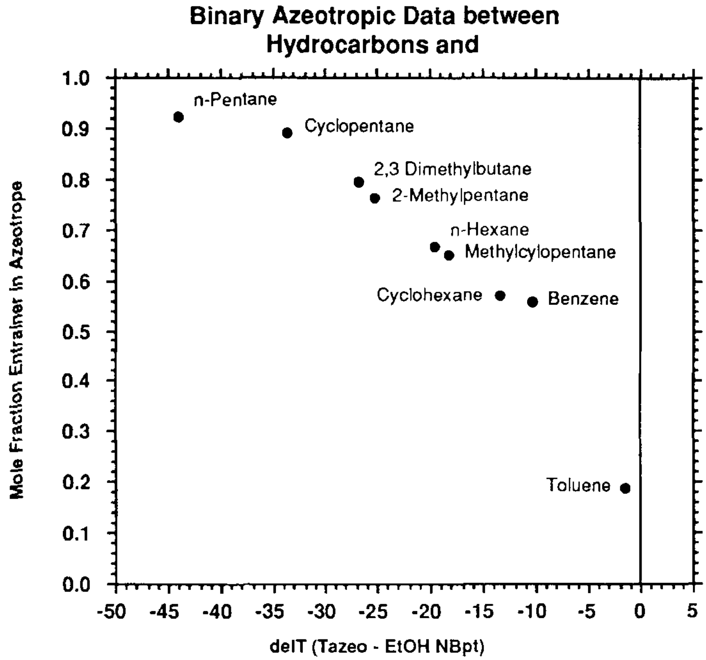

FIGURE 3. Binary azeotropic data of several hydrocarbon-ethanol pairs.

as the entrainer and compare the result with those of n-pentane and benzene. Thus, a similar method of simulation that is detailed in Rion and Van Brunt (6) was applied for 2-methylpentane.

SIMULATION RESULT COMPARISON

The simulation results for benzene, n-pentane, and 2-methylpentane are shown in Figures 4a-c and an overall comparison is shown in Table 1. From the table it can be seen, in terms of staging and energy requirements, that pentane and 2-methylpentane entrainers are better than benzene. The larger staging for benzene is because the temperature

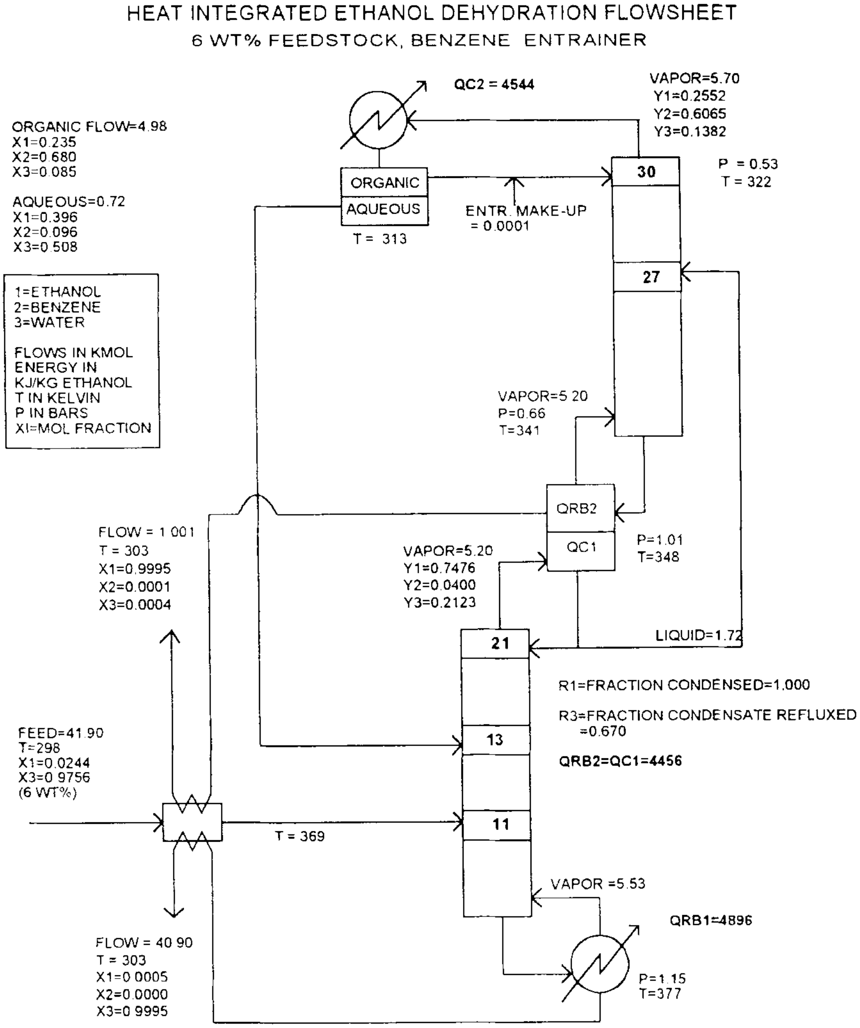

FIGURE 4a. Simulation results for 6 wt.% ethanol feed with benzene entrainer.

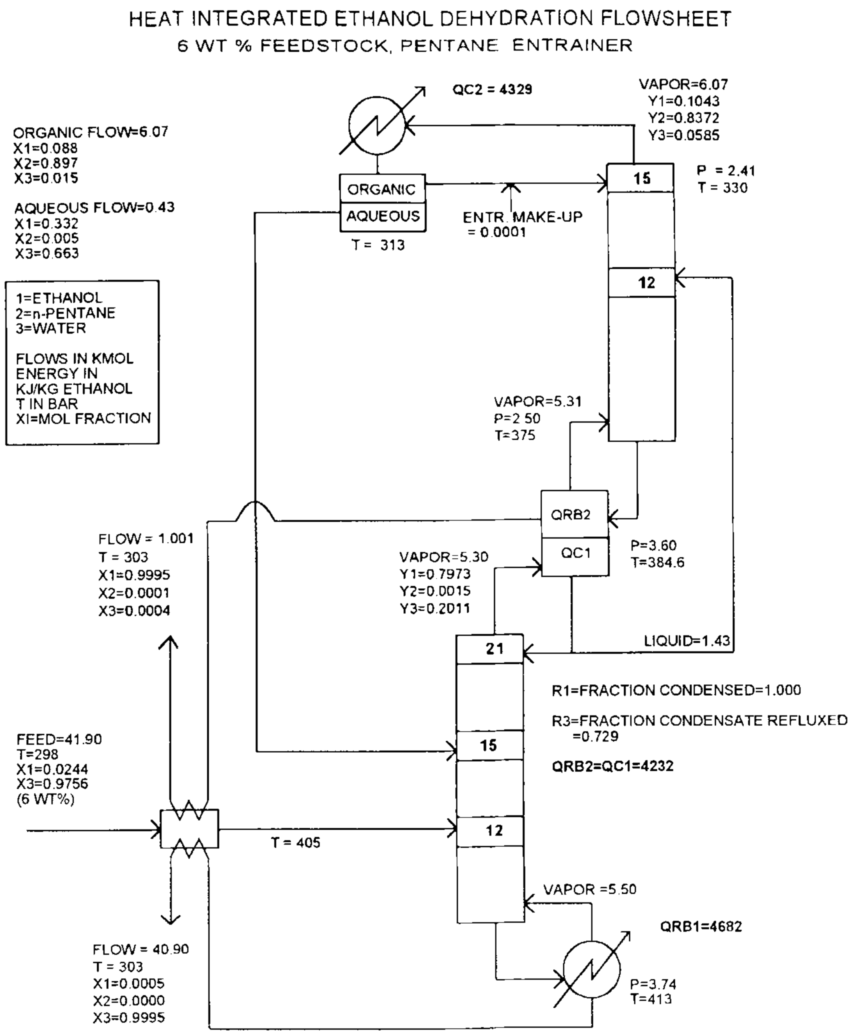

FIGURE 4b. Simulation results for 6 wt.% ethanol feed, pentane entrainer.

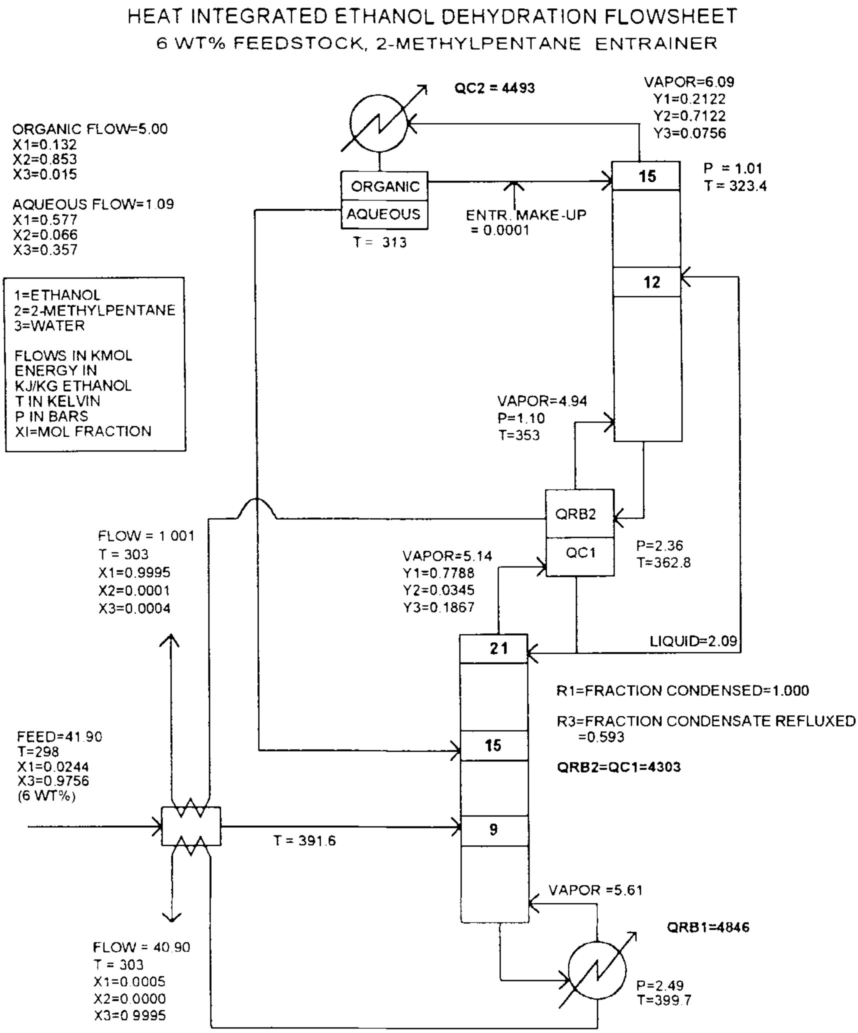

FIGURE 4c. Simulation results for 6 wt% ethanol feed, 2-methylpentane entrainer.

TABLE 1. COMPARISON OF SIMULATION RESULTS

Feed (%)	Entrainer	Stages Con.	Stages Deh.	Y_E Con.	Heat (kJ/kg EtOH) QRB1	QRB2	QC2	Pressure, bar Con.	Deh.
10	Benzene	28	40	0.804	3747	3470	3539	1.01	0.53
10	Pentane	28	22	0.835	3413	3117	3205	3.60	2.40
10	2-me.pentane	28	20	0.769	3500	3118	3292	2.26	1.01
6	Benzene	21	30	0.748	4896	4456	4544	1.01	0.53
6	Pentane	21	15	0.797	4682	4226	4329	3.60	2.40
6	2-me.pentane	21	15	0.779	4846	4303	4429	2.40	1.01

Con.=concentrator; Deh.=dehydrator; Y_E=ethanol concentration in the vapor phase of the last stage; QRB1 and QRB2 = reboiler duty of concentrator and dehydrator, respectively; QC2=condenser load of dehydrator.

difference (Figure 3) for benzene is significantly smaller than that for n-pentane or 2-methylpentane. Consistent with Figure 3, n-pentane is a better entrainer than 2-methylpentane. However, although the temperature difference is significantly larger than that of 2-methylpentane, the results exhibit no significant differences in staging.

COST COMPARISON

In terms of energy and staging requirements, n-pentane and 2-methylpentane entrainers are superior to benzene. However, for a better comparison, it is worthwhile to calculate detailed costs for each flowsheet and for other process alternatives. In doing this, we utilized other process configurations by Lynn and Hanson (7), namely, multieffect extractive distillation with 4 and 5 column sequences with ethylene glycol as the extractive agent. In their creative work, they found that by applying multieffect operation, the energy required for dehydrating 6% ethanol can be reduced from 5880 kJ/kg ethanol, with ordinary (single-effect) extractive distillation, to 3300 and 2540 kJ/kg ethanol, with two- and three-effect operation, respectively; (or to 2730 and 2110 kJ/kg ethanol if 10% ethanol feed was used).

Assuming a column efficiency of 65% and an annual production of 38000 metric tons (as in Lynn's work), the dimensions of the columns for each case of heat integrated azeotropic distillation were calculated. This was done using the method and corresponding empirical correlations that appear in Henley and Seader (8). In this case the stage height was specified as 2 ft/stage while column diameters were determined by assuming vapor velocities as 85% of flooding velocities which were obtained by using the Fair correlation (8). The column diameters were calculated with :

$$D = \left(\frac{4\ V\ M_V}{0.85\ U_f\ \pi\ (1 - \frac{A_d}{A})\ \rho_V} \right)^{0.5} .$$

The flooding velocities were obtained from empirical relationship below.

$$U_f = F_{ST}\ F_F\ F_{HA}\ C_F \left(\frac{\rho_L - \rho_V}{\rho_V} \right)^{0.5} ,$$

where:

D = diameter

V = vapor molal flowrate

M_V = vapor molecular weight

U_f = flooding velocity

A_d = down flow area

A = total tray area

$F_{ST} = (\tau/20)^{0.2}$; τ = surface tension, dyne/cm

F_F = foaming factor, assumed to be 1.0 (non foaming)

F_{HA} = hole area factor; depends on the ratio of slot area to active area

C_F = flooding factor obtained from Fair correlation

ρ_L, ρ_V = liquid, vapor densities.

The diameter was taken as the largest stage diameter. Table 2 shows the calculated size of each column. The heat exchangers were assumed to be shell and tube where the areas were estimated using overall heat transfer coefficients equal to 100 Btu/(ft^2 °F hr) for ethanol vs. feed, and 500 Btu/(ft^2 °F hr) for water vs. feed (9). The size of the decanter was estimated so that it would have sufficient residence time for the phases to separate.

The cost components considered were steam cost as operating cost, and equipment cost as capital cost, which included sieve tray columns, heat exchangers, steam ejectors (for vacuum columns), and decanters. The material of the equipment was chosen as 316 stainless steel. Capital costs were calculated using cost factors suggested by Walas (10) with 6% interest per year. The annual total cost of each case is shown in Figures 5a-b where steam price of $2.4 per 1000 lb (11) was applied and depreciation-life of 10 years was considered (12). Concentrator stages (Nc) and dehydrator stages (Nd), which were different for each entrainer, were varied by decreasing and increasing them by one stage. Thus, the figures also show the linear sensitivity to the number of stages. The cost sensitivity to steam price is illustrated in Figures 6a-b where the annual costs of each flowsheet were plotted against the steam price.

From Figures 5 and 6, it is apparent that 2-methylpentane and pentane entrainers are much more economical than benzene. While

TABLE 2. COLUMN SIZES OF HEAT INTEGRATED AZEOTROPIC DISTILLATION OF ETHANOL-WATER CAPACITY: 38000 METRIC TONS ETHANOL ANNUALLY

	6% Ethanol Feed		10% Ethanol Feed	
	Concen-trator	Dehy-drator	Concen-trator	Dehy-drator
Benzene				
trays	32	46	43	61
diameter (m)	1.60	2.04	1.46	1.86
n-Pentane				
trays	32	23	43	34
diameter (m)	1.28	1.92	1.10	1.65
2-Methylpentane				
trays	44	23	43	27
diameter (m)	1.37	2.16	1.16	1.86

azeotropic distillation with n-pentane is the least expensive, the cost difference with that of 2-methylpentane is not large. Compared to multieffect extractive distillation, azeotropic distillation using n-pentane or 2-methylpentane is still more economical. For 6% feedstock, with steam price \$ 2.4/1000 lb, the cost of three-effect extractive distillation (Lynn's 5-column system) is about the same as that of azeotropic distillation using 2-methylpentane. We notice also, for extractive distillation, that three-effect operation is more economical than two-effect operation (Lynn's 4-

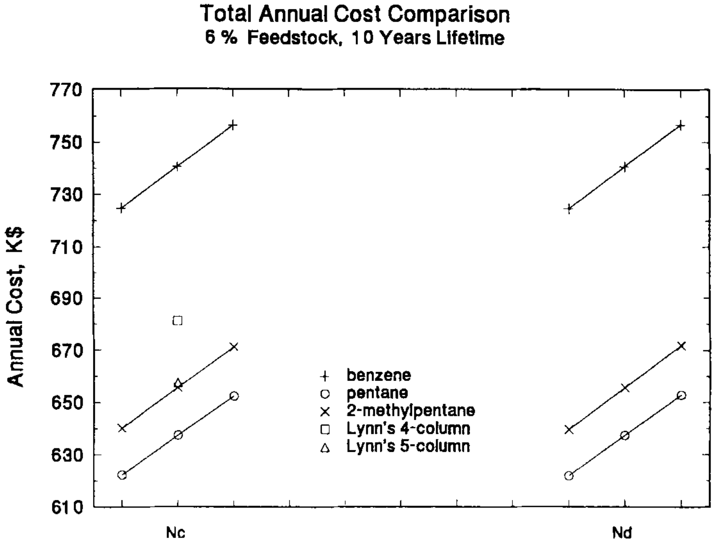

FIGURE 5a. Annual total cost comparison for 6 wt% ethanol feed.

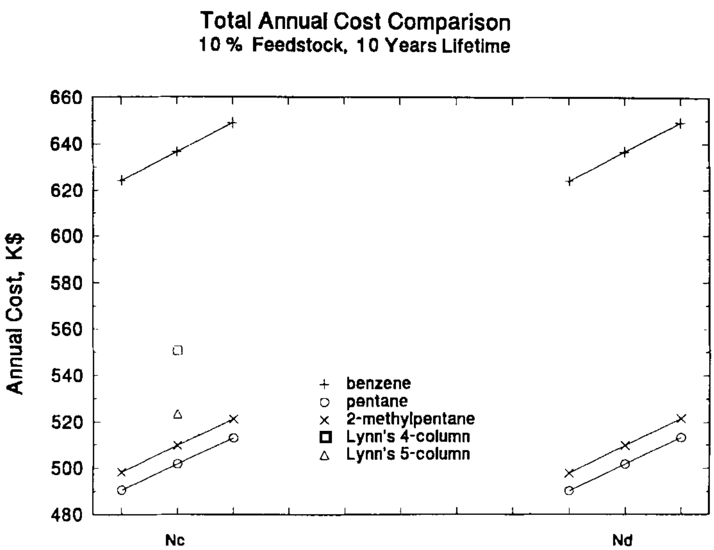

FIGURE 5b. Annual total cost comparison for 10 wt.% ethanol feed.

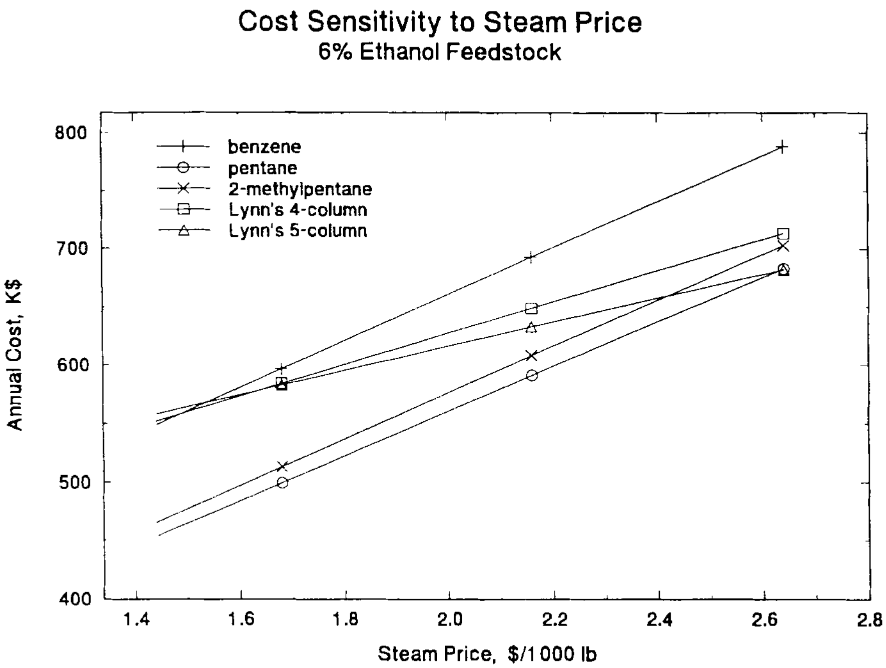

FIGURE 6a. Cost sensitivity to steam price for 6 wt.% ethanol feed.

column system). This implies that operating cost (steam cost) is more dominant than equipment cost; that is, the increase in capital cost caused by the addition of another column is less than the reduction in operating cost as a result of energy saving.

Figures 6a-b show that the azeotropic distillation is more sensitive to steam price than extractive distillation. Thus, multieffect extractive distillation will be more beneficial as the steam price increases. From Figure 6a, for example, we can observe that the azeotropic distillation using 2-methylpentane entrainer is more economical than three-effect extractive distillation if steam price is lower than $2.4/1000 lb.

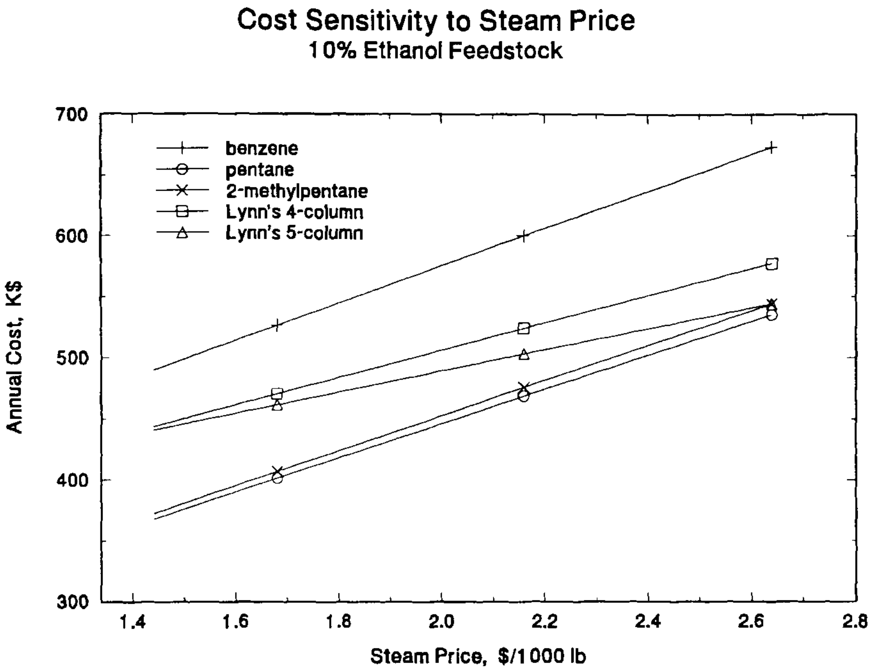

FIGURE 6b. Cost sensitivity to steam price for 10 wt.% ethanol feed.

What is striking here is that, basically, the simulation results are qualitatively consistent with what we have predicted previously. This further implies that the simple method for selecting an entrainer based on minimum information of ethanol-entrainer azeotropes (temperature differences between azeotrope's boiling temperatures and ethanol's normal boiling point, and azeotrope compositions) can provide a good estimation of relative costs. The fact that azeotropic distillation is more beneficial than multieffect extractive distillation implies that the size of azeotropic distillation columns is also a significant contributor to reduced capital cost.

CONCLUSION

Heat integrated azeotropic distillation using 2-methylpentane or n-pentane as the entrainer is an economical alternative to multieffect extractive distillation. Both n-pentane and 2-methylpentane are significantly more economical than benzene.

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