Preliminary Evaluation of Alternative Ethanol/Water Separation Processes

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May 1981

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INTRODUCTION

Much of the recently publicized attention directed at producing liquid transportation fuels from renewable resources has centered on the fermentation process for ethanol production. The process basically consists of a fermentation step that produces a low-grade ethanol/water mixture containing roughly 10% ethanol. This mixture is then upgraded by a two-step distillation process that yields absolute ethanol for use in gasohol.

Conventional "dual" distillation uses thermal energy to produce an azeotrope of 95% ethanol, followed by azeotropic distillation to produce 100% ethanol. While this process is quite effective in terms of degree of separation obtained, it is also very energy intensive. Combined, the distillation to 95% ethanol followed by azeotropic distillation to absolute ethanol accounts for greater than 50% of the energy required for ethanol production.

Alternatives to this conventional method are being investigated by staff members at the Pacific Northwest Laboratory (PNL) as well as by others involved in ethanol research, development or production. This study, conducted by PNL for the U. S. Department of Energy, is a preliminary evaluation of various ethanol/water separation techniques to determine which method or methods warrant further development and support.

The evaluation compared various techniques to conventional distillation and each techniques' energy requirements. Several potential methods for ethanol/water separation were investigated, including:

- ethanol extraction with CO₂ (the A. D. Little process)
- solvent extraction of ethanol
- vacuum distillation
- vapor recompression distillation
- dehydration with fermentable grains
- low temperature blending with gasoline
- molecular sieve adsorption
- reverse osmosis.

Following the Summary and Conclusions, each process is outlined and discussed in detail. Background information, other research efforts and relative costs are examined, including recommendations for future applications.

SUMMARY AND CONCLUSIONS

Our preliminary evaluation indicates that separation of ethanol and water can be accomplished with less energy than is now needed in conventional distillation processes. The state of development for these methods varies from laboratory investigation to commercially available processes. The processes investigated were categorized by type of separation depending on their ability to achieve varying degrees of ethanol/water separation. A summary of the projected energy requirements for these alternatives is shown in Table 1.

TABLE 1. Summary of Projected Energy Requirements for Alternative Ethanol/Water Separation Processes

Type of Separation	Ethanol Concentration Initial	Final	Process	Energy (Btu/gal)
Complete	10%	100%	Conventional "Dual" Distillation	27,400
			CO ₂ Extraction	8,000 - 10,000(a)
			Solvent Extraction	3,600(a)
			Vacuum Distillation	37,000
to Azeotrope	10%	95%	Conventional Distillation	18,000
			Vapor Recompression	6,400(a)
			"Multi-Effect" Vacuum	7,200
Azeotropic	95%	100%	Conventional Azeotropic Distillation	9,400
			Dehydration	1,200(b)
			Low Temperature Blending with Gasoline	3,000(c)
			Molecular Sieve	4,700 - 6,270
Other	5%	10%	Reverse Osmosis	500

(a) Figure given is the thermal energy required to provide mechanical energy for the process.

(b) For drying with CaO, energy requirements using fermentable grains would be considerably less.

(c) Results directly in production of gasohol.

The A. D. Little CO₂ extraction process uses approximately one-third the 27,400 Btu/gal required by conventional distillation processes. This process will be demonstrated in a pilot plant that is currently being constructed by the A. D. Little Co. in cooperation with the A. E. Staley Corp. While capital cost of this system is high, the much lower operating cost would provide a rapid pay back.

Although CO₂ extraction is a form of solvent extraction, high pressures (900 to 1,000 psi) are required to liquefy the CO₂ for the extraction step. With an "ideal" solvent, the process could be operated at moderate pressures, thereby further reducing the energy requirements (from 8,000 to 10,000 Btu/gal to about 3,600 Btu/gal). Additional research and development is required to find this optimal solvent. Neither CO₂ extraction nor solvent extraction require specialized equipment.

Both vapor recompression and "multi-effect" vacuum distillation are state-of-the-art technologies that could be designed into ethanol production processes. Use of vapor recompression and "multi-effect" vacuum distillation also requires considerably less energy than conventional distillation to reach the azeotrope. Vapor recompression, used for many years in the petroleum industry, is an energy conservation technology readily adaptable to distillation of ethanol and water. Capital costs would be 50% higher than for conventional methods, but total distillation costs are 55% less than for conventional steam-fired distillation.

Straight vacuum distillation can be used to "break" the azeotrope and produce 100% ethanol. However, its energy requirements are actually higher than for conventional distillation, at about 37,000 Btu/gal. While the equipment required is conventional, the system would be costlier than conventional processes.

The dehydration method employs a solid dehydration agent to remove the water from ethanol/water solution at up to 95 wt% ethanol. The dehydration agent may be either discarded, regenerated or in the case of grains can be sent to the fermenter for conversion into ethanol. The energy needed for dehydration using CaO, for example, is estimated to be 1,200 Btu/gal as thermal

energy. Although some investigations have been conducted using fermentable grains as the dehydrating agent, additional work is required to find the ethanol/water distribution between the grain and the ethanol product. If successful, this process would use even less than the 1,200 Btu/gal projected with CaO and is simpler and cheaper than conventional distillation.

The energy required to produce gasohol directly by low temperature blending of 95% ethanol with gasoline is projected to be 3,000 Btu/gal of ethanol. Cooling a mixture of gasoline, water and ethanol forms a separate aqueous phase of mostly water and some ethanol. Formation of this separate phase is the reason anhydrous ethanol is used for producing gasohol. This principle can be used in producing gasohol by adding an ethanol-water mixture to gasoline, cooling the solution, then drawing off the aqueous phase. Energy requirements for cooling are minimal (~700 Btu/gal ethanol) and the total process energy depends mostly on the need to redistill the aqueous phase for ethanol recovery. Additional laboratory investigations are needed to determine the actual distribution of ethanol in the aqueous phase, which will in turn determine the energy requirements for redistillation.

The molecular sieve adsorption technology, available from companies such as W. R. Grace and Schoof, Inc., reduces by one-half to one-third the energy required to produce anhydrous ethanol from conventional distillation. Although no commercial ethanol plants now use this technology, several are reported to have it at the drawing board stage. This process uses conventional equipment, and capital costs for installation would be equivalent to those for the azeotropic distillation process.

Reverse osmosis is impractical for concentrating dilute ethanol/water solutions to high concentration because of the high osmotic pressure needed and the poor ethanol retention. Although energy requirements for RO processing are fairly modest, application of the process is limited. Our investigation showed that RO has little or no potential in the ethanol/water separation area for fermentation beers, but might possibly be used in operation of a continuous fermenter. It is doubtful, however, that the economics of RO would be an improvement over conventional distillation methods.

RECOMMENDATIONS

Based on the results discussed in this report, the following recommendations are made:

- Ethanol at concentrations in or lower than the 75% to 85% range should be promoted as an independent fuel rather than using it for production of gasohol, which requires 100% ethanol. The energy required to produce these lower concentrations is roughly one-half that required to produce pure ethanol. In the case of on-farm production and use of ethanol, this approach also allows more ethanol to be used as a fuel closer to the source of production.
- Vapor recompression and "multi-effect" vacuum distillation are commercially ready processes that can be easily adapted to ethanol production. Pilot- or commercial-scale demonstrations of these technologies are recommended.
- Further laboratory investigations are recommended for:
 - a) the solvent extraction process, to determine the "ideal solvent, process pressures, volume of solvent required, toxicity of solvent and solvent losses,
 - b) the grain dehydration process, to determine the amount of water and ethanol taken up by the grain, final ethanol concentration, and material extracted from the grain by ethanol, and
 - c) the low temperature blending with gasoline process, to determine the actual distribution of ethanol in the gasoline and water as a function of the gasoline/water ratio and temperature used during blending.

DISCUSSION

Alternative processes for separation of ethanol and water were evaluated in this project and compared to the conventional "dual" distillation method. The first step in distillation uses thermal energy to selectively separate the more volatile ethanol from water. This process produces 95 wt% ethanol, at which concentration the ethanol and water form an azeotrope. At this point, the vapor has the same concentration as the liquid, thus preventing further separation by simple distillation.

In the second step, a third component (e.g., benzene, cyclohexane or pentane) is employed in the azeotropic distillation process to yield 100% ethanol, allowing a ternary mixture of ethanol/water/solvent to be distilled from the ethanol.

Although this "dual" process is efficient in the degree of separation obtained, it is also energy intensive and can account for more than 50% of the total energy used in ethanol production. The process and energy requirements for conventional distillation, plus alternatives studied in this report, are discussed below.

CONVENTIONAL DISTILLATION

A simplified conventional distillation process is shown in Figure 1. This process is capable of producing ethanol at the azeotrope composition of about 95%. The high energy required for this process results from the need for the high reflux rate required to achieve the degree of ethanol/water separation. The reported energy required for this "conventional" process varies from manufacturer to manufacturer (Rocket Research Co. 1980). The energy required for the azeotropic distillation process shown in Figure 2 also varies depending on the manufacturer. However, for a highly efficient conventional system the energy required to reach the azeotrope starting with about 6 1/2 wt% ethanol is about 21,850 Btu/gal of ethanol, and for azeotropic distillation the energy required is about 9,400 Btu/gal (Black 1980). The relationship between the initial concentration of the ethanol/water mixture fed to the distillation

FIGURE 1. Conventional Ethanol Distillation Process

process and the energy required to produce various ending ethanol concentrations is shown in Figure 3. The absolute energy required is expected to change depending on the exact equipment used. However, the relative trends shown in the figure are expected to be valid. From an energy standpoint it is important to have the feed (fermentation beer) to distillation process near 10% ethanol, as can be seen in Figure 3. Also noticeable from the figure is the significant energy reduction if a 75% to 85% ethanol solution is produced that could be used as a fuel by itself without making gasohol. If the initial ethanol concentration of the solution fed to distillation is 10%, the distillation energy required to reach 75% and 85% is about 10,500 and 12,500 Btu/gal of ethanol as compared to 27,400 to obtain 100% ethanol. The difference in energy required is even more pronounced if the feed concentration is higher. Because the same total amount of ethanol is produced whether it is concentrated to 75%, 85% or

FIGURE 2. Conventional Azeotropic Distillation Process

100%, the only difference in the total energy available is the energy required to vaporize the water in the fuel. This amounts to only 1.5 to 3% of the energy of the ethanol.

A. D. LITTLE CO₂ EXTRACTION PROCESS

Liquid CO₂ is used to extract ethanol from the ethanol/water solution as shown in Figure 4. This is conventional liquid-liquid extraction since the liquid CO₂ is not miscible with water. Ethanol partitions into the CO₂ phase under these conditions and is recovered by volatilizing the CO₂. The process operates under a total pressure of 900 to 1000 psi at approximately 90°F. The ethanol-rich CO₂ phase is flashed to approximately 700 psi, leaving ethanol and all of the fermentation components (e.g., fuel oils) in the liquid phase, which is then dropped to atmospheric pressure and recovered. Gaseous CO₂ is recycled to a compressor and condensed for reuse.

FIGURE 3. Ethanol Distillation Energy as a Function of Initial and Final Ethanol Concentration

FIGURE 4. Flow Diagram for Solvent or CO₂ Recovery of Ethanol

This process can be used to concentrate any ethanol/water solution. It is best adapted to concentrating dilute solutions, so the 10% or so typical fermentation beer is almost ideal. The product from this separation is anhydrous ethanol. The energy required by this process is mechanical, for recompression of vapor. This can be supplied by a turbine or an electrical drive. The thermal equivalent of energy required (according to A. D. Little contacts) is 8,000 to 10,000 Btu/gal, about one-third that required by conventional processes.

Because of the pressure systems involved and the need for gas compression, the overall capital cost of this type of system is about 20% greater than an

equivalent distillation system of conventional design. No commercial process is operating yet; however, a pilot plant is being built in cooperation with A. E. Staley Corp. Most of the equipment required is of conventional design, so commercial plants can follow soon after a successful pilot demonstration. Capital cost of the system is higher, however, the much lower operating cost will allow a rapid pay back. Industry is watching this process carefully and expects to be using it if the pilot plant is successful. Because the solvent (CO_2) is a byproduct of fermentation, solvent loss is not of critical concern. The process does operate in conjunction with the fermentation plant as a net producer of CO_2 .

SOLVENT EXTRACTION

Solvent extraction of ethanol from water involves the use of an organic solvent which is insoluble in water to selectively extract the ethanol. The process flow diagram (Figure 4) is the same as for the CO_2 extraction process, however the ideal solvent would not require the high pressures required for the CO_2 process. The ideal solvent also would have a high ethanol extraction coefficient and a boiling point different enough from ethanol to ease the solvent/ethanol separation step. The extraction step could be performed at moderate pressure (up to 50 psi), and following removal of the solvent/ethanol phase the pressure reduced to near atmospheric for "flash" distillation. The solvent should also be non-toxic to yeast so that the water phase can be returned to the fermenter for continuous fermentation purposes.

This process would take ethanol at low concentrations ideally suited to continuous fermentation (~5 wt%) and provide an end-product of 100% ethanol. The energy required for this process would be for compression and is estimated to be on the order of 1200 Btu/gal as mechanical energy or about 3600 Btu/gal as thermal energy for the "ideal solvent". Although several investigators (Othmer 1941; Scheibel 1950; Boeckeler 1948; Dreyfus 1936) have tried or suggested a number of solvents, no "ideal" solvent has yet been identified. Continued research in this area is recommended.

No highly specialized equipment is required for solvent extraction. The changes from conventional distillation are a solvent extraction column, flash

distillation and a compressor for solvent recycle. The main considerations are the exact pressures required, the volume of solvent recycle, solvent toxicity and solvent losses. All of these would be determined by the exact solvent used.

VACUUM DISTILLATION

Vacuum distillation could potentially take two alternative forms which include 1) use of vacuum to break the azeotrope and 2) use of a multicolumn system with each successive column operated at a lower pressure (similar to multi-effect evaporators) as shown in Figure 5. For the system using vacuum to break the azeotrope, the ending ethanol concentration would be at or near 100%; for multi-effect distillation, the ending concentration would be 95% as in conventional distillation. For both cases the initial concentration would be about 10%.

FIGURE 5. Multicolumn Vacuum Distillation (each successive column operated at lower pressure)

At pressures less than about 1.7 psi the ethanol/water azeotrope disappears (Black 1980). However, the distillation equilibrium line at high ethanol concentrations approaches the 45° diagonal (the vapor and liquid compositions are nearly equal). Therefore, in order to reach nearly 100% ethanol a very high reflux ratio of approximately 13 to 14 is needed, resulting in energy requirements estimated to be 37,000 Btu/gal, which is higher than for conventional distillation. For "multi-effect" vacuum distillation the energy required to get to 95% ethanol is projected to be a factor of 2.5 times less than conventional distillation or about 7,200 Btu/gal. Azeotropic distillation would still be required and would bring the total energy required to 16,600 Btu/gal. The energy required for providing the vacuum is expected to be negligible.

The equipment required for "multi-effect" distillation would be basic, commercially available items. The main difference is that three smaller diameter distillation columns would be used in place of one conventional column. The three-column "multi-effect" system is more expensive than conventional systems.

VAPOR RECOMPRESSION DISTILLATION

Vapor recompression distillation has been used for many years in the petroleum industry. Vapor recompression achieves significant energy savings by using the latent heat of overhead vapors in the column reboiler. The overhead vapors are compressed so condensation will occur at an adequate temperature difference in the reboiler. Compressors used for vapor recompression are reliable and cost effective if properly designed and maintained (see Figure 6).

An analysis of vapor recompression distillation was conducted to concentrate a 10% ethanol feed to 95 wt%, recovering 99% of the ethanol in the distillate product. Production capacity of the still was taken at 2,600 gal of 95% ethanol per hour. Using a 10°C temperature difference in the reboiler and an 80% compressor efficiency, only 1,932 Btu/gal is required for distillation. This compares to 18,000 Btu/gal for conventional distillation. The compressor energy for vapor recompression may be supplied by high pressure steam or electric power. Use of electric power requires a 2000 HP motor and

FIGURE 6. Vapor Recompression Distillation

would consume 0.58 kW/gal. Assuming a 30% conversion efficiency for thermal energy to electric power, the total energy requirement is 6,440 Btu/gal. It should be noted, however, that a steam driven turbine compressor may be efficiently employed because the low pressure waste steam may be used for sterilization and by-product drying. In this case the energy for vapor recompression distillation would again be 1,932 Btu/gal.

Capital costs for vapor recompression distillation are expected to be 50% higher than for conventional steam-fired distillation. The added cost is due to the high cost of the compressor. Total distillation costs are 55% less for vapor recompression distillation than for conventional steam-fired distillation.

DEHYDRATION

In dehydration, the first step is distilling a fermentation beer containing typically 5 to 12 wt% ethanol to yield a distillate containing 70 to 95 wt% ethanol. In the process proposed by Ladisch and Dyck (1979), the distillate would then be contacted with a solid dehydration agent where the remaining water would be adsorbed, producing an essentially anhydrous grade ethanol. Once spent, the dehydration agent would either be discarded, regenerated by heating, or if fermentable grain were used as the dehydration agent, sent to the fermenter to be converted into ethanol.

The energy required for dehydration using CaO is estimated to be 1,200 Btu/gal as thermal energy. Use of grain feedstocks for dehydration would reduce the energy required to essentially zero, because the drying agent does not need to be regenerated.

Further work is needed to determine: 1) the actual amount of water taken up by the grain, 2) the amount of ethanol taken up by the grain, 3) the final concentration of ethanol after dehydration and 4) material extracted from the grain by ethanol. In this process the grain would be used as the fermentable feedstock and therefore would not require energy to regenerate the dehydrating agent.

The equipment for this process would be less complex and less costly than current azeotropic distillation equipment. The dehydration studies conducted to date have used ethanol/water in the vapor state. There may be some advantages (particularly for small-scale operations) to dehydrating liquid ethanol/water mixtures.

LOW TEMPERATURE BLENDING WITH GASOLINE

Ethanol/water mixtures, when blended with gasoline and cooled, result in a separation of an organic and aqueous phase. The aqueous phase is mostly water containing minute quantities of ethanol, and the organic phase is the gasoline/ethanol mixture (gasohol). When separated from the water, this gasohol mixture is usable as an automobile fuel at temperatures above those at which low temperature blending was conducted. A simplified flowsheet for this process is shown in Figure 7.

FIGURE 7. Low Temperature Blending with Gasoline

We assumed that if 11.3 lb of 95% ethanol was blended with 90 lb of gasoline and cooled from 70°F to -30°F , two phases would separate. The organic phase would be 100 lbs of gasohol (10% ethanol) and the remaining 1.3-lb water phase was assumed to be 5.6% ethanol. The cooling process requires 665 Btu/gal of ethanol. In addition, 2,650 Btus are required to reconcentrate ethanol in the water phase to 95%. Since 0.6 thermal Btus are required per one cooling Btu, the net energy needed for cooling is 400 Btu/gal of ethanol. The total energy requirement is thus 3,050 Btu/gal of absolute ethanol produced by low temperature blending.

Further investigations are required to determine the actual distribution of ethanol in the gasoline and water as a function of the gasoline/water ratio and temperature.

MOLECULAR SIEVE ADSORPTION

In this process a 95% ethanol/water solution is passed through a molecular sieve column. The sieve adsorbs water preferentially. Approximately 3/4 of the sorbed material is water, while 1/4 of it is ethanol. The column produces anhydrous ethanol until it becomes saturated. At that point the stream is usually switched to a fresh column, and the saturated column is regenerated, producing a dilute stream containing about 25% ethanol in water. This dilute stream is fed back to the primary distillation column.

The energy required is thermal, to regenerate the molecular sieve. Generally speaking, the temperatures required are in the range of 400°F, so that direct thermal energy is normally used. The total amount of energy needed is 1/2 to 2/3 of that normally required to produce anhydrous ethanol via tertiary azeotropic distillation.

No commercial plant uses this technology; however, several are considering it. The process is conventional and uses completely conventional equipment. Capital cost for installation of the mol sieve process is almost exactly the same as for tertiary azeotropic distillation using benzene. Mol sieve life is usually excellent--normally lasting for a year or longer without replacement.

REVERSE OSMOSIS

In this process the RO membrane allows water to pass through it while retaining most of the ethanol. Because the permeate still contains some ethanol, this stream would be returned to the fermenter for a continuous fermentation process. Osmotic pressures for concentrated ethanol-water solutions are very high, reaching 1000 psi--the practical upper limit for RO units at about 14% ethanol. This makes RO an impractical method for concentrating dilute ethanol/water solutions to high concentration using conventional equipment, even if new membranes exhibiting excellent rejection characteristics are developed.

The only technically feasible application for RO would be to concentrate the ethanol in a fermentation beer by a few weight percent prior to distillation or use some concentrating unit operation. It is doubtful, however,

that this would improve either the economics or energy requirements for conventional dual-distillation processing. Energy requirements for RO processing would be fairly modest in this limited application.

Based on this analysis, it would seem that RO would have little or no potential in the ethanol/water separation area for fermentation beers, but could possibly be used in the operation of a continuous fermenter where an RO module could be used to maintain a constant ethanol concentration. In this case, the permeate containing a lower concentration of ethanol than the feed would be recycled back to the fermenter with the concentrate sent to distillation. It is likely, though, that extensive pretreatment of the fermentation beer would be necessary to remove suspended solids and/or organics that could foul the membranes. If so, the economics of RO in this application are questionable.

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