Membrane Technology in Carbon Dioxide Removal

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Summary

This study assessed the feasibility of membrane technology in carbon dioxide removal processes. The study was carried out in the framework of the dutch national programme on carbon dioxide removal. In this programme a number of gas streams, typical of gas streams occurring in future and present electricity generating plant, were defined. They consisted of flue gas from a coal fired plant, process gas from a plant incorporating a coal gasification unit and process gas from a plant incorporating a gas reformer. The described separation processes focused on mixtures of carbon dioxide and nitrogen and carbon dioxide and hydrogen.

Two possible applications of membrane technology were examined in detail, i.e. gas absorption membranes and gas separation membranes. Gas absorption membranes are used in conjunction with conventional absorption liquids where the essential element is the replacement of a conventional absorber by membranes. The quality of the separation will be depend on the absorption liquid. In case of gas separation membranes the intrinsic properties of the membrane material cause the separation of species. The driving force in the latter case is a pressure difference across the membrane.

The implementation of gas absorption membranes has several advantages over conventional absorber technology. Current membrane technology is useable in gas absorption processes at atmospheric pressure (flue gases). It will require, however, a module design different from the presently available modules in order to accommodate the high gas flows. At elevated pressures modifications to the modules will be necessary, as well as a process modification because the allowed pressure difference across the membrane is limited. The latter problem might also be solved by the use of dense membranes instead of microporous membranes.

The prospect for the use of gas separation membranes is less favourable due to the low selectivities of currently available membranes and the high costs of compression. Several options combining a conventional carbon dioxide removal process and gas separation membranes were compared with the conventional process alone. It appeared that energy consumption per mole carbon dioxide removed of the conventional process was always less. The application of currently available gas separation membranes to improve a conventional process appeared to be feasible in terms of energy consumption only if the selectivity of the membranes could be increased ten-fold. At present this ten-fold increase has already been realised at laboratory scale and therefore it can be expected that gas separation membranes will play a more important role in the future.

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1 Introduction

The carbon dioxide content in the atmosphere has risen considerably since the advent of the industrial revolution. It is widely believed that a continuation of this trend will lead to severe climatological changes. This is due to the fact that carbon dioxide absorbs infrared radiation, thereby trapping heat in the atmosphere. Lowering or stabilising the carbon dioxide content in the atmosphere appears to be of the utmost importance.

Reduction of atmospheric carbon dioxide emissions can be achieved by a wide variety of measures. One of these measures is the removal and subsequent storage of carbon dioxide. Carbon dioxide removal techniques are widely used in the ammonia plants and natural gas separation. The present study considers the use of membranes in carbon dioxide removal.

In the framework of the dutch national programme on carbon dioxide removal and storage four gas streams, typical of those occurring in present and future fossile fuel based electricity generating plant, have been defined. From these gasstreams, described in detail in Appendix 1, carbon dioxide needs to be removed. They are:

1. Flue gas from a coal-fired electricity generating plant in which nitrogen is the major component and water and carbon dioxide are the minor components.
2. Process gas in a coal gasification plant in which hydrogen, carbon dioxide and water are present in similar proportions.
3. Process gas from reformer after CO-shift in a gas-fired electricity generating plant in which hydrogen is the major component and water and carbon dioxide are the minor components. Of this gasstream two variations, differing in pressure level and slightly differing in composition are given.

In this report calculation results of carbon dioxide removal processes using membrane technology will be discussed, using the gasstreams mentioned above. The carbon dioxide product stream needs to possess a purity in excess of 95% and a extremely low water content (see Appendix 1). This necessitates special attention to the dehydration of the product carbon dioxide gas. Also it has to be delivered at pressure level of 110 bar in order to be fed into a pipeline transportation network.

2 Membrane technology

Membrane technology first became important during the sixties and seventies in the field of water treatment. Processes like reverse osmosis, ultrafiltration and electrodialysis are examples of this development. During the eighties membrane technology also entered the field of gas purification. In the case of carbon dioxide removal two membrane operations seem to be relevant, i.e. gas absorption and gas separation.

2.1 Gas Absorption Membranes

Gas absorption using membranes is a separation technique which was developed fairly recently. The essential element in the process is a microporous hydrophobic membrane as a result of which the gas phase remains separated from the liquid absorbent. The process is illustrated in Figure 1. A gas stream is fed along one side of the membrane. The components to be removed from the gas stream will diffuse through the gas filled pores of the membrane. On the other side of the membrane they will be absorbed in the liquid.

The main operational advantages of membranes over conventional gas/liquid apparatus are:

- Gas- and liquid flow independent;
- No entrainment, flooding, channelling or foaming;
- Compact apparatus through the use of hollow fibre membranes.

The separation characteristics of the process are determined by the choice of absorbent. Therefore the energy- and mass balance will be identical to a conventionally operated absorption process. A list of carbon dioxide absorbing fluids is given in section 3. All of these have high selectivities for carbon dioxide over other gases like hydrogen, carbon monoxide, nitrogen and methane. Hence, they are in principle suitable to treat any of the gas streams given in Appendix 1. For dehydration purposes triethyleneglycol appears to be the most suitable absorption liquid.

Gas absorption membranes are usually operated at atmospheric pressure and the transmembrane pressure should not be higher than approximately 1 bar. Operation at higher pressure levels seems possible but has yet to be demonstrated to be feasible. In theory there does not appear to be a constraint to operate gas separation membranes at elevated pressures as long as the transmembrane pressure is not allowed to exceed 1 bar. Obviously, the membrane module housing needs to be altered to accommodate the higher absolute pressures.

2.2 Gas Separation Membranes

Gas separation using membranes relies on differences in solubility and diffusion of gases in polymers or other type of membrane materials. The permeability P of a material for a certain gas is defined as:

$P = D * S$
where D: diffusion coefficient
S: solubility

The driving force for this separation process is a difference in partial pressure over the membrane (see Figure 2). Unlike membrane based gas absorption, in membrane based gas separation the properties of the membrane result in a separation of species. The separation characteristics of a binary mixture can be expressed in terms of a selectivity, i.e. the quotient of the respective permeabilities. Commercial gas separation membranes usually have an asymmetric structure, consisting of an ultrathin top layer made from a polymer and a support layer. The thin top layer will result in high fluxes. Very often these fluxes give rise to a considerable pressure drop in the membrane support layer. The resulting loss in driving force leads to a decrease in selectivity of the membrane compared with the characteristics of the polymer.

For the flue gas and process gas mixtures given in Appendix 1 the desired separations are carbon dioxide-nitrogen (flue gas in coal fired plant), carbon dioxide-hydrogen (process gas in coal gasification and reforming of natural gas) and to a lesser extent, hydrogen-carbon monoxide/methane (reforming of natural gas). Apart from these separations one might consider the enrichment of oxygen in the air to improve the combustion (oxygen-nitrogen separation).

2.2.1 Separation of Carbon Dioxide and Nitrogen

Table 1 lists a number of polymers which are suited to establish the separation of carbon dioxide and nitrogen.

Table 1 Polymer materials suitable for carbon dioxide nitrogen separation and typical selectivity values at room temperatures [SLU91]

Polymer	CO_2-N_2 Selectivity
Polyphenylene-oxide	19
Cellulose Acetate	66.5
Polysulfone	30
Polyimide	23.3

All of the polymers listed in Table 1 have been incorporated into commercial modules and the carbon dioxide - nitrogen membrane separation process is thus based on currently available technology. Section 4 will discuss whether or not it is economical to employ gas separation membranes in carbon dioxide removal.

2.2.2 Separation of Carbon Dioxide and Hydrogen

Table 2 lists polymers selective to carbon dioxide over hydrogen.

Table 2 Carbon dioxide selective polymer materials suitable for carbon dioxide hydrogen separation and typical selectivity values at room temperatures [EGL84, PAU89, HWA74]

Polymer	CO_2-H_2 Selectivity
Polybutadiene	3.3
Polydimethylsiloxane	5.1
Polyisopren	2.6
Polyethylene	3.3
Vulcaprene	3.0

Of the polymers listed in Table 2 polydimethylsiloxane (PDMS or silicon rubber) looks the most promising. The material is being used in commercial membrane modules and can be regarded as state of the art technology.

Table 3 lists polymers selective to hydrogen over carbon dioxide.

Table 3 Hydrogen selective polymer materials suitable for carbon dioxide - hydrogen separation and typical selectivity values at room temperature [EGL84, PAU89, HWA74]

Polymer	H_2-CO_2 Selectivity
Teflon	4.3
Polyvinylchloride	4
Polypropylene	4.5
Polamide	6.7
Polyimide	10

Polyimide looks the most promising membrane material. It is being used in commercial membrane modules and as a consequence can be regarded as state of the art technology.

Table 2 and 3 show fairly low selectivities for the carbon dioxide - hydrogen separation problem. It is therefore impossible to achieve the desired carbon dioxide purity and recovery in a single stage process with presently available gas separation polymer membrane technology. Nevertheless gas separation membranes may still be important for this particular separation problem as a carbon dioxide concentration technique prior to a carbon dioxide removal unit.

2.2.3 Separation of Hydrogen and Methane/Carbon Monoxide

This separation has received much attention in recent years in the following areas [SPI89]:

— Adjustment of H_2-CO ratio of synthesis gas in the petrochemical industry;
— Recovery of H_2 from purge streams in the ammonia industry and refineries.

Selectivities of the membranes can be in excess of 100 with hydrogen being the fast gas. In the framework of this study there is a need for methane/carbon monoxide rich stream in case of the natural gas fired electricity plant to fuel the reformer. As hydrogen is the fast gas and thus will be at a lower pressure after the gas separation it does not seem of interest to use gas separation membranes in this case.

2.2.4 Separation of Oxygen and Nitrogen

The production of purified nitrogen and oxygen from air using membranes is an important area of research. Current membrane technology has selectivities between 3.5 and 5.5 with oxygen being the fast gas. Using gas separation membranes one can achieve oxygen concentrations of up to 50% in a single stage. Coal gasification plants, however, use a mixture in which the oxygen molar fraction needs to be at least 95%. A multistage membrane process or hybrid process needs to be employed to achieve this.

3 Conventional carbon dioxide removal techniques

It has appeared from Section 2 that gas separation membranes are not very selective, especially for the carbon dioxide -hydrogen separation. For this reason it must be expected that gas separation membranes alone are not capable to produce carbon dioxide economically at the desired purity. Nevertheless they could play a role in a hybrid plant, e.g. a condensation unit in which the incoming stream is first concentrated by membranes. In addition to this, gas absorption membranes also need conventional carbon dioxide removal technology i.e. absorption processes. As a consequence it has become imperative to discuss these conventional carbon dioxide removal techniques.

Conventional carbon dioxide removal techniques are based on chemical and/or physical absorption. Carbon dioxide is absorbed by a solvent at low temperature and/or high pressure and released at high temperature and/or low pressure. Typical solvents used are:

- Chemical:
    - Monoethanolamine (MEA)
    - Di-ethanolamine (DEA)
    - Tri-ethanolamine (TEA)
    - Hot Potassium Carbonate (Benfield)
    - Hot Sodium Carbonate
- Physical:
    - Methanol (Rectisol)
    - N-methyl-2-pyrrolidone (Purisol)
    - Polyethylene Glycol (Selexol)
    - Propylene Carbonate (Fluor Solvent)
- Physical/Chemical
    - Sulfolane/Diisopropanolamine/Water (Sulfinol)

Figure 3 [ULL83] shows roughly the operating regimes of several solvents. Chemical solvents are mostly used in case of low carbon dioxide content in the feed and high purity carbon dioxide of the product. In case of high carbon dioxide content feed gas and lower purity requirements physical solvents are favoured. Following Figure 3 one can now choose the preferred conventional processes for each of the gasstreams defined in Appendix 1:

1. Flue gas from coal fired electricity generating plant: Amines,
2. Process gas from a coal gasification plant: Physical solvent,
3. Process gas in gas fired electricity plant after CO-shift: Hot potassium carbonate, concentrated amines, a physical solvent or a mixture of these.

Apart from these absorption techniques one might also consider direct condensation of carbon dioxide from gas mixtures by compression and/or cooling. This is favourable if the carbon dioxide content of the gas mixture is fairly high. Also the solubility of the other gases present in the mixture in liquid carbon dioxide is of importance as this might prevent one from reaching the desired carbon dioxide purity. Figure 4 shows the saturation vapour pressure curve of carbon dioxide. Cooling below the triple point (-56.6 ^{o}C) will result in solid carbon dioxide being formed. Operation above the critical temperature (31.1 ^{o}C) of carbon dioxide will not result in condensation. Hence the operation of the condensation unit should be limited to the temperature region between, say, -50 ^{o}C and +30 ^{o}C. In the final design of the condensation unit one has to take into account that carbon dioxide will be condensing from a mixture of gases. The thermodynamic properties of the mixture will be different from the pure substances.

4 Carbon dioxide removal from flue gases using membranes

4.1 Gas Absorption Membranes

Due to the low partial pressure of carbon dioxide in flue gases a MEA absorption process is the most economical. The process flow diagram is shown in Figure 5. Replacing the absorber by gas absorption membranes will not change the high energy requirement of the process but will have the additional advantages mentioned in Paragraph 2.1. The required membrane area is estimated to be 250000 m^2 based on hollow fibre membranes (internal diameter = 0.6 mm, length = 0.2 m, gas velocity through fibres = 3 m/s). These fibres are at presently available commercially and cost Dfl 1000 per m^2. The total cost of the membranes (Dfl $250*10^6$) is comparable to the costs of a complete absorption unit according to the costs given by Blok et. al. [BLO89]. As one might expect the membrane cost to drop significantly if the production volumes increase, a membrane based absorption unit could have an edge in terms of cost-effectiveness. Currently, dialysis membranes are produced in large volumes costing Dfl 50 per m^2. Using this price for membrane area the total cost for the absorber is reduced to Dfl $12.5*10^6$. The impact on the cost per unit carbon dioxide avoided, however, is limited as the contribution of the absorber to the total cost is around 5%.

4.2 Gas Separation Membranes

Van der Sluijs et. al. [SLU91] have considered the use of commercially available gas separation membranes in the recovery of carbon dioxide from flue gas. It appeared that with a single stage the desired carbon dioxide purity could not be reached. A two stage system seemed to be able to produce high purity carbon dioxide at the required recovery but a cost which was at least double the cost of a conventional MEA-absorption process. This was mostly due to the high energy input needed to compress the gas flows. Finally, they investigated a hybrid system in which the carbon dioxide was recovered by condensation. This system still gave rise to a cost per unit avoided carbon dioxide emission which was 50% higher than a conventional MEA-absorption process. Despite the favourable membrane selectivities of the carbon dioxide - nitrogen separation, gas separation using membranes is uneconomical due to high energy requirements of the compressors. Depending on required purity, recovery etc. it is expected that gas separation membranes with CO_2/H_2-selectivities in excess of 200 will be competitive with conventional amine absorption technology. This represents a ten-fold increase in selectivity compared to the currently available membranes.

5 Carbon dioxide removal from coal gasification process gas

5.1 Conventional Carbon Dioxide Removal Techniques

The carbon dioxide partial pressure in the coal gasification process gas is such that there are two conventional methods which appear to be well suited to perform the task of removing the carbon dioxide, i.e. direct condensation of process gas stream and physical absorption. The general specification for the carbon removal unit is given in Figure 6. For reasons of simplicity it was assumed that both processes were capable of producing pure carbon dioxide. In practice the product carbon dioxide stream will be contaminated but it is expected that the purity will be in excess of 95 vol%. In Figure 6 it was also assumed that the water vapour present in the feedstream was removed by cooling to ambient temperature.

5.1.1 Direct Condensation by Cooling and/or Compression

If, for example, the feed stream is cooled to -44 ^{o}C at the given pressure of 77 bar the amount of condensated carbon dioxide will meet the recovery requirements. The energy requirement for this cooling process will be equal to 13.6 kJ$_e$/mol CO_2 (see Appendix 2). The low temperature of the gas stream will probably remove the need of an extra dehydration stage.

5.1.2 Physical Absorption

The partial pressure of carbon dioxide at ambient temperature will be equal to 31.6 bar. A physical absorption process (e.g. Selexol) will benefit from this high carbon dioxide partial pressure. As a first approximation one might regard the absorbent recirculation rate and thus the energy requirement per mole carbon dioxide to vary inversely proportional to the carbon dioxide partial pressure. Extrapolation of the energy requirements per mole carbon dioxide given in SHA88/1 and HEN90 gives for the carbon dioxide partial pressure in this stream 1.3 kJ$_e$/mole CO_2 if the rich absorbens is flashed to atmospheric pressure resulting in a near complete carbon dioxide recovery. Flashing to say 5 bar will result in 70% recovery of carbon dioxide and a energy requirement of 1 kJ$_e$/mole CO_2 [SHA88/2]. As the carbon dioxide is to be delivered a pressure of 110 bar the compression energy requirements have to be added to the energy requirement for the separation process. These are, respectively, 15.3 kJ$_e$/mole CO_2 for compression from 1 to 110 bar resulting in a total energy requirement of 16.6 kJ$_e$/mole CO_2 and 10.2 kJ$_e$/mole CO_2 for compression from 5 to 110 bar resulting in total energy requirement of 11.2 kJ$_e$/mole CO_2.

It appears that the energy requirements per mole carbon dioxide removed for the condensation process and for the physical absorption process are roughly similar.

5.2 Gas Absorption Membranes

Chemical absorption techniques are ruled out because of the high carbon dioxide partial pressures. This leaves us with a physical absorption as described in Section 5.1. Although some reservations have to be made regarding the technical feasibility of the combination of a physical absorption process and gas absorption membranes (see Section 2.1) one can make some tentative calculations as regards the required membrane area.
The estimated membrane area is 9000 m^2 based on hollow fibre membranes (internal diameter = 0.2 mm, length = 0.2 m, gas velocity through fibres = 1 m/s). These hollow fibres are at present available and cost Dfl 1000 per m^2. This will lead to a total cost of Dfl $9*10^6$ for the membranes. Production volume increases can be expected to lead to a ten-fold decrease in membrane cost. It must be stressed that these calculations are of a preliminary nature as the application of gas absorption membranes at high pressure has yet to be demonstrated on the laboratory scale.

5.3 Gas Separation Membranes

Despite the presence of a considerable driving force in the process gas stream to operate a gas separation membrane, carbon dioxide removal based on this technique alone was not considered to be economical. This is due to the low membrane selectivities for CO_2/H_2 and vice versa (see Paragraph 2.2.2). It would involve a plant with several stages involving many compression steps and therefore the energy consumption of this process would prohibit economical operation.

Next the attention was focused on a hybrid plant, i.e. a plant in which the gas separation membranes were combined with a condensation unit or a physical/ chemical absorption unit (Figure 7). The object of the gas separation membranes was to separate the feed stream into a stream enriched in carbon dioxide and a stream enriched in hydrogen. As a result of this it was thought that it was easier to recover the carbon dioxide.

In Figures 8 and 9 the permeate molar fractions of, respectively, carbon dioxide and hydrogen are given as a function of the stage cut (= permeate mole flux through membrane / feed mole flux), for three different pressure ratios r_{pf} (=permeate pressure / feed pressure). These calculations are based on the cocurrent gas permeation model given by Shindo et. al. [SHI85] and are based on permeabilities for PDMS given by manufacturers (GKSS). It appears that although an enrichment in carbon dioxide does occur the permeate flux is rather high, resulting in a considerable hydrogen loss. It is therefore necessary to recover the hydrogen after the carbon dioxide removal unit. This is illustrated by Figure 10 and 11 which show, respectively, the total carbon dioxide permeate flux and the total hydrogen permeate flux.

5.3.1 Hybrid Plant involving Chemical Absorption

Carbon dioxide mole fractions in the permeate in case of a pressure ratio of 0.1 (absolute pressure = 7.7 bar) are such that only chemical absorption can be used to remove the carbon dioxide. The energy consumption of these processes is between 130 and 200 kJ_{th}/mole CO_2 which makes them uncompetitive compared with the two options given in Paragraph 5.1. At higher pressure ratios chemical absorption is not feasible because of the high carbon dioxide partial pressure.

5.3.2 Hybrid Plant involving Physical Absorption

The carbon dioxide partial pressure of the permeate is always lower than the carbon dioxide partial pressure of the feed which makes a physical absorption process more energy intensive compared with a physical absorption process operating directly on the feed stream. In order to raise the carbon dioxide partial pressure one must compress the permeate to the feed pressure level. An increase in the carbon dioxide partial pressure will lead to lower energy consumption per mole carbon dioxide of the physical absorption. Also, compression of the permeate allows the recovery of the lost hydrogen. The process flow diagram is shown in Figure 12. Table 4 shows the permeate flux above which there is sufficient carbon dioxide present in the permeate to meet the recovery requirements for three pressure ratios. It is assumed that all of the carbon dioxide present in the permeate can be recovered. Also shown is the recompression energy requirement in relation to moles carbon dioxide recovered.

Table 4 *Stage cut, carbon dioxide molefraction and recompression energy requirement (to 77 bar) for three pressure ratios for a carbon dioxide recovery equal to 81.8%*

Pressure ratio r_{pf}	Permeate flux	Carbon dioxide molefraction	Compression energy [kJ_e/mole CO_2]
0.1	0.551	0.610	12.19
0.2	0.600	0.560	9.34
0.5	0.728	0.462	4.83

Using the tentative calculations in Paragraph 5.1.2 one may expect a reduction of less 50% in the energy requirement for the physical absorption process. Since this was estimated to be around 1 kJ_e/mole CO_2 this energy saving is dwarfed by the recompression energy requirement. Incorporating a gas separation membrane into a physical absorption unit leads to a higher energy consumption due to the recompression energy requirements of the permeate.

5.3.3 Hybrid Plant involving a Condensation Unit

Figure 13 shows a process flow diagram of a hybrid plant in which the permeate flow of a gas separation membrane unit is recompressed and fed to a condensation unit. The condensation unit works by cooling only and is chosen to operate at 77 bar which allows the gaseous exit stream (mainly hydrogen) to be merged with the retentate of the membrane unit. The gas separation membrane unit will separate the feed stream into two streams, one enriched in hydrogen, the other enriched in carbon dioxide. As a result of this separation it was thought that savings could be made on the energy consumption of the cooling unit. Figure 14 shows the cooling temperature necessary to achieve the required recovery, as a function of the stage cut g (r_{pf}=0.1, 0.2). It appears that, compared with direct cooling of the feedstream (Paragraph 5.1.1) the gain in cooling is minimal for a pressure ratio equal to 0.1. For a pressure ratio equal to 0.2 one has to employ even lower cooling temperatures compared to direct cooling of the feedstream in order to achieve the required recovery. Taking this into account it must be concluded that the plant shown in Figure 13 must not be regarded as feasible with the CO_2/H_2 selectivities of current gas separation membrane. In fact it appears that the CO_2/H_2 selectivities should be increased to 150 in order to avoid the use of a cooling unit. Compression of the permeate to 77 bar would then result in condensation of sufficient carbon dioxide to meet the recovery requirements.

Figure 15 shows a second option of a carbon dioxide removal plant incorporating a cooling unit and a membrane based gas separation unit. This type of plant is suited for flows whose properties are such that direct condensation is fairly easy. The feedstream is cooled and part of the carbon dioxide will condensate. The remaining gas stream is fed to the gas separation membranes where the resultant permeate is enriched in carbon dioxide. This stream is recompressed and fed to the entrance of the cooling unit. The object of the gas separation membrane unit is to save on the energy requirements of the cooling unit. Figure 16 shows the energy consumption per mole CO_2 needed for cooling and permeate recompression as a function of the membrane flux for a pressure ratio equal to 0.2. These results are based on permeability data obtained from manufacturers (GKSS). It appears that there is a reduction in cooling energy requirement but that this reduction is offset by the requirements of the permeate recompression. As an example Figure 17 shows the total energy requirement per mole CO_2 for the same plant design but assuming a CO_2/H_2 selectivity of 50. It appears that with these membrane properties the energy consumption per mole CO_2 is reduced.

5.3.4 The Bi-Membrane Module

Conventional membrane modules contain one type of membrane which possesses a selectivity towards one component of a binary gas mixture.

Along the length of the module the concentration of the preferentially permeating component will be reduced. As a result of this, two effects, detrimental to the performance of the separation process, occur:

- the driving force for the fast component decreases along the length of the membrane module due to the decreased concentration of the permeating component, giving rise to a reduction in flux;
- the permeate concentration of the slow component will increase.

These effects are very pronounced in case of membranes possessing a low selectivity. CO_2/H_2 mixtures for which membrane selectivities vary between 5 and 10 are a typical example of this problem.

Stern et. al. [STE84] proposed the bi-membrane module to resolve this. If one manufactures a membrane module with two types of membranes of opposite selectivities it appears to be possible to maintain the concentrations in the retentate. A loss in driving force will therefore not occur. As a result the two permeate streams will be purer when compared with a single membrane module. This type of membrane module, using a silicon rubber and a poly-imide membrane is well suited to perform the carbon dioxide - hydrogen separation. Table 5 shows results from some calculations for this module.

Table 5 Permeate flux (related to feed flow) and purity (molar fraction) of a bi-membrane module based on a CO_2/H_2 mixture (molar fractions: 0.44, 0.56, respectively), feed pressure = 77 bar

r_{pf}	CO_2-rich		H_2-rich	
	permeate flux	CO_2-molar fraction	permeate flux	CO_2-molar fraction
0.05	0.51	0.79	0.49	0.08
0.1	0.51	0.78	0.49	0.09
0.5	0.57	0.63	0.43	0.19

It follows from table 5 that, compared to a single membrane module, the carbon dioxide concentration is higher for a given pressure ratio. Nevertheless, the purity is insufficient to allow direct compression and delivery to the gate. This means that a separate carbon dioxide removal unit is still necessary to obtain the carbon dioxide at the purity desired (>0.95). The design of this unit will be similar to the units discussed in Paragraphs 5.3.2 and 5.3.3. In these paragraphs it was concluded that the energy savings possible due to the carbon dioxide concentration increase were offset by the energy expenditure due to recompression. In addition to this one has to take into account that also the hydrogen will be available at low pressure. This will lead to a much reduced power output of the electricity plant.

6 Carbon dioxide removal from a gas reformer plant

6.1 Conventional Carbon Dioxide Removal Techniques

Cooling of the two streams given in Appendix 1 to ambient temperature will lead to carbon dioxide partial pressures of 4.4 bar and 3.3 bar respectively. At these levels a high performance chemical absorption technique or a mixed physical/chemical absorption technique appears to be the most suitable choice.

6.2 Gas Absorption Membranes

Absorption can in principle be carried out by gas absorption membranes with the advantages described in Section 2.1. Present gas absorption membranes, however, are designed to operate at near atmospheric pressure and ambient temperature. Also the trans-membrane pressure should not be allowed to exceed 1 bar. Therefore, the application of gas absorption membranes in this stream is possible with changes in module- and process design which allow the membranes to be operated at the given pressure levels (23.5 bar, 18 bar respectively).

6.3 Gas Separation Membranes

Gas separation membranes can be used to separate the incoming gas stream into two streams, one of which is enriched in carbon dioxide, the other enriched in hydrogen. Figure 18 shows the permeate mole fractions of carbon dioxide for several pressure ratios as a function of the stage cut (membrane flux relative to the incoming stream). Due to the low CO_2/H_2-selectivity the enrichment at the high recovery requirements specified is only limited. Figure 19 gives the hydrogen flux through the membranes relative to the incoming gas stream. This flux is quite considerable which means that recompression to the feed pressure levels is necessary to avoid a loss in hydrogen. This will require a substantial amount of energy.

The carbon dioxide partial pressure of the permeate is such that only a chemical absorption process (MEA) will be useable to recover the carbon dioxide. As these processes are more energy intensive than the processes chosen for treatment of the original stream, there does not seem to be any benefit in employing a gas separation membrane.

7 Miscellaneous topics

7.1 Dehydration

The product specification as regards the water content is less than 10 vppm. This is equivalent with a dewpoint of -20 ^{o}C at the pressure of 110 bar. With the exception of a cooling process, every carbon dioxide removal process will need a separate unit to meet this requirement. Triethylene glycol (TEG) is an often used absorbent for dehydration purposes. Instead of using a conventional absorber one can opt for a gas absorption membrane. The process flow diagram is shown in Figure 20. It is assumed that the product carbon dioxide is first compressed to 55 bar to allow the bulk of the water to be condensated out. It is not economical to compress further because this will lead to an increase in the water content of the product carbon dioxide [HEN91, PRI84]. This stream is the input stream for the gas absorption process. Water will be absorbed by the TEG and the temperature of the absorbent will lead to a temperature increase. The resulting heat is exchanged with the lean TEG entering the top of the gas absorption membranes. TEG is stripped of its water by a hot air stream. The total heat requirement of the process is 116 kW. Based on a flow velocity of 3 m/s through the fibres the estimated area is 165 m^2 which leads to a unit costing Dfl $165*10^3$ at current membrane costs and Dfl 8250 based on expected costs.

7.2 Ceramic Membranes

The process gases defined in Appendix 1 are generally available at temperatures far above ambient. Polymeric membranes are not suited for operation at these temperatures but ceramic membranes are. The development of commercial ceramic membranes is less advanced then polymeric membranes but at present a large research and development effort is being made. The processes for which ceramic membranes are being developed are, for example, hydrogenation and removal of nitrous oxides. Their selectivity characteristics for a carbon dioxide - hydrogen mixture have not been determined but they are expected to be better than polymeric membranes [VEL91]. Due to its small size hydrogen will be the fast component. As a result the hydrogen will be available at a lower pressure. This has to be taken into account when designing the gas turbine. A stream enriched in carbon dioxide will be available at high pressure and temperature. If the purity does not meet the 95% requirement the carbon dioxide needs to be recovered using an additional condensation unit. As the selectivities are expected to be much larger than for current polymeric membranes future developments are of great interest for the carbon dioxide - hydrogen separation problem.

7.3 Metallic Membranes

It has been known for quite some time that at temperatures between 300 and 400 ^{o}C hydrogen will dissociate at palladium surfaces and diffuse through the metal layer. This principle can be used to develop a separation process. The main problems encountered in this development concern the mechanical and thermal stability of the membrane. Also the presence of impurities like hydrocarbons might poison the membranes. Although commercial membrane modules based on palladium membranes do exist their costs have prevented use on a large scale. As hydrogen is the fast component it will become available at a lower pressure with the resultant loss in free energy.

7.4 Facilitated Transport

The CO_2/H_2-selectivities of commercial polymeric membranes are insufficient to perform an economical separation. Improvements of separation characteristics are possible with facilitated transport, e.g. by incorporation of a suitable complexing agent in the polymer matrix. Membranes based on this concept exhibit selectivities in excess of 200. For example, Bhave et. al. [BHA86] measured a CO_2/N_2-selectivity of 202.5 for an immobilised liquid membrane containing potassium carbonate, Pellegrino et. al. [PEL90] measured CO_2/H_2-selectivities between 280 and 446 for gel ion-exchange membranes. In both cases carbon dioxide is the fast component. Selectivity values in excess of 200 allow all of the separations to be achieved in a single step. As facilitated transport techniques are still in the laboratory phase there is virtually no information on aspects like durability, stability and costs. Their prospects, however, are promising, provoking a worldwide research interest.

8 Conclusions

The aim of this study was to assess the feasibility of membrane operations in the removal of carbon dioxide from flue gases and process gases. In Paragraphs 8.1, 8.2 and 8.3 the conclusions are summarised for each of the gas streams defined in Appendix 1.

8.1 Flue Gas

8.1.1 Gas Absorption Membranes

Conventional carbon dioxide removal technology based on mono-ethanolamine can be improved upon through the use of gas absorption membranes. Future investment costs could be lower than those of conventional absorbers. Currently available membrane modules need to be scaled up to be able to treat the large volumes of flue gas. There do not seem to be any major problems related to the scale-up procedure.

8.1.2 Gas Separation Membranes

The CO_2/N_2-selectivity characteristics of presently available gas separation membranes are favourable but insufficient to allow an economical operation of a carbon dioxide removal unit. A hybrid plant incorporating gas separation membranes and a condensation unit is more economical but is still more expensive than conventional carbon dioxide removal based on amines.

8.2 Process Gas from a Coal Gasification Plant

8.2.1 Gas Absorption Membranes

Gas absorption membranes combined with a physical absorption process has several operational advantages. Gas absorption membrane modules, however, operate at near atmospheric pressure. To enable these modules to work at the pressure levels encountered in the coal gasification stream they need to be housed in pressure vessel. Also, measures need to be taken to avoid a large pressure difference across the membrane. To overcome this problem one can also use dense membranes.
Scale up will require the development of larger membrane modules.

8.2.2 Gas Separation Membranes

The CO_2/H_2-selectivities of commercially available membranes are too low to allow economical operation of a carbon dioxide removal plant based on gas separation membranes alone. In case of a carbon dioxide selective membrane this will lead to a substantial loss of hydrogen which needs to be recovered by recompression. In case of a hydrogen selective membrane a major part of the hydrogen will be available at low pressure, giving rise to a substantial loss in free energy.

A hybrid process, i.e. a process based on conventional carbon dioxide technology combined with a membrane gas separation unit does not lead to a reduction in energy expenditure per mole carbon dioxide removed when compared with a process based on conventional carbon dioxide removal technology alone.

8.3 Process Gas from a Gas Reformer

8.3.1 Gas Absorption Membranes

Gas absorption membranes combined with a physical, chemical or physical/chemical absorption process has several operational advantages. Gas absorption membrane modules, however, operate at near atmospheric pressure. To enable these modules to work at the pressure levels encountered in the reformer gas stream they need to be housed in a pressure vessel. Also, measures need to be taken to avoid a large pressure difference across the membrane. To overcome this problem one can also use dense membranes.

Scale up will require the development of larger membrane modules.

8.3.2 Gas Separation Membranes

The CO_2/H_2-selectivities of commercially available membranes are too low to allow economical operation of a carbon dioxide removal plant based on gas separation membranes alone. As the hydrogen needs to be delivered at pressures only slightly smaller than the feed pressures a carbon dioxide selective membrane seems to be the only option. This will lead to a substantial loss of hydrogen which needs to be recovered by recompression.

A hybrid process will not benefit from gas separation membranes as the carbon dioxide partial pressure is substantially lower than that of the incoming stream.

9 Recommendations

From the study a number of points requiring attention in future studies has emerged. In the following they have been grouped according to the carbon dioxide removal method.

1. Chemical absorption using membranes

   - The energy consumption per mole of carbon dioxide removed in case of MEA-absorption is high. This is due to the large amount of heat needed in the reboiler. In other parts of the plant large amounts of heat are removed by cooling water, e.g. in the condensor installed after the stripper. Therefore it must possible to improve on the energy-efficiency of the process.

   - The absorption step can be carried out using gas absorption membranes, but, in principle, it must also be possible to replace the stripper step by a membrane process, e.g. pervaporation. The desorption in the latter process is carried out by a pressure difference across the membrane. It would be benefical if this membrane was preferentially permeable to carbon dioxide over the absorption liquids. In case of regeneration using a pressure swing the heat exchanger for the lean/rich absorbent exchange might not be needed.

   - In current gas absorption membranes (hollow fibres) the gas flow is inside the fibres. As a consequence the frontal area of the unit is much larger than needed for the flow. This is a disadvantage and could be removed if the gas flow were to be on the outside of the fibres. Operation in a cross-flow mode would then be possible, resulting in an increase in the mass transfer. Development of hollow fibre membrane modules employing cross-flow is at present receiving worldwide attention. TNO has a patent pending which is based on the idea of cross-flow and allows easy scale-up because of its modular design. Cross-flow operation could also result in integration of the membrane based carbon dioxide unit with a chimney stack.

2. Physical absorption using membranes

   - Current gas absorption membranes are microporous and are therefore useable at atmospheric pressure. Physical absorption processes operate at elevated pressures and this requires dense membranes. Current gas separation membranes might be employed for gas absorption at high pressures.

   - Regeneration of a physical absorbent loaded with carbon dioxide is carried out by pressure release. Recompression of the lean absorbent is the largest component of the total energy requirement. If one carries out the regeneration step using pervaporation with a CO_2-selective membrane the rich absorbent can be kept at pressure, thus avoiding the need for recompression. This would increase the energy-efficiency of the absorption process.

3. Gas separation membranes

   - Gas separation membranes can become of interest provided their selectivity ratios, especially for CO_2/H_2, can be increased. As the process gases are at high temperature future membranes need to be temperature resistant.

4. General

   - The amount of membrane area needed for the removal of carbon dioxide from a single 600 MW electricity generating plant is sizable when compared with the current production capacity. Manufacturing processes would have to be made more efficient in order to be able to produce the membrane area needed.

   - A sizeable amount of the energy needed in the carbon dioxide removal process and the consequent carbon dioxide storage is needed for the compression of carbon dioxide. Alternative methods to deal with the carbon dioxide stream might be more energy-efficient.

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August 1991 - December 1991

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Appendix 1 Input and output conditions of carbon dioxide removal unit

1. Product carbon dioxide stream

Delivery pressure:	110 bar
Delivery temperature:	10-20 °C
Composition	
Water content:	<10 ppm
Molar fraction permanent gases:	<0.05

2. Flue gas coal-fired plant

Mass flow:	650 kg/s
Pressure:	atmosferic
Temperature:	50 °C
Molar Mass:	29.09 kg/kmole

Composition (molar fraction)

N_2	0.71353
CO_2	0.13
H_2O	0.12
O_2	0.028
Ar	0.0084
SO_2	0.00007
Solids	<20 mg/Nm^3

Mass flow carbon dioxide product: (Derived from emission ceiling of 62 g CO_2/ MJ_e) (Equivalent to 70.9% carbon dioxide removal)	>90.2 kg/s

3. Process gas coal gasification plant

Mass flow:	2 * 90 kg/s
Pressure:	77 bar
Temperature:	310 °C
Molar mass:	20.11 kg/kmole

Composition (molar fraction)

H_2	0.38
CO_2	0.30
H_2O	0.27
CO	0.03
CH_4	0.01
N_2+Ar	0.01
H_2S	1800 ppm
COS	90 ppm

Mass flow carbon dioxide product: (Derived from emission ceiling of 62 g CO_2/ MJ_e) (Equivalent to 81.8% carbon dioxide removal)	>80.7 kg/s

4. Process gas from reformer (case 1)

Mass flow:	247 kg/s
Pressure:	23.5 bar
Temperature:	210-230 °C
Molaire mass:	13.864 kg/kmole
LHV:	9561 kJ/kg

Composition (molar fraction)	
H_2	0.4632
H_2O	0.37
CO_2	0.1178
CH_4	0.0245
N_2	0.0229
CO	0.0016

Mass flow carbon dioxide product: (Explicitly given in specifications) (Equivalent to 82.3% carbon dioxide removal)	>76 kg/s

LHV CH_4/CO stream:	<700 MW
Pressure CH_4/CO stream:	>5 bar
Pressure H_2 stream:	>22 bar

5. Process gas from reformer (case 2)

Mass flow:	169 kg/s
Pressure:	18 bar
Temperature:	210-240 °C
Molar mass:	12.919 kg/kmol
LHV:	12860 kJ/kg

Composition (molar fraction)	
H_2	0.5658
H_2O	0.2236
CO_2	0.1418
CH_4	0.0349
N_2	0.0290
CO	0.0049

Mass flow carbon dioxide product: (Explicitly given in specifications) (Equivalent to 82.3% carbon dioxide removal)	>76 kg/s

LHV CH_4/CO stream:	<650 MW
Pressure CH_4/CO stream:	>5 bar
Pressure H_2 stream:	>16.5 bar

Appendix 2 Calculation of energy requirement for CO_2-removal by cooling for coal gasification process gas

The recovery requirement as given in Figure 6 is equal to 0.336 as a fraction of the feed flow. In order to meet this requirement the gas stream needs to be cooled to -44 ^{o}C (CO_2 partial pressure = 8.7 bar). If one assumes the evaporator in the cooling unit to operate at -49 ^{o}C, the condensor to operate at 15 ^{o}C and furthermore the efficiency of the cooling unit to be equal to 0.5, the coefficient of performance of the cooling unit is given by:

$$COP = 0.5 \cdot 288.15/(288.15 - 224.15) = 2.25$$

The amount of heat to be transferred is equal to the sum of the sensible heat required for cooling of the gas stream (32 J/(mole K)) and the latent heat released by the condensation of carbon dioxide (25 kJ/mole CO_2). Hence per mole feed flow:

$$Q = 0.032 \cdot (288.15 - 229.15) + 0.336 \cdot 25 = 10.3 \text{ kJ/mole}$$

The amount of work required can now be calculated using the COP:

$$W = Q/COP = 4.57 \text{ kJ/mole}$$

When expressed in terms of the energy requirement per unit carbon dioxide, this becomes:

$$W = 4.57/0.336 = \underline{13.6 \text{ kJ/mole } CO_2}$$

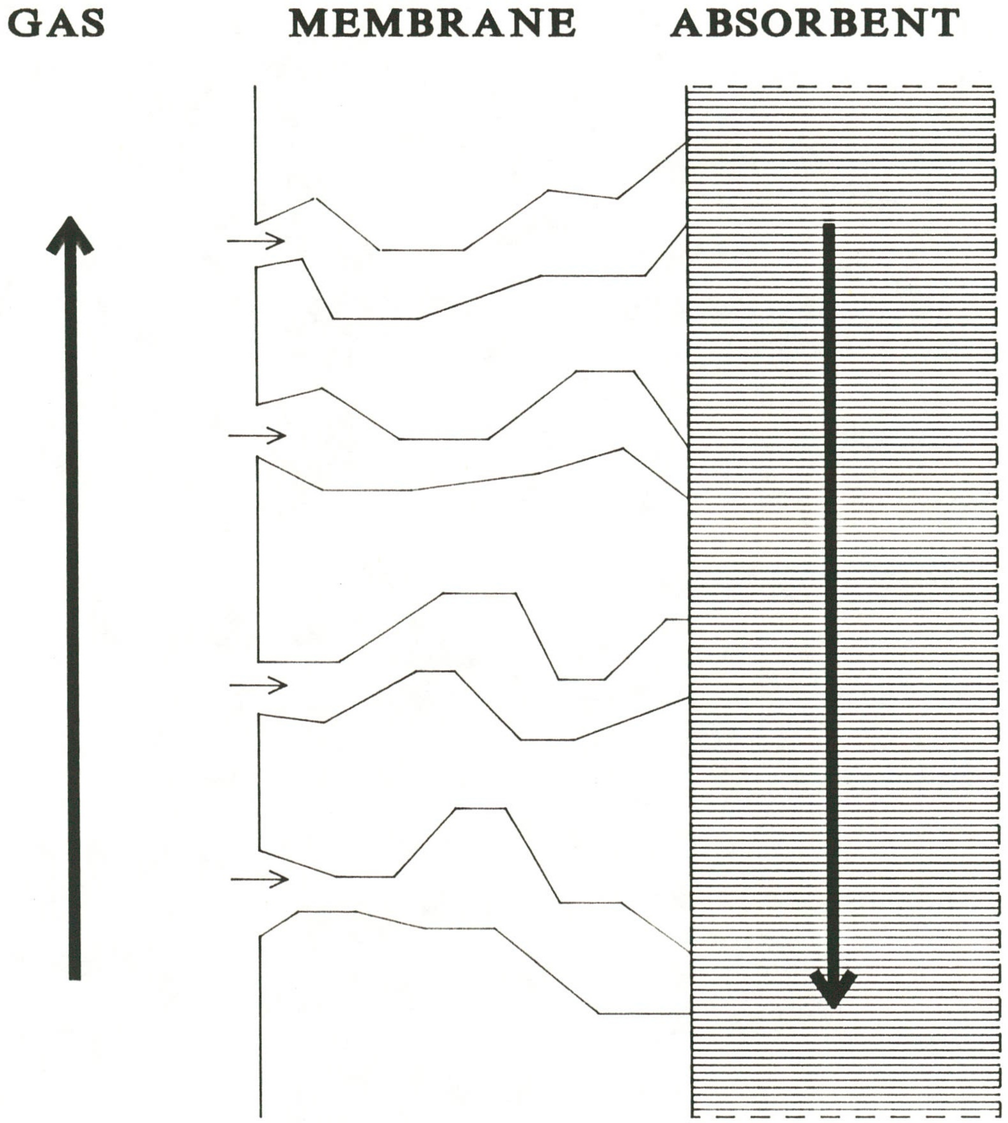

Figure 1: Gas Aborption Membranes

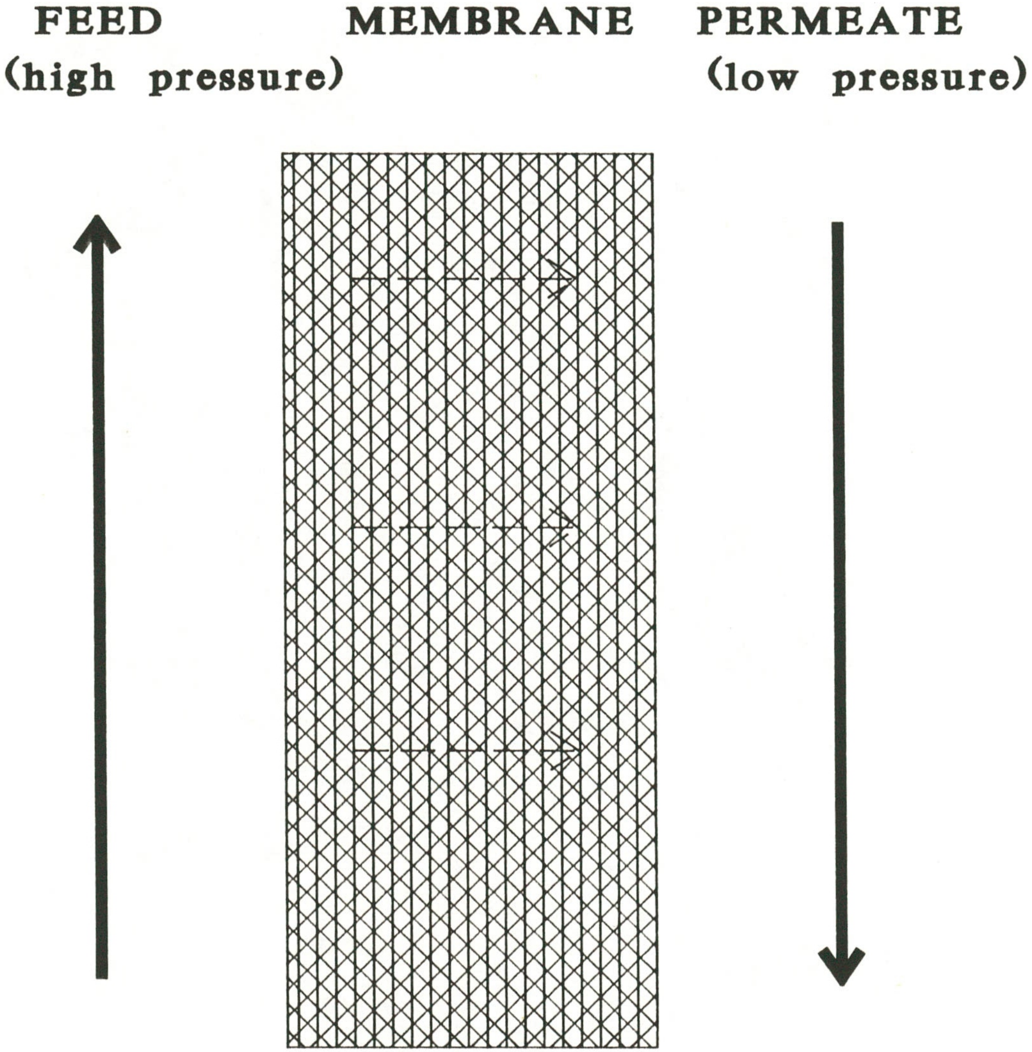

Figure 2: Gas Separation Membranes

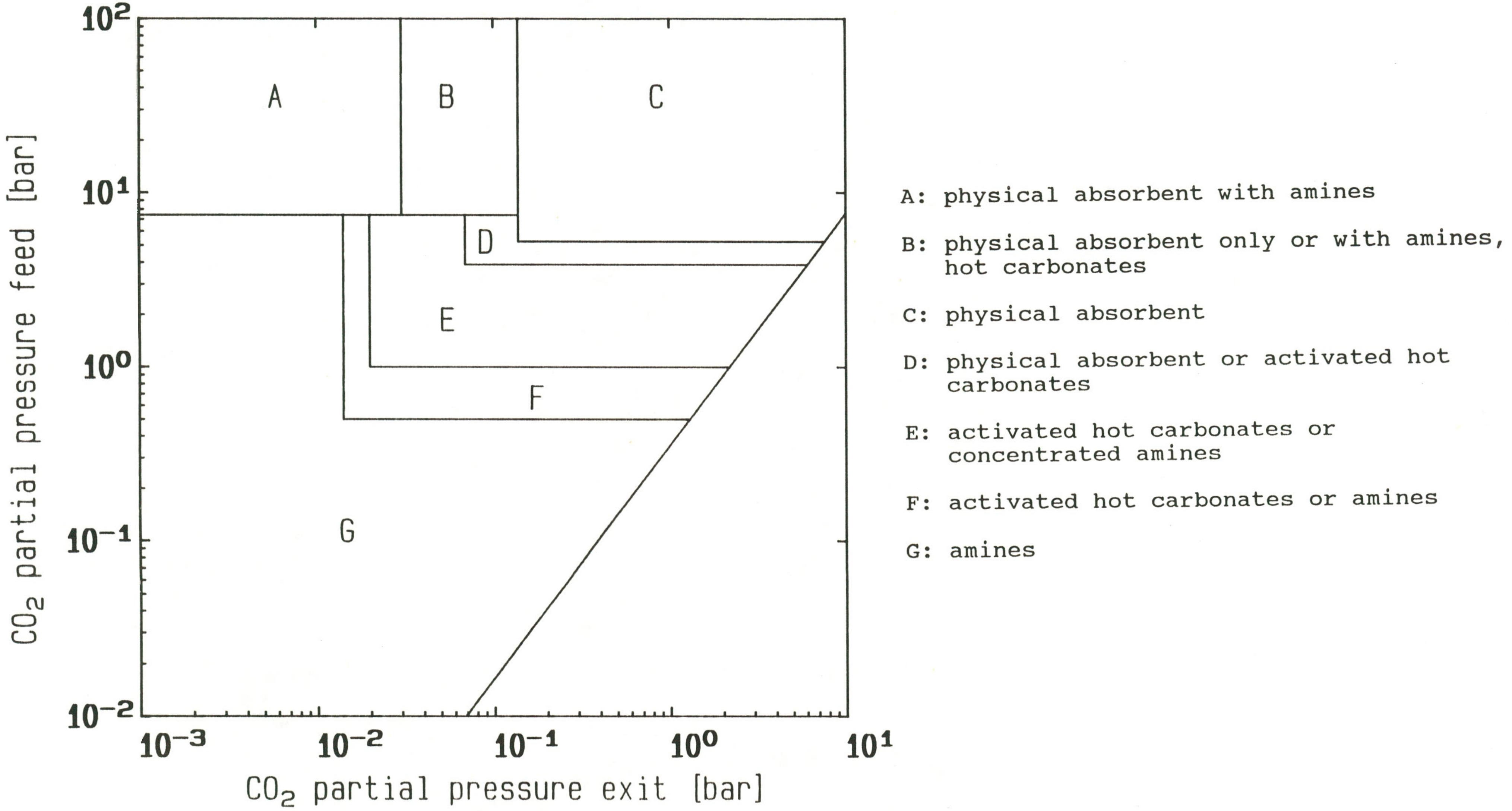

Figure 3: Selection Diagram for CO2 Absorption Processes [ULL83]

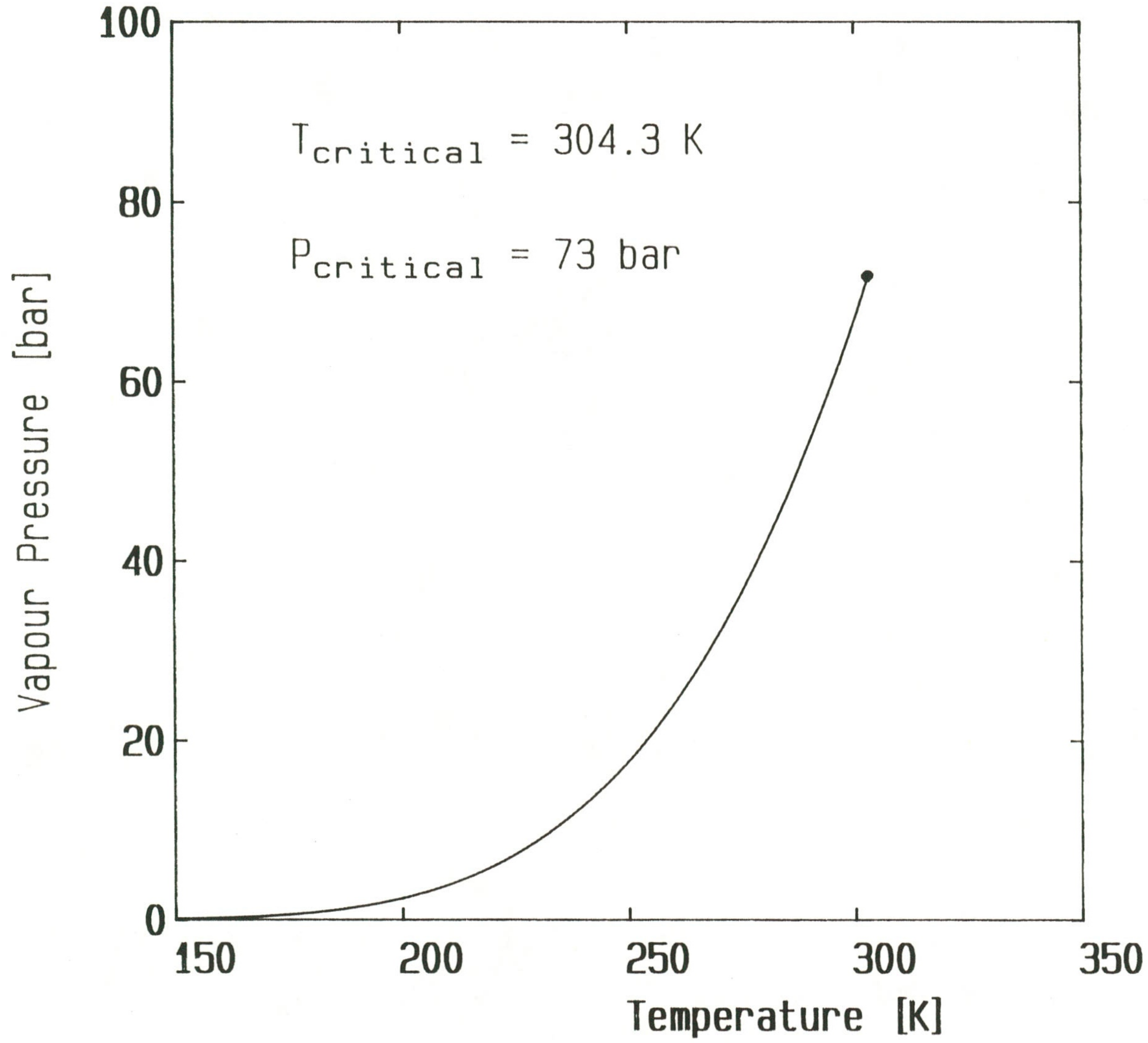

Figure 4: Carbon Dioxide Vapour Pressure

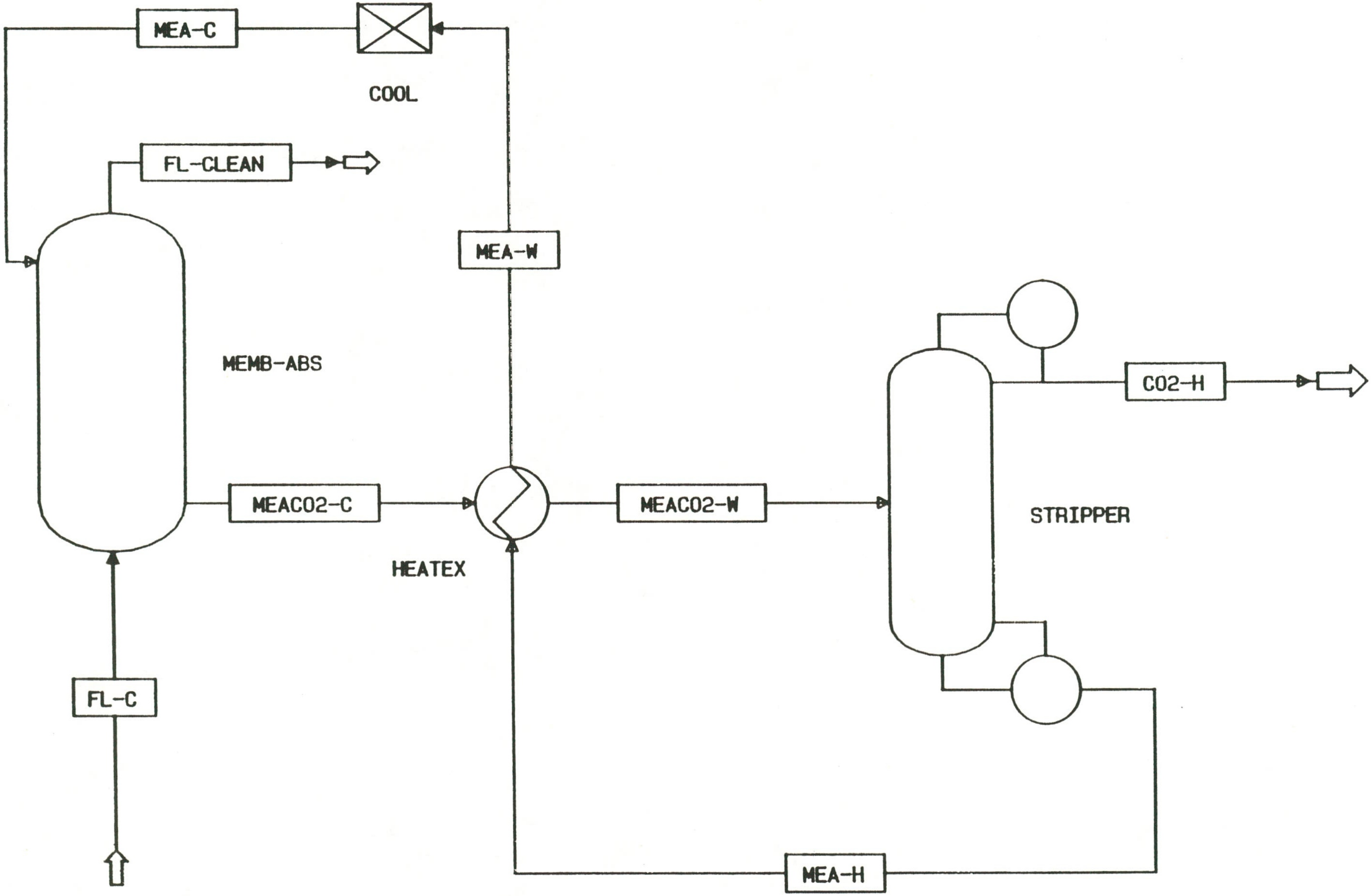

Figure 5: Process Flow Diagram for MEA Absorption

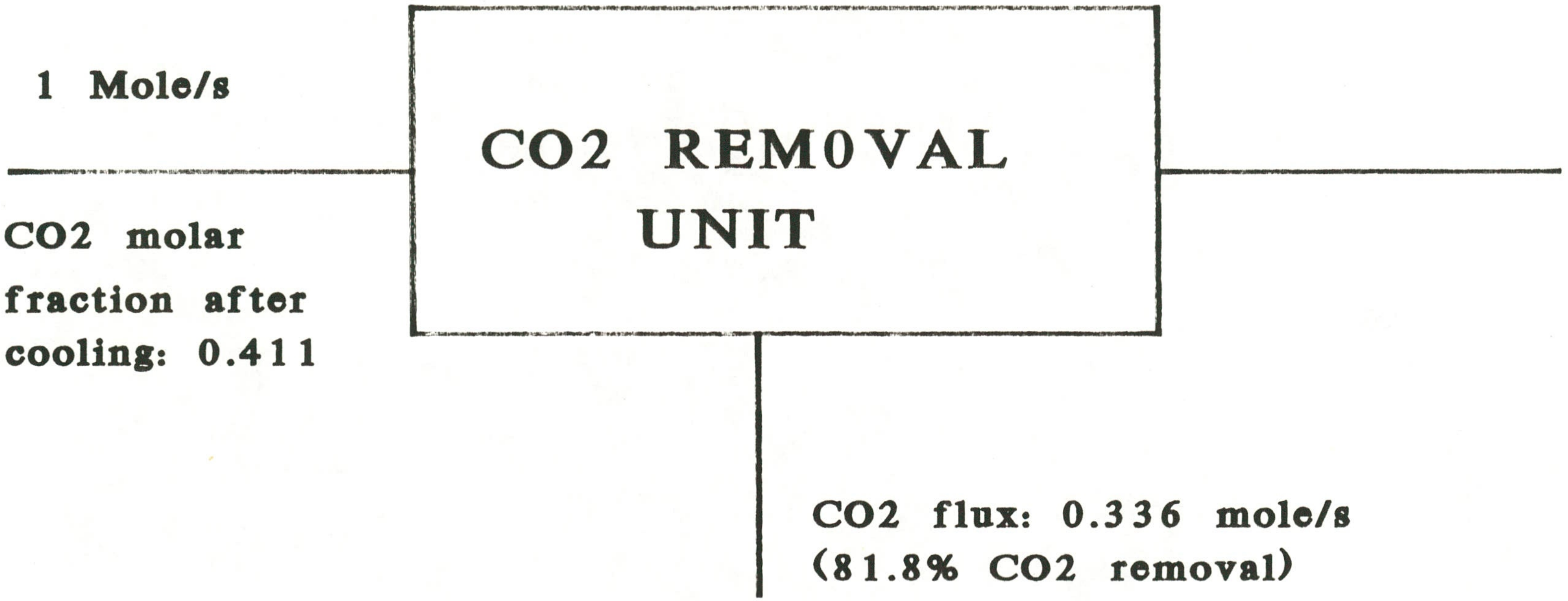

Figure 6: Specification CO2 removal unit; coal gasification

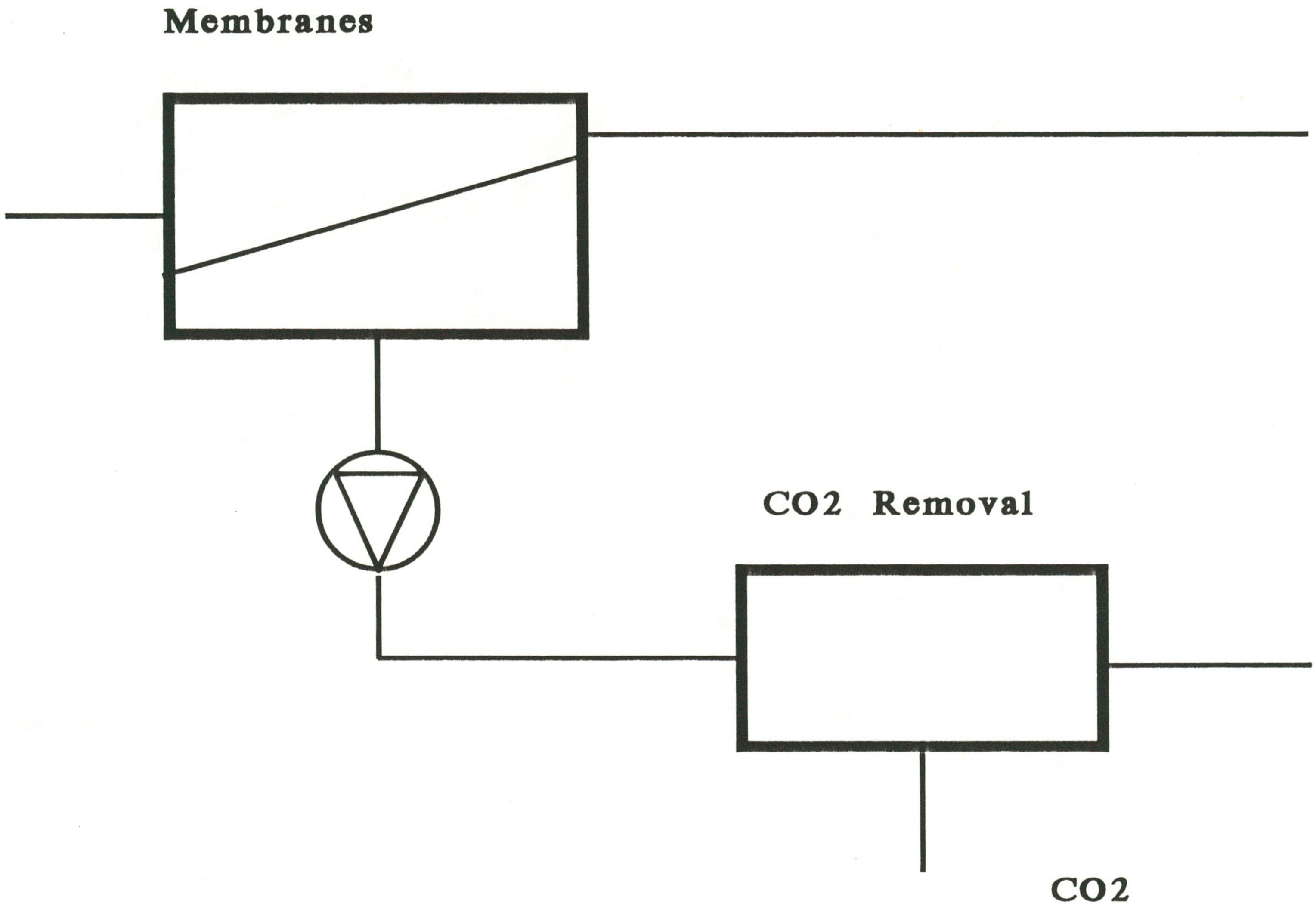

Figure 7: Hybrid Plant with Preconcentration Using Gas Separation Membranes Followed by CO2 Removal

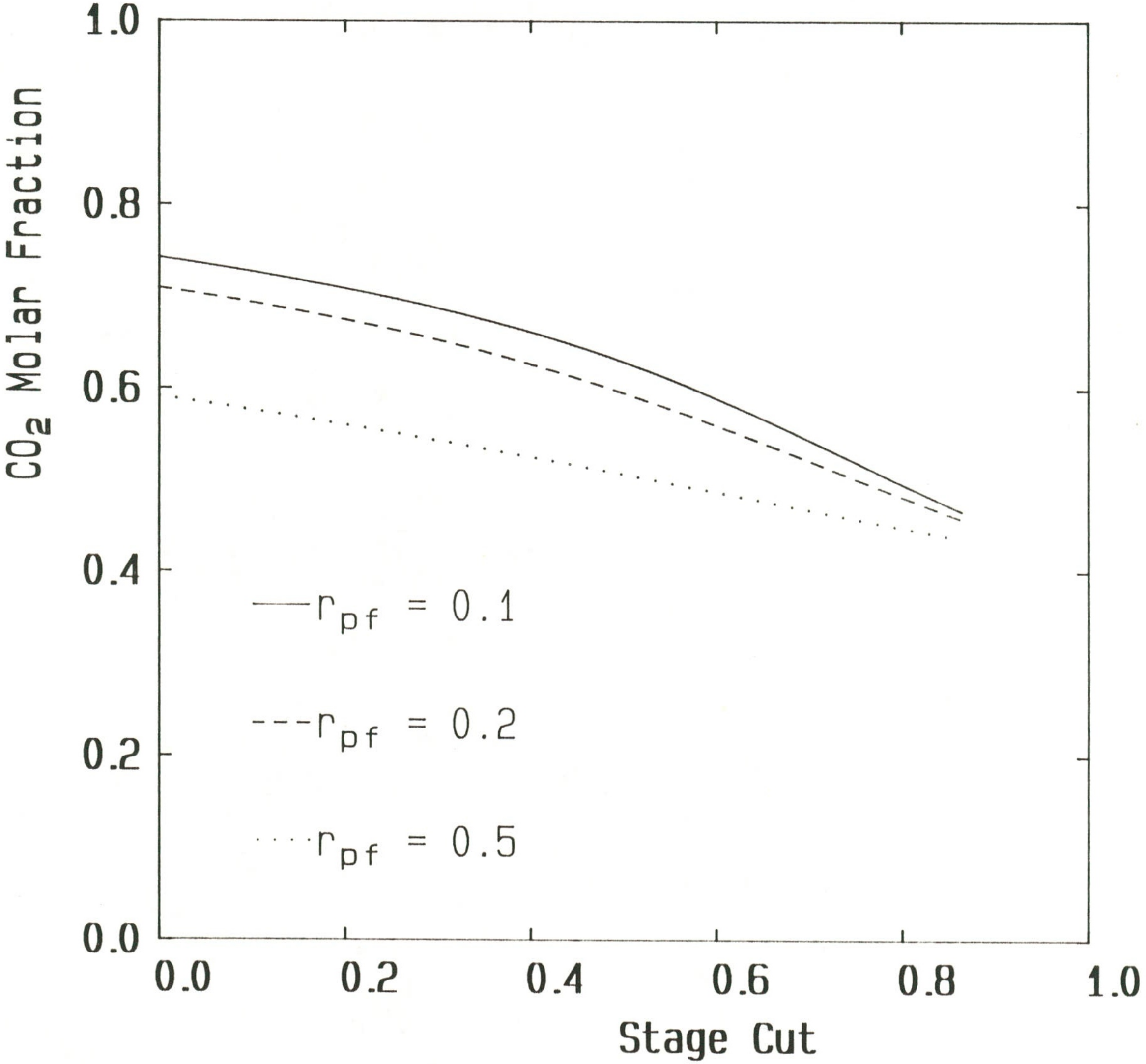

Figure 8: CO2 Molar Fraction in Permeate as A Function of Stage Cut; PDMS Membranes

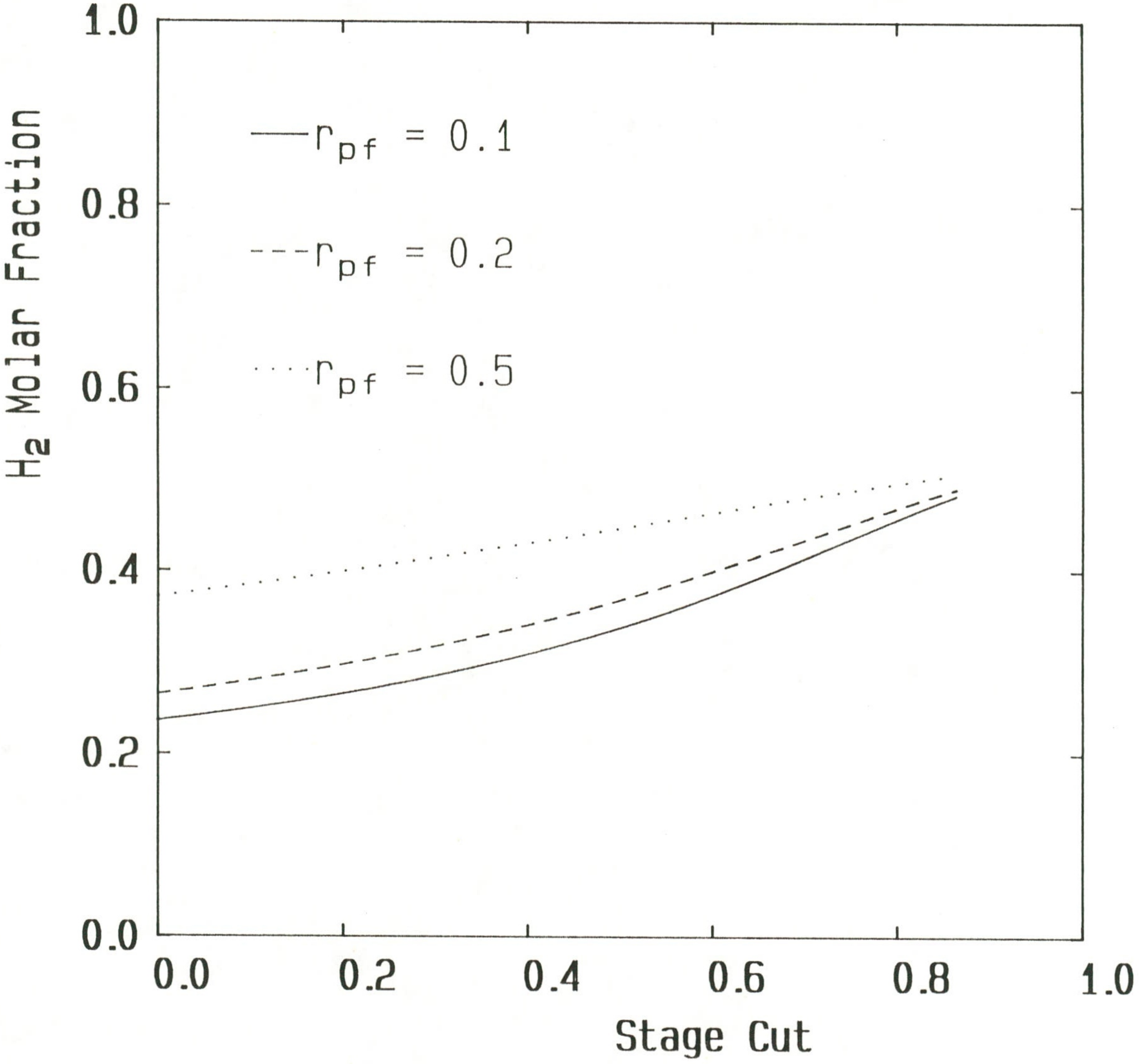

Figure 9: H2 Molar Fraction in Permeate as A Function of Stage Cut; PDMS Membranes

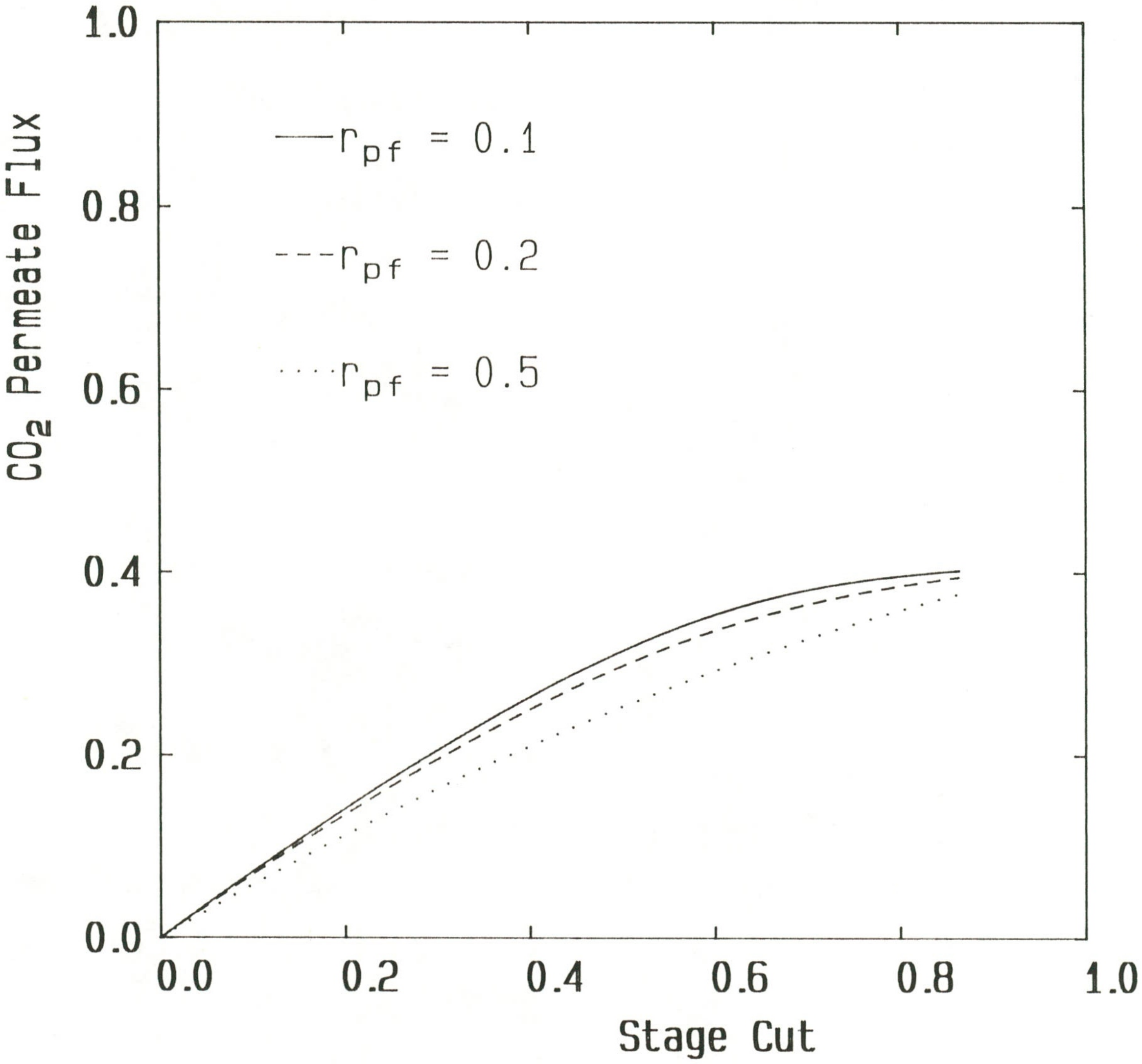

Figure 10: CO2 Permeate Flux Relative to Total Feed Flow as A Function of Stage Cut; PDMS Membranes

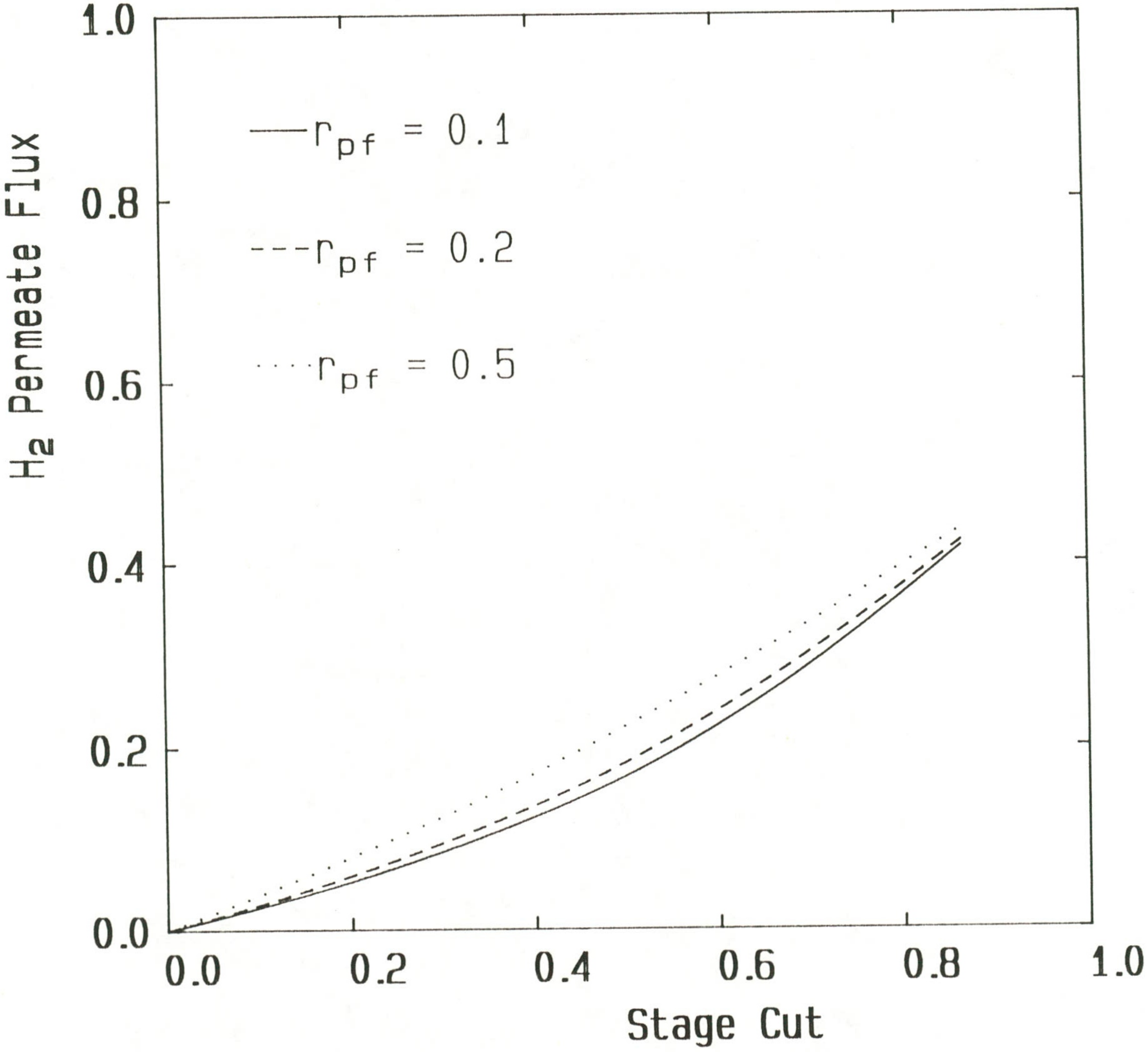

Figure 11: H2 Permeate Flux Relative to Total Feed Flow as A Function of Stage Cut; PDMS Membranes

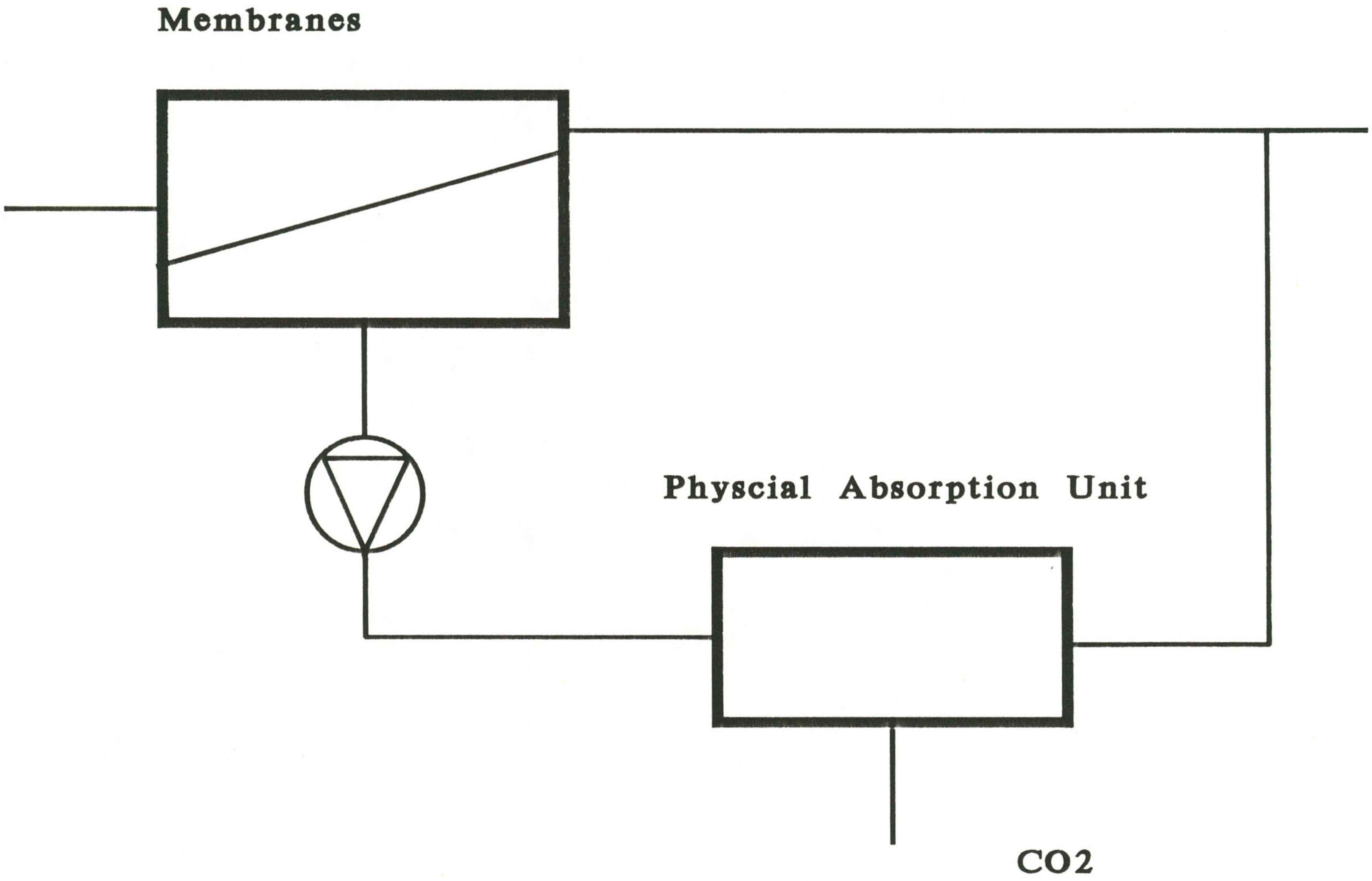

Figure 12: Hybrid plant with Preconcentration Using Gas Separation Membranes Followed by A Physical Absorption Unit

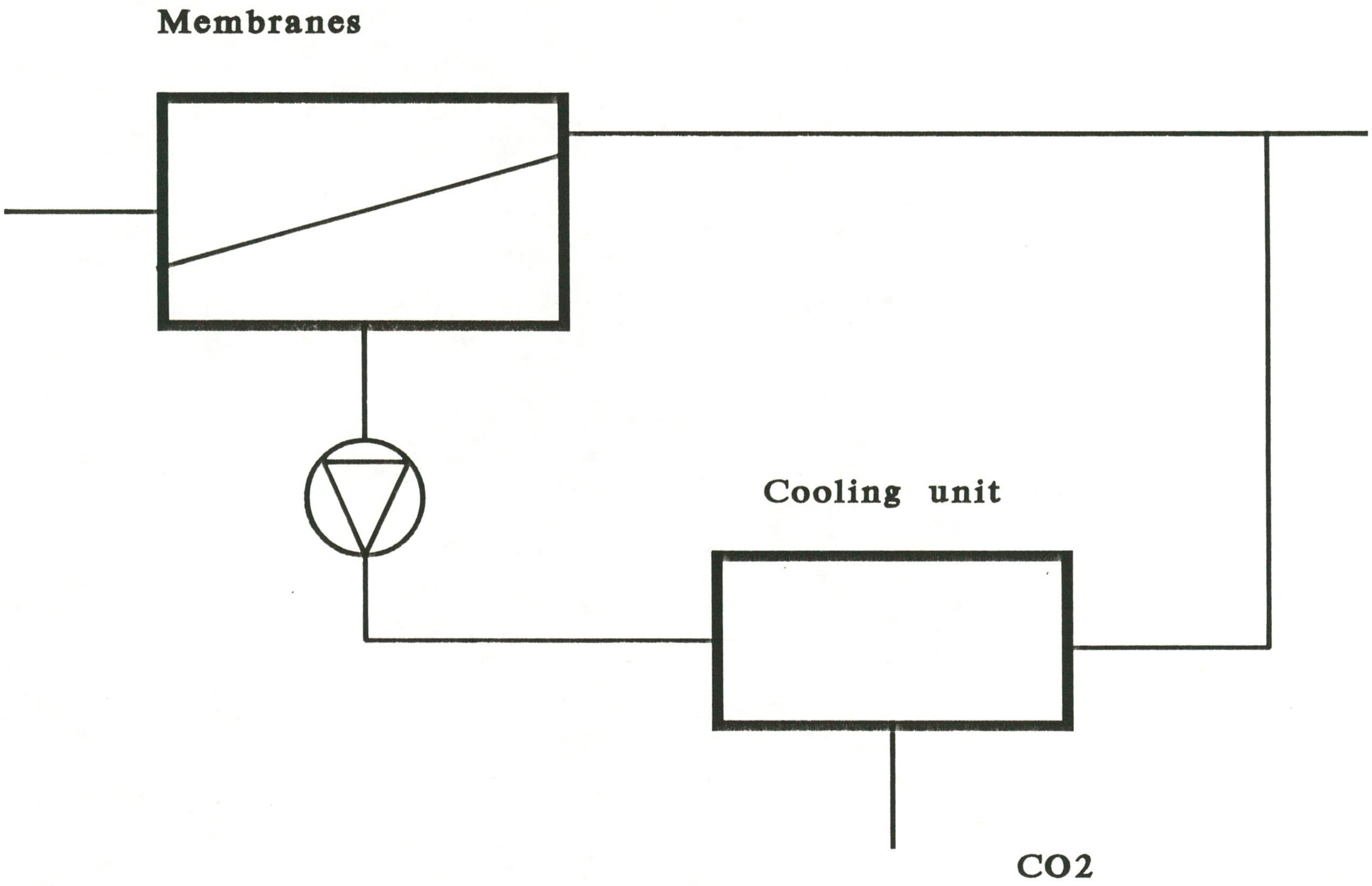

Figure 13: Hybrid plant with Preconcentration Using Gas Separation Membranes Followed by A Cooling Unit

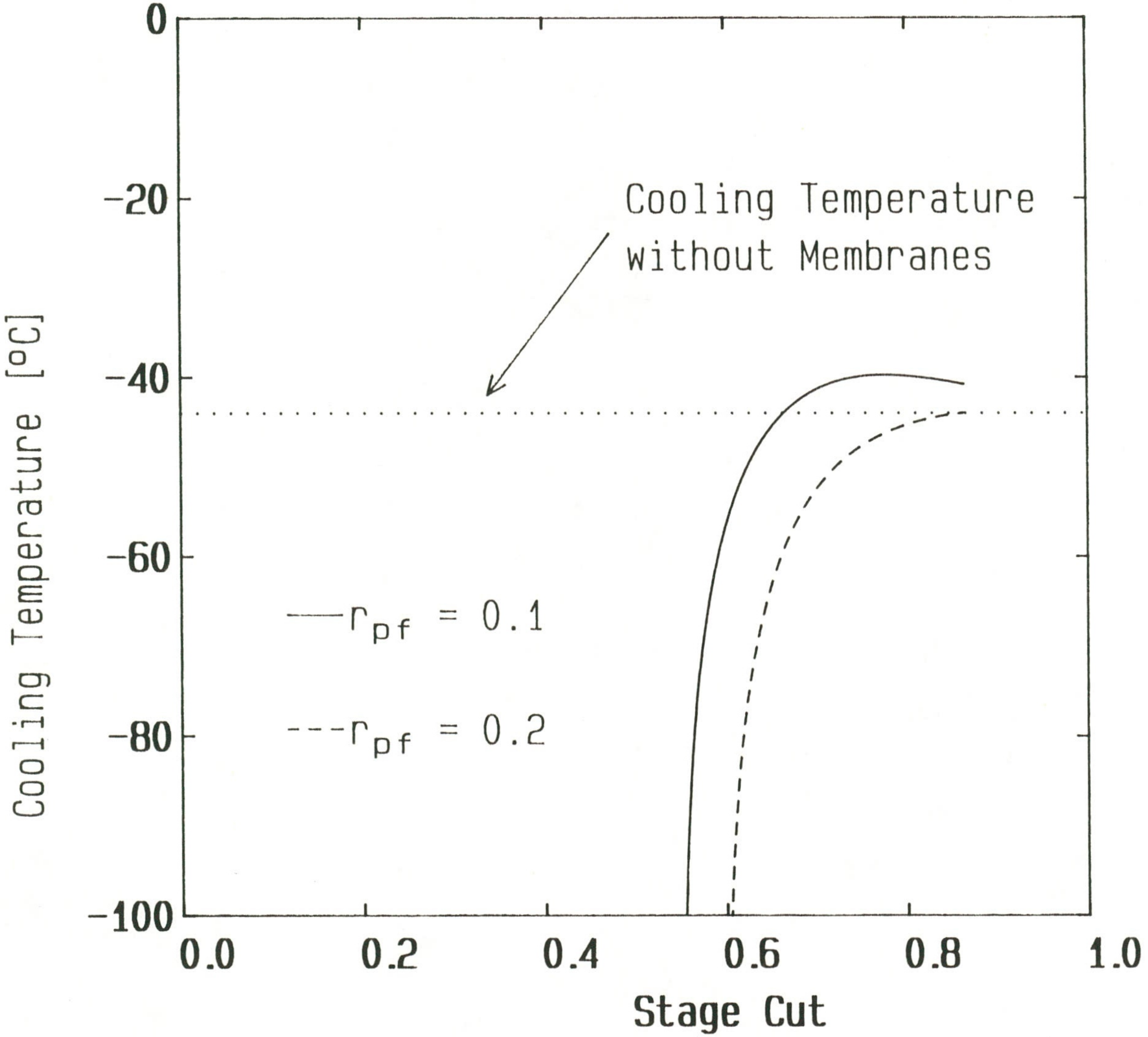

Figure 14: Required Cooling Temperature as A Function of Stage Cut For Cooling Unit in Figure 13

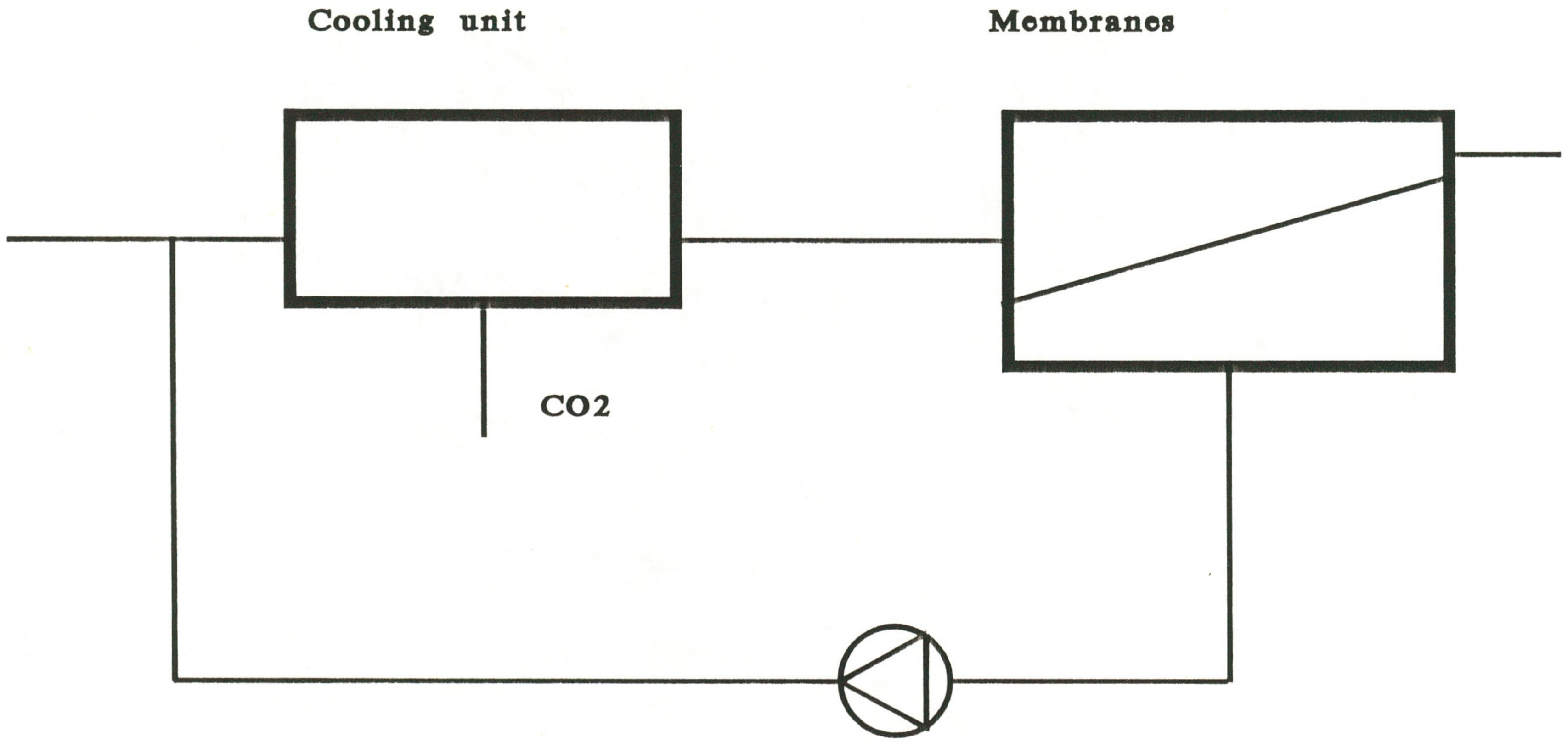

Figure 15: Hybrid Plant with Cooling Unit and Recycle of Residue Concentrated by Gas Separation Membranes

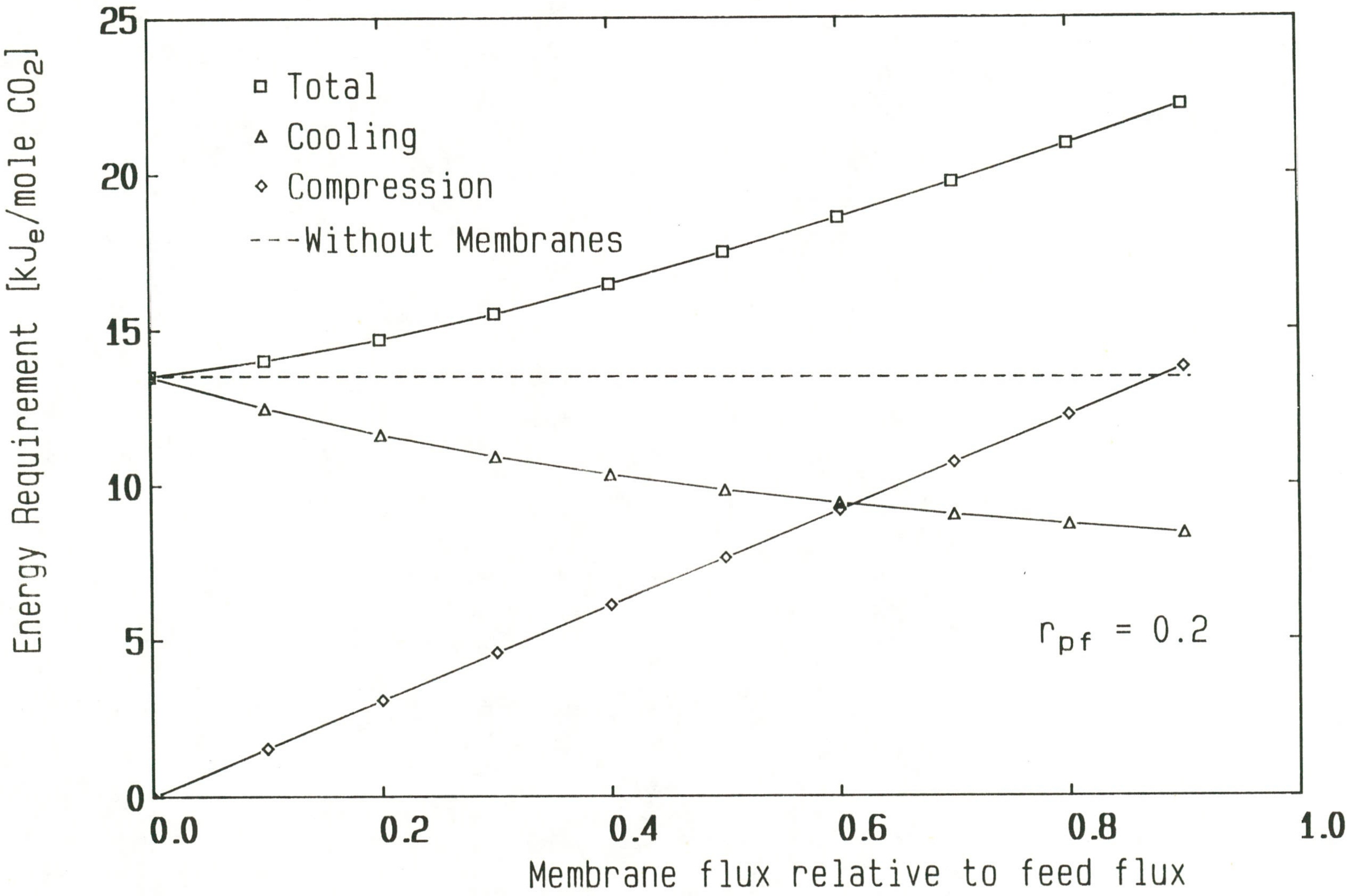

Figure 16: Total Energy Requirement of Hybrid Plant in Figure 15 as A Function of Membrane Flux PDMS-membranes

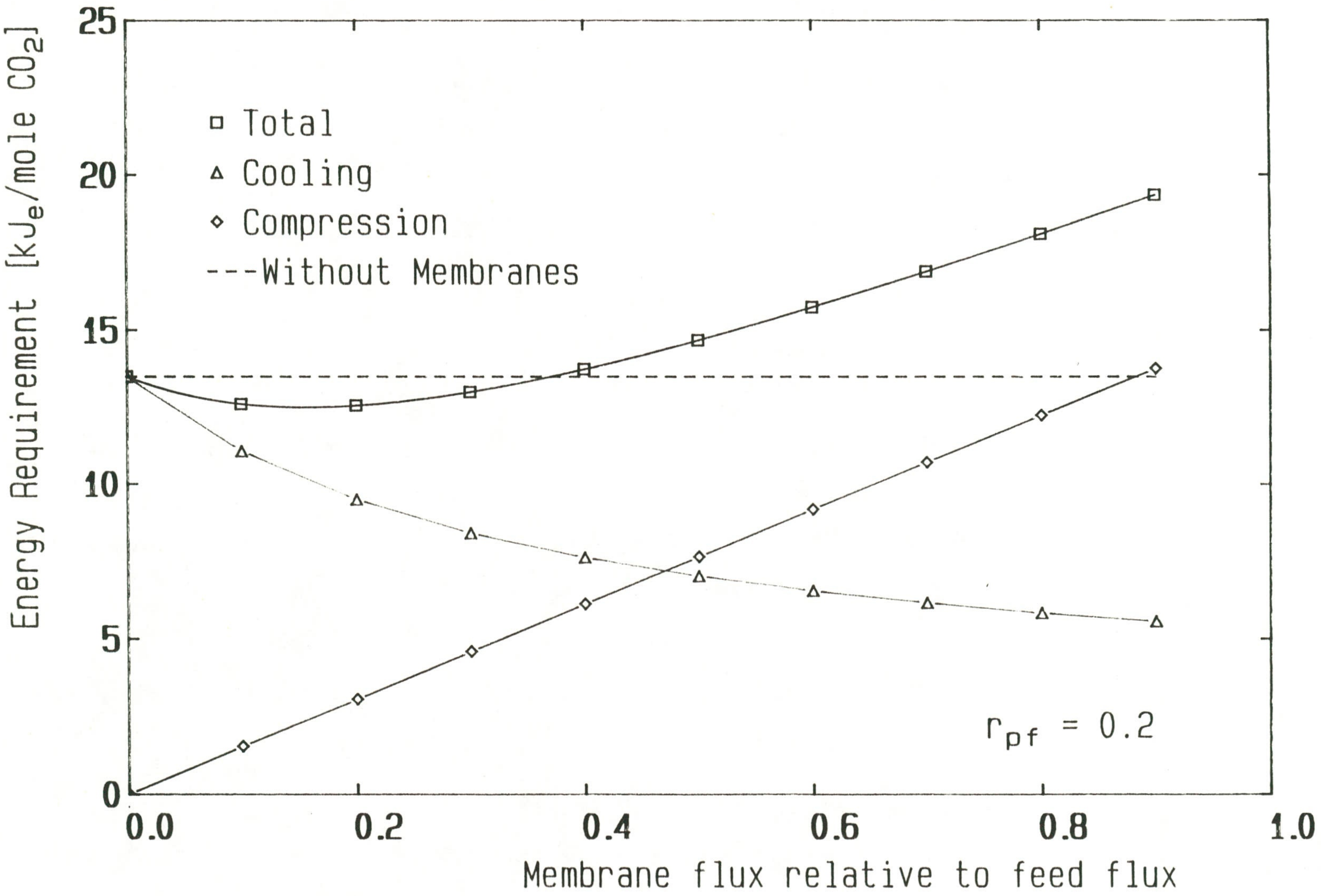

Figure 17: **Total Energy Requirement of Hybrid Plant in Figure 15 as A Function of Membrane Flux (CO2/H2-Selectivity = 50)**

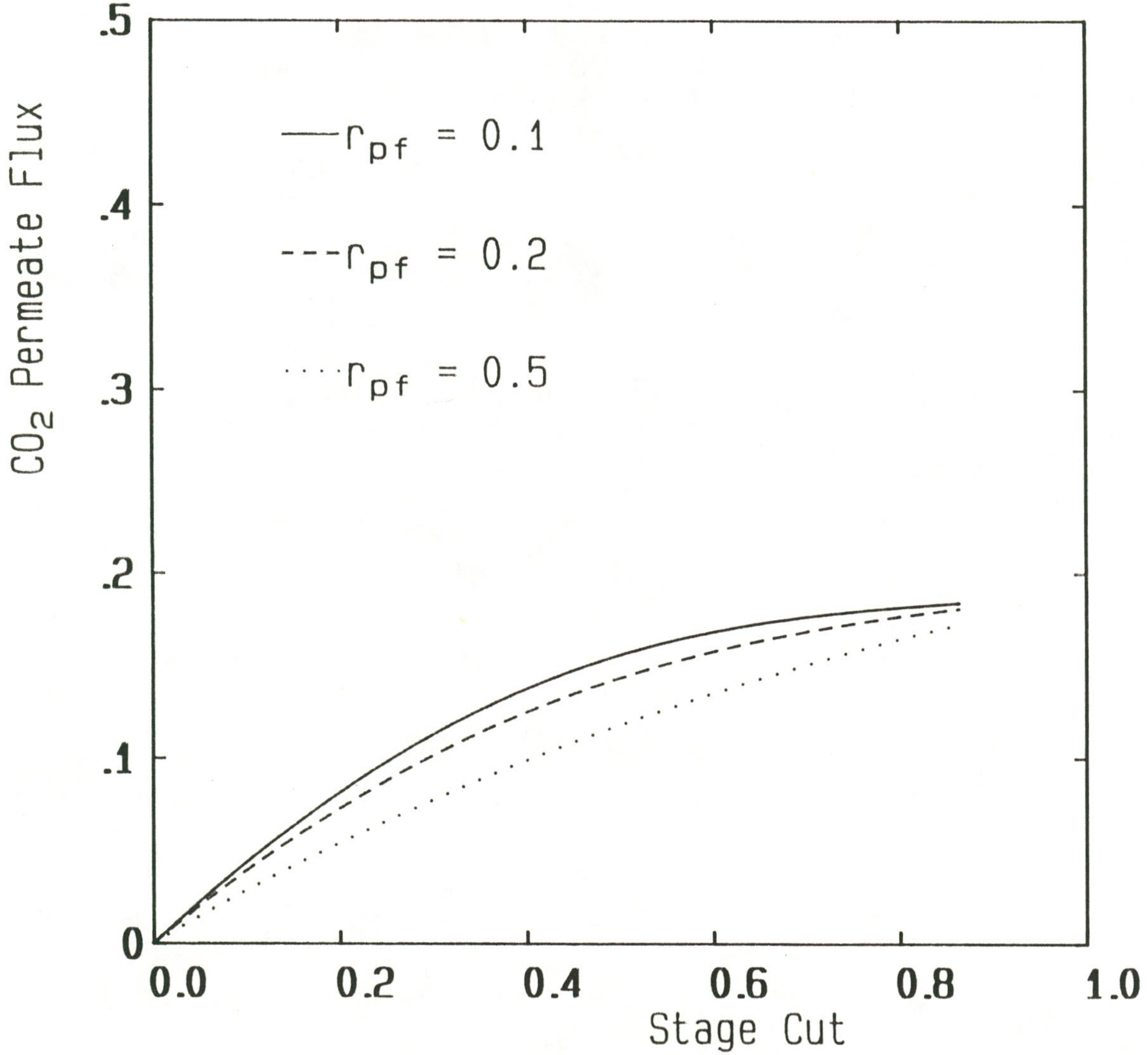

Figure 18: CO2 Permeate Flux Relative to Total Feed Flow as A Function of Stage Cut; PDMS Membranes

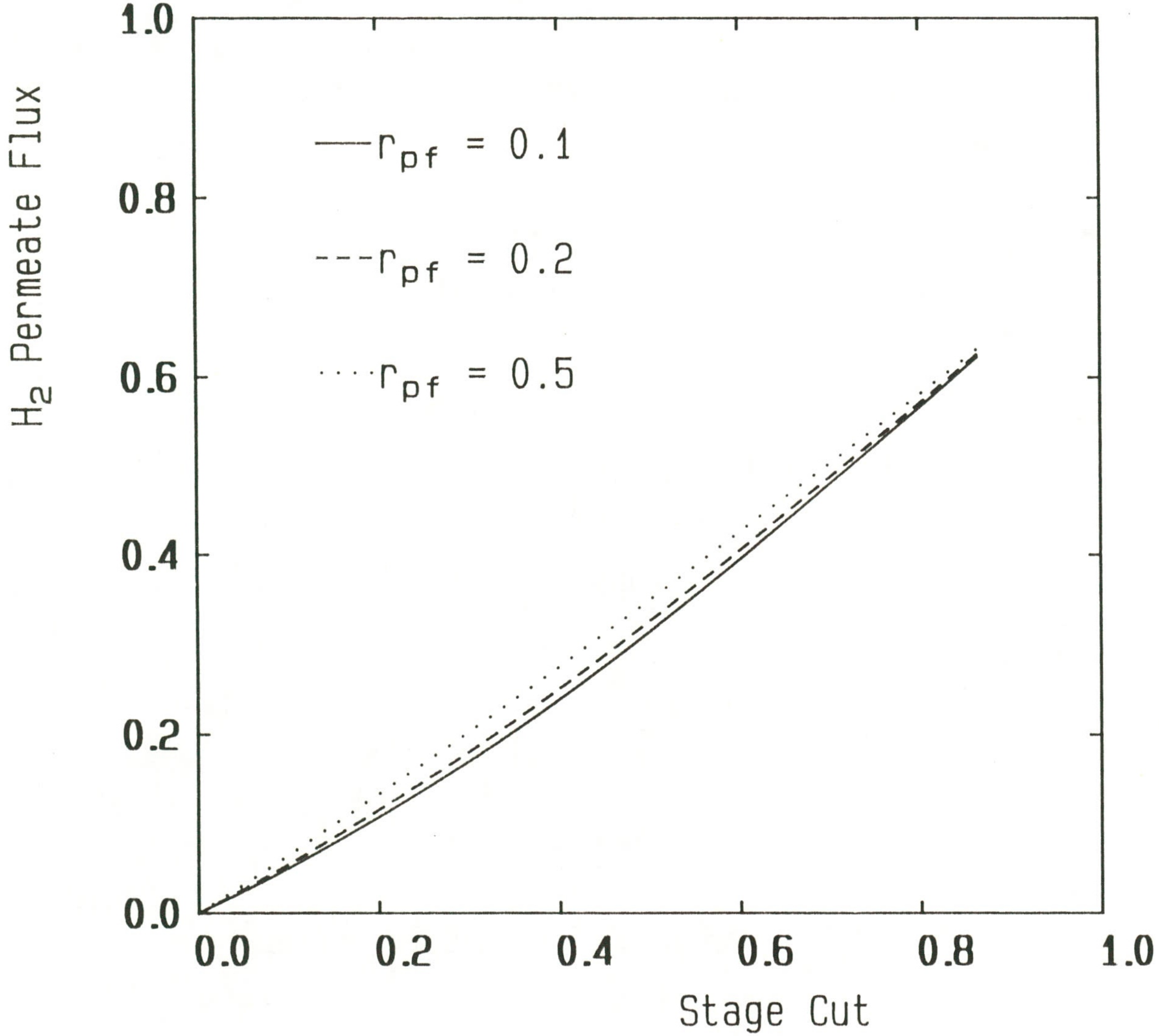

Figure 19: H2 Permeate Flux Relative to Total Feed Flow as A Function of Stage Cut; PDMS Membranes

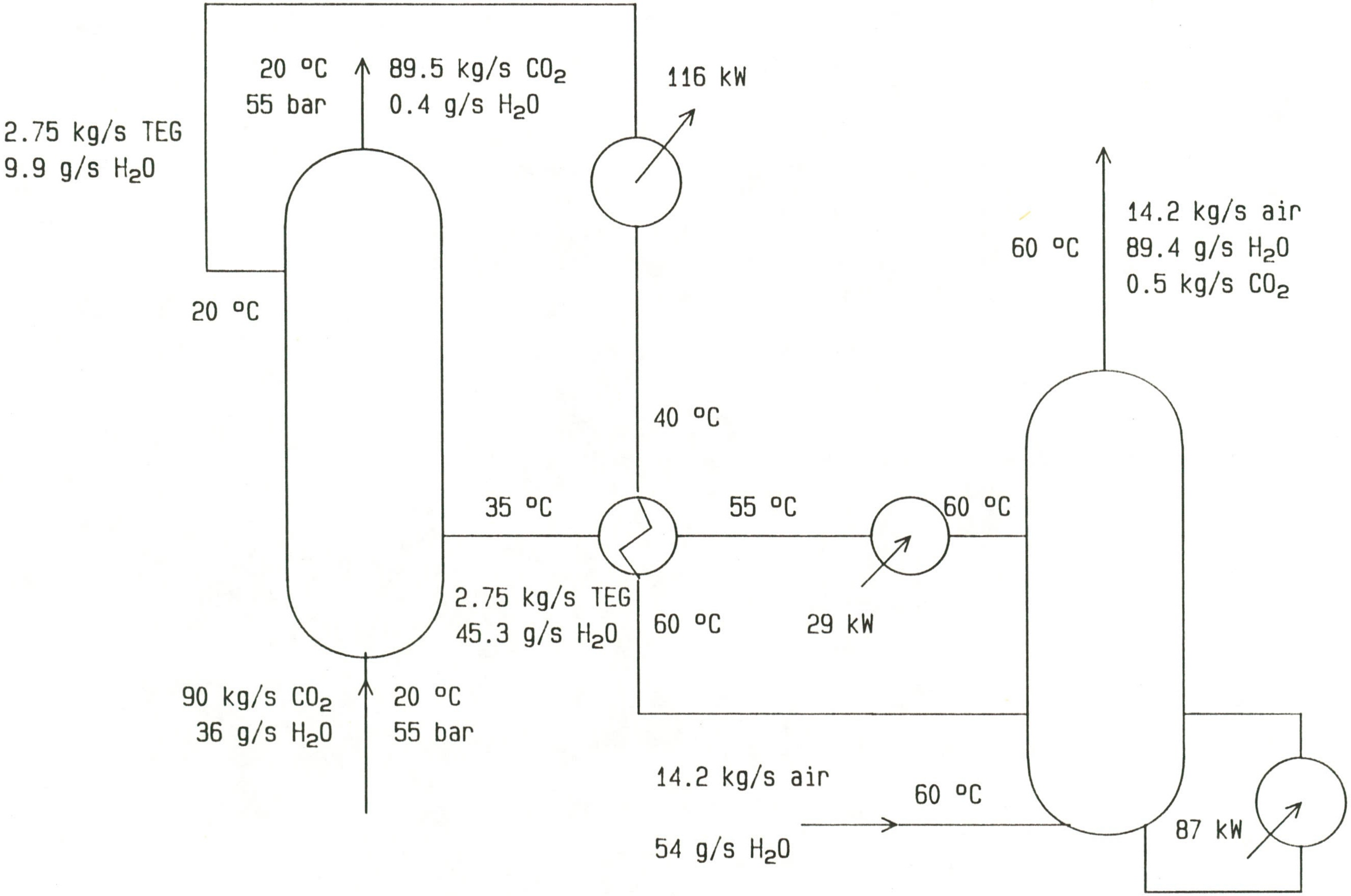

Figure 20: Process Flow Diagram for Dehydration