

DOE/PC/92118--T14

Bench-scale Demonstration of Biological Production of Ethanol from Coal Synthesis Gas

Topical Report 5: Process Analysis

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Contract No. DE-AC22-92PC92118

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ACKNOWLEDGEMENT

Financial support for this work was provided by the U.S. Department of Energy, Pittsburgh Energy Technology Center, on contract DE-AC22-92PC92118

ABSTRACT

The economics of converting coal to ethanol by a biological process is quite attractive. When processing 1500 tons of coal per day, the plant generates 85 million gallons of ethanol per year. The return on investment for the process is 110 percent and the payout is 0.9 years.

1.0 INTRODUCTION

The United States currently imports about 50 percent of its crude oil requirements and is expected to import 66 percent of its petroleum by the year 2010 (Hyd. Proc., 1991). The U.S. has become a debtor nation largely because of its dependence upon imported petroleum. Alternate sources of liquid fuels must be developed to alleviate this problem.

This nation has massive reserves of coal, which represent our largest fossil energy resource, equivalent to about 750 billion barrels of oil, or a 300 year supply of petroleum at the current rate of consumption (Speeks and Klusmann, 1982). Sub-bituminous coal, with an energy content of 5500-8000 BTU per pound, represents about 60 percent of this reserve (Hammond *et al.*, 1973). This abundant resource must become the predominant source of liquid fuel in this nation.

Processes to convert coal into liquid fuels have been under intensive investigation for the last 20 years. Several processes for the direct liquefaction of coal and for the conversion of coal synthesis gas into liquid fuels by Fischer-Tropsch reactions are being developed. These processes operate at elevated temperatures and pressures, which impair the thermodynamic and economic efficiency. Sulfur gases are also a catalyst poison, and the removal of these gases is expensive. Yields of liquid fuels are not high and the removal of heteroatom compounds requires large quantities of hydrogen. More economical processes for producing liquid fuels are needed to compete with imported petroleum.

A simple biological process may also be used to convert coal synthesis gas into ethanol. In general, microbial processes offer certain advantages over chemical conversions. Micro-organisms function and carry out reactions at ambient temperatures and pressures, which usually results in substantial energy and capital savings. Also, yields from biological conversions are quite high, since the microorganism utilizes only a small fraction of the substrate for energy and growth. Under proper conditions, microbial reactions are quite specific, generally converting a substrate into a single product, simplifying product recovery systems. Microorganisms can usually tolerate small quantities of sulfur gases without cumulative effects. Also, the irreversible nature of biological reactions allows complete conversion without thermodynamic equilibrium constraints and the resultant recovery and recycle of unconverted components.

These advantages are offset, somewhat, by slower reaction rates and special reactor considerations, such as sterility and nutrient provision. For the conversion of synthesis gas, sterility is insured by the substrate, CO, which is toxic to most organisms. Biological reactions are usually slow and require retention times of hours, which would be impractical for coal synthesis gas conversion. Synthesis gas components are only slightly soluble in the medium surrounding the microorganism and mass transfer is usually limiting in these reactions. Nevertheless, equivalent retention times of only a few minutes have been achieved for similar conversions (Klasson *et al.*, 1991; Gaddy, 1991; Vega *et al.*, 1989a; b), which makes this technology very attractive.

An anaerobic bacterium has been isolated from natural sources that converts the components of synthesis gas (CO, H₂, CO₂) into ethanol (Vega *et al.*, 1989c). This organism, the only one known at that time to produce ethanol from synthesis gas, has been identified as a new clostridial strain and has been named *Clostridium ljungdahlii*, strain PETC. Ethanol is produced according to the following stoichiometry:



Preliminary economics indicate that the capital cost for the bioprocess to handle synthesis gases from a gasifier processing 1000 tons coal per day would be only \$30 million. The revenue generated from ethanol at current market prices is \$66 million per year, based on current yields of about 150 gallons per ton. These economics are quite promising and demonstrate that commercialization can be achieved in the near term. In order to reduce this technology to practice over the next four years, a two-year bench scale demonstration is necessary, followed by a two-year larger scale prototype demonstration.

The purpose of this research project is to develop and operate a bench scale unit for production of ethanol from coal synthesis gas. This unit will include a bioreactor, cell recycle membrane, extraction unit and distillation column, as is shown in Figure 1.1. Recycle loops for culture medium and solvent will be incorporated. Studies will be conducted to select the best ethanol culture, *C. ljungdahlii* or a new isolate; and to identify the best solvent for ethanol extraction. Prolonged operation of the unit over a one year period will be conducted to demonstrate the viability of the culture and product recovery system and to identify potential problems with recycle loops. Scale-up parameters for the system will be developed for design of the prototype and commercial units. The economics for a commercial facility will be projected to identify high cost areas for concentrated research.

1.1 Purpose

The purpose of this report is to present the results from a design and economic analysis performed on the biological syngas to ethanol process. The plant size in this analysis is sized to process 1500 tons per day of coal and produce 85 million gallons per year of ethanol. Process alternatives with and without electricity generation are compared.

2.0 BIOLOGICAL ETHANOL PRODUCTION FROM SYNGAS - BACKGROUND

In 1987, enrichment studies utilizing natural inocula resulted in the isolation of a new anaerobic mesophilic bacterium that is capable of converting CO and H₂O or CO₂ and H₂ into a mixture of ethanol and acetic acid (Barik *et al.*, 1987; Barik *et al.*, 1988; Vega *et al.*, 1989d). The microorganism utilizes these gases for growth and also grows on arabinose, xylose, fructose, ethanol and pyruvate. This rod shaped bacterium has been named *Clostridium ljungdahlii*, strain PETC. The cells are flagellated and have an internal membrane structure, uncommon in other clostridial species. DNA composition studies have shown that *C. ljungdahlii* contains 22 mole percent guanine plus cytosine, which clearly identifies this strain as a new clostridial species.

Clostridial fermentations with sugars yield a wide variety of end-products which include two to five carbon acids and/or solvents, as well as H₂ and CO₂. The amount of reduced versus neutral or oxidized product is always balanced with the amount of H₂ and ATP produced and, consequently, has a great deal of natural variation. Consequently, early experiments with *C. ljungdahlii* were focused on increasing the ethanol productivity and followed work with other clostridial species to increase solventogenesis by adjusting growth-limiting factors, such as pH and nutrients, addition of reducing agents, induction of sporulation, etc. (Bahl *et al.*, 1982; Bahl and Gottschalk, 1984; Gottschalk and Morris, 1981). Limiting yeast extract with *C. ljungdahlii* resulted in doubling the ethanol productivity. Low pH and low dilution rates in continuous culture resulted in increasing the ETOH:HAC ratio to

3.0. The use of reducing agents, such as cysteine hydrochloride or benzyl viologen in low concentrations, was also found to increase ethanol productivity.

The following sections summarize the early results from experiments with *C. ljungdahlii* to further improve the performance and increase ethanol productivity. The basis for the development of the conceptual ethanol process, including bioreactor selection, is also discussed.

2.1 Effect of Growth Parameters

A series of experiments have been conducted in batch culture with *C. ljungdahlii* to examine the effect of growth parameters on ethanol productivity. All experiments were conducted at the optimal pH of 4.0 with small amounts of cysteine hydrochloride. Minimal yeast extract was found to improve the ethanol production and yeast extract was, therefore, eliminated from the medium. Studies were then initiated to vary the other medium constituents, Pfennig minerals, Pfennig trace metals and B-vitamins. Varying the minerals or metals had little effect on the ethanol/acetate ratio. However, reducing the B-vitamins to one-half had a pronounced influence, as shown in Figure 2.1. As is noted, the cell growth was rapid for the first 100 hours, when the ethanol and acetic acid production were about equal. When the cell growth slowed, the ethanol concentration rose quickly, while acetic acid slowly declined. A maximum ratio of ETOH:HAC of 11 resulted.

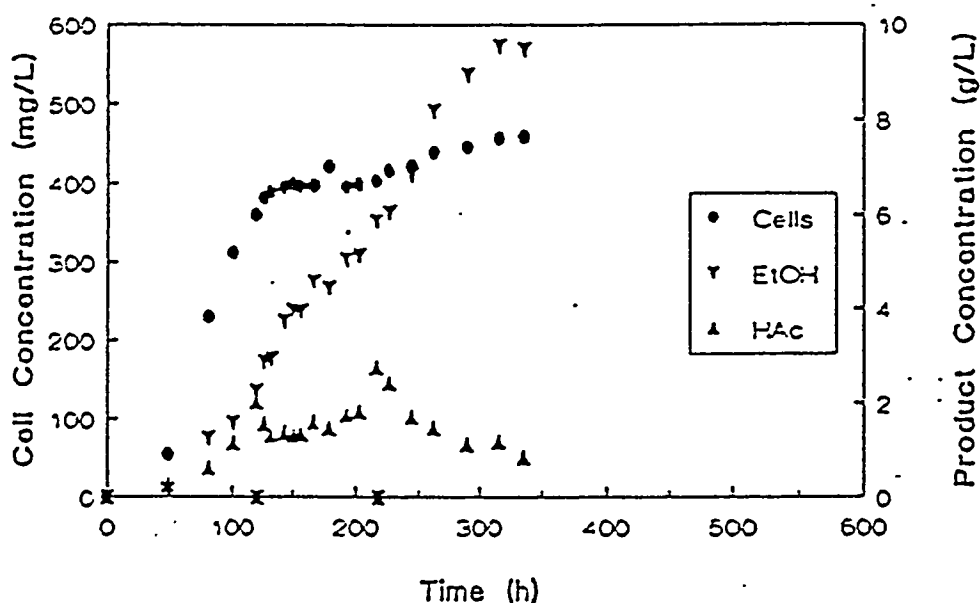


Figure 2.1. Cell and Product Concentration Profiles for *C. ljungdahlii* with Half B-Vitamins

Additional experiments were conducted with lower concentrations of B-vitamins and showed that cell growth, and thus ethanol concentration, became limited. Therefore, studies were made to examine the individual B-vitamins to determine which might be essential for good growth. These experiments used 8 percent of the normal B-vitamins, supplemented with biotin, thiamine or calcium pantothenate. Biotin and thiamine both were found to stimulate growth and increase ethanol

productivity, as shown in Figures 2.2 and 2.3. As is noted, fast growth was achieved and was accompanied by high ethanol production. Also, production of acetic acid was essentially eliminated. Higher cell concentrations were achieved with calcium pantothenate, but acetic acid levels were also much higher.

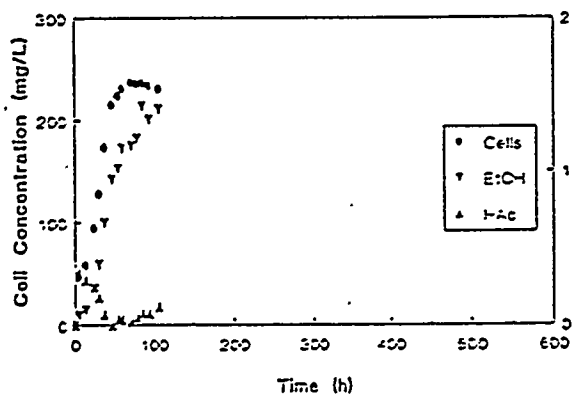


Figure 2.2. Cell and Product Concentration Profiles with Biotin (*C. ljungdahlii*)

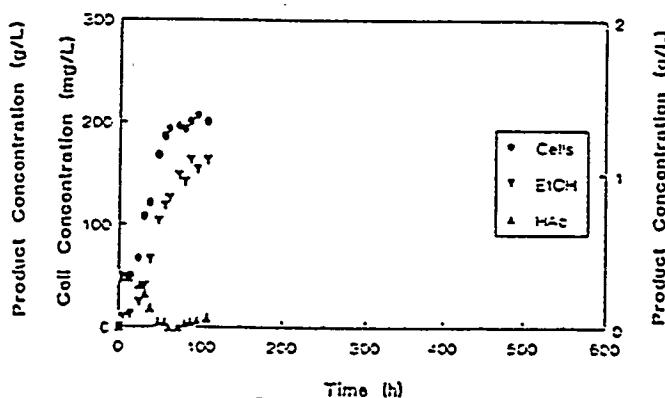


Figure 2.3. Cell and Product Concentration Profiles with Thiamine (*C. ljungdahlii*)

2.1.1 Significance

The regulation of growth parameters in the culture medium is the key to controlling the ethanol productivity. In particular, low B-vitamins, supplemented with biotin and thiamine, has been found to effectively eliminate acetic acid production. It is postulated that these growth parameters regulate enzymatic activity within the cell that favors ethanol production.

2.2 Continuous Culture Performance

Experiments in continuous culture are necessary to demonstrate the viability of the culture to produce large amounts of ethanol for extended periods. Nutrient limitation in continuous reactors is controlled by the dilution rate, or liquid flow rate. Since the gas retention time controls the reactor volume in synthesis gas fermentations, the liquid retention time can be varied to give high ethanol productivities. Low liquid flow rates give higher cell concentrations and are desirable. In order to achieve high cell concentrations, a system to remove cells from the reactor effluent has been developed. This system consists of a small filter in the liquid effluent, which is periodically backwashed to prevent clogging. Cells that are removed by backwashing are recycled to the bioreactor. An improved system has been developed at BRI using a hollow fiber membrane (HFM) and will be used in the bench scale studies proposed herein. The HFM removes permeate radially, allowing the cells to flow through the fibers without backwashing.

The results of experiments with a continuous stirred tank reactor (CSTR) using a New Brunswick Bioflo chemostat with cell recycle are shown in Figure 2.4. These experiments were run with low B-vitamins at pH 4. Over the 40 day experiment the cell concentration increased from 0.4 to 1.2 g/L. As the cell concentration gradually increased, the ethanol concentration increased to about 15 g/L, while the acetic acid concentration remained about 3 g/L. Similar studies at high cell concentrations have resulted in eliminating acetic acid production. Therefore, high cell concentrations, coupled with nutrient limitations, result in high ethanol concentrations and minimal acetic acid production.

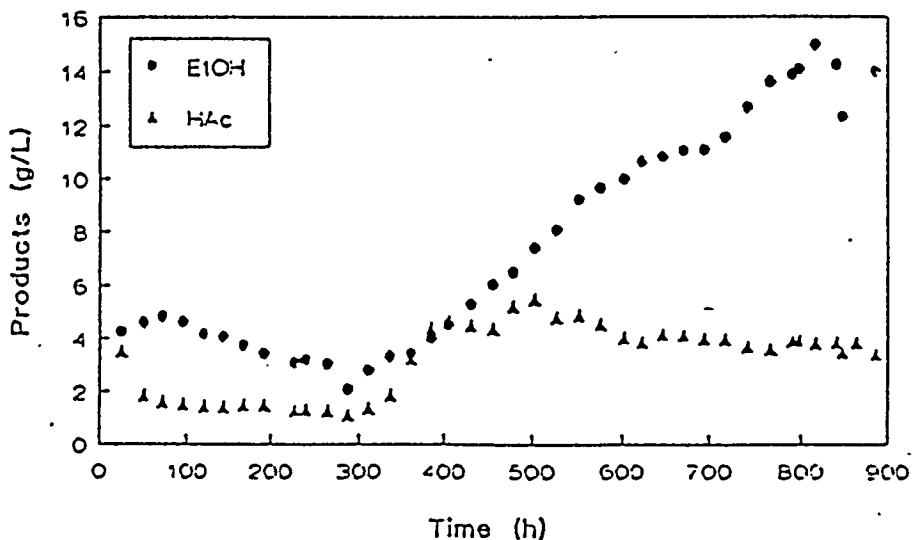


Figure 2.4. Ethanol and Acetate Concentration Profiles in the CSTR with Cell Recycle.

Experiments have also been conducted at various agitation rates in the CSTR. The results of increasing the agitation from 300 to 480 are shown in Figure 2.5. As is noted, at low agitation rates, the ethanol production was quite low. However, at higher rates ethanol becomes the predominant product. These experiments were not conducted with nutrient limitation and, consequently, the ratios of ethanol/acetate are low. However, the data illustrate a very important principle, i.e., when the system becomes mass transfer limited at low agitation rates, the ethanol productivity is low. This result will be discussed in more detail later, but indicates that ethanol may be consumed by *C. ljungdahlii* when the gaseous substrate becomes limiting.

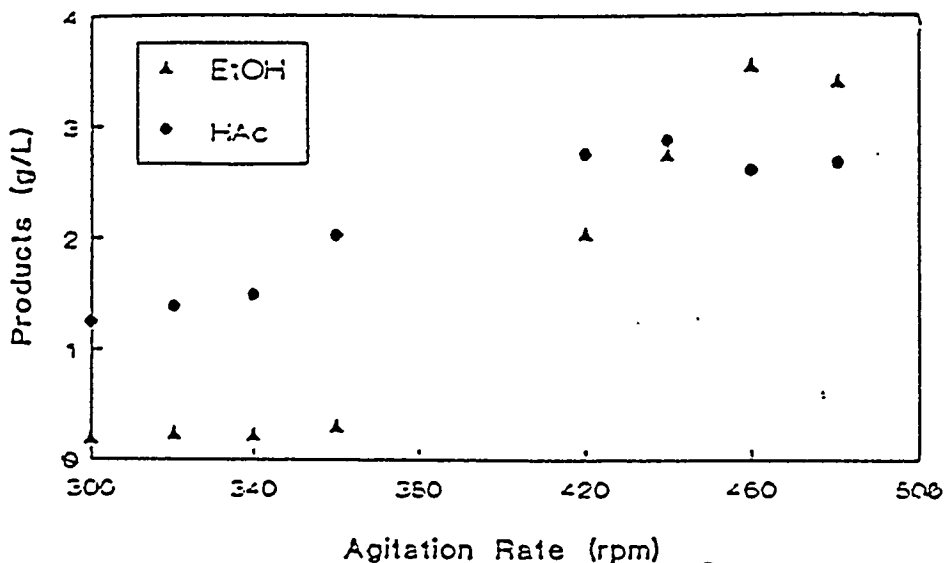


Figure 2.5. The Effect of Agitation Rate on Product Concentrations with C. ljungdahlii

2.2.1 Significance

Continuous reactors have been operated for extended periods with nutrient limitation to produce high ethanol concentrations and high ethanol/acetate ratios. High cell concentrations are essential to good performance. The ethanol productivity is maximized when adequate amounts of gaseous substrates are available to the culture at high agitation rates.

2.3 Effect of Sulfur Gases on C. ljungdahlii Performance

Coal synthesis gases will contain small quantities of sulfur gases, primarily H_2S and COS . These components cause rapid deactivation of chemical catalysts and must be removed. Many biochemical catalysts are less sensitive to these gases. Experiments to determine the tolerance of *C. ljungdahlii* to H_2S and COS were conducted with a culture not previously acclimated to sulfur gases. The effects of H_2S concentrations up to 10 percent on growth and gas consumption are shown in Figures 2.6 and 2.7. Concentrations up to 2.7 percent do not affect growth or gas utilization rates. There is a slight effect at 5.2 percent and no growth or gas utilization occur at 10 percent H_2S . Similar results were obtained for COS at the same concentration levels. The H_2S concentration in a high sulfur coal would not exceed 2 percent (Simbeck *et al.*, 1983). Also, the performance of the cultures will improve after acclimation to sulfur gases, and concentrations of 20 percent have been used in our laboratories with similar cultures with no adverse effects (Vega *et al.*, 1990; Smith *et al.*, 1991). Therefore, removal of sulfur from the synthesis gas is not necessary for use with *C. ljungdahlii*.

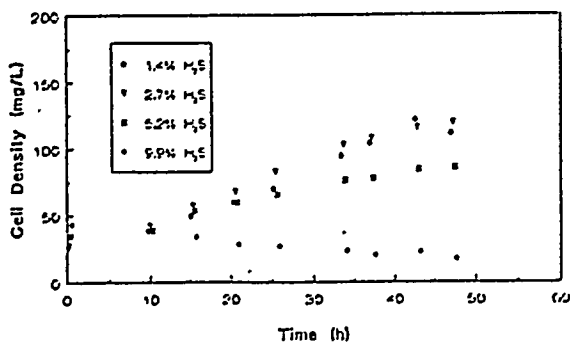


Figure 2.6. The Effects of H₂S on the Growth of *C. ljungdahlii*

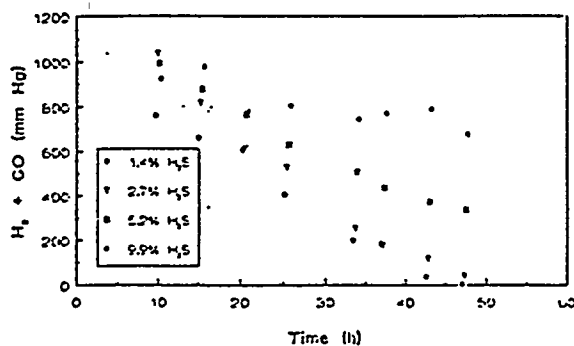


Figure 2.7. The Effects of H₂S on CO and H₂ Uptake by *C. ljungdahlii*.

2.3.1 Significance

C. ljungdahlii is not inhibited by H₂S or COS in concentrations in synthesis gas from high sulfur coal. Therefore, the raw synthesis gas can be introduced directly into the bioreactor, where a co-culture with another microorganism may be used for sulfur removal.

2.4 Effect of Pressure

Synthesis gas fermentations require the transport of the gaseous substrate from the gas phase, through the liquid phase, and into the solid microorganism for reaction. This heterogeneous system is generally mass transfer limited due to the very low solubilities of H₂ and CO. The transport rate (and the reaction rate), dN_s^G/dt , under mass transfer limited conditions is given by:

$$\frac{dN_s^G}{V_L dt} = \frac{K_L a}{H} P_s^G \quad (2.1)$$

where N_s^G = moles of gas transported from the gas phase
 V_L = liquid volume of the reactor
 t = time
 $K_L a$ = mass transfer coefficient
 H = Henry's law constant
 P_s^G = partial pressure of substrate in the gas phase

As noted in Equation (2.1), the reaction rate is directly proportional to the partial pressure of the gaseous substrates. Hence, the reaction rate and reactor size are proportional to total pressure in the fermenter. There will, of course, be an upper hyperbaric limit of pressure that the microorganisms can withstand. Also, the cell concentration must be increased as the transport rate is increased. A high pressure fermenter has been built and is now being tested. This system is being used to determine the upper limits of pressure for these fermentations and this maximum pressure will be used in this study.

Earlier experiments were conducted in our laboratories at pressures up to 10 atm and confirmed that the reaction rate is proportional to pressure up to this level (Ko *et al.*, 1989). Most

microorganisms will withstand much higher pressure and it is hoped that a pressure of 40 atm, equal to most gasifier operating pressures, can be achieved, which would reduce retention time to seconds.

2.4.1 Significance

The reaction rate and reactor volume are directly proportional to the total pressure in the bioreactor, up to an upper limit fixed by the hyperbaric restrictions of the cells. Experiments up to 10 atm have been successfully performed and higher pressure experiments are being conducted to determine the upper limits. The bench scale system to be used in these studies will be operated at this maximum pressure.

2.5 Other Isolates

BRI has isolated a number of other bacteria from natural sources that also produce ethanol by Equations (1.1) and (1.2). These new isolates have not yet been identified, but are not the same culture as *C. ljungdahlii*, as evidenced by substrate comparison studies, as well as microscopic analysis. At least two of these cultures, in fact, are superior to *C. ljungdahlii* in their ability to produce ethanol in favor of acetate. Isolate O-52 is particularly impressive in terms of CO uptake and tolerance, and its ability to produce high concentrations of ethanol.

2.6 Mechanism for Ethanol Production

Although *C. ljungdahlii* is the first microorganism to demonstrate the production of ethanol from CO, H₂ and CO₂, several other clostridial species produce ethanol from sugars (Rogers, 1986). In all cases, acetic acid, and sometimes lactic acid, are produced as by-products. The ethanologenic clostridia convert sugars to pyruvate via the fructosebiphosphate pathway producing two moles of ATP and two moles of NADH per mole of hexose. The majority of the pyruvate is converted to acetyl-CoA with small amounts going to lactate or CO₂ and H₂. Acetyl-CoA can then be reduced to acetaldehyde and then to ethanol, or it can be converted into acetate with stoichiometric production of ATP.

The production of ethanol requires reducing power, while the production of acetic acid generates energy for the cell. Studies with other clostridial species have found that ethanol productivity could be maximized by minimizing cell growth through manipulation of nutrients, such as phosphate and nitrogen (Bahl *et al.*, 1982; Bahl and Gottschalk, 1984; Gottschalk and Morris, 1981). It was concluded that solventogenesis (production of ethanol) is a metabolic response to unbalanced growth where a utilizable energy source is available, but growth factors are limited.

The pathway utilized by acetogenic bacteria and some clostridial species to autotrophically grow on CO₂/H₂ or CO was recently established as involving acetyl-CoA (Wood *et al.*, 1982). The mechanism involves the reduction of one molecule of CO₂ to a methyl group and then its combination with a second molecule of CO₂ (or a molecule of CO) and CoA to form acetyl-CoA. The reduction of CO₂ to a methyl group in the tetrahydrofolate pathway requires one molecule of ATP and one molecule of NADH per molecule of CO₂ reduced. It is important to note that the conversion of acetyl-CoA to acetate is the only source of substrate level phosphorylation in the acetogenic clostridia during unicarbonotrophic growth (Ljungdahl, 1983). When terminating in acetate, the pathway is

balanced in ATP and the production of ethanol would result in a net consumption of ATP, which would not support growth.

2.7 Summary of Research Needs

Since the discovery of *C. ljungdahlii* in 1987, significant progress has been made in developing a process to biologically produce ethanol from coal synthesis gas. Ethanol productivity has been substantially increased and the production of acetate nearly eliminated by controlling certain B-vitamins and pH. The viability of the culture to produce ethanol over extended periods has been demonstrated in the CSTR. High cell concentrations and good gas transport facilitate high ethanol productivity. The tolerance of the culture to high concentrations of sulfur gases has been demonstrated. The preliminary economics of the process for the biological production of ethanol are quite promising. This technology is nearing the stage of commercialization and requires only bench scale and prototype demonstrations prior to full scale operation.

This research project will seek to demonstrate this process at the bench scale for an extended period of operation. The new conceptual design to remove ethanol as it is produced should lead to even higher ethanol productivities. This system, including bioreactor, cell recycle, solvent extraction, and ethanol distillation, must be operated continuously at the bench level for a prolonged period to measure the ethanol productivity and demonstrate the viability of the culture and product recovery system. The best culture must first be selected for these experiments. High cell concentrations will be achieved by use of a hollow fiber membrane to retain cells in the bioreactor. Fast gas transport is essential and the bioreactor will be operated at the optimal pressure found in current experiments. Appropriate solvents for ethanol recovery must be screened for high distribution coefficient and compatibility with the culture. Parameters for scale-up of this system to prototype and commercial facilities must be developed. Design and economic projections must be prepared for analysis and to guide the development efforts.

3.0 ECONOMIC PROJECTIONS

Economic projections for a plant to process 1500 tons per day of coal to ethanol have been prepared based upon the results of the research program. Figure 3.1 shows an updated flow diagram for the process including both acetic acid and ethanol recovery. Acetic acid is recovered by extraction and distillation, and ethanol is recovered by distillation and pervaporation. This plant is projected to produce 85 million gallons of ethanol and 15 million pounds of acetic acid per year.

Table 3.1 presents the capital cost summary for the plant without electricity generation, and Table 3.2 presents an economic analysis. As is noted, the plant requires a capital investment of \$36 million, including a boiler, fermenters and facilities for acid and ethanol recovery. The total operating costs including raw materials, utilities, labor, overhead, maintenance, taxes and insurance and depreciation are \$53.5 million/yr. With revenue from ethanol and acetic sales totaling \$114 million/yr, a pre-tax profit of \$60.2 million/yr results. After taxes, the net profit is \$36.1 million/yr which results in a cash flow of \$39.7 million/yr. The return on investment is 110 percent and the payout is 0.9 years.

Tables 3.3 and 3.4 present the capital cost summary and economic analysis for the process with electricity generation. The capital costs increase slightly to \$39.4 million, and the total operating costs decrease to \$52.4 million/yr. The cash flow is \$44.6 million/yr, the return on investment is 113 percent and the payout is 0.88 years.

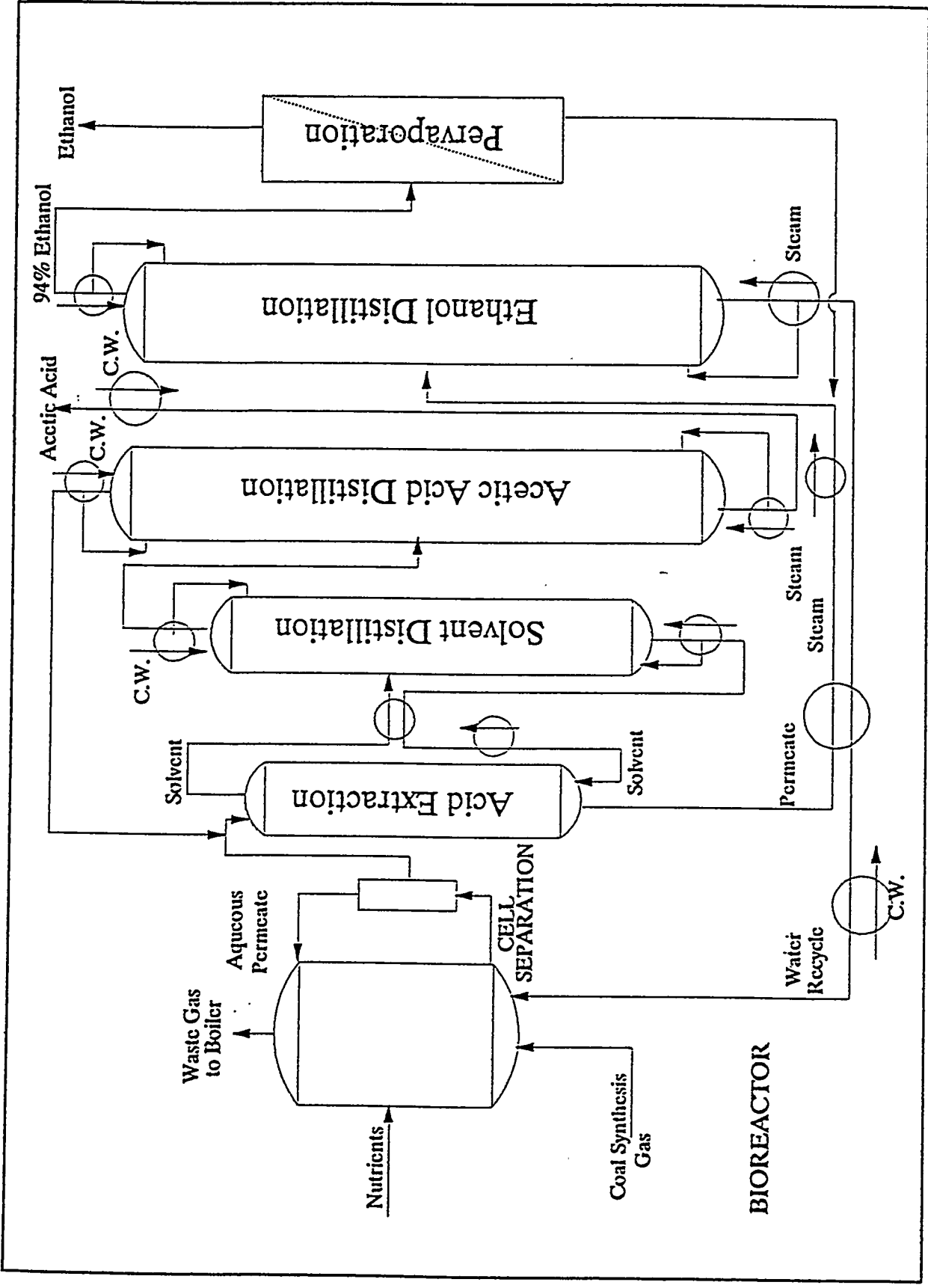


Figure 3.1 Process for Ethanol and Acetic Acid Production from Coal Synthesis Gas

Table 3.1. Capital Cost Summary
1500 tons/day coal - 85 million gal/yr ethanol
No Electricity Generation

System	Installed Capital Cost, \$
Reactor System	8,223,000
Cell Recycle System	1,731,000
Acetic Acid Extraction	2,334,000
Solvent Distillation	3,234,000
Acetic Acid Distillation	1,062,000
Ethanol Distillation	8,688,000
Pervaporation Unit	4,374,000
Pervaporation SubSystem	2,544,000
Waste Heat Boiler	3,821,000
Total Fixed Capital Investment	36,011,000

**Table 3.2. Economic Evaluation
1500 tons coal/day - 85 million gallons ethanol/yr
No Electricity Generation**

	Annual Cost, M\$
Revenue	
Ethanol, \$1.30/gallon	104,356
Acetic Acid, \$0.28/lb	4,273
Steam, \$2.00/MM Btu	5,099
Total Revenue	113,728
Raw Materials/Supplies	
Coal Synthesis Gas, \$1.00/1000 SCF	41,160
Medium, \$9.00/M ³	2,598
Process Water, \$0.20/M gallon	2
Solvent Makeup	241
Total Materials Cost	44,001
Utilities	
Electricity, \$0.05/kwhr	1,929
Cooling Water, \$0.05/M gallon	678
Total Utilities	2,607
Labor	378
Overhead & Supervision	378
Depreciation, 10% FCI	3,601
Maintenance, 5% FCI	1,801
Insurance, 2% FCI	720
Total Operating Cost	53,486
Pretax Profit	60,242
Income Taxes (40%)	24,097
Net Profit	36,145
Cash Flow	39,746
Return (%)	110.37
Payout Period (years)	0.91

Table 3.3. Capital Cost Summary
1500 tons/day coal - 85 million gal/yr ethanol
With Electricity Generation

System	Installed Capital Cost, \$
Reactor System	8,223,000
Cell Recycle System	1,731,000
Acetic Acid Extraction	2,334,000
Solvent Distillation	3,234,000
Acetic Acid Distillation	1,062,000
Ethanol Distillation	8,688,000
Pervaporation Unit	4,374,000
Pervaporation SubSystem	2,544,000
Waste Heat Boiler	1,735,000
Generator	5,520,000
Total Fixed Capital Investment	39,445,000

**Table 3.4. Economic Evaluation
1500 tons coal/day - 85 million gallons ethanol/yr
With Electricity Generation**

	Annual Cost, M\$
Revenue	
Ethanol, \$1.30/gallon	104,356
Acetic Acid, \$0.28/lb	4,273
Electricity, \$0.05/kwhr	11,594
Total Revenue	120,223
Raw Materials/Supplies	
Coal Synthesis Gas, \$1.00/1000 SCF	41,160
Medium, \$9.00/M ³	2,598
Process Water, \$0.20/M gallon	2
Solvent Makeup	241
Total Materials Cost	44,001
Utilities	
Cooling Water, \$0.05/M gallon	678
Total Utilities	678
Labor	504
Overhead & Supervision	504
Depreciation, 10% FCI	3,945
Maintenance, 5% FCI	1,972
Insurance, 2% FCI	789
Total Operating Cost	52,393
Pretax Profit	67,830
Income Taxes (40%)	27,132
Net Profit	40,698
Cash Flow	44,643
Return (%)	113.18
Payout Period (years)	0.88

4.0 CONCLUSIONS

The economics of converting coal to ethanol by a biological process is quite attractive. When processing 1500 tons of coal per day, the plant generates 85 million gallons of ethanol per year. The return on investment for the process is 110 percent and the payout is 0.9 years.

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