(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) **World Intellectual Property Organization**
International Bureau

(43) **International Publication Date**
4 November 2010 (04.11.2010)

PCT

(10) **International Publication Number**
WO 2010/126985 A1

(51) **International Patent Classification**:
B01D 53/22 (2006.01) ***B01D 61/58*** (2006.01)

(21) **International Application Number**:
PCT/US2010/032732

(22) **International Filing Date**:
28 April 2010 (28.04.2010)

(25) **Filing Language**: English

(26) **Publication Language**: English

(30) **Priority Data**:
12/433,695 30 April 2009 (30.04.2009) US

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(81) **Designated States** *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) **Designated States** *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report (Art. 21(3))*

(54) **Title**: MEMBRANE-BASED PROCESS FOR CO_2 CAPTURE FROM FLUE GASES GENERATED BY OXY-COMBUSTION OF COAL

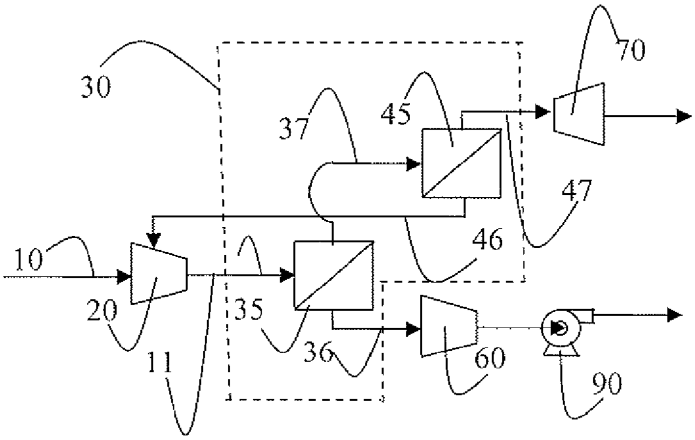

FIGURE 1

(57) **Abstract**: Disclosed is a membrane-based method and system for treatment of flue gases from an oxy-combustion coal-fired boiler to recover approximately 90% (vol/vol) to approximately 95% (vol/vol) of the carbon dioxide in the flue gas and produce a carbon dioxide product having a carbon dioxide concentration of approximately 90% (vol/vol dry basis) to approximately 97% (vol/vol dry basis).

Membrane-Based Process for CO_2 Capture from Flue Gases Generated by Oxy-Combustion of Coal

Background

Coal is often used as an energy source for industrial processes such as power generation. Coal combustion produces CO_2, a greenhouse gas. Concerns of global warming have prompted calls for reduction in CO_2 emissions. Processes have been proposed to extract CO_2 from the flue gases of industrial processes and sequester the CO_2 in the ground, thus preventing this greenhouse gas from being released into the atmosphere.

The US Department of Energy (DOE) currently defines a “clean coal” power plant as one that captures at least 90% of the CO_2 generated by the power plant. The captured CO_2 stream must be at a desired purity, temperature, and pressure in order to be suitable for transportation and storage. The CO_2 stream may also be used for enhanced oil recovery, enhanced gas recovery, or other applications, such as storage or sequestration. However, these applications may impose additional specifications on the CO_2 stream.

If coal is combusted in air, the flue gas contains about 12-20% CO_2 (vol/vol dry basis), with the majority of the remaining gas being N_2. Acidic gases like oxides of sulfur (SO_x) and nitrogen (NO_x) are also present in the flue gas. Conventional processes like amine absorption have been utilized to extract CO_2 from such flue gases. However, amine systems cannot tolerate sulfur compounds, thus requiring a sulfur removal system. The extent of sulfur removal prior to amine processing may be more than mandated by transport or storage specifications of CO_2-containing streams, thus increasing the overall cost of the CO_2 capture unit. Amine units also have a large footprint which further increases the cost of these systems. Finally, amine systems lose a portion of amine in the effluent gas stream, thus requiring make-up amine. More importantly, amine vapors are a hazardous waste, thus causing pollution or requiring additional treatment processes for amine capture.

Membrane-based processes have been proposed for CO_2 capture from coal combustion in air. However, these processes are not economical for processing dilute streams of CO_2. The large volume of the flue gas stream requires a great deal of compression energy to compress the stream to high pressures for membrane separation and a large amount of membrane area to separate the components of the gas.

Oxy-coal combustion has been proposed as an option to simplify the CO_2 purification process by removing N_2 from air before the combustion process. Oxygen having a relatively high concentration may be used instead of using air as the oxidant in the combustion of coal. This results in a flue gas stream that is highly concentrated in CO_2. Additionally, the flue gas volume decreases significantly. Some exemplary flue gas components produced by the oxy-coal combustion process, based on the type of coal, follow:

Estimated Flue Gas Compositions For Pulverized Coal Oxy-Fired Power Plant						
	Lignite		Sub-Bituminous		Eastern Bituminous	
Constituents	weight %	mol %	weight %	mol %	weight %	mol %
H_2O	8.61	17.41	8.64	17.42	8.70	17.43
CO_2	72.08	59.63	72.20	59.57	70.96	58.18
N_2	12.32	16.01	12.59	16.32	13.68	17.63
O_2	3.60	4.10	3.53	4.00	3.64	4.11
SO_2	0.67	0.38	0.24	0.14	0.18	0.10
Ar	2.63	2.40	2.75	2.50	2.77	2.50

Cryogenic processes have been proposed to purify the flue gas produced by the oxy-coal combustion process, while still capturing enough CO_2 to meet clean coal requirements. This overall process is economically competitive with amine scrubbing. However, cryogenic processes require removal of water vapor from the flue gas stream to prevent water condensation/freezing at cryogenic temperatures. These water vapor removal requirements are stricter than current specifications on water content for pipeline transport of CO_2. For example, the widely recognized Kinder Morgan specification limits water content in CO_2 pipelines to 30 lb/MMCF (pounds/million cubic feet) or ~ 600ppmv (parts per million volume), while cryogenic systems require water removal below 10 ppmv. This introduces additional costs to the purification process.

Also, cryogenic processes are limited in the combinations of CO_2 purity and recovery combinations that they can provide because the process relies upon the vapor-liquid equilibrium phenomena to separate mixtures. Therefore, cryogenic processes offer little availability to change operating conditions and/or compression costs.

Thus, there is a need for a system and process to capture CO_2 in desirable and adjustable purity-recovery combinations at low cost and that are flexible to the changing conditions, such as flue gas compositions and volumes, in the oxy-coal combustion process.

Summary

Disclosed is a method of purifying carbon dioxide from a gaseous mixture by obtaining a gaseous mixture from a flue gas of an oxy-coal combustion process at a low pressure. The gaseous mixture is compressed by a first compressor train and flowed into a gas separation membrane system having two or more stages, each stage selectively permeating a carbon dioxide enriched stream. The gas separation membrane system recovering approximately 90% (vol/vol) to approximately 95% (vol/vol) of the carbon dioxide from the gaseous mixture and produces a carbon dioxide product having a carbon dioxide concentration of approximately 90% (vol/vol dry basis) to approximately 97% (vol/vol dry basis).

Embodiments of the method may further include one or more of the following aspects:

- the gaseous mixture is at a pressure of approximately 0.8 to 1.2 bar.
- the gaseous mixture comprises greater than approximately 65 % (vol/vol dry basis) carbon dioxide.
- the first compressor train compresses the gaseous mixture to a pressure of approximately 3 to approximately 30 bar.
- the gas separation membrane system having two stages, with the compressed gaseous mixture flowing into the first stage to produce a first retentate and the carbon dioxide product as a first permeate; the first retentate flowing into the second stage to produce a second permeate and a second retentate; the second retentate is vented to atmosphere; and the second permeate is directed to the first compressor train for combination with the gaseous mixture.
- the gas separation membrane system having three stages and a second compressor train, with the compressed gaseous mixture flowing into the first stage to produce a first permeate and a first retentate; the first retentate flowing into the second stage to produce a second permeate and

a second retentate; the second retentate being vented to atmosphere and the second permeate flowing into the second compressor train; the compressed second permeate flowing into the third stage to produce a third retentate and a third permeate; the third retentate being combined with the first retentate at the second stage to produce the second permeate and the second retentate; and the third permeate being combined with the first permeate to produce the carbon dioxide product.

Also disclosed is a carbon dioxide purification system for recovering carbon dioxide from a source of low pressure gaseous mixtures obtained from the flue gas of an oxy-coal combustion process. The gaseous mixture contains greater than approximately 65 % (vol/vol dry basis) carbon dioxide and is at a pressure of approximately 0.8 to 1.2 bar. A first compressor train compresses the gaseous mixture and a gas separation membrane unit receives the compressed gaseous mixture from the first compressor train. The gas separation membrane unit has two or more stages, each stage selectively permeating a carbon dioxide enriched stream. The gas separation membrane unit recovers approximately 90% (vol/vol) to approximately 95% (vol/vol) of the carbon dioxide in the gaseous mixture and produces a carbon dioxide product having a carbon dioxide concentration of approximately 90% (vol/vol dry basis) to approximately 97% (vol/vol dry basis).

Embodiments of the method may further include one or more of the following:

- the gas separation membrane unit having two stages, a first stage adapted to separate the compressed gaseous mixture into a first retentate and the carbon dioxide product as a first permeate, a second stage adapted to separate the first retentate into a second permeate and a second retentate, the second stage having a retentate outlet in selective communication with ambient to vent the second retentate to atmosphere, the second stage having a permeate outlet in fluid communication with the first compressor train, and the first compressor train being further adapted to receive a combination of the gaseous mixture and the second stage permeate.
- A second compressor train, the gas separation membrane unit having three stages, a first stage adapted to separate the compressed

gaseous mixture into a first permeate and a first retentate; a second stage adapted to separate the first retentate into a second permeate and a second retentate; the second tage having a retentate outlet in selective fluid communication with ambient to vent the second retentate to atmosphere; the second compressor train adapted to receive the second permeate; a third stage adapted to separate the compressed second permeate into a third retentate and a third permeate; the second stage having an inlet in fluid communication with a retenate outlet of the third stage for combination of the third retentate and the first retentate; and a product line in fluid communication with a permeate outlet of the first stage and a permeate outlet of the third stage for combination of the third permeate and the first permeate to produce the carbon dioxide product.

Brief Description of the Drawings

For a further understanding of the nature and objects of the present invention, reference should be made to the following detailed description, taken in conjunction with the accompanying drawings, in which like elements are given the same or analogous reference numbers and wherein:

FIG 1 illustrates a schematic of an exemplary embodiment of the system disclosed.

FIG 2 illustrates a schematic of another exemplary embodiment of the system disclosed.

Detailed Description of the Invention

The method and system disclosed provide a relatively low cost and more flexible method and system to extract carbon dioxide from the flue gas of an oxy-coal combustion process than the traditionally proposed amine or cryogenic methods and systems. An oxy-combustion process is a combustion process in which nitrogen is largely eliminated from air before its use in combustion. The resulting nitrogen deficient air typically contains an oxygen concentration greater than approximately 80% (vol/vol), and more preferably greater than approximately 90% (vol/vol). An oxy-coal combustion process is an oxy-combustion process that produces heat from the combustion of coal with the nitrogen-deficient air. The nitrogen-deficient air may be diluted with carbon dioxide, such as from the flue gas, prior to combustion.

Increasing the CO_2 content of the flue gas by oxy-combustion of coal concentrates the flue gas sufficiently that membranes become attractive. Membrane processes can capture CO_2 from the flue gases of oxy-combustion of coal with similar compression energy expenditure as cryogenic processes. Additionally, by optimization of membrane surface area, flue gas compression requirements may be adjusted. Membrane processes can also provide CO_2 purity and CO_2 recovery combinations that may not be achievable with a cryogenic process. Additionally, membranes can tolerate greater amounts of water in the feed gas stream than cryogenic processes. As a result, membrane processes do not require an additional water removal process other than that mandated by the product gas specification. Membranes are also modular in nature and therefore their capacity is easily scalable to changes in feed flowrate. Thus, changes in the load produced by an oxy-coal combustion process can be easily accommodated by changing the number of membrane modules in operation.

Except for the removal of fly ash or other solid components, membranes do not require flue gas pretreatment in excess of any specified requirements for the final product. Thus, depending on the final CO_2 application, pretreatment costs may be avoided. For example, membrane materials may be selected that are resistant to SO_x and NO_x.

In the exemplary embodiments illustrated in **FIGS 1 & 2**, the gaseous mixture **10** obtained from the flue gas of an oxy-coal combustion process is compressed by a first compressor train **20** from a low pressure range of approximately 0.8 to approximately 1.2 bars to a high pressure range of approximately 3 to approximately 30 bars. One of ordinary skill in the art will recognize that a compressor train includes one or more compressors depending upon the ultimate pressure increase desired for the gaseous mixture. If multiple compressors are used, heat exchangers may be located between the compressors to cool the compressed gas. Depending on multiple factors in the oxy-coal combustion process, the gaseous mixture **10** normally contains a carbon dioxide content of at least 65% (vol/vol dry basis). The carbon dioxide content of the gaseous mixture is also normally less than the concentration required by the carbon dioxide product, thereby necessitating purification. Preferably, fly ash and other solids that may be contained in the flue gas are removed from the gaseous mixture **10** by methods such as electrostatic precipitation prior to compression of the gaseous mixture **10** by the first compressor train **20**.

Depending on the intended use of the carbon dioxide product, sulfur compounds, such as SO_2 and SO_3, contained in the flue gas may also be removed from the gaseous mixture **10** by methods such as flue gas desulfurization prior to compression by the first compressor train **20**. However, it is also envisioned that SO_x and NO_x contained in the flue gas may be processed by the gas separation membrane unit **30**. In that case, the SO_x and NO_x would preferably be sequestered with the CO_2 product.

From the first compressor train **20**, the compressed gaseous mixture **11** flows into a gas separation membrane unit **30**. In the embodiment illustrated in **FIG 1**, the gas separation membrane unit **30** has two stages of membrane-based gas separation, **35** and **45**. In the embodiment illustrated in **FIG 2**, the gas separation membrane unit **30** has three stages of membrane-based gas separation, **35**, **45**, and **55**. Each stage utilizes one or more gas separation membrane modules (not shown). Suitable gas separation membrane modules include any gas separation membranes known in the art that preferentially permeate carbon dioxide over nitrogen and oxygen. Non-limiting examples of such membranes include the membranes disclosed in U.S. Pat. Nos. 7,422,623 and 6,860,920, and 5,015,270, incorporated herein by reference in their entireties. One of ordinary skill in the art will recognize that the level of compression necessary to separate the gaseous mixture **10** may be decreased by increasing the surface area of the membranes, which may be accomplished by adding more membrane modules. Similarly, less surface area, and accordingly less or smaller membrane modules, may be required if the level of compression is increased.

Additionally one of ordinary skill in the art will recognize that the gas separation membrane unit **30** may utilize alternate membrane stage arrangements, provided the gas separation membrane unit **30** utilizes at least two stages of membrane-based gas separation. However, the embodiments depicted in **FIGS 1 & 2** are preferred embodiments for the disclosed method and system.

A drying step may be conducted before processing of the gaseous mixture **10** by the gas separation membrane unit **30** to remove water from the gaseous mixture **10**. Alternatively, if the water content of the gaseous mixture **10** is tolerated by the gas separation membrane unit **30** but remains higher than the carbon dioxide product specification, the water vapor may be processed by the gas separation membrane unit **30** and collected with the first permeate **36**. Removing the water vapor from the first permeate **36** in the product compression step should be cheaper due to the smaller size of the first permeate **36** and therefore of any drying equipment.

In **FIG 1**, the first stage of membranes **35** separates the compressed gaseous mixture **11** into a first retentate **37** and a first permeate **36**. The first permeate **36** is the carbon dioxide product of the gas separation membrane unit **30**. The first stage **35** is operated to produce a first permeate **36** having a concentration of at least 90% (vol/vol dry basis), preferably 95% (vol/vol dry basis), and more preferably 97% (vol/vol dry basis). Based on this, the first stage **35** will recover less than 90% (vol/vol) of the CO_2 originally contained in the gaseous mixture **10**. The first retentate **37** contains the additional CO_2 that is required to achieve the targeted recovery.

The second stage of membranes **45** separates the first retentate **37** into a second permeate **46** and a second retentate **47**. The second retentate **47** contains less than approximately 10% (vol/vol) of the carbon dioxide originally contained in the gaseous mixture **10**. Therefore, if this embodiment were utilized in conjunction with a coal power plant, the second retentate **47** may be vented to the atmosphere in compliance with current DOE definition of a clean coal power plant. To help recover energy from the disclosed method and system, an expander **70** may recover energy from expansion of the second retentate **47** to a lower pressure.

The second permeate **46** is recycled to the feed of the first stage of membranes **35** at the appropriate point in the first compressor train **20** depending on the pressure of the second permeate **46**.

Preferably, after system start up, the first permeate **36** contains approximately 90% (vol/vol) to approximately 95% (vol/vol) of the carbon dioxide originally contained in the gaseous mixture **10** at a purity of approximately 90% (vol/vol dry basis) to approximately 97% (vol/vol dry basis). By changing the operating conditions of the process and the number of membrane modules in operation, other purity-recovery combinations can be achieved. Depending upon the intended use of the carbon dioxide product, the first permeate stream **36** may be further compressed to liquid form by third compressor train **60** and pumped to its intended destination by liquid pump **90**.

In **FIG 2**, the first stage of membranes **35** separates the compressed gaseous mixture **11** into a first retentate **37** and a first permeate **36**. The first permeate **36** forms a portion of the carbon dioxide product. The first stage **35** is operated to produce a first permeate **36** having 90% (vol/vol dry basis), preferably 95% (vol/vol dry basis), and more preferably 97% (vol/vol dry basis) purity. Based on this, the first stage of membranes **35** will recover less than 90% (vol/vol) of the CO_2 originally contained in the gaseous mixture **10**. The first retentate **37** contains the additional CO_2 that is required to achieve the targeted recovery.

The second stage of membranes **45** separates the first retentate **37** into a second permeate **46** and a second retentate **47**. The second retentate **47** contains less than approximately 10% (vol/vol) of the carbon dioxide originally contained in the gaseous mixture **10**. Therefore, if this embodiment were utilized in conjunction with a coal power plant, the second retentate **47** may be vented to the atmosphere in compliance with current DOE definition of a clean coal power plant. To help recover energy from the disclosed method and system, an expander **70** may recover energy from expansion of the second retentate **47** to a lower pressure.

In the embodiment depicted in **FIG 2**, the second permeate **46** is compressed by the second compressor train **80**. The third stage of membranes **55** then separates the compressed second permeate **46** into a third permeate **56** and a third retentate **57** rather than recycling the second permeate **46** back to the first stage of membranes **35** as shown in **FIG 1**. The third permeate **56** and the first permeate **36** are combined to produce the carbon dioxide product. For some conditions, the embodiment of **FIG 2** will prevent dilution of the second permeate **46** by the lower purity gaseous mixture **10**. Additionally, this embodiment may result in lower energy costs than the embodiment of **FIG 1**. For example, the third stage of membranes **55** may require a lower feed pressure than required by the first stage of membranes **35** to provide the same purity carbon dioxide product, thereby reducing the compression energy requirements of the second compressor train **80**. Alternatively, the third stage **55** may produce a third permeate **56** having a concentration greater than 95% (vol/vol dry basis). In that case, the first stage **35** may be configured to produce a first permeate **36** having a concentration less than 95% (vol/vol dry basis), so long as the combined first permeate **36** and third permeate **56** produce a carbon dioxide product having a concentration of 90% (vol/vol dry basis), preferably 95% (vol/vol dry basis), and more preferably 97% (vol/vol dry basis) and recover approximately 90% (vol/vol) to approximately 96% (vol/vol) of the carbon dioxide from the gaseous mixture **10**. At lower purity, the first stage **35** will provide a greater recovery and hence push less of the gas stream to the subsequent stages where further compression is required, resulting in compression energy savings.

It will be understood that many additional changes in the details, materials, steps, and arrangement of parts, which have been herein described and illustrated in order to explain the nature of the invention, may be made by those skilled in the art within the principle and scope of the invention as expressed in the appended claims. Thus, the present invention is not intended to be limited to the specific embodiments in the examples given above and/or the attached drawings.

What is claimed is:

1. A method of purifying carbon dioxide from a gaseous mixture, said method comprising:

   (a) obtaining a gaseous mixture from a flue gas of an oxy-coal combustion process at a low pressure, the gaseous mixture containing a percentage of carbon dioxide;

   (b) compressing the gaseous mixture with a first compressor train; and

   (c) flowing the compressed gaseous mixture into a gas separation membrane system having two or more stages of membrane-based gas separation, each stage selectively permeating a carbon dioxide enriched stream, the gas separation membrane system recovering approximately 90% (vol/vol) to approximately 95% (vol/vol) of the carbon dioxide in the gaseous mixture and producing a carbon dioxide product having a carbon dioxide concentration of approximately 90% (vol/vol dry basis) to approximately 97% (vol/vol dry basis).

2. The method of claim 1, wherein the low pressure of the gaseous mixture is approximately 0.8 to 1.2 bar.

3. The method of claim 1, wherein the gaseous mixture comprises greater than approximately 65 % (vol/vol dry basis) carbon dioxide.

4. The method of claim 1, wherein the first compressor train compresses the gaseous mixture to approximately 3 to approximately 30 bar.

5. The method of claim 1, wherein the gas separation membrane system comprises two stages, wherein:

the compressed gaseous mixture flows into the first stage to produce a first retentate and the carbon dioxide product as a first permeate;

the first retentate flows into the second stage to produce a second permeate and a second retentate;

the second retentate is vented to atmosphere; and

the second permeate is directed to the first compressor train for combination with the gaseous mixture.

6. The method of claim 1, wherein:

the gas separation membrane system comprises three stages and a second compressor train;

the compressed gaseous mixture flows into the first stage to produce a first permeate and a first retentate;

the first retentate flows into the second stage to produce a second permeate and a second retentate;

the second retentate is vented to atmosphere and the second permeate flows into the second compressor train;

the compressed second permeate flows into the third stage to produce a third retentate and a third permeate;

the third retentate is combined with the first retentate at the second stage to produce the second permeate and the second retentate; and

the third permeate is combined with the first permeate to produce the carbon dioxide product.

7. A carbon dioxide purification system for recovering carbon dioxide from low pressure gaseous mixtures comprising:

a source of a gaseous mixture obtained from a flue gas of an oxy-coal combustion process, wherein the gaseous mixture contains greater than approximately 65 % (vol/vol dry basis) carbon dioxide and is at a pressure of approximately 0.8 to 1.2 bar;

a first compressor train adapted to receive the gaseous mixture; and

a gas separation membrane unit adapted to receive the compressed gaseous mixture from the first compressor train, the gas separation membrane unit having two or more stages of membrane-based gas separation, each stage selectively permeating a carbon dioxide enriched stream, the gas separation membrane unit recovering approximately 90% (vol/vol) to approximately 95% (vol/vol) of the carbon dioxide in the gaseous mixture and producing a carbon dioxide product having a carbon dioxide concentration of approximately 90% (vol/vol dry basis) to approximately 97% (vol/vol dry basis).

8. The carbon dioxide purification system of claim 7, wherein the gas separation membrane unit comprises two stages, wherein:

the first stage is adapted to separate the compressed gaseous mixture into a first retentate and the carbon dioxide product as a first permeate,

the second stage is adapted to separate the first retentate into a second permeate and a second retentate,

the second stage has a retentate outlet in selective communication with ambient;

the second stage has a permeate outlet in fluid communication with the first compressor train; and

the first compressor train is further adapted to receive a combination of the gaseous mixture and the second stage permeate.

9. The carbon dioxide purification system of claim 7, further comprising a second compressor train, wherein

the gas separation membrane unit comprises three stages;

the first stage is adapted to separate the compressed gaseous mixture into a first permeate and a first retentate;

the second stage is adapted to separate the first retentate into a second permeate and a second retentate;

the second stage has a retentate outlet in selective fluid communication with ambient;

the second compressor train is adapted to receive the second permeate;

the third stage is adapted to separate the compressed second permeate into a third retentate and a third permeate;

the second stage has an inlet in fluid communication with a retentate outlet of the third stage for combination of the third retentate and the first retentate; and

a product line in fluid communication with a permeate outlet of the first stage and a permeate outlet of the third stage for combination of the third permeate and the first permeate to produce the carbon dioxide product.

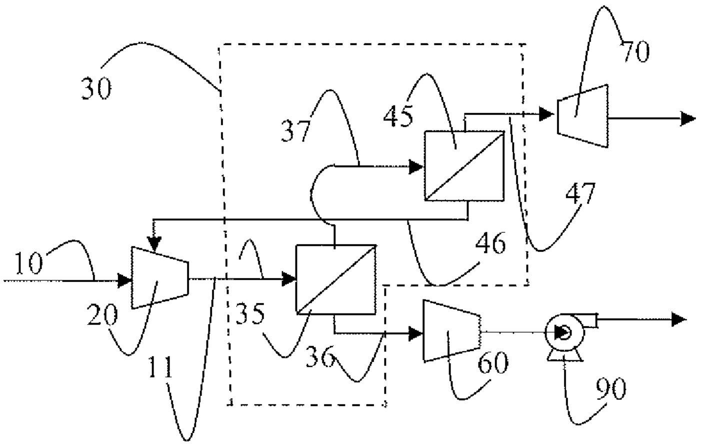

FIGURE 1

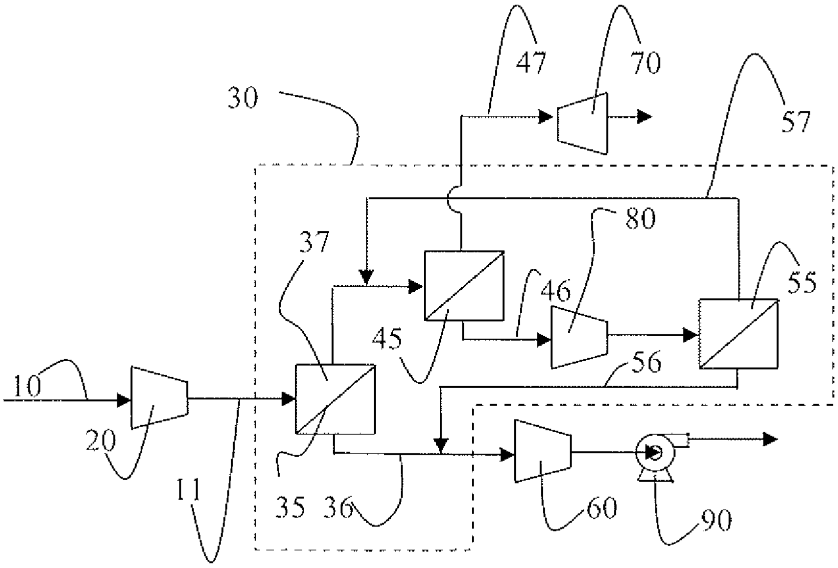

FIGURE 2

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2010/032732

A. CLASSIFICATION OF SUBJECT MATTER
INV. B01D53/22 B01D61/58
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 130 403 A (COOLEY T E ET AL) 19 December 1978 (1978-12-19)	7,9
Y	column 11, line 61 - column 12, line 8; figure; table 1	6
X	FAVRE ET AL: "Carbon dioxide recovery from post-combustion processes: Can gas permeation membranes compete with absorption?" JOURNAL OF MEMBRANE SCIENCE, ELSEVIER SCIENTIFIC PUBL.COMPANY. AMSTERDAM, NL LNKD- DOI:10.1016/J.MEMSCI.2007.02.007, vol. 294, no. 1-2, 5 April 2007 (2007-04-05), pages 50-59, XP022021879 ISSN: 0376-7388	1-4
Y	pages 50, 51	5,6,8
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☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search	Date of mailing of the international search report
27 July 2010	03/08/2010
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Persichini, Carlo

International application No
PCT/US2010/032732

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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