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Evaluation of Nondistillation Ethanol Separation Processes

L. Douglas
D. Feinberg

SERI

Solar Energy Research Institute
A Division of Midwest Research Institute

1617 Cole Boulevard
Golden, Colorado 80401

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PREFACE

This report was prepared by the Solar Energy Research Institute (SERI) under Task Nos. 1267.00 (FY 1982) and 1372.24 (FY 1983). Both conventional and nonconventional ethanol purification processes are reviewed. It is apparent that at least some of these nonconventional processes can compete with or suppplement distillation systems to favorably affect the overall economics of ethanol production. The results and conclusions presented in the report should be useful in evaluating and planning future separations research goals within SERI, the Department of Energy, and other institutions.

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SUMMARY

Objective

Several ethanol/water separation processes under past and present SERI-sponsored development are evaluated. Energy and capital-cost requirements for these processes are compared with the requirements of distillation systems. The potential for these processes to replace all or a portion of a distillation system is assessed.

Discussion

Distillation has been the conventional method of separating ethanol and water. An integrated two- to three-column vapor reuse distillation process requires approximately 18,000 Btu/gal to produce anhydrous alcohol from a 6 wt % feed. This is over 20% of the heating value (84,000 Btu/gal) of the ethanol product. Vacuum distillation without fully integrated heat recovery systems consumes even larger amounts of energy. For a given distillation process, the energy requirement can be reduced by decreasing the external reflux ratio, but the resulting increased tower capacity requires a trade-off between capital and operating costs to minimize total system costs. Several nonconventional separation processes have been investigated for their commercial potential. The Cantrell-Petrek Associates diffusion/carrier gas distillation process uses nitrogen gas at partial pressures sufficiently high that, in principle, no ethanol/water azeotrope is formed. Liquid extraction processes, developed by Intertechnology/Solar/Science Applications, Inc., and Georgia Tech, can offer energy savings since the amount of material that must be vaporized is small. The Intertechnology/Solar/Science Applications, Inc., process uses a light paraffin oil, while Georgia Tech's two-stage process employs 2-ethyl hexanol (EHOH) followed by ISOPAR-L. Adsorption processes of two distinct types are under consideration. In the Hydrocarbon Research, Inc., process, the ethanol/water mixture is first fed in the liquid phase to activated carbon columns and then to molecular sieves. In the Purdue University process, the mixture is vaporized and then adsorbed on either grain or calcium sulfate. Besides the distillation in the Purdue University process, energy is only required in these processes to regenerate the adsorption beds. Shock Hydrodynamics has formulated a synthetic polymer that selectively absorbs ethanol from ethanol/water mixtures. Southern Research Institute is developing ethanol-selective membranes that will separate liquid ethanol/water mixtures. A private German firm also uses membranes, but their mixtures are fed into the vapor phase into a process called pervaporation.

We tried to maintain as much consistency as possible when evaluating these processes. Capacities ranged from 1 gal/h to 27 million gal/yr; but with processes where the equipment and economics were completely defined, the capacities were about 25 million gal/yr. Input concentration of ethanol to each process must also be specified. If the experimental work and system designs used feed concentrations greater than the 10-15 wt % ethanol (which is commonly available in fermentation beers), an additional factor was added to account for the production of the specified concentration, starting from 10%. In some cases, detailed equipment specification and/or cost estimates were unavailable, and it was not reasonable to prepare them from poor data. Continuing work on some of the processes should alleviate this shortcoming.

Conclusions

Distillation is still the front runner in ethanol/water separation because of its long history of use. But such nonconventional processes as liquid extraction, vapor-phase adsorption, and membrane pervaporation can reduce or eliminate the need for distillation, both in terms of energy and cost. Further development, equipment design, and scale-up will be necessary to prove the commercial worth of these nondistillation processes for ethanol/water separation.

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SECTION 1.0

INTRODUCTION

Distillation of the ethanol formed in fermentation is a highly energy-intensive process that consumes between 25% and 50% of the heat content of anhydrous alcohol. Reducing the cost of energy is a key to improving the energy efficiency and cost-effectiveness of biomass-to-ethanol processes.

In recent years, the Department of Energy/Solar Energy Research Institute (DOE/SERI) Alcohol Fuels Program has supported several research contractors who have developed promising processes that address reduced energy and cost requirements. The processes included molecular sieve adsorption, solid/vapor adsorption, liquid/liquid extraction, and vacuum distillation. Energy balance was such an important part of cost-effective ethanol production that it was appropriate to expand this area of the Alcohol Fuels Program. Consequently, a solicitation was issued requesting energy-efficient ethanol separation processes. As a result of this solicitation, five additional approaches (and subcontractors) were selected to improve ethanol enrichment. These contractors and approaches were

- Cantrell-Petrek Associates—diffusion/carrier gas distillation
- InterTechnology/Solar/Science Applications, Inc.—temperature-dependent liquid extraction
- Hydrocarbon Research, Inc.—selective adsorption
- Shock Hydrodynamics—selective absorption
- Southern Research Institute—ethanol-selective membranes.

The effectiveness of these separation approaches will be assessed and compared with standard distillation in Section 3.0.

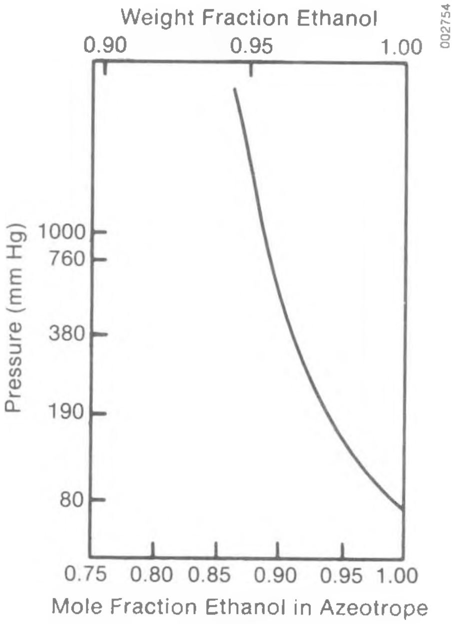
The objective of the program is to select several advanced ethanol/water separation processes to put on a fast-track—to identify the approaches with technical and economic potential. The high potential processes will then be subjected to advanced bench-scale evaluation. The selection of technically sound and economically feasible processes would, therefore, come from a broad pool of conceptual ideas. The energy efficiency and capital cost of each process will be evaluated. By 1984, the program hopes for 35% energy reduction; 1987 projections call for 70% energy reduction with a cost reduction of 50%. The purpose of this fast-track approach is to advance these target dates by one and two years, respectively.

The purpose of this report is to evaluate these five novel separation processes. The degree of detail included depends on the quality of information available from each subcontractor. The report will, where possible, present process flowsheets, capital and operating cost summaries, and energy requirements. For comparison, the state-of-the-art in conventional and vacuum distillation will first be outlined.

SECTION 2.0

DISTILLATION

The use of a single distillation column operated at atmospheric pressure limits the distillate ethanol concentration to 95.6 wt %, the concentration where the azeotrope boils (172.7°F). Figure 2-1 shows that the azeotrope remains until very low pressures (approximately 0.1 atm) are reached. Whether the presence of 4.4% water can be tolerated depends on the end uses of the ethanol. If the ethanol will be blended with gasoline, for example, it must be dehydrated to about 0.25% water. Whatever the end use or desired product concentration, it is useful to examine the distillation process in two steps: concentration up to the production of 95% (or less) ethanol, and then the dehydration of the azeotrope to 99% and above.



SOURCE: Black 1980.

Figure 2-1. Azeotropic Composition for Ethanol and Water

2.1 CONCENTRATION UP TO THE AZEOTROPE

Conventional distillation equipment includes a multiple-tray or packed column, plus a reboiler and reflux condenser. The system under consideration here incorporates some modifications as discussed by Lawrence Berkeley Laboratory (LBL) (Maiorella et al. 1982). As shown in Figure 2-2, separation is accomplished with two columns rather than one. This enables all of the corrosive organic acids in the fermentation beer to be removed in the small, stainless steel beer stripper and associated reboiler. The 95%-alcohol column (the larger column), its condenser, and the intercolumn exchanger can then be constructed of carbon steel. The resultant cost savings are significant, based on equipment cost ratios (stainless steel equipment cost/ carbon steel equipment cost) of 1.6 for the reboiler and 2.0 for the column. The intercolumn exchanger acts as the reboiler for the 95%-alcohol column, while condensing the stripper vapor to saturated liquid. The 50-psig steam to the stripper reboiler thus constitutes the entire energy input for the process. The LBL team developed computer programs for vapor-liquid equilibrium (VLE) data, equipment design, and cost estimation; the team then tried to optimize the system by varying the 95%-alcohol column pressure. They determined that the capital cost would be minimized with a column pressure of 170 mm Hg (0.224 atm). Purchased equipment costs and operating costs are summarized in Tables 2-1 and 2-2, respectively; costs are related to production rate in Figure 2-3. Note that this optimized system is designed to minimize costs, so that the steam loading of 12,010 Btu/gal is not the minimum energy requirement.

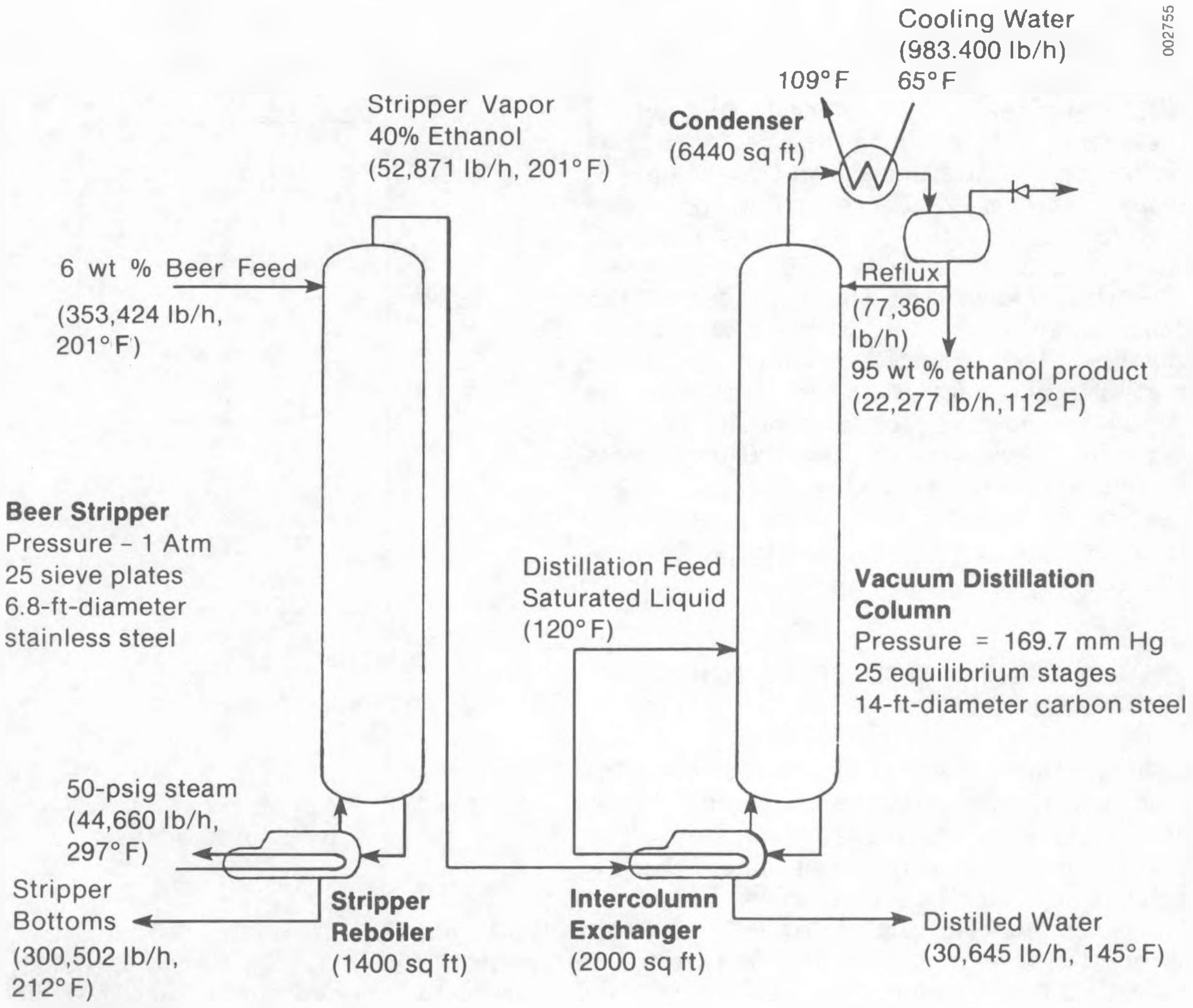


Figure 2-2. **Optimized Base Case Design for Two-Column Distillation**
(6 wt % feed to 95 wt % product)

In lieu of the LBL computer programs, SERI could further analyze the two-column system only when both the stripper and the 95%-alcohol column were operated at 760 mm Hg. As in the LBL analysis, ethanol recovery was assumed to be 100%; that is, the bottom product of both columns contains no alcohol. Specifications of the feed and bottoms concentration, along with the operating pressures, also fix the temperatures along each column. The objective of this portion of the analysis was to determine the energy requirements for the range of product concentrations below the azeotrope. It may eventually be more economical to distill the fermenter beer to an intermediate concentration, and then use one of the newer processes discussed in Section 3.0.

Table 2-1. Purchased Equipment Summary (25 million gal/yr, 6 wt % ethanol feed, 95% ethanol product)

Equipment	Purchased Cost
Stripping Column 25 Sieve trays 6.8-ft. diameter, 54 ft tall stainless steel	$105,500
Stripper Reboiler 1400 sq ft stainless steel	36,600
Distillation Column 25.2 Stages (Raschig ring packing) 14-ft diameter, 35 ft tall carbon steel	136,700
Intercolumn exchanger 2,000 sq ft carbon steel	27,900
Condenser 6,440 sq ft carbon steel	53,200
Total Purchased Equipment Cost	$359,900
Fixed Capital Investment (4.13 x Purchased Cost)	$1,486,400
Total Capital Investment (4.89 x Purchased Cost)	$1,759,900

Table 2-2. Operating Cost Summary (25 million gal/yr, 6 wt % ethanol feed, 95% ethanol product)

	Basis	Charge (¢/gal)
Direct Costs		
Labor and supervision	1.81 workers/shift	0.681
Utilities		
Steam [50 psig ($3.15/1000 lb)]	13.4 lb/gal	4.221
Cooling water [65° C (23¢/100 gal)]	29.5 lb/gal	0.812
Maintenance	6% Fixed capital	0.337
		6.042
Fixed Costs		
Depreciation	18 year, straight line	0.314
Local taxes	3% Fixed capital	0.170
Insurance	1% Fixed capital	0.057
		0.541
Gross Profit		
Net profit	15% Simple return on investment	0.844
Income tax	50% Rate	0.844
		1.688
	Total Charges	8.271

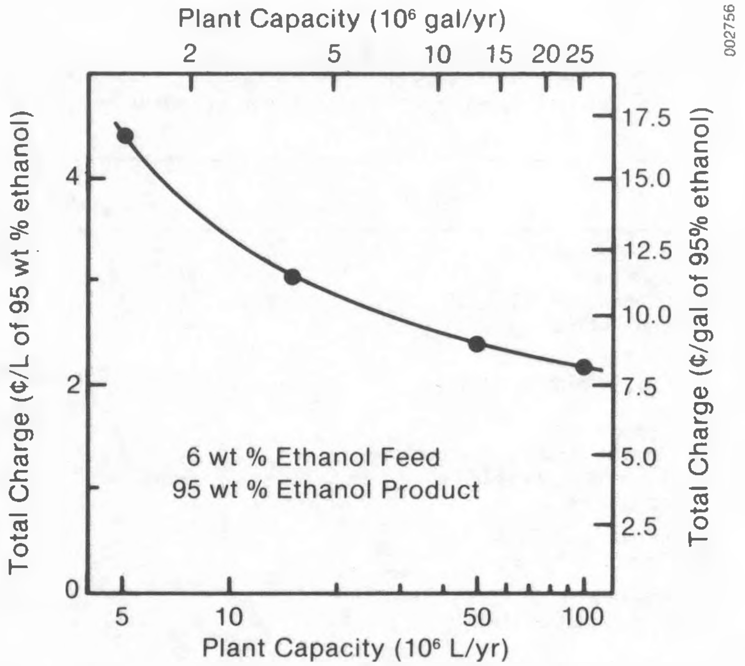
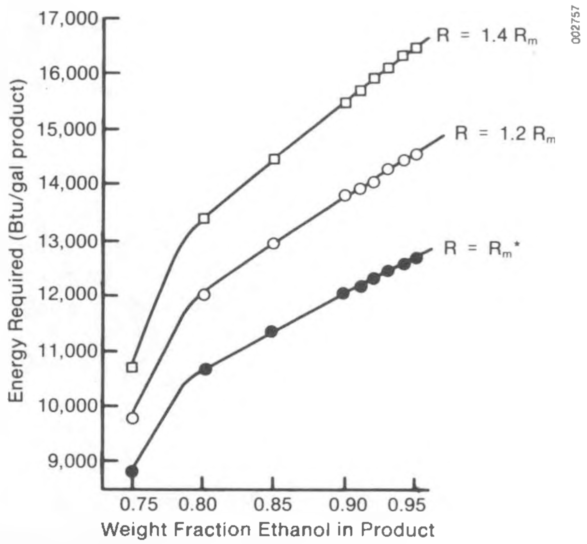


Figure 2-3. Effect of Plant Scale on Distillation Cost

The trade-off between capital costs and energy costs is better understood from Figures 2-4 and 2-5. In the former, the energy required to produce one gallon of the specified concentrations is plotted for various reflux ratios (R). The minimum reflux ratio R_m is the ratio at which an infinite number of trays is required to achieve the separation. Extensive operating experience has shown that the optimum reflux ratio commonly occurs between 1.15 and 1.5 R_m. Figure 2-4 shows that for a specified reflux ratio, more energy will be required to form a more concentrated product.

Compare the LBL value of 12,010 Btu/gal of 95% ethanol with the higher values shown in Figure 2-4; the temperatures of the 95% column are 60°F lower for vacuum operation than for atmospheric. Figure 2-5 shows the number of theoretical plates required to produce the specified product concentrations. First, the minimum number of plates N_m (at total or infinite reflux) was stepped off from the VLE curve at each product concentration. The required number of trays at various (finite) reflux ratios was then determined using the Erbar-Maddox correlation, which is an updated version of the Gilliland correlation (Perry 1973). Increasing the reflux ratio increases the energy requirement, but also decreases the number of plates required for the separation. At $R = 1.4\ R_m$, approximately 1.8 N_m plates are required while at $R = 1.2\ R_m$, more than 2.3 N_m plates are required. The LBL vacuum column is operated at $R = 3.47$, or about 1.5 R_m, and 25 theoretical plates are required to produce 95% ethanol.

Thus, the number of theoretical plates is primarily an indicator of equipment cost and, to some degree, operating cost. (Taller towers require more liquid handling capacity but lower reflux ratios.) Similarly, the reflux ratio contributes first to operating cost (steam requirement) and then to equipment cost (as larger reflux ratios necessitate larger reboilers but smaller towers). Figures 2-4 and 2-5 are instructive in quantifying these relationships, although finding the cost minimum for a particular system requires more detailed design on the columns, as LBL has done.



*Number of Theoretical Plates (N_t) = ∞.

Figure 2-4. Energy Requirement vs. Product Concentration at Various Reflux Ratios
(6 wt % feed, 760 mm Hg)

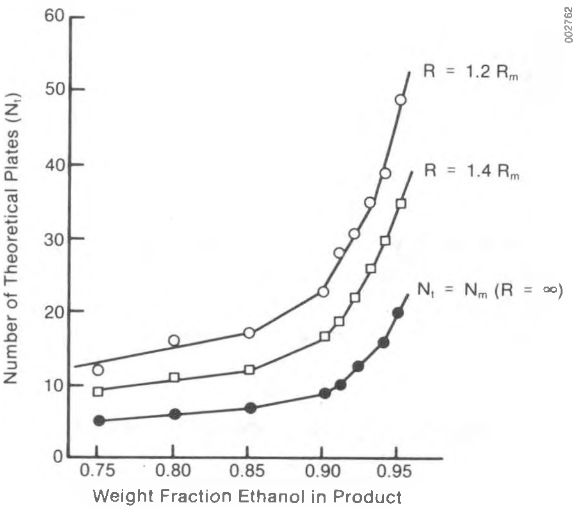


Figure 2-5. Number of Theoretical Plates vs. Product Concentration at Various Reflex Ratios
(6 wt % feed, 760 mm Hg)

2.2 DEHYDRATION OF THE AZEOTROPE

As discussed in detail by Black (1980), anhydrous ethanol can be produced by distillation in one of three methods. According to Figure 2-1, operation of a distillation column below about 86 mm Hg would produce anhydrous ethanol directly, since the minimum boiling azeotrope no longer forms. Based on rigorous tray-to-tray analysis, however, Black concluded that equipment and energy costs were prohibitive. Using a 20-tray column to produce a 92.5 wt % distillate, which was in turn fed to a 65-mm Hg vacuum column, Black found that even with an exorbitant reflux ratio of 9.7 (about 6 R_m), a 60-tray column produced no better than 98% ethanol. Further, this incomplete dehydration consumed over 15,300 Btu/gal. This value, as well as the accompanying value of 22,000 Btu/gal needed to produce 92.5% ethanol from a 6.4% feed (in Figure 2-4), would certainly drop substantially for lower reflux ratios. But if the desired product concentration cannot be provided with the extremely high reflux and number of trays, other processes should be investigated.

Since it is impractical to completely dehydrate binary ethanol/water, even under vacuum, the other two alternatives use ternary or pseudo-ternary systems. In azeotropic distillation, a third component is chosen that enhances the volatility of water while inhibiting that of ethanol. This component, called an entrainer, can be pentane, benzene, or diethyl ether. When the entrainer is fed to the second column along with the 92.5%

ethanol, it carries off the water as the overhead product, leaving anhydrous ethanol as the bottom product of this column. A small stripper is then required to recover the entrainer from the overhead. Black found that the 19-tray azeotropic distillation column and the 10-tray stripper consumed only 9700 Btu/gal when producing ethanol with only 0.001% water, suitable for industrial or gasohol use. Note that LBL chose diethyl ether as the entrainer (see Section 2.3).

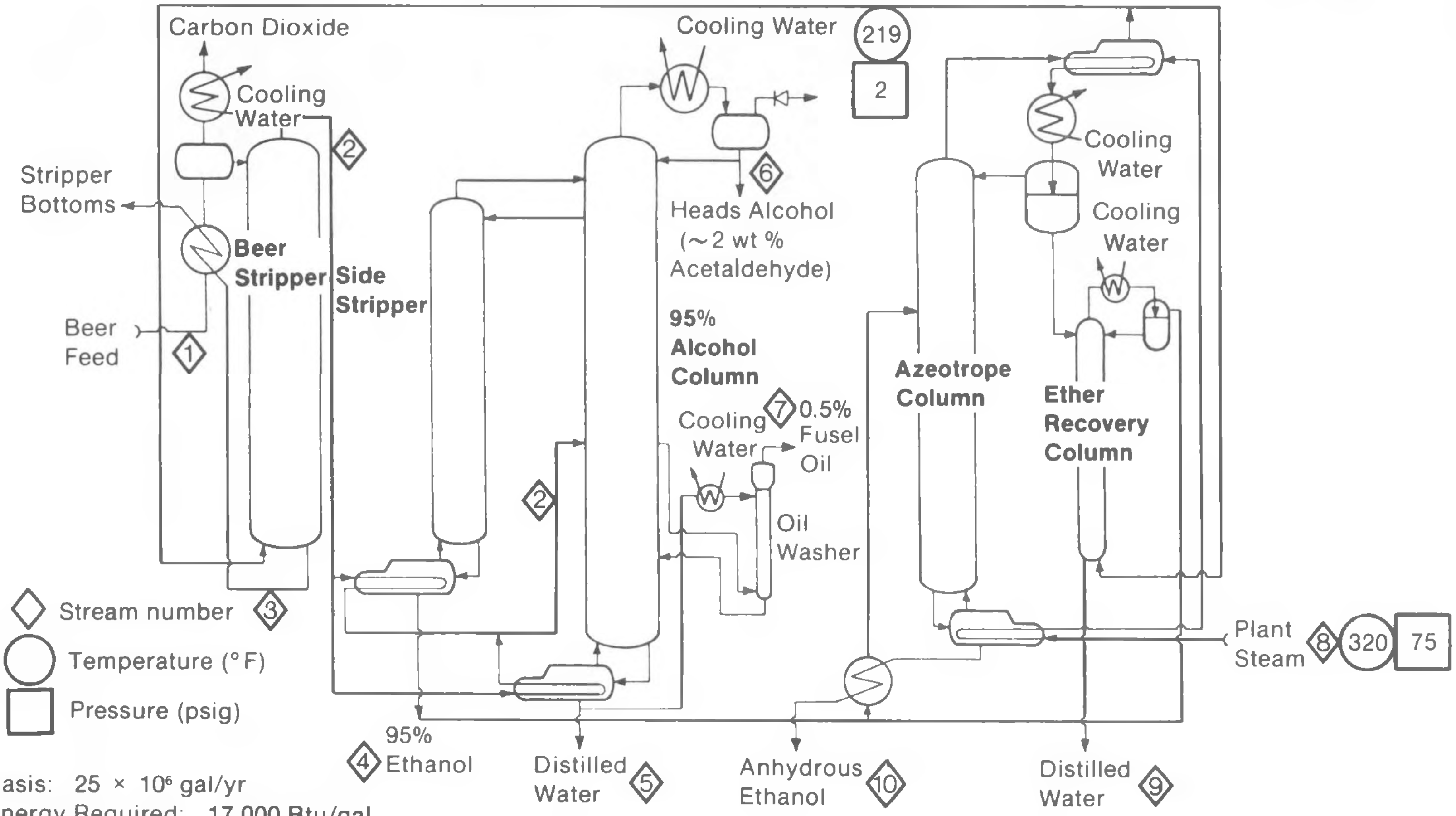
The third process for production of anhydrous ethanol is termed extractive distillation. Depending upon the solvent, ethanol may be removed as an overhead vapor (as when ethylene glycol is used) or as a bottom product. Using a light gasoline as the solvent, resulting in a pseudo-ternary system, Black determined that 7800 Btu/gal was required to produce gasohol containing 35% ethanol and no water. This process is limited to gasohol production, however; industrial alcohol production is not feasible.

2.3 VAPOR REUSE DISTILLATION USING DIETHYL ETHER

The integrated process for anhydrous ethanol production from fermenter beer is illustrated in Figure 2-6. This process is designed to deliver 25 million gallons per year of anhydrous ethanol operating 8000 hours per year. The atmospheric pressure stripper has 25 sieve plates and a 6.8 ft diameter. It is constructed of stainless steel to prevent corrosion by the organic acids in the beer. This allows the rest of the system to be made of carbon steel. The vacuum (170 mm Hg) distillation unit has a 14 ft diameter and is packed with 2-in. Raschig rings to affect 25 equilibrium stages. The azeotrope column has 30 trays, operates at 130 psig, and has an atmospheric pressure ether recovery column with 20 stages.

Diethyl ether azeotropic distillation has one distinct advantage over benzene and heptane: a ternary azeotrope does not form, and all of the ethanol is recoverd at the bottom of the azeotrope column. The two-column vacuum distillation system incorporates fusel oil and acetaldehyde separation. The vapor reuse methods are extended so that the azeotrope column condenser generates steam for the beer stripper. Dilute beer is degassed and fed to the stripper to give a concentrated (40 wt %) ethanol vapor for rectification and a bottom stillage product essentially exhausted of ethanol (0.03 wt %). The stripper vapor is condensed in the reboiler of the main vacuum distillation column and its side stripper. This concentrated liquid is then fed to the vacuum column. Pure water is removed at the bottoms. A small side draw low in the rectifying section is cooled to separate and remove fusel oils (a mixture of amyl and propyl alcohols and their isomers). At the column head, a small stream of technical grade alcohol (contaminated with roughly 2 wt % acetaldehyde) is removed. A large side draw, which is a few plates below the head, feeds the side stripper. The 95 wt % ethanol bottom product from the side stripper is fed to the azeotropic column. Anhydrous ethanol is produced as the bottom product, and the constant boiling mixture of diethyl ether and water (4.4 wt %) is condensed and refluxed at the column head. A portion of this is cooled, and an aqueous layer is separated. The aqueous layer, saturated with traces of diethyl ether, is fed to a very small column to strip the ether and produce a pure water bottoms product.

The path of heat flow in this system is as important as the mass flow. Plant steam at 75 psig (320°F) is fed to the azeotropic column reboiler and boils up pure ethanol. The azeotropic column distillate (303°F) is condensed boiling up steam (2 psig, 218°F) for the main beer stripper and the ether recovery column. Vapors from the beer stripper are condensed in the main vacuum column reboiler and its side stripper ethanol reboiler.



Basis: 25 × 10⁶ gal/yr
Energy Required: 17,000 Btu/gal

lb/h	1	2	3	4	5	6	7	8	9	10
Ethanol	20,654	20,562	92	20,562	—	1,028	—	—	—	20,562
Water	323,579	30,843	292,736	946	29,897	—	—	57,180	946	—
Acetaldehyde	—	—	—	—	—	21	—	—	—	—
Fusel Oil	—	—	—	—	—	—	103	—	—	—
Total	344,233	51,405	292,828	21,508	29,897	1,049	103	57,180	946	20,562

Figure 2-6. Integrated Vapor Reuse Process for Anhydrous Ethanol Production

Finally, the vacuum column vapors (104°F) are condensed with cooling water. The original steam heat is thus used in three successive operations.

The large reflux (and hence steam) requirement of the diethyl ether system compared to other entrainers is not a disadvantage. The steam flow rate to the azeotropic column was chosen to balance the steam requirements of the successive distillation operations. The number of plates in the azeotropic column was chosen to give the required separation with the available reflux. In a modern plant that has reduced steam requirements to produce 95 wt % ethanol, trays should be added to the azeotropic column to reduce its steam consumption and maintain balanced operation.

The selling price of anhydrous ethanol is 10¢-12¢/gal higher than 95% ethanol. This is greater than the entire production cost for 95% ethanol from fermenter beer (see Table 2-2). Thus, the most common method of producing anhydrous ethanol from fermenter beer costs twice as much as producing 95% ethanol. The potential for cost and energy savings is greatest at the highest concentrations of ethanol. Some of the nonconventional ethanol purification processes try to spread the savings over the entire range of concentrations, while others include a distillation step, placing all the capital and energy cost savings in the upper concentration range.

SECTION 3.0

ALTERNATIVES TO DISTILLATION

3.1 DIFFUSION/CARRIER GAS SYSTEM

The diffusion/carrier gas system is shown in Figure 3-1. The system was designed to deliver 99.5% ethanol from a 10 wt % ethanol/water solution using approximately 6500 Btu/gal of product based on diffusion of the ethanol into the carrier gas (nitrogen) at low pressures (about 1.5 psia). Large-scale equipment specifications were not defined, but the laboratory apparatus was built and operated. The lab-scale version has the humidifier, evaporator, and partial condenser stacked one on top of the other to operate in unison. This results in essentially a 1-plate still with a partial condenser. Even in the presence of nitrogen, the process is governed by the ethanol/water, vapor/liquid equilibrium. Cantrell-Petrek Associates contend that the process is operated under sufficient vacuum so that an azeotrope is not formed, but no such evidence is presented.

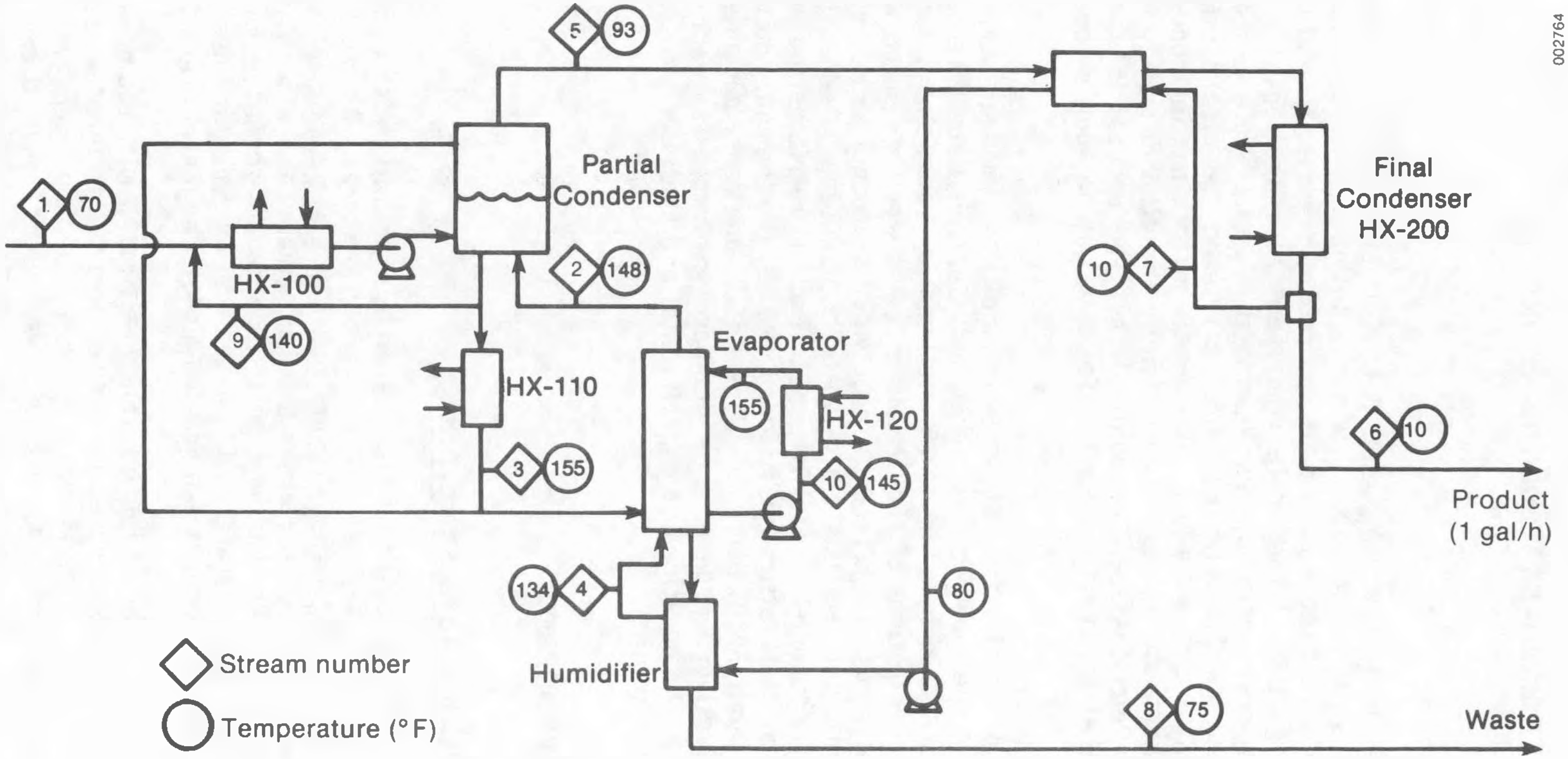
With regard to the lab data, no greater than 85 wt % ethanol was produced during the contract period. In addition to the shortcomings mentioned, Cantrell-Petrek Associates used a denatured alcohol feed that included up to 7% methanol by volume as a denaturant. Further, the ethanol concentrations of the various streams were measured with a hydrometer, which provides no means to differentiate between methanol and ethanol in the product. The energy requirement was 6000 Btu/gal as thermal energy added to the evaporator and about 1300 Btu/gal as electrical energy to run the refrigeration unit in the final condenser. With a heat-to-electricity efficiency of 0.33, the thermal equivalent of the electrical energy became 3900 Btu/gal. This total energy requirement of 9900 Btu/gal for an 80%-85% ethanol product does compare somewhat favorably to a standard distillation. However, the questionable experimental and analytical techniques discourage further pursuit of this project.

3.2 SOLVENT EXTRACTION PROCESSES

3.2.1 InterTechnology/Solar/Science Applications, Inc.

This solvent extraction process (Figure 3-2) uses the change in miscibility with temperature of ethanol in light paraffin oil to separate ethanol/water mixtures. Above 158°F, ethanol and paraffin oil form a homogeneous organic phase while the higher density water settles out. Subsequent cooling of the organic phase enables the ethanol to be removed. The ethanol product is greater than 99% pure with respect to water but contains 4.5% paraffin oil by weight. Since paraffin oil is in the fuel oil range and has a fuel value about twice that of ethanol, this product can be used directly as motor grade fuel.

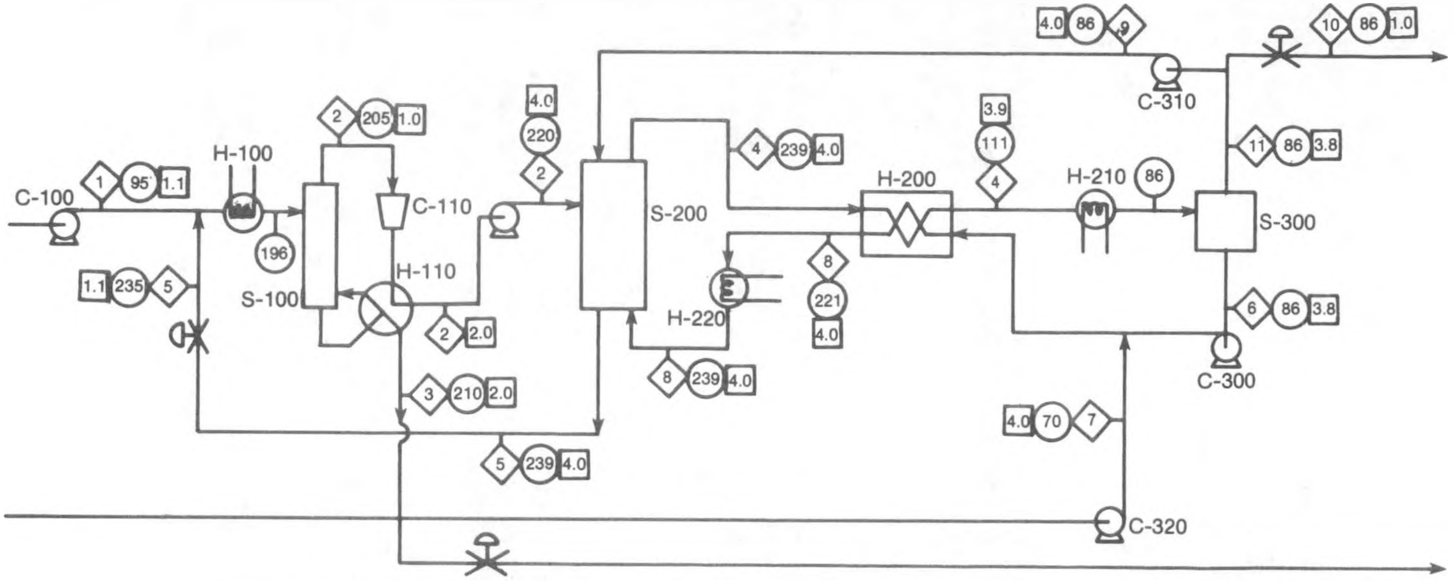
The ethanol feed concentrations investigated ranged from 20-45 wt %, with the base case at 30 wt %. A beer still was needed to remove any sludge from the fermenter effluent and concentrate it up to 30 wt %. A vapor recompressor and heat recovery boiler decreased the overall thermal energy requirement relative to the conventional fired reboiler. The beer still and compressor consumed about 7200 Btu/gal of product while removing about 70% of the water feed. The total energy required to concentrate a 10 wt % solution to essentially anhydrous ethanol is about 9550 Btu/gal. These energy



lb/hr	1	2	3	4	5	6	7	8	9	10
Ethanol	6.90	7.62	6.90	—	6.85	6.55	0.30	0.30	5.80	2.74
Water	61.67	7.04	61.67	3.73	0.03	0.03	—	61.69	52.27	544.62
Nitrogen	—	29.10	—	29.10	29.10	—	29.10	—	—	—
Total	68.57	43.76	68.57	32.83	35.98	6.58	29.40	61.99	58.07	547.36

HX-	Btu/h
200	-2,991
100	-3,290
110	+987
120	+5,474

Figure 3-1. Diffusion/Carrier Gas System



Basis: 5 × 10^6 gal/yr product

lb/h	1	2	3	4	5	6	7	8	9	10
Ethanol	4,335	6,996	426	14,059	3,093	5,469	—	5,458	4,699	3,963
Water	39,000	16,226	38,311	827	15,531	47	—	99	40	34
Extractant	—	99	—	150,514	99	150,784	281	150,282	223	188
Total	43,335	23,321	38,737	165,400	18,723	156,300	281	155,840	4,962	4,185

S-100 = Beer Still (with vapor recompression)
S-200 = Extraction column
S-300 = Gravity phase separator

◇ Stream number

○ Temperature (°F)

□ Pressure (atm)

Unit	10^6 Btu/h	Btu/gal	
H-100	1.70	2,650	
C-110*	2.92	4,550	(13,650)**
H-220	1.51	2,350	
		9,550	(18,650)** Total

* $\eta_{compressor} = 0.80$

** when $\eta = 0.33$ for converting heat to electricity is included.

Figure 3-2. Solvent Extraction System
(Based on ITC/SAI process)

requirements are based on 100% conversion efficiency from thermal to electrical energy; if the more realistic figure of 33% is used, the energy requirement is 18,650 Btu/gal.

The design of the continuous extractor was based on very scattered lab data taken from a batch system. Since the design of the extraction system is empirical, this data must be refined before a reasonable design can be performed. The major trade-off would be the cost of the extractor versus the energy necessary to bring the fermenter product up to an acceptable concentration. The extractor performance should be more precisely defined and the range of feed concentrations extended down to 10 wt % ethanol so that this capital versus energy trade-off can be accurately assessed. The absence of generalized correlations also makes the design of the full-sized extractor from lab data less straightforward than stills or absorbers.

3.2.2 Georgia Institute of Technology

In the first two years of research, experimental data suggested that substances that exhibited high ethanol distribution coefficients had low ethanol/water selectivities. (The distribution coefficient is the ratio of the weight fraction of ethanol in the solvent phase to its weight fraction in the aqueous phase. The selectivity is the ratio of the ethanol/water ratio in the solvent phase to the ethanol/water ratio in the aqueous phase.) Therefore, the present system was based on a two-cycle recovery concept in which a solvent like 2-ethyl hexanol (EHOH) was used first to recover a 50-80 wt % product. This intermediate product was then fed to a drying cycle that contains a more selective solvent like ISOPAR-L (a branched paraffinic refinery cut). A simplified process flow diagram is shown in Figure 3-3. Heat exchangers are not shown but the major energy requirement to heat stream 6 is about 5,000 Btu/gal.

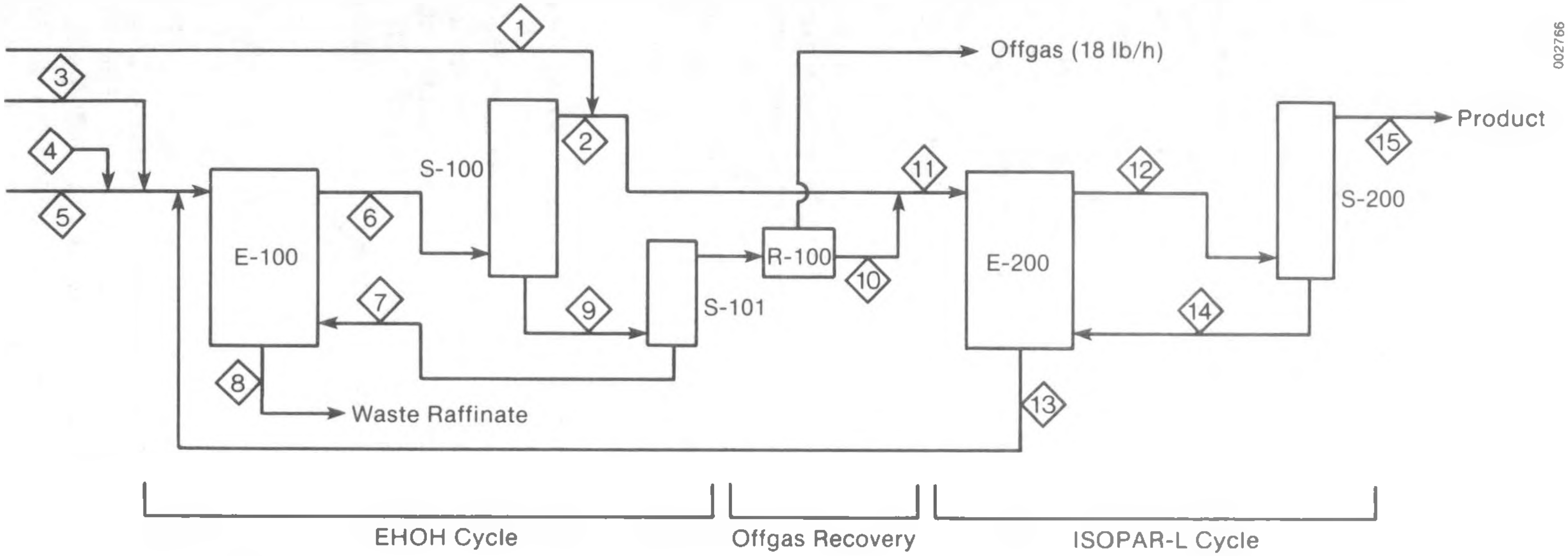
At the front end of this process is a continuous vacuum (55 mm Hg) fermentation with cell recycle, based on a process used by Lawrence Berkeley Laboratory (Cysewski and Wilke 1978). Operation of the fermenter under vacuum enables most of the product to be withdrawn as vapor, with an ethanol content as high as 40%. The actual fermentation beer, stream 5, is 8% ethanol after centrifugation; this would be the only product of a conventional fermentation. Instead, the feed to the Georgia Tech solvent extraction process is 26 wt % ethanol.

Georgia Tech reports that 12,600 Btu/gal is needed to produce 98 wt % ethanol from these feeds. This number includes 876 Btu/gal as electrical energy which has a thermal equivalent of 2600 Btu/gal. To produce 26% ethanol from a more commonly available feed (about 10%), an additional 5000 Btu/gal or more would be required. The installed equipment costs were estimated at $3.7 million to produce 27 million gal/yr. The degree to which energy savings of the solvent extraction process might be offset by increased capital and operating costs of the vacuum fermentation process should be investigated in the coming year.

3.3 ADSORPTION/ABSORPTION PROCESSES

3.3.1 Hydrocarbon Research, Inc.

This process was designed to concentrate 15 wt % ethanol in water up to 70 wt % ethanol, followed by dehydration to 99.9 wt %. In the first step, ethanol was concentrated



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Equipment:
E - Extractors
S - Strippers
R - Refrigeration Units
(Heat exchangers omitted for clarity)

Flow Rates (10^3 lb/h) for 27 × 10^6 gal/yr plant

	F	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Ethanol	22.1	13.4	12.2	2.4	4.4	1.9	19.0	0.2	0.7	6.8	6.1	32.2	23.0	10.5	1.3	21.7
Water	62.5	19.4	8.1	4.3	17.5	21.3	12.7	62.3	0.5	4.6	4.2	32.3	0.5	32.0	0.2	0.3
Sugar	1.4	—	—	—	—	1.4	—	—	—	—	—	—	—	—	—	—
EHOH	—	—	—	—	—	—	501.0	—	501.0	501.0	—	—	—	—	—	—
ISOPAR-L	—	—	—	—	—	—	—	—	—	—	—	—	696.0	—	696.0	—
Total	86.0	32.8	20.3	6.7	21.9	24.6	534.0	62.5	502.5	512.4	10.3	64.5	719.5	42.5	697.5	22.0

F = Composite Feed (streams 1, 3, 4, 5).

Figure 3-3. Simplified Two-Stage Solvent Extraction Process
(Based on Georgia Tech system)

from dilute solutions by preferential adsorption on carbon. Then, after a vapor mixture further enriched in ethanol was produced by desorption from the carbon column, dehydration was completed in a molecular sieve column. Ethanol was recovered from the dried vapor by condensation. Figure 3-4 is a simplified outline of stream flows and regeneration energy requirements, while Figure 3-5 shows all the equipment and the other details of the system.

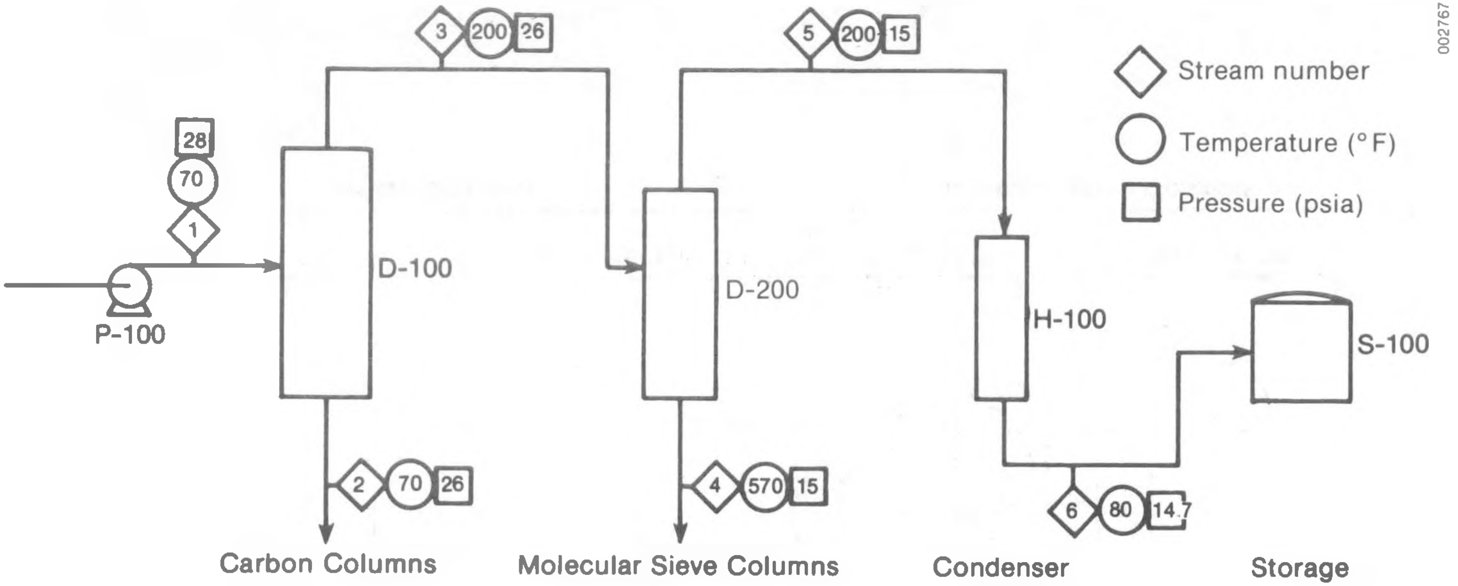
Dilute ethanol solution was pumped through a carbon column until ethanol appeared in the effluent. The column contents drained back into the feed tank so that interstitial solution could be removed. The column was heated by a steam coil and purged with carbon dioxide to effect desorption and vaporization of an ethanol/water mixture. The vapors passed to a molecular sieve column as long as the ethanol content was relatively high. When desorption from a second carbon column began, the lower-grade vapors leaving the first column are routed to the second column. After ethanol was exhausted, the first carbon column was cooled in preparation for a new adsorption cycle. In the molecular sieve column, water was adsorbed, and dry ethanol vapor was passed through. Dry ethanol was separated from the vapor stream in a condenser, and carbon dioxide (saturated with ethanol) was recycled to the carbon column desorption step. When a molecular sieve column becomes saturated with water, the ethanol/water vapor stream was switched to a second column. The first molecular sieve column was regenerated by purging with hot air until its water content fell to a predetermined level. The column was cooled by purging with ambient air in preparation for a new adsorption cycle.

A cycle time of eight hours for each carbon column and six hours for each molecular sieve column was chosen. Operation in the carbon column was broken down to three hours adsorption, one hour draining, two hours desorption (one hour each for first and second periods), and two hours cooling. Operation in the molecular sieve column included one hour adsorption, four hours regeneration, and one hour cooling. To meet the design capacity of the plant, 24 carbon columns and 12 molecular sieve columns are needed. They are grouped into three eight-carbon-column trains and two six-molecular-sieve-column trains so that each train matches the corresponding cycle time, permitting continuous, cyclic operation.

Regeneration of the adsorbents requires 12,400 Btu/gal as thermal energy and 4830 Btu/gal as electrical energy to run the regeneration gas compressors. If a heat-to-electricity efficiency of 0.33 is used to adjust the electrical requirements, the necessary thermal energy jumps to around 25,000 Btu/gal. The equipment costs are outlined in Table 3-1 including the adsorbent costs, which come to almost one quarter of the total equipment costs. Even if this system could be made more energy efficient, the improvements could probably not compensate for the higher capital costs when compared with standard distillation ($4 million vs. $1.6 million, respectively).

3.3.2 Purdue University

Purdue is investigating a process that uses grain as a dehydrating agent. (The work is being conducted for the USDA.) They have produced anhydrous ethanol from 91 wt % ethanol using approximately 2000 Btu/gal for vapor feed and about 4500 Btu/gal for liquid feed. The unit holds 40 lb of grain and currently produces 10,000 gal/yr; future outputs should reach 40,000 gal/yr. Operation is simple, equipment is cheap, and regeneration can be accomplished using less than 200°F air. The grain has gone through 85 cycles in 3 months and is still functioning up to specifications.



lb/h	1	2	3	4	5	6
Ethanol	20,655	—	20,655	—	20,655	20,655
Water	117,045	108,193	8,852	8,831	21	21
Total	137,700	108,193	29,508	8,831	20,676	20,676

Column Regeneration Thermal Energy

D-100	27.550 MBtu/h	Carbon Columns
D-200	11.631 MBtu/h	Molecular Sieve Columns
Total	39.181 MBtu/h	= 12,400 Btu/gal

Design Basis: 25 × 10^6 gal/yr 99.9% ethanol, 330 days/yr, 24 h/day

Figure 3-4. Hydrocarbon Research, Inc., Adsorption Process—An Overview

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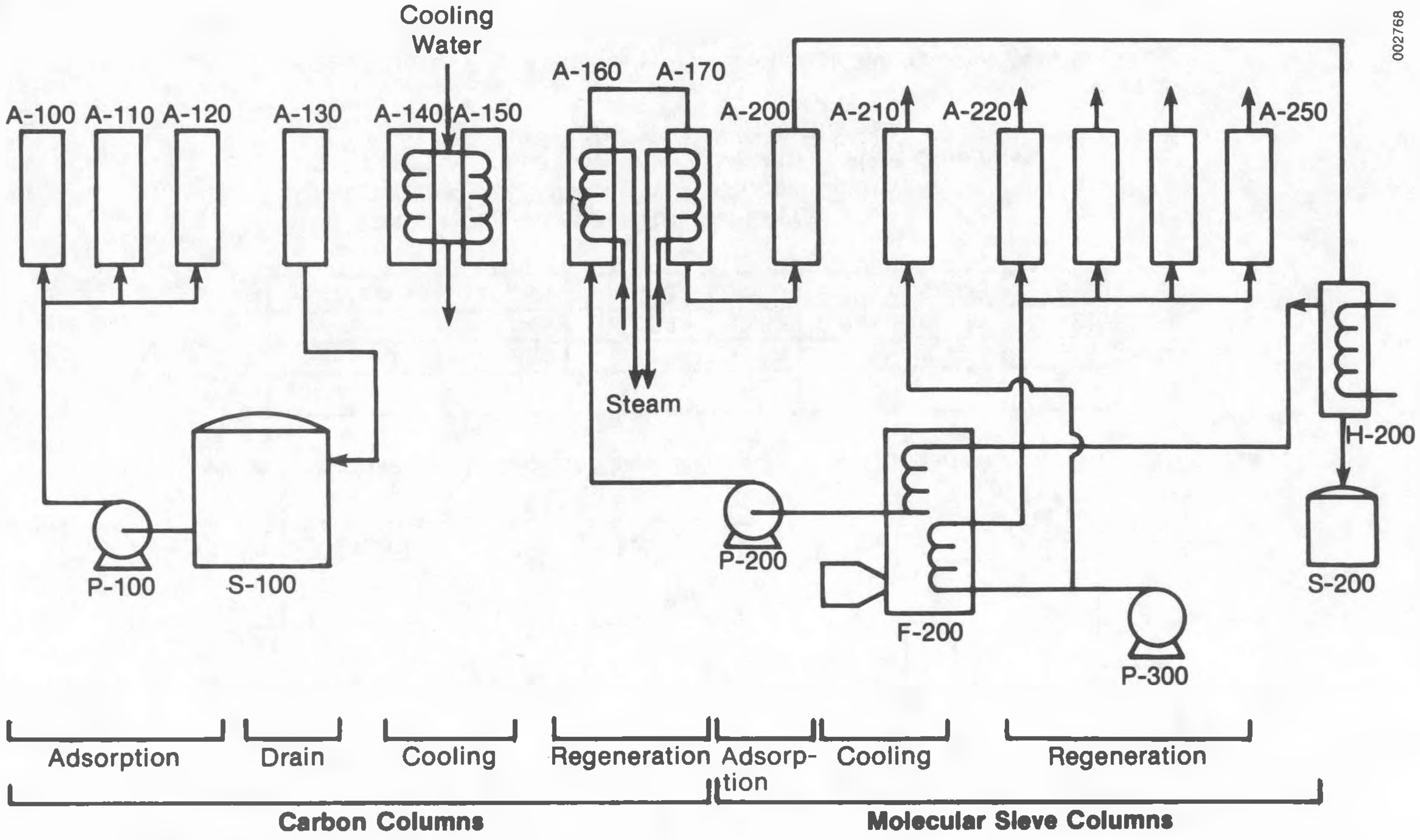


A — Adsorption Columns
F — Furnace
H — Condenser
P — Compressors, Pumps
S — Storage Tanks

Figure 3-5. Hydrocarbon Research, Inc., Adsorption Process—Flow Diagram

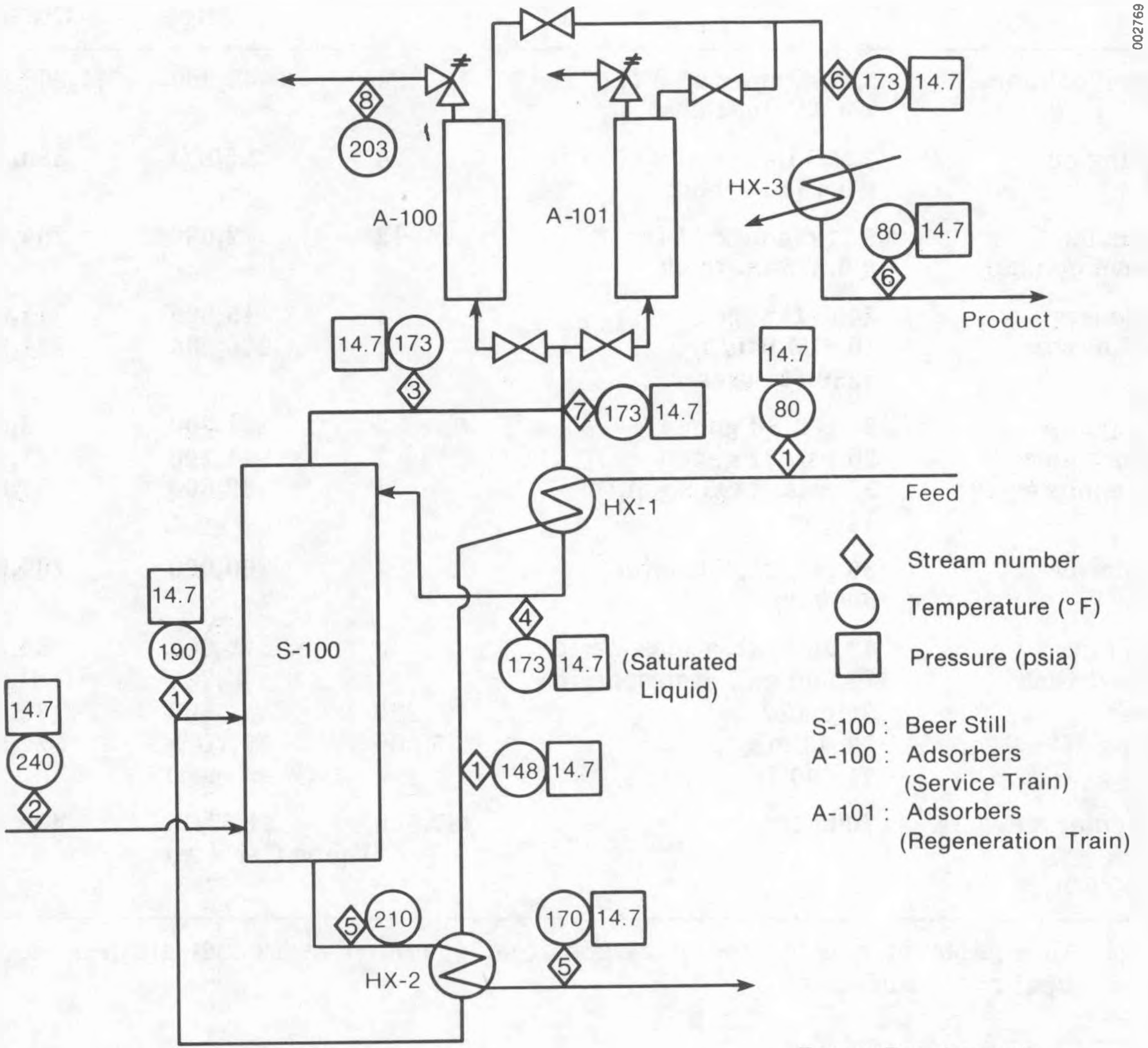
Table 3-1. Equipment Costs for the Hydrocarbon Research, Inc., Adsorption Process

Item	Specifications	Quantity	Unit Price	Total Cost
Carbon column	5 ft diam. x 56.6 ft x 0.375 in. thick	24	42,000	\$1,008,000
Heating coil	2.375 in. diam. x 9675 ft x 0.154 ft thick	24	2.50/ft	580,500
Molecular sieve column	5 ft diam. x 29 ft x 0.375 in. thick	12	22,000	264,000
Condenser	2400 ft^2 area	1	15,000	15,000
Fired heater	10.5 M Btu/h, 5250 ft^2 area	1	220,000	220,000
Feed pump	26 psi, 90 gpm	3	1,200	3,600
Product pump	20 psi, 32 gpm	1	1,200	1,200
CO_2 compressor	37 psia, 1496 scfm, 125 hp	1	32,000	32,000
Air blower	50 psi, 32,000 scfm, 5050 hp	1	700,000	700,000
Feed tank	43,200 gal, atmospheric	3	28,000	84,000
Product tank	76,000 gal, atmospheric	1	41,700	41,700
Valves	Solenoid	220	600	132,000
Nuchar WV-W	12-40 mesh, 21,000 ft^3	735,000	\$0.71/lb (Westvaco)	522,000
Molecular sieve 3A	7000 ft^3	287,000	\$1.50/lb (Union Carbide)	430,500
TOTAL				\$4,034,500

Notes: All equipment constructed of carbon steel. Fermenter product clean-up equipment not included.

The overall process is shown in Figure 3-6. Purdue used a packed distillation tower to produce 90% ethanol vapor from 10% feed. The distillate was fed directly to one of the grain filled adsorbers for dehydration. Water adsorption was accompanied by an increase in adsorber temperature from 180°F to 205°-210°F. The adsorber was usually removed when the increased temperature was measured about 75% of the distance downstream of the adsorber inlet. With 212°F air, the time required for regeneration was roughly the same as the service (adsorption) time.

Purdue has also used calcium sulfate as a dehydrating agent. Researchers have been able to concentrate 91-92 wt % ethanol to anhydrous ethanol while consuming 2000-3000 Btu/gal. The design is nearly identical to the grain system design but has gone through only 14-15 cycles to date while maintaining its activity. Purdue has overcome calcium sulfate degradation by increasing the temperature to 140°C (284°F) for the regeneration cycle.



Energy Requirements

Process Block	Btu/gal
Distillation	10,000
Adsorber Regeneration	2,000
Total	12,000

Basis: 10 × 10^6 gal/yr

lb/h	1	2	3	4	5	6	7	8
Ethanol	8,342	—	19,870	11,579	42	8,300	8,300	—
Water	75,081	10,931	2,208	1,287	85,099	—	922	922
Total	83,432	10,931	22,078	12,865	85,141	8,300	9,222	922

Figure 3-6. Purdue Grain Adsorption Process

Both these systems have great potential. Further laboratory data were obtained, and a more detailed engineering design is being performed. The reported energy requirements for this system were 12,000 Btu/gal, which is quite competitive with the other processes discussed.

3.3.3 Shock Hydrodynamics

The researchers at Shock Hydrodynamics studied a new and potentially more energy-efficient method for separating alcohol from fermentation beers. The approach proposed involves synthesis of imbibitive polymer beads that would selectively absorb rather than adsorb the organic component from an aqueous solution. The method had previously been demonstrated in absorbing acetone from a 6% water solution using a polymethyl acrylate polymer bead system. This system would absorb its own volume of acetone under these conditions. The same system would absorb 30% of its volume of ethanol from a 37.5% solution by volume (75 proof). Based on these preliminary results, they proposed that by modifying the base polymer system, a selective absorbent could be prepared that would extract ethanol from fermentation beers.

Thirty-three different polymer and copolymer formulations were prepared and evaluated during the course of this project. The first screening was accomplished by measuring (via photomicroscopy) the volume change in the polymer beads when equilibrated against either a 95% ethanol solution or distilled water. In the best case, the beads showed a preference for ethanol by swelling (imbibing) to 11 times their normal volume and involved only 8%-40% for water. When this system was subjected to dilute alcohol solutions, essentially no ethanol separated from water. These results indicate that alcohol imbibition was not a sufficient criterion for extraction capability, and further testing was done by measuring the partition or distribution coefficient K of ethanol between the polymer and water. The maximum K (at 22°C) was 0.26, a value too low to be useful in a practical separation system.

The researchers also tried to determine the capability of these polymer beads to absorb butanol from dilute (2%) aqueous solution. The K values ranged from about 1.5 to 3.1 as the temperature of the absorption system was changed from 25° to 60°C. There is good potential for using these beads in an extractive fermentation process, such as with butanol, to remove the product from the reaction mixture and increase productivity by limiting product imbibition. This application is being investigated.

While the approach proposed on this project is very interesting, very little progress was made during the year toward producing a feasible system for separating ethanol and water. Prospects are better for separating butanol and water; whether or not there are any cost advantages over membrane or liquid/liquid extraction separation techniques has not been determined.

3.4 MEMBRANE PROCESSES

3.4.1 Southern Research Institute

Southern Research Institute conducted a 12-month research project to develop ethanol-selective membranes for use in separating ethanol/water mixtures. The objective of the project was to fabricate a membrane that could separate a 50:50 wt % alcohol/water solution and produce a near anhydrous product. The general approach for producing a membrane was to form thin selective layers on low-resistance (high flux) substrates because polymers that exhibit selectivity generally have high resistance to flow (low fluxes) if they are thick enough to have acceptable mechanical strength. If the layer of selective polymers is made thin enough and is attached to a low-resistance substrate for mechanical strength, then it should be possible to combine high selectivity with low resistance and physical strength.

The overall objective was to develop an energy-efficient process that separated ethanol from water. If ethanol-selective membranes could be used in direct contact with fermentation beers, the energy required to separate ethanol from water would probably be minimized. But membranes of all kinds are subject to fouling, and there are materials in fermentation beers that will almost certainly foul membranes and reduce the transfer rate. A method of eliminating membrane fouling is to use the membranes to treat vapors, or liquids condensed from vapors, obtained from the fermentation beers. Because the theoretical number of plates and the energy input required to produce an overhead product containing 80 wt % ethanol are relatively low, SERI used this composition as a standard for establishing goals for the membrane process.

The cost of separating ethanol from water by a membrane process depends primarily on the separation factor, the composition of the material in contact with the high-pressure sides of the membranes (membrane feed), the throughput rate as a function of pressure drop, the operating lifetime of the membranes, and the installed cost of the membranes. The separation factor is

$$\frac{\text{wt \% ethanol/wt \% water (in permeate)}}{\text{wt \% ethanol/wt \% water (in feed)}} .$$

A comparison of feed composition versus permeate composition for different separation factors shows that with a separation factor less than 10, the desired composition of the permeate (99 wt % ethanol) cannot be obtained even with a feed composition of 80 wt % ethanol. Obviously, higher separation factors will give better results.

The membrane feed composition can be maintained in the desired range (73-80 wt % ethanol for a membrane with a separation factor of 10) by several methods. The simplest method is to mix fresh material from the distillation column (80 wt % ethanol) with recycled, depleted membrane feed (73 wt % ethanol). If the membrane feed is a liquid, the energy needed for recycling the depleted feed will be small because the pressure drop required to maintain the desired feed velocity past the high-pressure sides of the membrane is not expected to be high.

It was more difficult to establish goals for membrane lifetimes, costs, and throughput rates. However, if membranes were used that had throughput rates of 2 gal/ft^2/day, lifetimes of 3 years, and a cost of \$10/ft^2, the cost of membrane replacement would be less than \$0.006/gal of ethanol. Hollow-fiber reverse osmosis and ultrafiltration membranes cost even less and have lifetimes of 3 years or more. Throughput rates up to 15 gal/ft^2/day have been achieved with existing membranes in other applications. Therefore, the research goals were a minimum throughput rate of 2 gal/ft^2/day, a 3-year operating lifetime, and a maximum cost of \$10/ft^2 were established.

High separation factors and high throughput rates represent opposing goals. Generally in membrane-transfer processes, highly selective membranes offer high resistance to transfer. To combine high selectivity with low resistance to transfer, two-layer membranes were developed. A very thin layer of selective material was applied on the surface of a thicker substrate material that was characterized by low resistance to transfer and acceptable strength. The resistance of the selective layer can be made low if the layer is sufficiently thin.

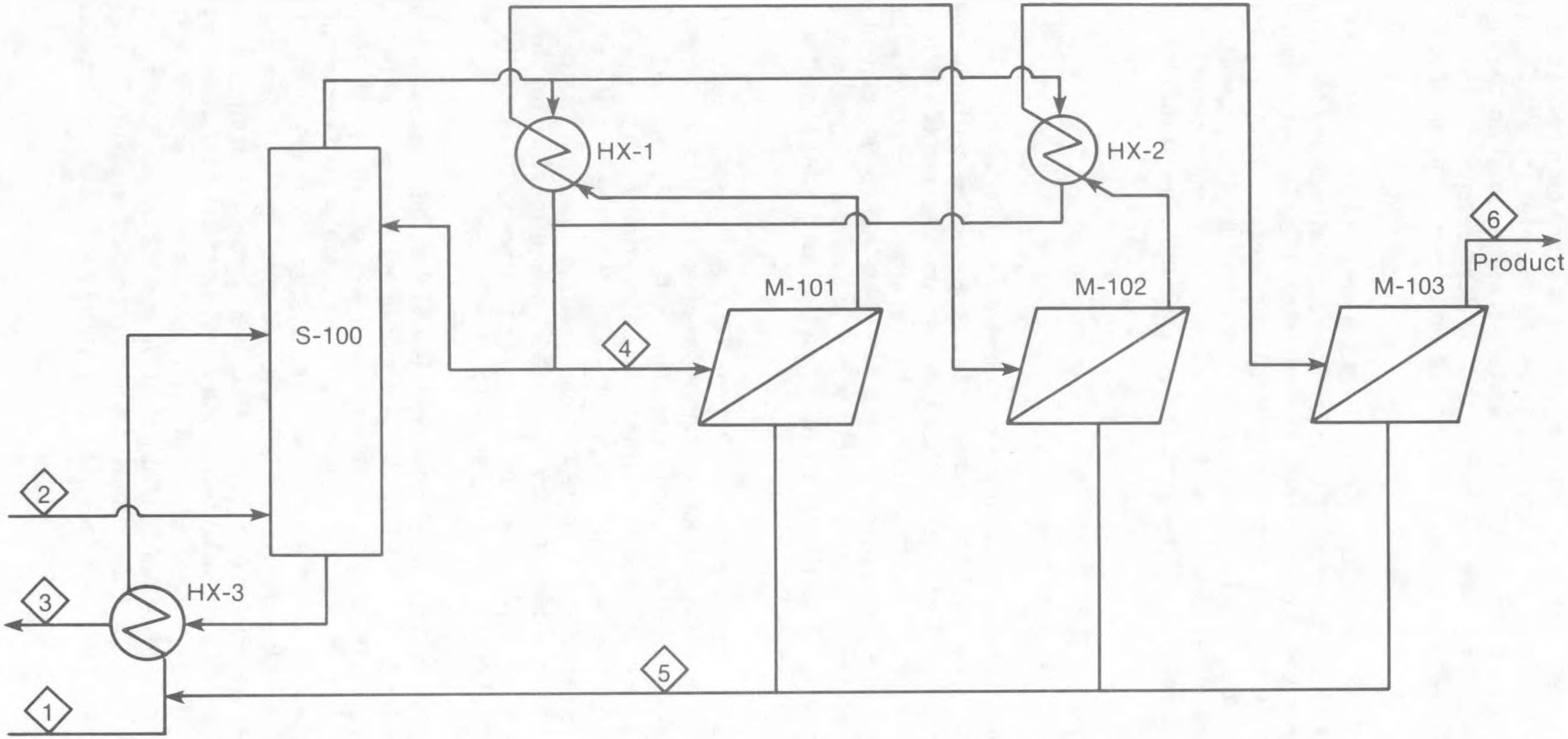
While the program goal of producing a totally ethanol-selective membrane was not accomplished during this 12-month period, substantial progress was made. Researchers prepared structural composites and developed an understanding of the mechanisms operating in doped polymer films that promote alcohol and water selectivity. However, energy requirements must be significantly reduced in membranes that operate in low and intermediate alcohol concentration ranges (10-70 wt %). The fundamental information developed during this contract should be applicable to membranes in this lower concentration range.

3.4.2 GKSS

GKSS, a private firm, has developed a unique dehydration system using the principle of membrane pervaporation. The liquid to be separated is contacted with one side of the membrane, and a vacuum is applied to the other side. If the vacuum is sufficiently low, the components of the feed will evaporate through the membrane. However, the relative rates at which the components evaporate are determined by their physicochemical interaction with the membrane rather than by their vapor pressures. Therefore, the azeotropic points observed in normal distillation vanish, and ethanol/water mixtures over all concentration ranges can be separated.

A summary of the GKSS process is shown in Figure 3-7. A beer still concentrated the 8 wt % feed to 80%. The overhead vapor was condensed to its boiling point and fed to the sequence of membranes. GKSS achieved throughput rates as high as 1.07 gal/ft^2/day, with separation factors above 15. GKSS defined the separation factor <u>beta</u> in terms of water concentration rather than ethanol; their <u>beta</u> was thus the reciprocal of that in Section 3.4.1. The three-membrane process had an overall separation factor of 5.03. If one assumes that equal amounts of water are removed in each membrane, then the individual <u>betas</u> are calculated as 5.03, 6.99, and 12.71, respectively, because the feed concentrations are 80, 84.6, and 90.9 wt %, respectively.

As shown by Figure 3-7, the only thermal energy required for this process was steam for the distillation column—about 11,200 Btu/gal. The electrical energy required to run the vacuum pumps on the permeate side of the membranes was estimated at 2000 Btu/gal, based on the heat-to-electric conversion efficiency of 0.33, and a vacuum in the range of 30-35 mm Hg (0.6-0.7 psia). With an estimated total energy requirement of 13,200 Btu/gal, the complete distillation/pervaporation system appeared quite promising from an energy standpoint. The same was true for capital costs. Figure 3-8 compares the capital costs for a conventional azeotropic distillation plant with partial heat recovery to costs of the GKSS process in Figure 3-7. The distillation process used benzene as the azeotropic agent and consumed roughly 20,000 Btu/gal. (The actual dollar figures for the cost estimates cannot be used because the estimates were prepared for plants in Brazil.) This analysis indicates that pervaporation has cost and energy advantages over azeotropic distillation.



Basis: 10 × 10^6 gal/yr

lb/h	1	2	3	4	5	6
Ethanol	8,269	—	—	10,279	2,010	8,269
Water	95,056	14,719	109,736	2,602	2,563	39
Total	103,325	14,719	109,736	12,881	4,573	8,308

Energy Requirements

Process Block	Btu/gal
Distillation (S-100)	11,200
Membranes (M-101,102,103)	2,000
Total	13,200

Figure 3-7. Integrated Distillation and Membrane Pervaporation Process for Anhydrous Ethanol Production

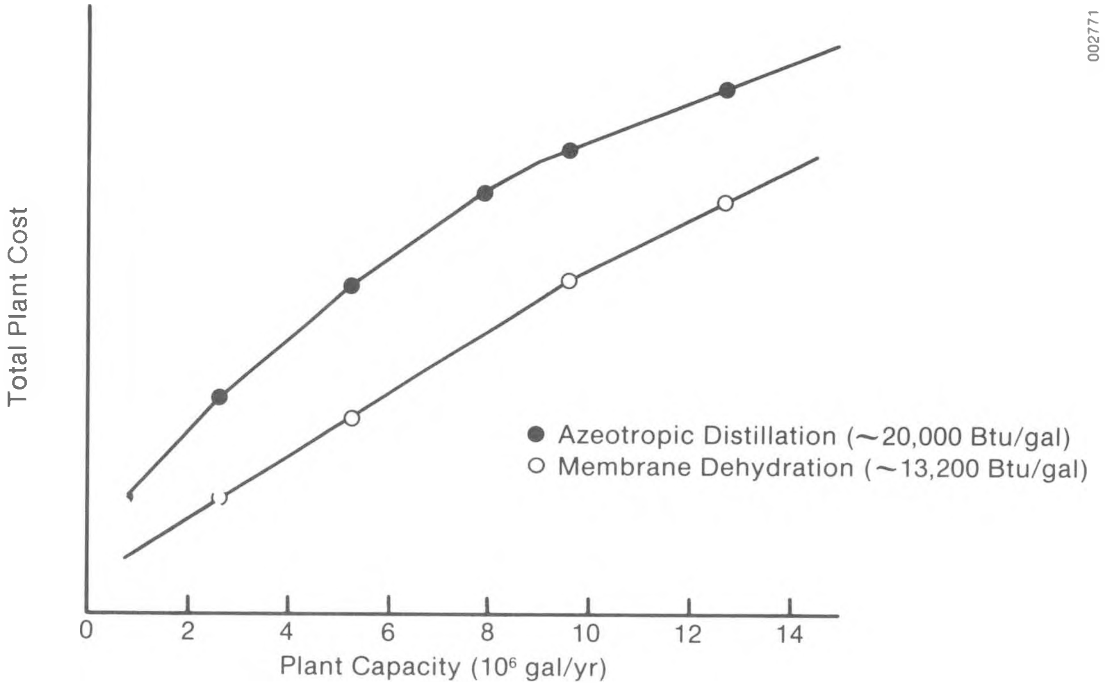


Figure 3-8. Plant Cost vs. Capacity for Anhydrous Ethanol Plants That Include Distillation as the First Step

SECTION 4.0

CONCLUSIONS AND RECOMMENDATIONS

The comparative energy and equipment costs for the ethanol/water separation processes under consideration are shown in Table 4-1. Certain of these processes have technical and economic potential for displacing all or portions of the distillation units in an alcohol plant. Summaries of three processes that have demonstrated some promise follow, along with recommendations for future action.

Table 4-1. Summary of Energy and Equipment Costs

System	Concentration (wt %)	Energy Requirement (Btu/gal)*	Installed Equipment Cost [$M ($10^6$ gal/yr)]
Conventional distillation	10-99.9	27,000	1.6 (25)
Vapor reuse distillation	10-99.9	18,000	2.0 (25)
Cantrell/Petrek—Diffusion/carrier gas	10-85	9,900 (3900)	—
Intertechnology/Solar/Science Applications, Inc.—Solvent extraction	30-99.9	2350 7200 (10-30)**	— —
Georgia Tech—Solvent extraction	26-98	12,600 (2,600) 5,000 (10-26)**	3.7 (27)
Hydrocarbon Research Inc.—Ethanol selective adsorption	15-99.9	25,000 (12,600)	4.0 (25)
Purdue—Ethanol selective absorption	91-99.9	2000-3000	—
Shock Hydrodynamics—Ethanol selective absorption	—	—	—
Southern Research Institute—Ethanol selective membranes	—	—	—
GKSS—Membrane dehydration system	8-99.5	13,200	—

*The number in parentheses represents the thermal equivalent of the electrical energy requirement with a heat-to-electricity efficiency of 0.33.

**Approximate energy requirement to concentrate ethanol from 10 wt % to the concentration shown.

4.1 SOLVENT EXTRACTION

In two years, Georgia Tech has made significant progress toward developing an energy-efficient system. The EHOH/ISOPAR-L process is almost energy- and cost-competitive with the other systems under consideration. However, the 12,600 Btu/gal requirement must be increased by at least 5000 Btu/gal to compensate for their higher feed concentration. Georgia Tech is apparently ready to build an entire pilot plant, including fermenters, so that further energy and cost improvements could then be achieved.

4.2 GRAIN ADSORPTION

The dehydration systems being researched at Purdue appear to have great potential. The system using grain is being funded by the USDA, while the calcium sulfate system is funded by SERI. These systems are simple, inexpensive, and economical over a wide range of plant sizes. After thorough analysis of laboratory data, a large-scale test facility could be designed and integrated with a promising, low-end separation system as part of an overall ethanol production plant.

4.3 MEMBRANE PERVAPORATION

The researchers at Southern Research Institute did not produce a membrane that would concentrate a 50 wt % ethanol/water solution to above 97 wt %. However, they did produce a valuable body of data useful in understanding the mechanisms in alcohol enrichment via membrane processes. During the course of their investigations, they developed five new membrane fabrication techniques and demonstrated the feasibility of their technical approach. Based on the very promising performance data reported by GKSS, the emphasis of Southern Research Institute's project, as well as any new work, should be directed toward pervaporation and toward lower ethanol concentrations (i.e., 10-75 wt %).

The energy reduction targets discussed in Section 1.0 of this report do appear technically feasible, especially with the latter two processes. Cost reduction targets pose a greater challenge. Without further technical breakthroughs, the prospects for a short-term, 50% cost reduction are not likely, which is all the more reason to intensify efforts on the process that has potential.

SECTION 5.0

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The purpose of this report is to present an assessment of several alternatives to distillation as a means of separating ethanol from fermentation beers. Process flowsheets, capital and operating cost summaries, and energy requirements are presented. Each process is assessed in terms of nearness to commercial adaptability and ranked according to energy-efficiency and cost using a state-of-the art distillation process for comparison.

While this review is based on preliminary data in some cases, it is clear that certain of these processes have technical and economic potential for displacing all, or portions of, the distillation units in an alcohol plant.

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